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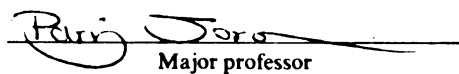
**Reinforcement of Thin Cement Products
with Recycled Wastepaper Fibers**

presented by

Zahir Shah

has been accepted towards fulfillment
of the requirements for

Ph.D. degree in Civil Engineering


Major professor

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REINFORCEMENT OF THIN CEMENT PRODUCTS WITH RECYCLED WASTEPAPER FIBERS

By

Zahir Shah

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ABSTRACT

REINFORCEMENT OF THIN CEMENT PRODUCTS WITH RECYCLED WASTEPAPER FIBERS

By

Zahir Shah

The main intent of this research was to determine the technical feasibility of utilizing magazine wastepaper fibers, obtained through dry processing of paper, as reinforcement in thin cement products. Dry-processed magazine papers have high levels of non-cellulosic impurities, and the recycling process also breaks and damages the fibers.

In order to produce wastepaper fiber-cement composites, first the influential variables in slurry-dewatering method of processing the composites were identified in an experimental study based on fractional factorial design. Among the proportioning and processing variables investigated, fiber mass fraction, level of substitution of virgin fibers with recycled ones, and fiber refinement conditions were found to have statistically significant effects on the flexural performance of composites. Subsequently, response surface analysis techniques were used to devise an experimental program which helped determine the optimum combinations of the selected influential variables based on flexural performance and cost. The optimized recycled composites were then technically evaluated versus virgin composites. They were shown to possess acceptable flexural strength, dimensional stability, density, water absorption and moisture content. Specific size distribution of recycled fibers (with higher fine contents) were used to justify their differences with virgin fibers in cement composites.

The effects of moisture and weathering on the performance of recycled wastepaper fiber-cement composites were investigated through accelerated laboratory tests simulating the

effects of wet-dry and freeze-thaw cycles as well as carbonation and chemical interaction, in natural weathering. Microstructural studies were conducted in order to establish the mechanisms of ageing in the composite material. These mechanisms provided the basis for selection of certain refinements in the matrix composition, which were successfully evaluated for the control of weathering effects on the composite material structure and properties. The effects of ageing and moisture on composites were best controlled by measures which reduced the calcium hydroxide content of hydration products and improved the water-tightness and the structure of interface zones; these refinements were made using relatively high levels of replacing cement with silica fume or through full substitution of Portland cement with a special cement.

A cost analysis was performed on the optimized wastepaper fiber-cement composites versus alternative siding building materials. The optimized recycled composites were found to present the best initial and life-cycle cost positions among commercially available siding materials.

Dedicated to my mother “Hussan Bano”

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CHAPTER 1

INTRODUCTION

1.1 ENVIRONMENTAL ASPECTS OF WASTEPAPER

1.1.1 Magnitude of Solid Waste Problem

Approximately 250 million tons of residential, commercial and industrial wastes are generated in the U.S. each year [1]. The corresponding number for Michigan is 11.8 million tons per year [2]. Leaving industrial discard aside, EPA estimates that residential and commercial wastes account for around 160 million tons. This figure is projected to reach about 193 million tons by the year 2000 [3].

In 1986 only 10 percent of all municipal solid waste was recycled and 10 to 15 percent was incinerated (mostly with energy recovery), while almost 80 percent - about 130 million tons - was disposed of in landfills. The Michigan recycling (about 12 percent) and incineration (about 19 percent) levels roughly correspond to the national values [4].

1.1.2 Wastepaper

1.1.2.3 Contribution of the Paper and Paper Products to the Solid Waste Stream: The available data on different materials excavated from landfills indicate that paper and paper products have increased steadily and now comprise approximately 55 percent by volume (and almost one-half by weight) of the materials excavated [3]. Information on specific types of paper products excavated from landfills suggest that glossy magazine paper has increased steadily to comprise 2.5 percent of landfilled municipal solid waste.

U.S. paper industry represents 30% of world capacity [5]. U.S. primary paper and paper board products in 1989 were 82.44 million tons, and are expected to be 91.07 million tons in 1992 [5]. The increase in waste paper use is expected to rise twice as fast as other fibers [5]. Within two years Michigan is expected to enjoy a rise in total paper and paper board capacity from 17.9 to 22.3%. Annual waste paper use in Michigan will increase from 20.87 million tons to 24.98 million tons in 1992 [5]. In Michigan there are total of 35 paper mills producing 9621 tons of paper per day of which 27 mills use recycled paper utilizing 4986 tons per day [6].

On the world-wide basis, recycled waste paper is already the largest fiber stream used for paper making. Fiber consumption by grade in 1987 was: recycled fiber 31%; bleached kraft 26%; un-bleached kraft 15%; mechanical pulp 14%; semi-chemical pulp 4%; sulfite pulp 5%; and non-wood 5%. Recycling rates are low -- around 10% -- in fiber rich countries such as Finland and Canada, and range upward to 50 percent in fiber poor countries, such as Europe and Japan. The U.S. falls in between these extremes, at about 27%.

Although there are over 50 different grades of wastepaper, these grades are generally divided into five categories [7]: (1) News-predominantly old newspaper (ONP), (2) Corrugated- includes old corrugated containers (OCC), (3) Mixed- color papers, magazine, and envelopes, (4) pulp substitutes, and (5) High grade deinking. Paper's inherent qualities (strength, durability, printability etc.), and ultimately its utility, are determined by its fiber composition e.g., softwood or hardwood, bleached or unbleached, virgin or recycled. Softwood fibers, for example, impart strength and tear resistance and printability. Newsprint generally includes 80-85% ground pulp (mechanical wood pulp) [8]. Fiber length is shortened in the grinding process and lignin, which remain in the unprocessed pulp, hastens its deterioration. Papers made from the ground wood pulp are weak to begin with and deteriorate easily upon ageing. Magazine paper fibers are mostly from chemical pulp. Generally, a mix of softwood and hardwood fibers are used to get the desired performance.

Recycling is the first step to reduce the solid waste dilemma, because both the landfills and incinerators are becoming increasingly costly because of new requirements and siting difficulties. The important point to recognize is that recycling is not the only solution for the solid waste problem. Recycling is only part of what must be a comprehensive plan to battle solid waste.

Magazine papers are often coated for aesthetic reasons. Coated paper is smoother, has a finer pore structure and, therefore, the quality of print is much enhanced. Conventional coatings consist of clay and pigment particles bound with a latex or a soluble binder such as starch [8,9]. The use of coated waste papers in the paper mills results in the production of large amounts of sludge, which can amount to as much as 30 percent of the input by weight [1]. This sludge then becomes industrial waste.

1.1.2.2 Landfill Crisis: Landfilling has been the most available disposal method, but many areas of the country are experiencing shortfalls of permitted landfill capacity and rising landfill costs. The “capacity crises” has become a significant concern around the country, particularly in the Northeast (Figure 1.1). According to EPA, for example, more than half of our existing landfills will reach their capacity within eight years.

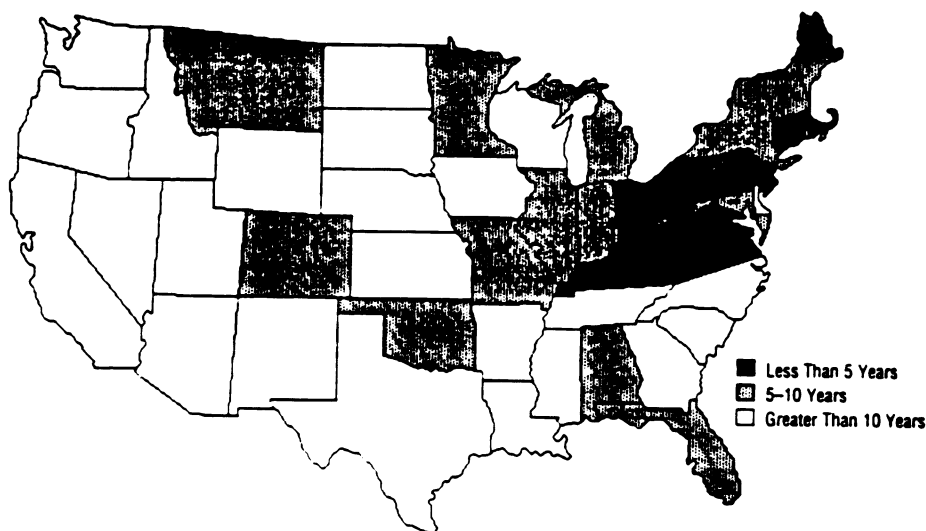


Figure 1.1 State Landfill Capacity Map [3].

Assuming that the existing landfills close at their current rate (11.2 million tons per year of lost capacity) and the new facilities are built at the same pace as in the recent years (4 million tons annually), at today’s recycling and recovery levels, our disposal requirements

will exceed existing capacity by around 1998 [1,3]. For every ton of recycled paper produced, landfill space is reduced by three cubic yards [7]. To bring our disposal needs roughly into balance with the supply of landfill space, other steps must be taken, such as meeting EPA's national recycling goal (25 percent, which is also the median statewide goal in Michigan) within the next two or three years, and tripling the number of waste-to-energy plants (Figure 1.2).

1.1.2.3 Recycling of Paper: Paper and paperboard account for a larger fraction of municipal solid waste than any other single category of material. In 1987, total wastepaper recovery (including pre-consumer waste) in the United States reached 24 million tons, a recovery rate of 28.5 percent [1]. Recovered wastepaper, or secondary fiber, is used to produce new paper products, construction materials, animal bedding, insulation, etc. Currently in Michigan old corrugated cardboard is recovered at the highest rate, 59 percent of the generation [10]. The recovery rates are much less for glossy magazine paper, coated old corrugated cardboard, and mixed paper [10]. Certain trends in recovery and utilization of wastepaper together with some technical and economic factors, as described below, are making it necessary to look beyond paper mills for the utilization of various grades of wastepaper.

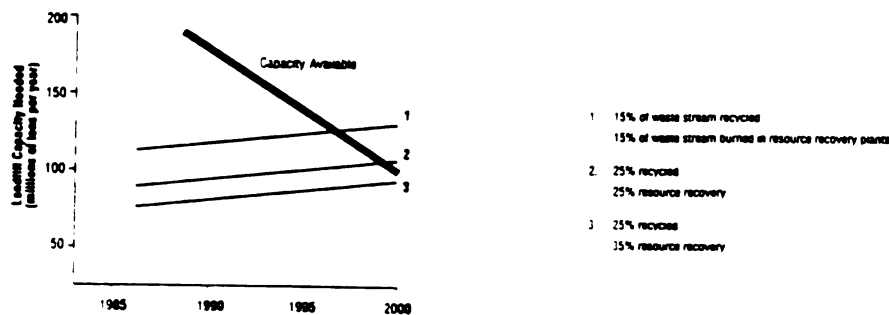


Figure 1.2 Landfill Capacity in the Year 2000 [3].

In North America, as more local recycling programs come on line, the old newsprint market is being saturated. Within the next five years, the supply of old newsprint will reach 20

million tons annually. About one-fourth of this total will be recycled by domestic mills. Combined with export of old newsprint, this means recycling old news into new paper only accounts for one-third of the total output [11].

As higher amounts of old newsprint are being recovered, this relatively cleaner wastepaper is substituting other grades (e.g., mixed paper) in mills producing boxboard and roofing products, and also in the export markets [11]. True progress in solid waste management can not be made by substituting 1 ton of mixed paper for 1 ton of old newsprint in our landfills.

Paper is not a closed-loop recyclable like glass. It can not be used indefinitely for its original use, because the cellulose fibers break down into smaller pieces each time the paper is recycled [11, 12].

The use of glossy magazines and coated old corrugated cardboard for the reproduction of paper products in paper mills results in the production of large amounts of sludge, which can amount to as much as 30 percent of the input by weight [1]. The wet sludge, produced while separating the coating material currently has no commercial value and has been removed for disposal in landfills [13]. This generally makes the use of glossy magazines and coated old corrugated cardboard in paper mills uneconomical.

Markets for paper are historically volatile, causing major fluctuations in the prices of virgin wood pulp; prices for wastepaper are even more volatile than those for wood pulp (this kind of relationship is typical of secondary materials) [1,14].

Other barriers to increased recycling of various grades of paper for reproducing paper products include capital investment in equipment, competitiveness of market, higher levels of contaminants found in news supplies of wastepaper, and restrictive specifications and rules [1,10]

Given the above constraints against recycling wastepaper into paper products, and the increased collection of wastepaper, we have to look beyond the paper industry and broaden the base of paper recycling. Advantages can be taken of the insulating, absorptive, and reinforcing qualities of recycled wastepaper to produce a whole range of products from insulation to cement boards [11, 12, 13]. These non-paper applications can grow in importance as increasing supplies of recovered wastepaper becomes available.

1.2 WASTEPAPER APPLICATIONS IN CEMENT PRODCUTS

1.2.1 Why Look into Cement Products Than Other Forms of Recycling

Recycling of wastepaper into non-paper products generally starts with grinding up the paper with augers or hammer mills and screening them to various sizes for different products. In some applications chemicals are added to give cellulose fibers different properties. Non-paper products that can be produced using wastepaper include cellulose insulation, animal bedding, mulch, cat litter, fire logs, worm bedding, roofing materials, absorbent for hazardous wastes and sludges, and thin sheet cement products. These non-paper markets for secondary fibers are briefly analyzed below.

1.2.1.1 Cellulose Insulation is made by grinding newsprint and then adding some additives as flame retardants. Old newsprint seems to be more suitable than other grades of wastepaper (e.g., magazine) because of its insulation properties. The market for cellulose insulation has declined in the recent years due to the popularity of fiberglass insulation, and also because of the loss of a healthy retrofit market after the federal government eliminated the energy tax credits in December, 1985 [11]. Also cellulose insulation is inferior to glass fiber, rock wool and polyurethane insulations [15].

1.2.1.2 Mulch made from wastepaper is generally made by shredding and fluffing the paper, mixing dye and water with the fluff, and then adding the seed prior to spraying the mulch. All grades of wastepaper are reported to be used. Currently the majority of seeding uses straw mulch and, partly due to economic factors, it is highly unlikely that State's use of recycled mulch will increase much beyond the one to five percent (less than 1000 tons per year) currently being used [10].

1.2.1.3 Cat Litter is another non-paper product that can take advantage of the high absorbency and biodegradability of wastepaper; particularly old newsprint. Sand and wastepaper are the primary ingredients of the litter. The materials are combined, then formed into granules. The impact that this product could have on the Michigan waste stream appears insignificant, mainly because its potential market is very small [10].

1.2.1.4 Other Products: Fire Logs are also being produced from old newsprint to replace raw firewood for residential uses (Goldgerg, 1989). A new product made with wastepaper is **worm bedding**, which is used to fill boxes in which the "herds" are shipped. Some research has also been done on using old newsprint to clean up hazardous liquids and sludges. **Roofing products** such as shingles, ply felts, base ply and cap sheets are also made from wastepaper. This market, however, has declined sharply in the recent years mainly due to the popularity of fiberglass roofing products [13].

In summary, non-paper products, other than thin-sheet cement products, appear to have little impact on the solid waste stream [10, 11]. These markets are either too small or they are declining, and can only be effective in some small or concentrated areas. Many of these markets also use old newsprint as the dominant wastepaper grade, and will not be suitable for other grades such as glossy magazine papers. Increased recovered volumes of wastepaper and the limited capacity that can be offered by paper mills for wastepaper in general and coated grades of wastepaper (glossy magazine and coated corrugated cardboard) in particular, however, provide strong incentives for searching alternative markets for wastepaper [1,11]. The use of recycled paper as reinforcing fibers in thin-sheet cement products presents an attractive market for wastepaper. Relatively large volumes of different wastepaper grades can potentially replace some costly and energy-intensive virgin fibers currently used by the thin-sheet cement products industry, which is still suffering from the elimination of asbestos fibers from the U.S. market. Potentials for the utilization of secondary cellulose fibers in thin-sheet cement products are discussed below.

1.2.1.5 Thin-Sheet Cement Products: World use of hydraulic cements is close to one billion tons per year [16] and, along with steel and wood, they are the most important construction materials used today. It has been proposed that the use of cement could be doubled by the year 2000. The low cost and ready availability of raw materials (limestone, clay, etc.), the fact that the energy consumed for the manufacture of cement is considerably less than for metals and plastics, and that hardening takes place with water at ordinary temperatures, provide the incentives for widespread use of cement products. Cement-based materials, however, suffer from one common shortcoming, they fail in a brittle manner under tensile stresses or impact loads. An effective approach to resolve this problem involves the use of short, randomly distributed reinforcing fibers in cement-based materials.

The reason why brittle materials like cement are made stronger by very small addition of fibers is that cracks are stopped or deflected by the presence of fibers and, as a result, the toughness and tensile strength are dramatically increased. Fibrous cement materials, with their desirable toughness characteristics and cracking resistance, have found broad applications for the construction of various thin-sheet products including cladding panels, partition components, ceilings and walls, garden fencing, silo lining, green house panels, ducting, drainage and irrigation channels, tiles, pipes, water troughs and fittings, and laboratory surfaces. The global use of thin-sheet fibrous cement products is estimated at 2.5 million tons each year, consuming about 0.5 million tons of reinforcing fibers annually.

1.2.1.6 Replacing Asbestos: World-wide, the asbestos cement sheet industry has been searching for an alternative reinforcing fiber owing to the health risk associated with the use of asbestos [17]. Consumption of asbestos for cement reinforcement reached 1.5 million metric tons of fiber in 1970's [18]. Usage was principally for factory-made cement cladding panels and pipes produced in some 800-900 manufacturing units operating virtually in every country of the world. The asbestos replacement activity in recent years has resulted in vast world-wide research into alternative cement reinforcement fibers. Virgin cellulose, glass and polyethylene are among the fibers considered to substitute asbestos in cement products. Cellulose fibers derived from softwood and hardwood, being fairly strong and stiff as well as cheap and relatively energy-efficient, have emerged as the dominant fiber types currently used in non-asbestos thin-sheet cement products [19].

Preliminary research studies conducted in Japan and Australia have demonstrated the potentials of recycled fibers obtained from various grades of wastepaper to substitute virgin cellulose fibers in thin sheet cement products [20, 21]. While Japanese workers uses a combination of recycled cellulose and asbestos fibers[19, 20, 21], the research in Australia concentrated on full substitution of all virgin fiber types with recycled wastepaper (including coated magazine paper) in thin-sheet cement products [19]. Wastepaper fiber reinforced cement has shown to provide mechanical and physical performance characteristics close to those obtained with virgin cellulose fibers. The relatively high fine contents of recycled fibers act more like fillers than reinforcing fibers, and thus higher fiber contents were found to be needed in the case of recycled fibers to reach optimum levels of mechanical performance comparable to those obtained with virgin fibers. The promising results of the preliminary studies conducted so far on the use of recycled wastepaper as reinforcing

fibers in cement products encourage more thorough investigation of on such critical aspects of the material behavior as long-term durability, moisture-sensitivity and dimensional stability. The resulting technical information should be accompanied with economical feasibility studies in order to facilitate commercial applications of wastepaper fiber reinforced cement products in applications where recycled wastepaper presents a viable alternative to the virgin fiber types.

The uses of wood fiber reinforced cement sheets are diverse. They range from major components in industrial manufacturing to uses in commercial, residential and agricultural construction (Figure 1.3). The desirable flexural strength and toughness characteristics, dimensional stability, fire resistance and impact strength of wood fiber-cement composites suggest that they could be valuable in areas of application with demanding requirements on materials. Heat shields and spray booths, sound barriers and modular flooring, duct lining and air shafts, gaskets and seals, laboratory tops and splashbacks, and fire walls in dry kilns are some of the typical industrial components made of wood fiber reinforced cement composites. Commercial and residential uses of wood fiber reinforced cement is mainly for the production of flat and corrugated sheets roofing elements, exterior and interior wall paneling, equipment screens, fascias, facades and soffits, substrate for tiles, window sills and stools, stair treads and risers, substrate for applied coatings, and utility building cladding panels. Agricultural uses of wood fiber reinforced cement composites are mainly for farm buildings sidings, stalls and walls, poultry houses and incubators, green house panels and work surfaces, and fencing and sunscreens.

The use of recycled paper as reinforcing fibers in thin-sheet cement products, where currently about 0.5 million tons of virgin fibers are used annually, presents an attractive market for wastepaper, with potentials to divert about 10 percent of the waste magazine paper from U.S. landfills.

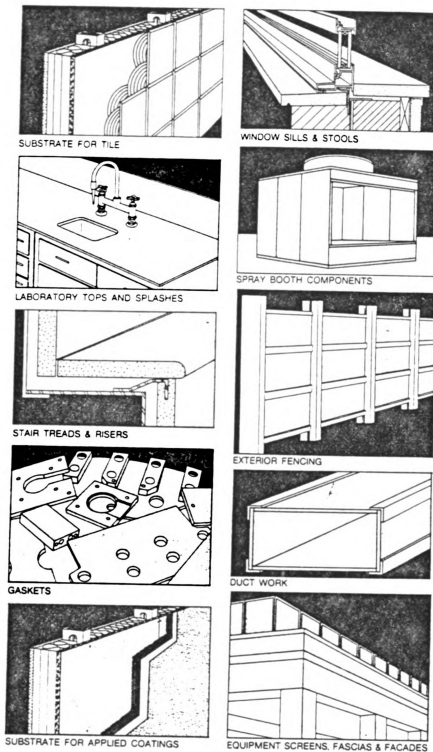


Figure 1.3 Application Areas of Cement Products Reinforced with Cellulose Fibers[21].

1.2.2 Concepts of Fiber-Reinforced Cement

Reinforcement with short, randomly distributed fibers presents an effective approach to the solution of problems with the brittle nature of failure in cement products [21, 22, 23]. These fibers are effective in stopping and deflecting the cracks propagating inside cementitious matrices, thereby substantially enhancing the toughness characteristics and cracking resistance of the material (Figure 1.4).

Fibrous cement products using virgin cellulose, polyethylene and glass fibers have found broad applications for the construction of various thin-sheet products such as cladding panels, sidings and soffits, roofing tiles, ducting, drainage and irrigation channels, tiles-substrate, pipes, water troughs and laboratory surfaces. The reinforcement action of the relatively low-cost cellulose fibers in cementitious matrices is quite good relative to other fibers such as glass (Figure 1.5).

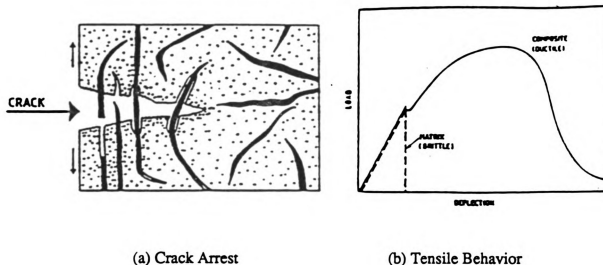


Figure 1.4 Reinforcement Action of Fibers in Cement-Based Materials [23].

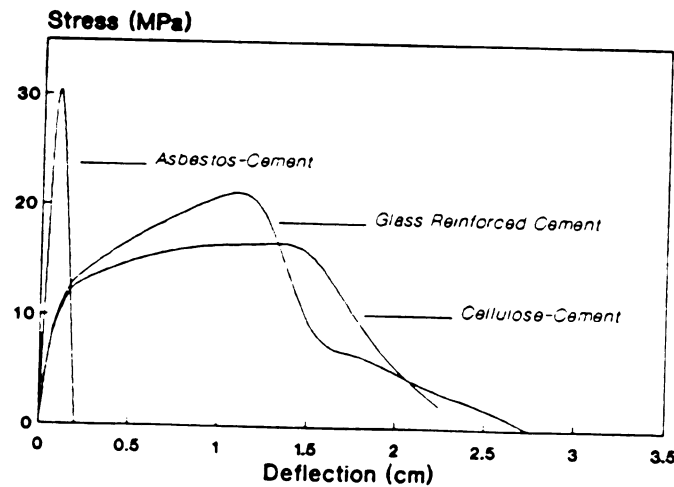


Figure 1.5 Flexural Performance of Cellulose Fiber-Cement Composites Compared with Glass Fiber Reinforced Cement and Asbestos Cement [18].

1.2.2.1 Fiber Types

Cementitious materials are relatively brittle with relatively low tensile strength. Different types of fiber have been used to overcome this shortcoming. Asbestos, steel, glass, carbon and synthetic and natural fibers are among those fiber types successfully used for the reinforcement of cement-based matrices. Properties of virgin cellulose fibers are compared in Table 1.1 with those of other fiber types used in cement products. It is evident from the ratio of cost to load carried by fibers that virgin cellulose fibers are highly cost effective. This illustrates why they are now dominating the thin cement-products market [19].

1.2.2.2 Cellulose Fibers.

Figure 1.6 briefly illustrates the structure of wood and wood fiber. If a piece of lumber is considered, it may have defects (knots, cracks etc.); by selection, a piece of clear wood (near macro defect-free) could be obtained with a tensile strength of say 70 Mpa (9.31 Ksi). However, single fiber which constitutes the reinforcing unit of bulk wood has been tested and found to have tensile strengths greater than 700 Mpa (93.1 Ksi [16]. If one considers cellulose as the basic molecule which makes up the fiber, and if one could express

the strength of the chemical bonds which make up the structure of cellulose in terms of tensile strength, an even greater value of around 7000 Mpa (931 Ksi) would be recorded.

Table 1.1 Comparisons of Cost and Strength of Wood Fibers with Other Fibers [19]

Fiber	Rel. Cost per Unit Weight	Specific Gravity (S_G)	Tensile Strength* (f_t , MPa)	f_t / S_G	Rel. Cost per unit Weight (f_t / S_G)
Wood (Kraft Pulp)	1	1.5	500	333	1
Glass Rav-ings	4	2.5	1,400	560	2.2
Steel	1.4	7.9	2,100	267	1.6
Kevlar Pulp	20	1.5	2,800	1,867	3.3
Asbestos (JM 5R)	1.2	2.6	700	269	1.3

* Realistic tensile strength values for commercial fibers

Trees serve as the major raw material for cellulose fibers. The trees harvested for the production of cellulose fibers are known commercially as “softwoods” and “hardwoods.” Among commercial trees, softwoods are the source of so-called ‘long fibers.’ The unbroken cellulose fibers in important softwoods range in length from about 2.5 mm (0.098 in.) up to 7 mm (0.28 in.), but the vast majority of these fibers average in length between 3 and 5 mm (0.12 and 0.20 in.). Even within the same tree species, fiber lengths can vary considerably. Softwood cellulose fibers have widths, or diameters, that range from about 15 to 80 microns (30 to 45 microns for most softwoods) [24]. Hardwoods yield cellulose fibers that, on an average are about 1/3 to 1/2 the length and about 1/2 the width of softwood fibers. Cellulose fibers produced from hardwood also have higher fines content when compared with those obtained from softwood.

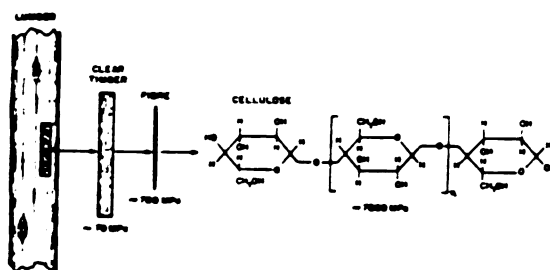


Figure 1.6 Structure of Wood and Wood-Fiber [16].

Figure 1.7 provides information on the geometry and appearance of the major fiber types in softwoods and hardwoods. All diagrams are at the same magnification to show the relative sizes of these elements.

The major chemical components of wood are cellulose, hemicellulose, lignin and a very small fraction of extractives. The cells in their natural arrangement in solid softwoods and hardwoods are bonded together by a layer of amorphous cementing material. It is this bonding that must be broken in the cellulose fiber production (pulping) process, by either chemical or mechanical means.

Pulping processes are classified as either chemical, semi-chemical, or mechanical. This classification refers to the nature of the defiberization process. In mechanical pulping, the reduction of logs or chips to fibers occurs by mechanical action which is usually aided by thermal softening of the lignin between wood cells. No chemicals are added in mechanical pulping to dissolve the lignin or any other wood component. Semi-chemical processes use a combination of both chemical reactions and mechanical power [24]. Chemical Pulps also called kraft pulps are commonly used in the production of book paper and writing, while mechanical pulps are regularly used for the manufacture of newsprint.

1.2.2.3 Recycled Coated Magazine Paper Fibers

Recycled coated magazine paper fibers are different from virgin cellulose fibers in some aspects. These have broader length distribution (mostly from 0.1 mm to 5 mm) and have fibers that are damaged and shortened as a result of recycling. In composition these “fibers” actually a combination of fibers and impurities (e.g. clay) [25]. Cellulose fiber is the major constituent comprising about 80% of mass. These fibers are formed by mechanical processing and are grey in color because of the coating pulverization. Recycled cellulose fiber have surface area of 6-7 sq.m /gm and the pH value in 5% slurry has been found to be about 7.2.

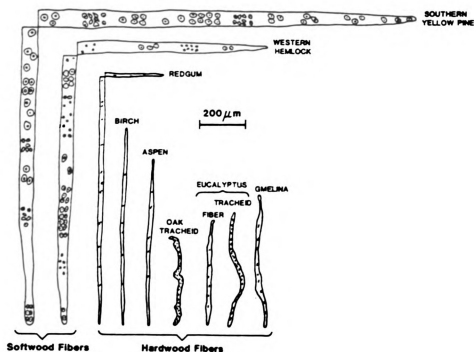


Figure 1.7 Geometry and Appearance of Major Fiber Types of Fibers in Softwood and Hardwood [24].

1.3 OBJECTIVE, SCOPE AND SIGNIFICANCE OF THIS RESEARCH STUDY

1.3.1 Objective: The main thrust of this research is to assess the technical feasibility of using recycled wastepaper as reinforcing fibers in thin-sheet cement products (to substitute more expensive virgin fibers such as glass, polyethylene and virgin cellulose). Recommendations are also made for the production and use of cement products reinforced with recycled paper fibers.

1.3.2 Scope: This research dealt with magazine wastepaper fibers, which cause problems in recycling back into papers due to their high level of impurities. Dry-processed magazine paper fibers were evaluated as reinforcement in thin-cement products. Short-term and long-term performance characteristics of the composites were evaluated, and microstructural changes associated with the ageing processes were investigated. Refined composites were produced with recycled magazine fibers with a desirable balance of short-term engineering properties and long-term weathering resistance.

1.3.3 Significance: Recycling in construction provides opportunities for long-term diversion of major volumes of market-limited (impure) waste from landfills, and also for the development of lower-cost and energy-efficient construction products. A large-volume component of the municipal solid waste stream (magazine paper) is targeted in this research, which concerns high-value utilization of this waste product in thin cement products for residential and commercial construction.

CHAPTER 2

LITERATURE REVIEW

Every composite of materials represents an individual chemical system with its own set of problems. In discussions on cement composites reinforced with recycled paper fibers we are focussing on cementitious environments which are alkaline in nature and a paper pulp (wood fiber) which may be acidic and thus unstable in alkaline environments (if excess of alum was used in its manufacture [9]) or basic. Whether paper lasts indefinitely or briefly depends on the materials and methods used in its manufacture as well as the environments in which it is stored. Since the early observations of Murray and the practical solutions suggested by Sutermeister and Barrow it has been repeatedly demonstrated that additives which create acidity within paper hasten its deterioration [9]. Acidic environments catalyze hydrolytic degradation of the polymeric cellulose molecules, reducing their chain length; even a few chain scissions per molecule cause a substantial loss of physical properties. Mildly basic environments such as calcium or magnesium carbonate minimize the acid concentration and therefore the rate of the acid hydrolysis reaction. The cellulose molecule can also suffer hydrolytic cleavage in an alkaline environment. Residual lignin in wood pulp can also accelerate the degradation of paper [9].

2.1 WASTEPAPER FIBERS IN CEMENT

Wastepaper is shredded mechanically by a dry process to get the wastepaper fibers. These fibers as compared to virgin cellulose fibers are shorter, splintered and flattened or collapsed. The use of recycled magazines and newsprint as source of cellulose fibers for the reinforcement of cement products has been reported by Coutts [26] and Hirajima et al

[22]. The main fiber constituents of the wastepaper fiber used by Coutts [26] were kraft pulped *P. radiata* and mixed eucalypts, thermomechanical pulped *P. radiata*, neutral sulphite semichemical pulped mixed eucalypts, and other fiber types present in small amounts in waste products. The distribution of fiber lengths in the wastepaper sample is shown in Table 1 along with the data for kraft *P. radiata* and kraft *E. regnans* virgin fibers. At least 10000 fibers were analyzed for the wastepaper and *E. regnans* pulps, and in excess of 5000 fibers for the *P. radiata* pulp. The wastepaper fibers are observed to have smaller average lengths, and to contain a relatively a relatively large fraction of fines. For the production of wastepaper fiber reinforced cement, the wastepaper was treated in the laboratory in a Valley beater; the matrix was prepared from ordinary portland cement.

Table 2.1 Fiber Length Data [26]

Length (mm)	Weighted Distribution (%)		
	<i>P. radiata</i>	<i>E. regnan</i>	Wastepaper
<0.2	2.6	2.0	6.6
0.2-0.6	4.0	12.8	21.2
0.6-1.2	9.1	74.2	40.0
1.2-2.0	18.0	9.0	15.3
2.0-3.0	28.0	1.5	11.0
3.0-4.0	25.0	-	5.2
4.0-5.0	11.0	-	1.0
>5.0	3.0	-	-
Weighted Average (mm)	3.2	1.0	1.9

Composites were prepared from the wastepaper fibers using different fiber fractions (2-16% fiber by mass). In earlier studies of cellulose fiber reinforced cement matrices, flexur-

al strength was at a maximum value at about 8-10% fiber mass fraction [27, 28, 29]. The wastepaper fiber reinforced cement (WPFRC) developed maximum flexural strength of 18 MPa (2,600 psi) at about 12% fiber content by mass, when tested in a controlled atmosphere (Figure 1). The effect of moisture on samples is to reduce flexural strength to approximately 47-75% of the dry strength (Figure 1). This is in general agreement with the reduction in strength observed with other air-cured wood fiber reinforced cement composites when tested both wet and dry [28, 29].

The wastepaper considered by Coutts [24] contained softwoods and hardwoods, but these fibers had experienced considerable damage due to processing and recycling as shown by high fines content (Table 2.1). Much of the damaged material acts more as a filler-diluent than as a reinforcing fiber. The flexural strength increased up to higher than usual fiber loadings before the maximum value was reached at 12% fiber content by mass. This could be due to the fact that much of the short material (say <0.6 mm) offers little reinforcement to the composite and so a greater mass of fiber must be added to achieve sufficient numbers of the longer reinforcing fibers. The flexural strength values were lower than those of cement composites reinforced with the short *E. regnans* fibers (Figure 2.1). This might be attributed to the fact that although the numbers of long fibers are sufficient to provide high strength, inefficient fiber packing had already taken place due to the total volume of fibrous material.

A similar shift of the maximum flexural strength value, with respect to fiber content, was noted for *P. radiata* thermomechanical pulp reinforced air-cured cement [30], the maximum being around 10% by mass. In that instance, the higher fiber content required and the lower flexural strength achieved were related to the lower number of fibers for a given mass (due to the extra mass of extractives not removed during the pulping process).

In addition to reduced fiber length, the altered surface properties of physically and chemically recycled fibers may also influence both flexural strength and fracture toughness properties of fiber-cement composites.

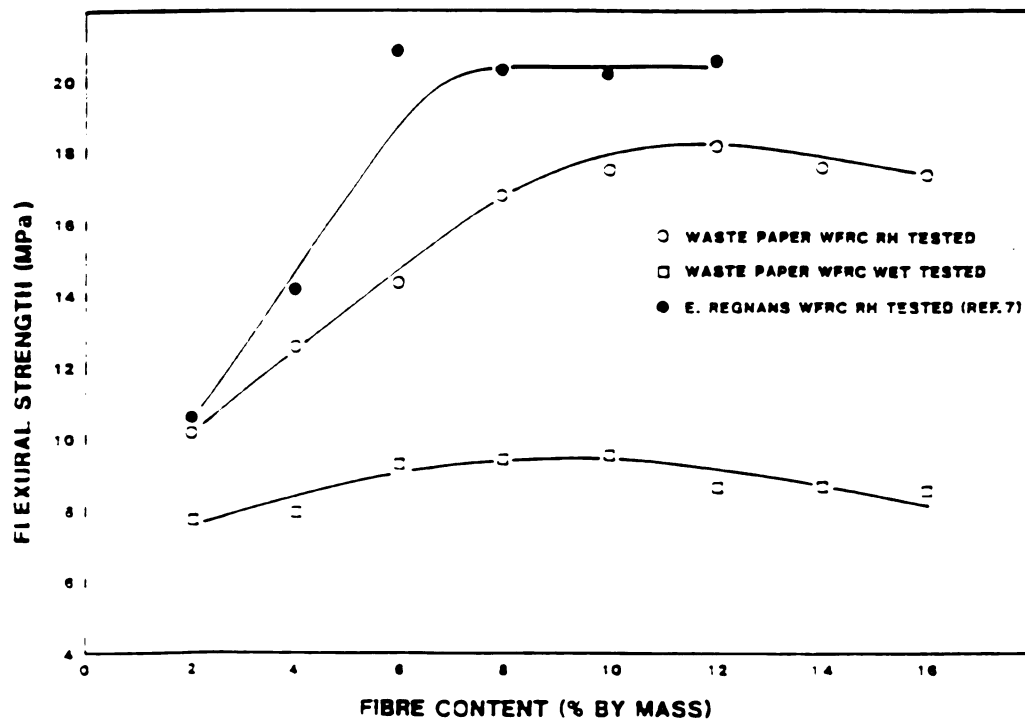


Figure 2.1 Flexural Strength Versus Fiber Content [26].

The mechanisms that take place during the fracture of a composite include fiber breakage and fiber pullout. The latter can have a considerable influence on the value of fracture toughness. If the fiber is short, then the energy used up in pulling the fiber through the matrix after the fiber to matrix bond is broken can contribute little to the dissipation of energy contained in the advancing crack. Therefore, the crack continues to propagate through the sample and the material appears brittle. In Figure 2.2 a comparison is made between the fracture toughness versus fiber content of wastepaper and E. regnans fiber reinforced cements. The E. regnans fiber provides fracture toughness values almost twice those of wastepaper fiber at comparable fiber mass fractions. This would confirm the presence of shortened fibers derived from wastepaper. Of course, other factors such as refining, which takes place during paper manufacture, would have an effect on fiber performance; this has been reported elsewhere [25]. Kraft E. regnans fiber reinforced cement composites [29] produce fracture toughness values which are almost half those of the P. radiata fiber reinforced cement [30]. This would support the hypothesis that fiber length is highly important

in increasing of fracture toughness, noting that the average fiber lengths of *E. regnans* and *P. radiata* being approximately 1.0 to 3.2 mm (0.04 to 0.1 in), respectively (Table 2.1). Although the average fiber length in the wastepaper pulp is 1.9 mm (0.07 in), the amount of fine material (<0.6mm) constitutes almost 28% of the mass of the fiber whereas *P. radiata* and *E. regnans* have values of only 6.6% and 14.8%, respectively (Table 2.1).

When the wastepaper fiber reinforced samples are tested wet, there is the expected increase in fracture toughness value [28, 29]. The increase is, however, very small, never exceeding 40% of the dry value. With *P. radiata* reinforced samples [29], increases of up to 150% of dry values have been recorded at certain fiber loadings (6-8% by mass).

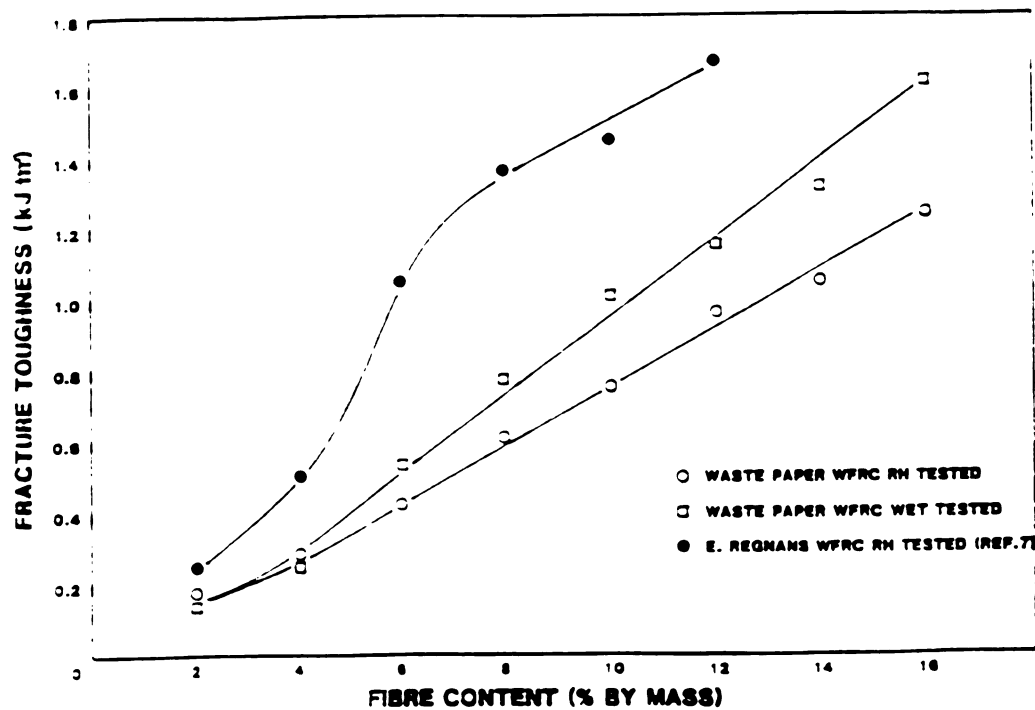


Figure 2.2 Fracture Toughness Versus Fiber Content [26].

The physical properties of air-cured cellulose-fiber reinforced cement containing *E. regnans* fibers are compared with wastepaper reinforced cement composites in Figure 2.3.

Density, water absorption and porosity are all inter-related in so far as their magnitude de-

depends upon the free space or void volume present in the material. The higher density of WPFRC could be attributed to the ability of the fine fiber fragments to pack more efficiently with the matrix and so to produce fewer voids. This in turn would account for the lower water absorption of WPFRC compared to the E. regnans reinforced material at any given fiber content.

Hirajima et al [20] have also used refined pulps recovered from municipal solid waste for the production of thin-sheet cement boards. The results showed that there was no need to modify or change the production process adopted for virgin cellulose fiber in the case of newsprint waste utilization as reinforcing fibers. Thin-sheet cement boards produced with wastepaper fibers fulfilled Japanese specifications. Refined wastepaper pulp proved to be a suitable reinforcing fiber for thin-sheet cement boards.

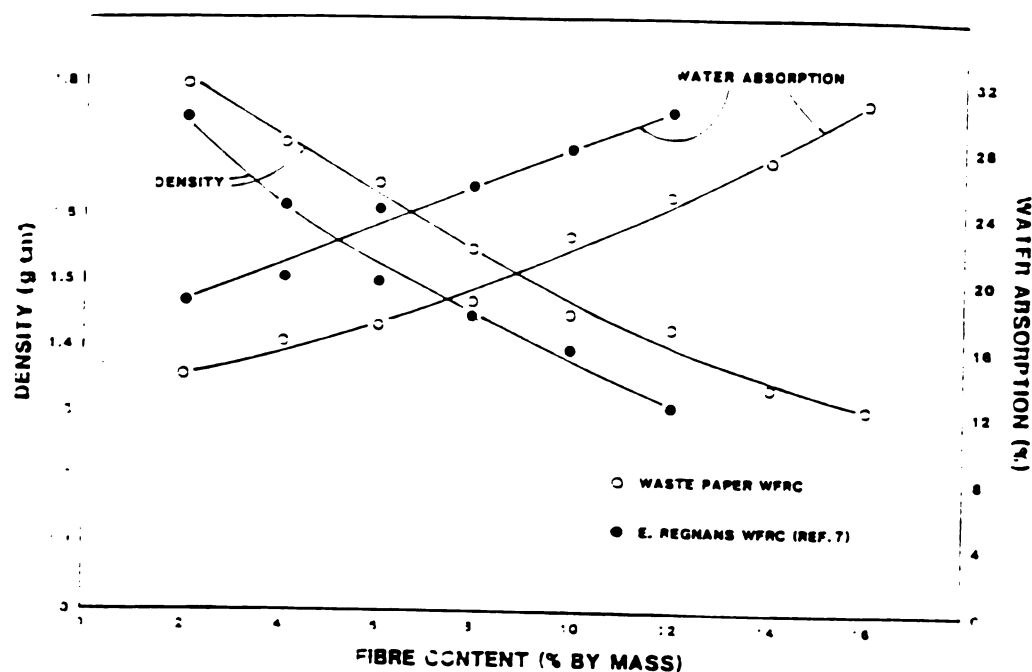


Figure 2.3 Water Absorption and Density [26].

2.2 POTENTIAL PROBLEMS - WEATHERING EFFECTS

It is important to ensure that the improvements achieved in the properties of cement based materials though recycled cellulose fiber reinforcement would be retained over a long period of time in actual exposure conditions. In particular, one should be careful about the affinity of cellulose fibers for moisture, their durability in the alkaline environment of cement, and the possibility of biological attacks on wood fibers. As far as the biological attacks are concerned, it is worth mentioning that no evidence is available to indicate that natural fibers can be decomposed biologically when used in cement materials [31]. There are no test data available on durability characteristics of wastepaper fibers reinforced cement composites. However, studies carried out on natural and virgin cellulose fibers in cementitious matrix provide insight into general durability characteristics of lignocellulosic fiber-cement composites. A brief review of the mechanisms of deterioration of natural and virgin cellulose fibers in cement-based matrices along with long-term performance of composites is presented below.

2.2.1 Mechanisms of Deterioration

Virgin kraft cellulose fibers are the key fiber type used in cement composites; they have minimum lignin content and, noting the susceptibility of lignin to alkaline attack, have been used in applications involving outside exposure. Different weathering conditions actually increase the flexural strength and modulus of elasticity of the composite. However, weathered kraft cellulose fiber reinforced cement composites are more brittle than the original composites [32, 33, 34].

Much of the available test data on long-term durability of wood fiber-cement composites deals with the use of natural fibers (e.g., sisal) in cement-based materials. In this case, unless measures are taken to reduce the alkalinity of cement matrix or to protect the natural fibers, the repeated action of wetting and drying results in the transport of alkaline pore water to fibers and the removal of neutralized pore water (which would be produced in the vicinity of fibers) as well as the decomposed products from these fibers, causing decomposition of some natural fibers like sisal [31]. This can lead to the embrittlement and loss of flexural strength in natural fiber reinforced cement composites. Repeated wetting and drying is a key factor accelerating this deterioration process of natural fibers, and it depends

on temperature and humidity history in the vicinity of the cement product. Interior exposure conditions or continuous immersion in water at ambient temperatures can not produce cycles of wetting and drying which are particularly harmful to the composite. Two years of exposure in such conditions did not lead to any embrittlement of sisal reinforced cement composites in test results reported by Gram (1983) [31].

A description of the mechanisms leading to the embrittlement of natural fiber reinforced concrete under the action of repeated wetting and drying (rain-heat) is presented in Gram (1983) [31]. According to this description, the alkaline pore water in concrete reacts with the lignin and hemicellulose existing in the middle of lamellae causing decomposition of these constituents of fibers. This leads to the weakening of the link between individual fiber cells in natural fiber (Figure 2.4). External changes in moisture and temperature which can provide a supply of water to and removal of water from concrete pores generate the moisture movement needed for alkaline pore water to reach and progressively decompose the natural fibers, leading to the embrittlement of the composite material.

Wood kraft fibers, as mentioned earlier, contain negligible amounts of lignin and thus they can withstand the alkaline pore water attack better than natural fibers. The reduced toughness accompanied with the strength gain in these composites with ageing may be associated with the petrification process (filling of the core of the fiber with hydration products) and the consequent changes in fiber failure mode [35, 36, 37, 38, 39]. The filling of fiber cores and possibly cell wall pores with hydration products is expected to result in an increase in strength and stiffness. In addition to that, it seems that the petrified fibers are more stable dimensionally and no separation and debonding between the fibers and the matrix could be observed. In short, this petrification process increases the stiffness, strength and bond strengths of fibers, but reduces their ductility; these conditions lead to improved stiffness and strength and increased brittleness of the composite.

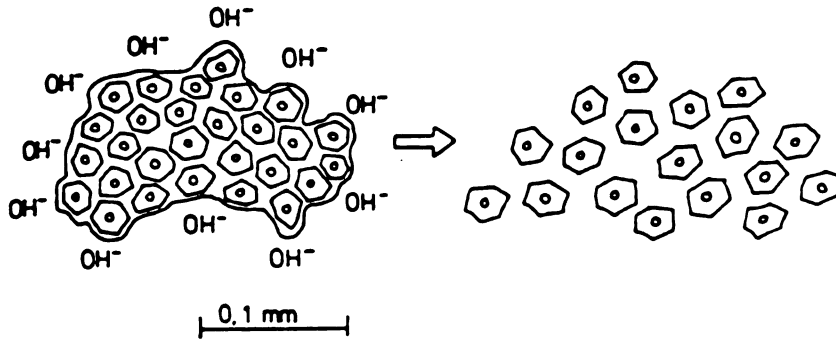


Figure 2.4 Schematic Sketch of the Decomposition of Natural (sisal) Fibers in the Alkaline Pore Water of Concrete [31].

2.2.2 Durability Test Results

2.2.2.1 Natural Fibers

Gram (1983) [31] has reported test results on the durability characteristics of sisal and coir fiber reinforced mortars, in which a typical matrix had 1:2:0.5 binder: aggregate: water proportions. Fiber content ranged from 0.5-4% by volume. Tests were performed under natural ageing and also accelerated ageing conditions. In order to reproduce weathering effects simulating the alkaline pore water attack on natural fibers (e.g. conditions involving repeated exposure to rain and sun shine), an accelerated wetting-drying test equipment was developed (Figure 2.5). The panel specimens used in this “climate box” measure 10 mm (0.4 in) in thickness and are subjected to half-cycles of moistening and cooling by spraying them with water, followed by half cycles of heating with the temperature reaching 105 deg. C (221 deg. F) and maintained at this level for a sufficiently long period so that the capillary pore system in the specimen dries out.

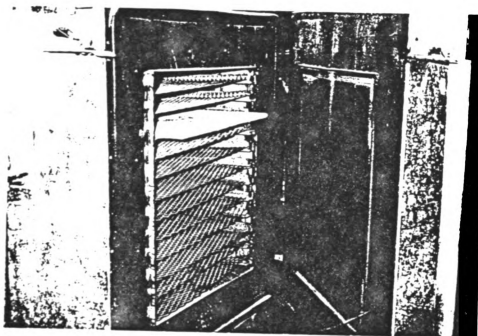


Figure 2.5 CBI Climate Box [31].

As a result of the above wetting-drying cycle, the capillary pore system of specimens is both filled with and emptied of water during the conditioning cycle that lasts about 6 hours. This means that the fibers embedded in specimens come in contact with the alkaline pore water of the concrete during the moistening phase, and that any decomposition products which are formed as a result of the reaction between the fiber components and the pore water can be transported away from the fiber during the drying phase. Figure 2.6 (a) presents the reduction in flexural strength of specimens incorporating sisal fibers due to ageing under repeated wetting-drying cycles. Considerable decrease in flexural strength is observed as the number of cycles increases. This can be attributed to the attack on natural fibers by the alkaline pore water of concrete.

The alkalinity of pore water in the matrix can be reduced and the weathering resistance of the composite can be improved by replacing part of the cement with silica fume. The pH value for the pore water reduces from 13.2 for a matrix with ordinary Portland cement to 12.0 for a matrix in which 33% of Portland cement by weight is substituted with silica fume. Figure 2.6 (b) shows the flexural strength of specimens reinforced with sisal fibers after 0 and 120 cycles of wetting and drying as a function of the percentage of cement by weight substituted with silica fume. Specimens in which up to 20% of Portland cement

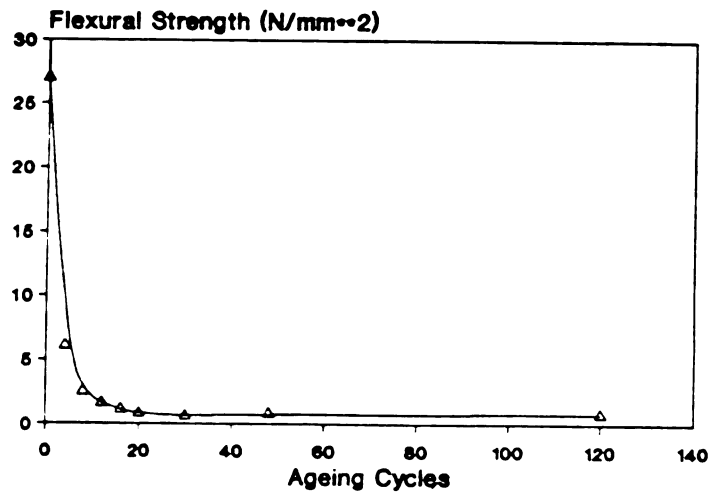
was replaced with silica fume show comparatively insignificant drops in strength after 120 wetting drying cycles when compared with specimens without silica fume. A dramatic improvement is obtained when 30-50% of the cement is replaced with silica fume. Hence, an effective approach to reducing the alkaline attack and thus enhancing the durability of natural fiber-cement composites is through reducing the alkalinity of concrete pore water by the use of silica fume [39, 40, 41].

2.2. 2.2 Processed Fibers

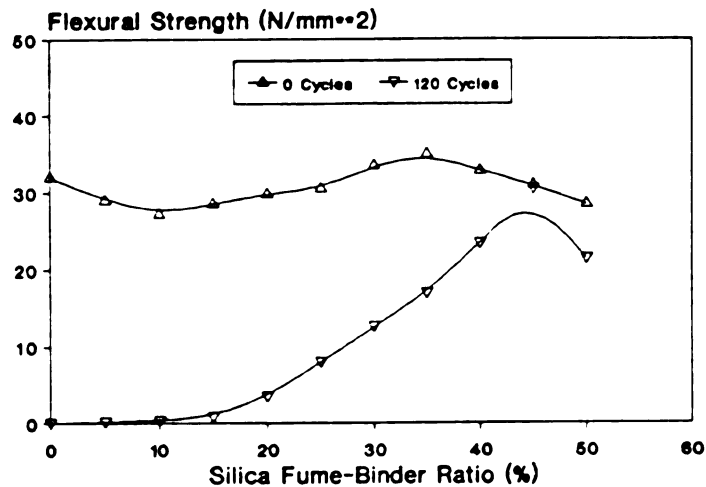
Earlier durability test results with processed cellulose fibers (paper pulp) suggest that cellulose fiber-cement composites may be prone to degradation in certain exposure conditions. Four potential ageing mechanisms were investigated by Sharman (1983) [32] and Sharman, et al. (1986) [32] for autoclaved cellulose fiber reinforced cement sheets. The ageing mechanisms considered were carbonation, microbiological attack, moisture stressing of wood fibers, and increase in fiber-to-matrix bond, acting independently or together.

Carbonation is an important ageing mechanism in asbestos cement, causing embrittlement. In the case of cellulose fibers, carbonation may also increase susceptibility to microbiological attack by reducing the alkalinity of the cement pore water and/or increasing the bonding of cementitious matrices to cellulose fibers. The rate and extent of carbonation depends on the physical conditions of the sheet (e.g. porosity) and local climatic conditions (relative humidity and temperature). Microbiological attack on cellulose fibers in cement products was studied by Mansur and Aziz (1982) [42], who found it to be unlikely in the highly alkaline environment of cementitious matrices. Long-term drop in the pH of cement products after weathering and carbonation effects, however, may have adverse effects on resistance to biological attack.

The behavior of cellulose fiber reinforced cement composites has been investigated under various accelerated ageing effects, including (1) hot water soak, (2) moisture cycling, (3) carbonation, and (4) fungal cellar exposure. Comprehensive ageing test data have been reported by Sharman et al. (1986) [32] for autoclaved cement sheets consisting of 8% kraft pulp (*P. Radiata*), 46% cement and 46% silica sand, and also by other investigators. A brief review is presented below.



(a) Pure Cement



(b) With Silica Fume

Figure 2.6 (cont'd) Flexural Strength of Composites Reinforced with Sisal Fibers After Wetting-Drying Cycles [31].

Hot Water Soak

Hot water soak tests have been used to accelerate the ageing process and the subsequent strength loss of glass fiber reinforced cement composites. In the case of cellulose fiber reinforced cement, this test has been used to investigate any ageing effects on fibers or

their bond to cementitious matrices. Effects of 350 days of immersion in 50 deg. C (122 deg. F) water on various aspects of the engineering properties of cellulose fiber reinforced cement composites are presented in Table 2.2. It should be noted that changes in material properties in warm water soak occurred dominantly in the first 20 days of immersion; thereafter, the material properties stayed practically constant. These test results indicate that warm water immersion, as an accelerated weathering condition, causes an increase in elastic modulus of wood fiber reinforced cement composites; other effects of accelerated ageing under warm water do not seem to be significant.

Harper (1982) [43] has observed similar effects of warm water immersion on wood fiber (kraft pulp) cement composites, implying that degradation of chemically processed wood fibers under weathering effects is unlikely. This can be explained by the fact that lignin, which is the wood fiber constituent most susceptible to alkali attack, is totally removed during the kraft pulping process (noting that kraft pulp is the main wood fiber type used in cement composites). The fact that mechanical properties of wood fiber reinforced cement sheets remained much the same throughout exposure indicate that there is no major change in the nature of fiber-to-cement bond. Failure in mechanical testing was predominantly by fiber pullout.

Table 2.2 Wood Fiber-Cement Composites - Hot Water (50 deg. C, 122 deg. F) Soak Test (see Appendix B for notation [32])

Number of Days Immersed	Mechanical Test Method	Moisture Movement (%)		Modulus of Elasticity(GPa)		Modulus of Rupture (MPa)	
		MD	CD	MD	CD	MD	CD
0	Mean	0.302	0.283	7.92	3.88	18.6	10.3
	S.D.	0.008	0.005	0.33	0.70	0.6	0.2
350	Mean	0.235	0.224	9.79	8.15	19.2	12.1
	S.D.	0.009	0.012	0.33	0.35	0.9	1.2

Accelerated Wetting and Drying

Repeated wetting-drying tests are widely used for accelerated weathering of particle-boards. Sharman et. al (1986) [32] have reported test results on wood fiber reinforced cement sheets subjected to 10, 20, 35 and 50 cycles of wetting and drying. The test results showed only a small increase in modulus of elasticity up to 10 cycles with no further changes after 10 cycles. In this work, possible embrittlement of the composite under repeated wetting-drying effects was not investigated.

Recent studies on repeated wetting and drying are reported by Akers et al. [34]. In this study, the effects of natural versus accelerated weathering conditions on the ageing behavior of wood fiber reinforced cement composites were also investigated. Two products manufactured on a standard Hatschek machine were used in this study. They were: (1) 8% wood fibers in a Portland cement-based matrix and cured at ambient temperature and relative humidity; and (2) 8% wood fibers in a Portland cement and silica based matrix and autoclaved. The dimensions of the flat sheet products were 600 x 400 x 6 mm (24 x 16 in x 0.23 in) for natural weathering and 200 x 100 x 6 mm (8 x 4 x 0.23 in) for accelerated ageing tests. The Portland cement-based and Portland cement silica-based products were exposed to natural weathering for 5 and 4 years, respectively. Similar products were also subjected to accelerated tests.

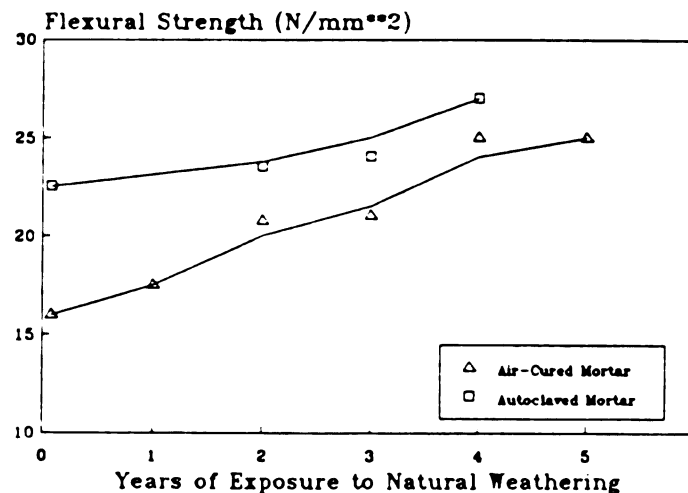
The accelerated test involved repeated cycles of wetting and drying, with each cycle consisting of the following steps over a 24-hour time period: (a) 9 hours submersion under water at 20 deg. C (68 deg. F); (b) 3 hours in air at 20 deg. C (68 deg. F); (c) 9 hour of infrared radiation at 80 deg. C (176 deg. F) in air; and (d) 3 hours cooling down to 20 deg. C (68 deg. F) in air. The natural and accelerated ageing effects on wood fiber-cement composites are discussed in the following.

The test results on specimens exposed to natural weathering (Figure 2.7) showed a general increase in strength and stiffness. This increase in strength is associated with an increase in density from 1750 to 1870 kg/m³ (109 to 117 lb/ft³) over a period of 5 years for Portland-based (air cured) specimens and from 1610 to 1790 kg/m³ (100 to 112 lb/ft³) over a period of 4 years for Portland cement/silica-based (autoclaved) specimens. The products were found to be well carbonated at the end of the natural weathering test period.

The degree of polymerization of wood fibers in the naturally aged products was found to

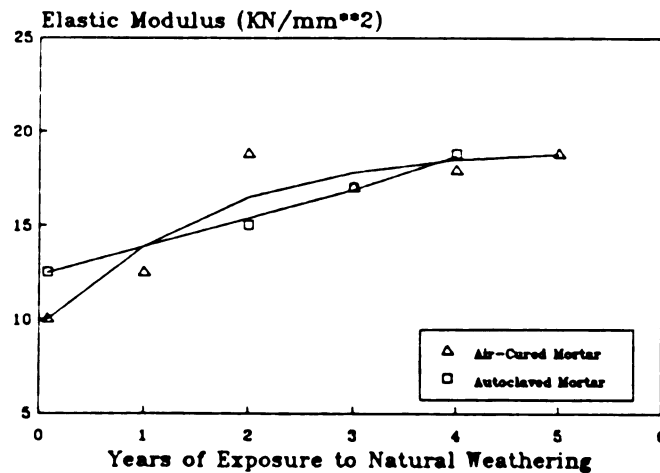
decrease with age by about 20% for air cured products and 35% for autoclaved products. This is in contrast to the tendency in strength to increase with age. Thus, the weakening effects which might have been expected upon reduction in the degree of polymerization are apparently more than compensated for by other processes, including the improvement in fiber-to-matrix bonding and overall densification of the composite under ageing effects.

The naturally aged specimens were found to be brittle. The embrittlement of these composites may have been due to the increase in fiber-to-matrix bond, which results in fiber rupture rather than pullout dominating failure at fracture surfaces; this ageing effect reduces the frictional energy absorption associated with fiber pullout. The specimens subjected to accelerated ageing (up to 3 months) in ambient environment (with minimal carbonation effects) showed drops in flexural strength (16.4 to 12.3 MPa, 2.38 to 1.78 ksi). This is in contrast to the increase in flexural strength shown by naturally aged composites. The increase in elastic modulus and the drop in degree of polymerization were, however, comparable.



(a) Flexural Strength

Figure 2.7 Flexural Strength and Young's Modulus of Air Cured and Autoclaved Products Exposed to Natural Weathering [34].



(b) Elastic Modulus

Figure 2.7 (Cont'd) Flexural Strength and Young's Modulus of Air Cured and Autoclaved Products Exposed to Natural Weathering [34].

Observation of the fractured surfaces of composites after accelerated ageing in ambient environment indicated invariably broken fibers with their hollow nature clearly shown (see Figure 2.8) [35]. In most cases the matrix around the fibers was quite dense (Figure 2.8). It was typical to observe debonding between wood fibers and the surrounding matrix.

Carbonation

Carbonation of the matrix by atmospheric carbon dioxide has been observed to cause loss of strength in asbestos cement composites. Since wood fiber cement sheets will also undergo carbonation in everyday use, it is desirable to account for this process in any accelerated ageing tests on wood fiber-cement composites.

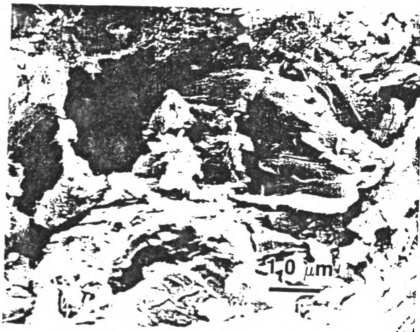
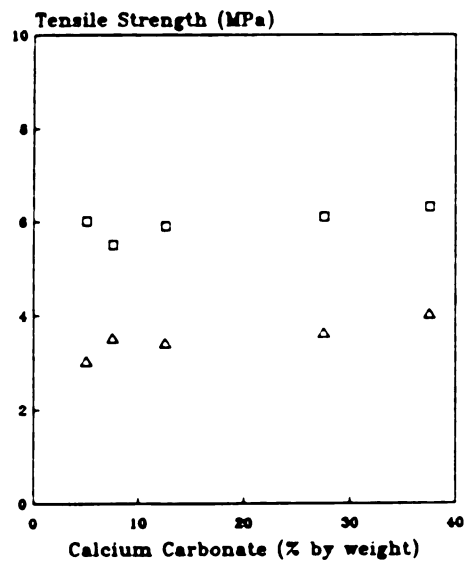
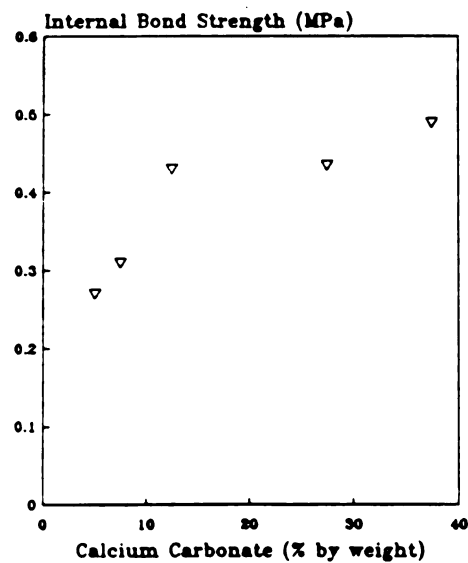


Figure 2.8 Scanning Electron Micrographs of Fractured Surfaces After Accelerated Weathering in Ambient Environment [35].

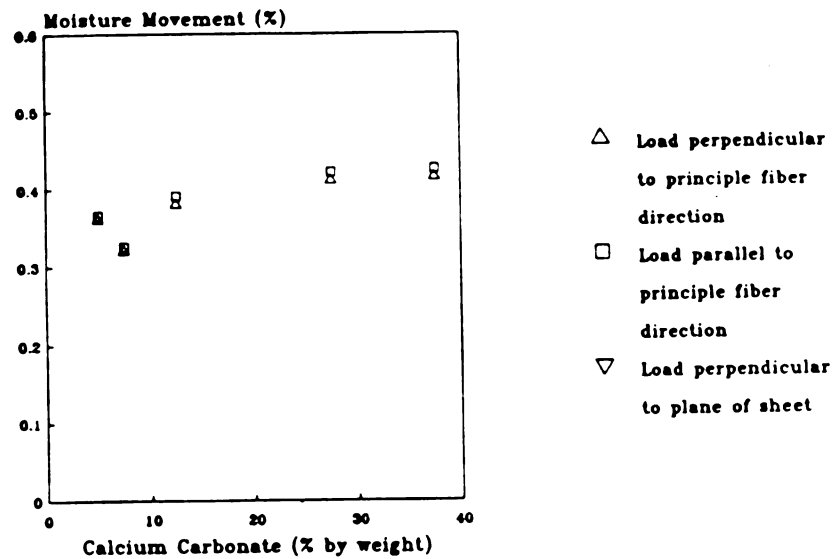
Sharman et al. (1986) [32] have conducted carbonation tests on autoclaved wood fiber reinforced cement composites. The specimens were stored in a tank (RH 65% and temperature 20 deg. C, 68 deg. F) which was supplied with carbon dioxide at a rate of $40 \text{ cm}^3/\text{min}$ ($2.5 \text{ in}^3/\text{min}$). The extent of carbonation was assessed by the measurement of CaCO_3 content, which reached levels ranging from 36 to 41%, when the absorption of CO_2 virtually ceased. The significant changes noted in the properties of carbonated wood fiber reinforced cement sheets were increased tensile strength, internal bond, and moisture movement (Figure 2.9). An increase in modulus of rupture was also observed, and the flexural stiffness showed a similar increase. It is worth mentioning that the increase in moisture movement seems to be the only potentially deleterious effect of carbonation observed in this investigation.



(a) Tensile Strength



(b) Internal Bond Strength



(c) Moisture Movement

Figure 2.9 Effects of Carbonation on Wood Fiber Reinforced Cement Sheets [32].

Akers et al. [34] in more recent studies on carbonation effects, subjected wood fiber-

cement composites to accelerated weathering conditions which also involved exposure to a carbon dioxide rich environment. Two composites (air cured cement-based and autoclaved Portland cement silica-based matrices) were subjected to the following accelerated weathering cycles.

- (a) 8 hours submersion under water at 20 deg. C (68 deg. F)
- (b) 1 hour in oven at 80 deg. C (176 deg. F)
- (c) 5 hours in oven at 20 deg. C (68 deg. F) in a saturated CO₂ environment
- (d) 9 hour in oven at 80 deg. C (176 deg. F)
- (e) 1 hour cooling down from 80 deg. C to 20 deg. C (176 to 68 deg. F)

The test cycles chosen were optimized by trial and error experiments based on the degree of carbonation and water penetration into the products. Table 2.3 shows the test results on the ageing of composites subjected to natural weathering and accelerated ageing tests. The results suggest that the development of mechanical properties of wood fiber reinforced cement composites when exposed to CO₂ rich accelerated test simulates more closely the behavior in natural weathering. Accelerated ageing in a CO₂ rich environment and natural weathering both led to an increase in strength and elastic modulus. Also, the increase in the degree of carbonation in a CO₂ environment compares favorably with the naturally weathered products. The increase in density of the product with age may be associated with carbonation of the matrix.

With respect to wood fiber properties, there is a breakdown in the molecular chain of the fibers with age, which may be directly correlated with the decrease in degree of polymerization (see Table 2.3). The drop in degree of polymerization should logically result in a drop in tensile strength of the wood fibers; however, other factors such as carbonation, which tend to increase the strength and elastic modulus of the composite overshadow any negative effects of the damage to wood fibers.

Table 2.3 Properties of Wood Fiber-Cement Products Exposed to Different Ageing Conditions [34]

Type of Ageing	Flexural Strength (N/mm ²)	Elastic Modulus kN/mm ²	Density Kg/m ³
Non Aged	16.4 ± 0.9	10.9 ± 1.3	1770 ± 20
Accelerated Aged 3 Months Ambient Environments	12.3 ± 1.1	14.8 ± 1.2	1780 ± 10
Accelerated Aged 3 Months CO ₂ Rich Environments	23.9 ± 2.2	18.9 ± 1.4	1800 ± 10
Natural Weathering	25.1 ± 1.6	18 ± 0.9	1830 ± 40

The mode of fracture after ageing was brittle with most of the fibers being broken at the fractured plane (Figure 2.10). Very frequently, the circular cross section of the broken fiber was filled up with dense hydration products (Figure 2.10) and there was no perimeter debonding. This microstructure is referred to as “brittle petrified.”

It is suggested that the increase in strength and rigidity of the petrified fibers, and the increase in their bond strength (due to matrix densification and elimination of shrinkage-debonding from the surrounding matrix) can account for the increase in strength and elastic modulus of the composite upon ageing.

Naturally aged composites produced microstructural features at failure surfaces similar to those of composites subjected to accelerated ageing in a carbon dioxide rich environment. This suggests that petrification takes place more readily under carbonating conditions, probably due to the lower pH and greater solubility of the hydration products.

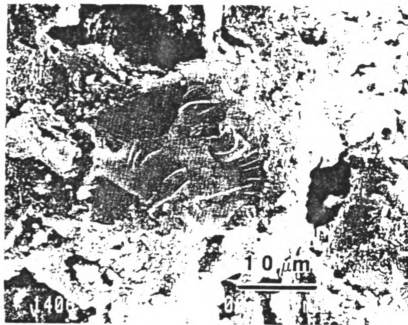


Figure 2.10 Brittle Fracture in a Composite After Accelerated Ageing in a CO₂ Rich Environment [34].

2.3 THEORETICAL CONSIDERATIONS

Basics of Fracture Mechanics

When the tensile strength of a brittle material is reached in a structure, cracking will occur. The study of the conditions around and in front of a crack tip is called fracture mechanics [44]. Fracture mechanics was first studied for brittle materials such as glass [45]. The first applications to concrete appear to have been made by Neville [46] and by Kaplan [47]. A historical review and an annotated bibliography of the applications of fracture mechanics to cement and concrete is given by Mindess [48].

The application of fracture mechanics to concrete structures has provided new ways of understanding and modeling phenomena which could only be treated empirically before. There is a growing international interest in these questions and this is reflected in recent

published literature. Some works covering the main parts of the development have been presented by Wittman [49], Shah [50], Carpinteri and Ingrassia [51], Sih and DiTommaso [52], Reinhardt [53], and Ewalds [54].

Fracture mechanics refers to the analysis of the fracture of materials by the rapid growth of pre-existing flaws or cracks. Such rapid (or even catastrophic) crack growth may occur when a system requires sufficient stored energy that, during crack extension, the system releases more energy than it absorbs. Fracture of this type (often referred to as fast fracture) can be predicted in terms of energy criterion [55,56,57].

If we consider an elastic system containing a crack and subjected to external loads, the total energy in the system, U , is

$$U = (-W_L + U_E) + U_S \dots \dots \dots (\text{Equation 2.1})$$

where

$-W_L$ = work due to the applied loads

U_E = strain energy stored in the system

U_S = surface energy absorbed for the creation of new crack surfaces

A crack will propagate when $dU/dc < 0$, where dc is the increase in the crack length. Using this theory, one can derive the Griffith equation, which gives the theoretical fracture strength for brittle, linearly elastic materials:

$$\sigma_f = (2E\gamma_s/\pi c)^{1/2} \dots \dots \dots (\text{Equation 2.2})$$

where

σ_f = Stress at first crack strain

c = one half of crack length

γ_s = surface energy of the material

This is the basic equation of linear elastic fracture mechanics (LEFM).

If we define a parameter $G_c = 2\gamma_s$ = critical strain energy release rate, then we may write

the criterion for catastrophic crack growth as

$$\sigma(\pi c)^{1/2} = (EG_c)^{1/2} \dots\dots\dots \text{(Equation 2.3)}$$

That is, fracture will occur when, in a stressed material, the crack reaches a critical size (or when in a material containing a crack of some given size, the stress reaches a critical value).

Alternatively, we may define a parameter $K_{Ic} = \sigma(\pi c)^{1/2}$ = critical stress intensity factor.

$$K_{Ic}^2 = EG_c \dots\dots\dots \text{(Equation 2.4)}$$

K_{Ic} has the units of $N/m^{3/2}$, and is often referred to as the fracture toughness (not to be confused with the term “toughness”, which is used to refer to the area under the load-deflection or stress-strain curve).

The LEFM parameters, G_c and K_{Ic} , are one-parameter descriptions of the stress and displacement fields in the vicinity of a crack tip. In much of the early work on the applications of fracture mechanics to cement and concrete, it was assumed that they provided an adequate failure criterion. However, later research showed that even for these relatively brittle materials, LEFM could only be applied to extremely large sections (e.g., mass concrete structures, such as large dams). For more ordinary cross-sectional dimensions, nonlinear fracture mechanics parameters provide a much better description of the fracture process.

Fibers enhance the strength and, more particularly, the toughness of brittle matrices by providing a crack arrest mechanism. Therefore, fracture mechanics concepts have also been applied to model fiber reinforced cement composites. Mindess [48] has reviewed the difficulties in modelling cement composites based on the fracture mechanics approach. LEFM might be adequate to predict the effects of the fibers on first cracking. However, to account for the post-cracking behavior (which is responsible for the enhanced toughness of fiber-cement composites), it is essential to resort to elastic-plastic or nonlinear fracture mechanics. A measure of toughness (i.e., the energy absorbed during fracture) can be obtained from the area under the stress-strain curve in tension. The fracture mechanics concepts which could provide a more precise measure of toughness of fiber reinforced cement composites include the crack mouth opening displacement (CMOD), R-curve analysis, the fictitious crack model (FCM), and various other treatments, all of which model (either implicitly or explicitly) a zone of discontinuous cracking, or process zone, ahead of the ad-

vancing crack. These approaches provide fracture parameters which are, at least, dependent on the fiber content, whereas the LEFM parameters (G_c or K_{Ic}) are most often insensitive to fiber content. It might be added here that, while the J integral has often been used to describe these systems, theoretically it cannot be applied to composite systems such as fiber reinforced concrete, where there is substantial stress relaxation in microcracked region in the vicinity of the crack tip.

In the investigation of the fiber-crack interactions using fracture mechanics concepts, three distinct issues must be considered:

- (1) Crack Suppression: This is the increase in stress, due to the presence of the fibers, required for crack initiation (i.e., the increase in the first-crack stress).
- (2) Crack Stabilization: This refers to the arrest of the cracks already generated, which have begun to propagate across the fibers.
- (3) Fiber-matrix Debonding: This process can be modelled as crack propagation along the fiber-matrix interface.

Crack Suppression

Romualdi and Batson [57] were the first to use LEFM concepts to analyze the mechanics of crack suppression in a cement matrix induced by the presence of fibers. In the absence of any cracks, the extensions of both the matrix and the fiber under tensile loading are the same. However, when cracks are present, the matrix tends to extend more than the fibers, because of the stress concentrations just ahead of the crack tip. The fibers oppose this tendency. Through interfacial shear bond stresses, they apply 'pinching forces' which effectively reduce the stress intensity factor at the crack tip. As a result, higher applied stresses are now required to produce a stress field ahead of the crack tip such that the maximum stress exceeds the critical stress intensity factor, K_{Ic} , of the cement matrix. The shear bond stress distribution which causes this pinching effect is shown in Figure 2.11; this permits a determination of the contribution of neighboring fibers to the stress intensity factor. This work led to the introduction of the concept of the spacing factor, S ; the stress required to cause matrix cracking was found to be inversely proportional to S .

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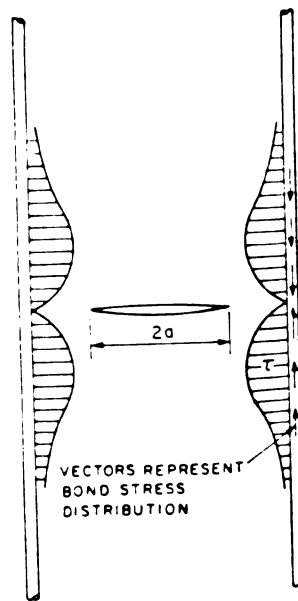


Figure 2.11 The ‘Pinching Effect’ and Interfacial Shear Stress Distribution Predicted by Romualdi and Batson in the Arrest of Crack Propagation by Fibers [57].

Romualdi and Mandel [58] calculated the effective spacing factor in a 3-dimensional random short fiber reinforced concrete:

$$S = 2.76r(1/V_f)^{1/2} \dots\dots\dots(\text{Equation 2.5})$$

They used this relationship to demonstrate the validity of the spacing factor concept for predicting the improvement in first crack stress due to the presence of fibers, as shown in Figure 2.12. A number of other numerical expressions for the spacing factor have since been developed, generally by considering the distances between fibers crossing a given plane in the composite [58]. Though the various spacing equations are similar in form, they do not properly account for the chief geometrical factors which define a fiber, i.e., length and diameter.

There are a number of limitations to the application of spacing factor equations for predicting first crack strains. For instance, some experimental results considered by Edginton et al. [60] showed considerable deviation from the theoretical predictions of Romualdi et

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al [59]. It appears that in calculations of S , one must account not only for length and diameter, but also for the effects of fiber orientation and the nature of the fiber-matrix bond. Bond has been generally assumed to be 'perfect' [59, 61], which is not realistic for short fiber-cement composites. Swamy et al [61] have suggested the concept of 'effective' spacing, which takes into account modifications due to both geometrical and bond considerations.

An alternative approach to the calculation of the first-crack stress was proposed by Aveston et al [62], based on energy balance considerations. For a crack to form under conditions of a fixed tensile stress, the energy changes to be considered are:

- (1) Work, ΔW , done by the applied stress, since the body length changes.
- (2) Work, γ_{db} , done in debonding the fiber from the matrix. This term can be calculated through fracture mechanics analysis of fiber-matrix debonding assuming that the debonding energy at the fiber-matrix interface, G_{db} , is less than or equal to the surface energy, γ_m , of the matrix.
- (3) Work, U_s , done by frictional slip after debonding.
- (4) The reduction in elastic strain energy of the fibers after matrix cracking, ΔU_m .
- (5) The increase in elastic strain energy of the fibers after matrix cracking, ΔU_f , due to load transfer from the cracked matrix to the fiber.

Hence, if the work expended in creating a new crack (i.e., the surface energy of the crack area) is γ_m , then a crack will only form if [55]:

$$2\gamma_m V_m + \gamma_{db} + U_s + \Delta U_f \leq \Delta W + \Delta U_m \dots\dots\dots \text{Equation 2.6}$$

On this basis, the first cracking strain of the matrix is:

$$\epsilon_{mu} = (12 \tau_{fu} \gamma_m E_f V_f^2) / (E_c E_m^2 r V_m) \dots\dots\dots \text{Equation 2.7}$$

Equation 2.7 predicts an effective increase in the matrix cracking strain for a high fiber volume content, a high interfacial frictional shear strength, and a small fiber diameter. In essence, this is another way of predicting the degree of crack suppression (i.e., the increase in first-crack stress), in accordance with the spacing factor concept. A significant increase in the matrix cracking strain should occur with well-bonded, small-diameter filaments. This has been observed with asbestos and glass fibers, which generally consist of filaments (often in bundles) less than $\sim 15 \mu m$ in diameter.

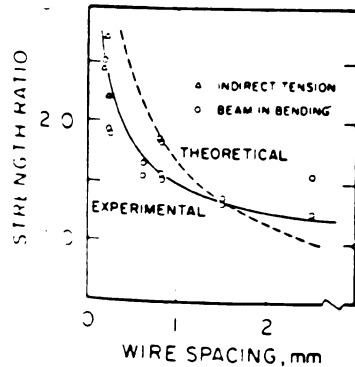


Figure 2.12 Effect of Fiber Spacing on First Crack Stress Ratio [61].

Crack Stabilization

Once first cracking has taken place in the brittle matrix, fibers serve to inhibit unstable crack propagation. At this stage, the cracking patterns are complex, with discontinuous microcracks present ahead of the principal crack. This can be deduced from various analytical models, and has also been observed microscopically (by Bentur and Diamond [63] in Figure 2.13). Thus, in cracked composite, it is difficult to define the 'true' crack tip. The simplistic definition of a traction-free crack (as assumed in LEFM) is not applicable to FRC. Stress is transferred across the crack by a variety of mechanisms, as can be seen from the idealization of a crack proposed by Wecharantana and Shah [64] in Figure 2.14. Three distinct zones can be identified [55]:

- (1) Traction Free Zone:
- (2) Fiber bridging zone, in which stress is transferred by frictional slip of fibers; and
- (3) Matrix process zone, containing microcracks, but with enough continuity and aggregate interlock to transfer some stress in the matrix itself.

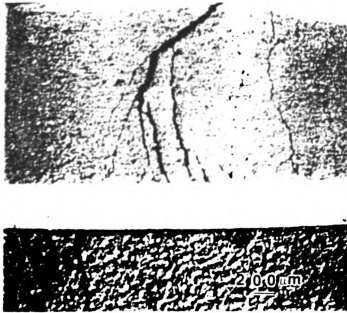


Figure 2.13 Complex Crack Patterns at the Interaction of an Advancing Crack and a Fiber Lying in its Path [63].

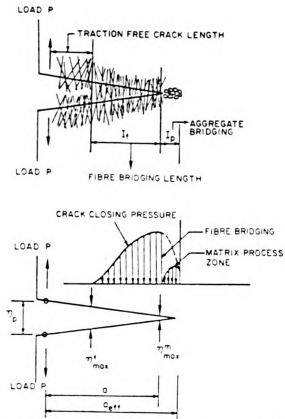


Figure 2.14 Idealized Representation of an Advancing Crack and the Stress Field Around it, in a Fiber Reinforced Cement [64].

Many of the analytical treatments of these effects involve either the assumption of specific stress field around the apparent crack tip, or the consideration of a traction free crack surface which is subjected to a closing pressure [65], as shown in Figure 2.15.

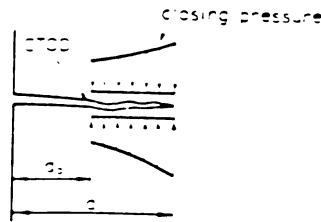


Figure 2.15 Schematic Description of a Traction Free Crack with a Closing Pressure, to Model the Fracture Behavior of Fiber Reinforced Cement [60].

There are, in addition, a number of models which take different approaches to fracture mechanics modeling of cement composites. Bazant and his co-workers have developed a smeared crack model, in which fracture is modelled as a blunt smeared crack band [66]. The fracture properties are characterized by three parameters: fracture energy, uniaxial strength, and width of the crack band. This approach lends itself particularly well to computer-based finite element modelling of cracks. For very large structures, this theory becomes equivalent to the LEFM approach. For smaller structures, however, the theory predicts a lower critical strain energy release rate, because the fracture process zone can not develop fully. This theory has been shown to provide a good fit to experimental data from the literature.

Hillerborg [67] has developed the fictitious crack model to describe the fracture of both plain and fiber reinforced concretes. In this model, the deformation of a specimen is given in terms of two diagrams:

- (1) The stress-strain (σ versus ϵ) diagram (including the unloading branch);
- (2) The stress deformation (σ versus w) diagram for the fracture zone itself.

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The σ versus w curve gives, the additional deformation in a test specimen due to the presence of a damage zone. Moreover, the area under the σ - w curve (called the fracture energy) equals the energy absorbed per unit (projected) area during the fracture process due to the additional deformation of the process zone.

However, none of the non-linear fracture mechanics approaches described above appears to provide a truly fundamental fracture parameter, independent of specimen geometry and loading conditions. It is also difficult to interpret the physical significance of the stress distributions ahead of the crack tip which are assumed (or implied) in various models. It is, therefore, difficult to disagree with Majumdar and Walton [68], who conclude that 'for composites which display the phenomenon of multiple cracking and rising stress-strain relationships it would appear that fracture mechanics approaches currently under consideration will have little prospect of success in producing parameters that will be useful in design' [55].

Fiber-Matrix Debonding

A fracture mechanics approach has also been applied to the problems of fiber debonding and pull-out, as an alternative to the treatment based on the analysis of elastic and frictional shear stresses. The object is to develop material parameters to account for debonding which are more reliable and easier to evaluate experimentally than the interfacial shear bond strength values. In this treatment, it is assumed that the debonded region is traction free ($\tau_f = 0$ in Figure 2.16), and this zone is treated as an interfacial crack of length b . Using the classical Griffith theory (of LEFM), the conditions leading to the propagation of this crack, and to spontaneous debonding, can be calculated.

Outwater and Murphy [69] calculated the fiber tensile stress (σ) required for catastrophic debonding as

$$\sigma = [(8 E_f G_{db})^{1/2}] / d \dots \dots \dots \text{Equation 2.8}$$

where G_{db} is the energy required to debond a unit surface area of fiber, and E_f and d are fiber elastic modulus and diameter, respectively.

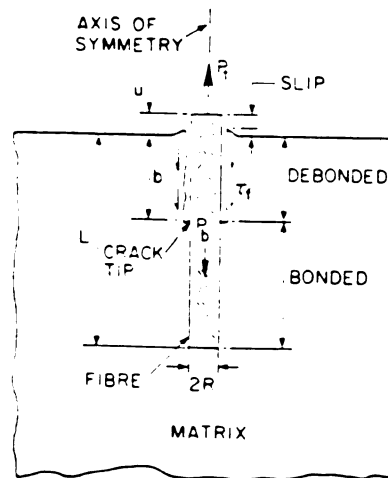


Figure 2.16 Schematic Description of the Model Used to Consider the Pullout Problem in Terms of Fracture Mechanics Concepts, With a Propagating Debonding Crack of Length b [65].

The above analysis considers only the energy balance in the fiber itself. Subsequently, a solution was developed which takes into account the compliance of the entire pull-out system. Morrison et al [70] further extended the analysis by taking into account also the frictional resistance in the debonded zone, which is the more realistic case for fiber reinforced cement composites. The critical strain energy release rates for debonding calculated by Morrison et al [70], that is 2.5 N/m, is similar to those calculated by Mandel et al, which is 4-4.7 N/m. these values are, however, lower than typical values of the critical strain energy release rate of plain mortars, which are typically 5-12 N/m. Thus, for a crack to follow the path of least resistance, it should propagate along the interface rather than through the matrix.

This fracture mechanics approach tends to confirm the conclusions reached previously. That is, the interface in fiber-cement composites is relatively weak. This leads to preferential crack propagation along the fiber-matrix interface.

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Theories Applied to Cellulose Fiber Reinforced Cement

Composite materials approach (rule of mixture) has been used by several researchers to predict the tensile, flexural strength and other characteristics of cellulose fiber reinforced cement composites [71]. In this approach, strength is assumed to be the sum of the effects of the matrix and the fibers; the fiber contribution is governed by pull-out (assuming that fiber-pull out dominates the behavior) and is a function of $\tau l/d$, while the matrix contribution is a function of the strength of a void free matrix, σ_{mo} , multiplied by its solid content, $(1-V_o)$. Therefore,

For tensile strength:

$$\sigma_{cu} = \sigma_{mo}(1-V_o)V_m + 2\eta\tau V_f l/d \dots \dots \dots (\text{Equation 2.9})$$

For flexural strength:

$$\sigma_b = \alpha/\beta (\sigma_b)_{mo} (1-V_o)V_m + 2\eta\alpha\tau V_f l/d \dots \dots \dots (\text{Equation 2.10})$$

Similarly, the modulus of elasticity in tension, E_t , and bending E_b , is:

$$E_b, E_t = E_{mo}(1-V_o)V_m + \eta E_f V_f \dots \dots \dots (\text{Equation 2.11})$$

To use Equations 2.9 through 2.11 for predicting strength and E modulus of cellulose fiber cement composites, it is necessary to determine the constants η , α , β , the interfacial bond strength (τ), the fiber aspect ratio (l/d) and the void free matrix properties, E_{mo} and σ_{mo} .

The efficiency factor (η) can be taken as 0.41 after Romualdi and Mandel[58]. The values of α and β , which are the ratios of bending strength to tensile strength of the composite and matrix, respectively, have been found to be 2.96 and 2.81, respectively. The Properties of the void free matrix have been determined in separate tests of the matrix only, with a void content of 23%, from which tensile strength, bending strength and modulus of elasticity for $V_o=0$ were calculated. Substituting these parameters in Equation (2.9-2.11) permits the prediction of the mechanical properties as a function of fiber content.

As shown in Figure 2.17, these predictions agree satisfactorily with experimental results. While these theories predict a continuous increase in tensile strength and bending strength with increasing fiber mass fraction, the experimental results show no strength improvement beyond 6% fiber mass fraction. This is probably a consequence of the relatively large void fractions at larger fiber mass fraction, which cause further reductions in the interfa-

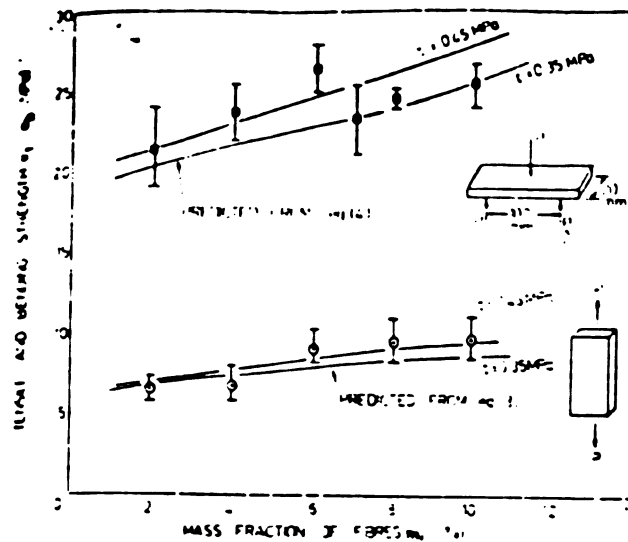
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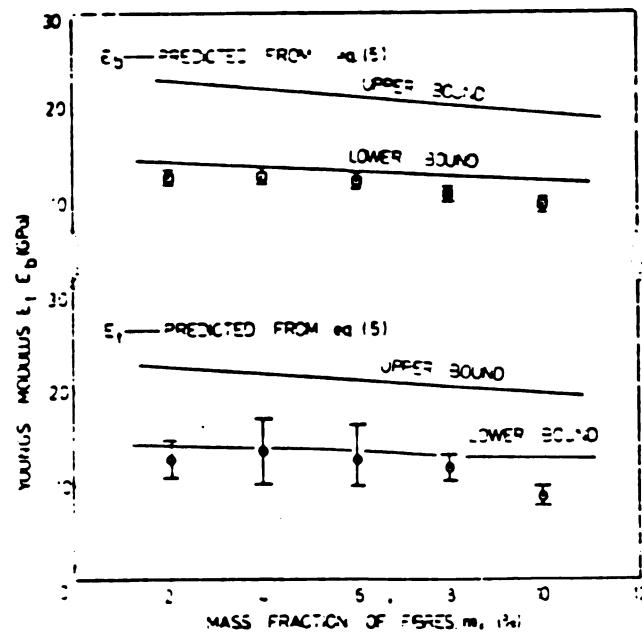
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Figure 2.
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cial bond strength below 0.35 MPa (50 psi). The large void fractions are also responsible for the low moduli in bending and in tension for fiber mass fractions greater than 0.08.



a. Variation of Tensile and Bend Strengths With Mass Fraction of Fibers



b. Variation of Young's Modulus in Bending and in Tension With Mass Fraction of Fibers

Figure 2.17 Experimental Vs. Theoretical Predictions of Different Properties of Cellulose Fiber Cement Composites [71].

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Das Gupta et al [72] applied a somewhat different composite material model to natural fiber cement composites. They included in their calculation the effects of fiber orientation and length, and developed different relations for fibers with lengths greater or shorter than the critical length. Their model was used to analyze pastes reinforced with short randomly distributed coir fibers. The average fiber diameter was 0.119 mm (0.004 in) and the fiber matrix bond determined by pull-out tests was found to be 1.5 MPa (220 psi). Comparison of their experimental and analytical results is provided in Figure 2.18. The agreement is reasonably good, up to a fiber content of 5%, but the model does not predict the decline beyond this fiber volume, which is probably associated with a reduction in bond and matrix strength due to poor compaction. Andonian et al [71] have reported on efforts to predict these effects by considering the influence of an increase in void content on the contribution of the matrix. Their data does not show a marked reduction in properties at high fiber contents, probably due to a different method of preparation. It should be noted that, while Andonian et al [71] assumed fiber pull out, dominating the behavior analysis of the fractured surfaces in a later work suggested that a large portion of fibers may have failed by fiber fracture [73].

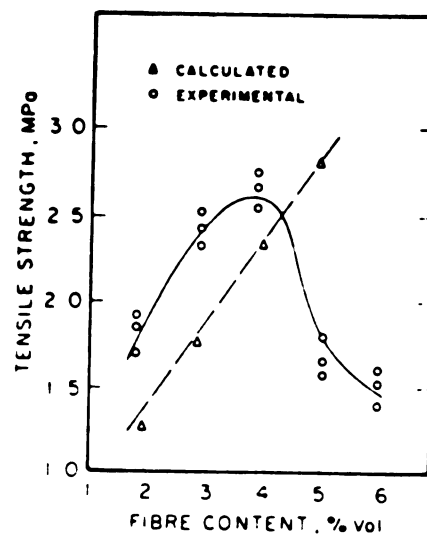


Figure 2.18 Relations Between Experimental and Calculated Tensile Strength in Coir Fiber Reinforced Cement[[72].

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Fracture mechanics concepts have also been applied to predict the behavior of cellulose fiber reinforced cement composites. Fracture parameters were found to be dependent on the moisture content, with wet cellulose fiber cement composites having higher toughness values (Mai et al [73]; and Mindess et al [74]). Wet composites were also found to be notch insensitive, suggesting that LEFM cannot adequately model the behavior of such composites. This was probably the result of the cracking mode, in which the crack path was tortuous, with some fibers failing by pull-out.

Hughes and Hannant [75] examined the effects of moisture on the first crack stress, by considering the reduction in the modulus of elasticity of the wet fiber, and using the concepts of crack arrest when the fiber spacing is less than the critical flaw size. Their results, assuming a fiber modulus of 4 and 40 GPa (580 and 5800 ksi) for wet and dry conditions, respectively, are presented in Table 2. 4. If the bond is assumed to remain constant once wet composites are dried, at or about 0.5 MPa (70 psi), (as suggested by Andonian et al [75]), the matrix failure strain should increase upon drying. If the bond increases from 0.5 MPa (70 psi) to an assumed value of 2.0 MPa, the matrix failure strain would be expected to double. The increase in bond on drying may be a result of hydrogen bonding, which is more readily generated in dry state. The transition of the fracture mode from fiber fracture in the dry state to fiber pull out in the wet state may be associated with the effects calculated in Table 2.4, in which a large stress can be developed in the fiber prior to matrix cracking, leading to the rupture of fibers failure matrix fails.

Mia et al [73] have considered slow stable crack growth (based on LEFM) as a prominent feature of the fracture behavior of cellulose fiber cement. Double cantilever beam specimens with side grooves were used to obtain resistance curves. Fiber reinforced composites containing 8% mass fraction of bleached fibers were tested in wet and dry conditions. Figure 2.19 shows typical load-displacement curves during slow crack growth in a dry and a wet sample of cellulose fiber cement. The load at which the load-deflection curve deviates from linearity is taken as the onset of crack growth and is used to calculate G and K values for the construction of slow crack growth resistance curve. It is apparent from Figure 2.19 that, upon unloading to zero load after crack extension, there is a permanent deformation (δ_r).

Table 2
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* Assur

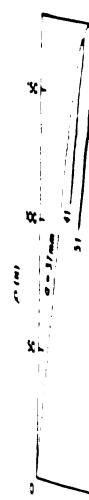
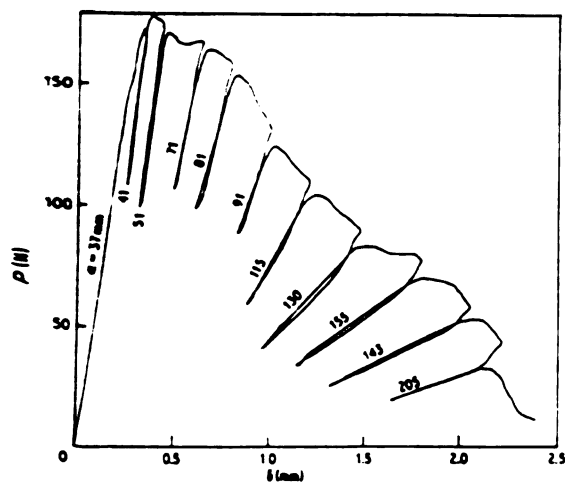


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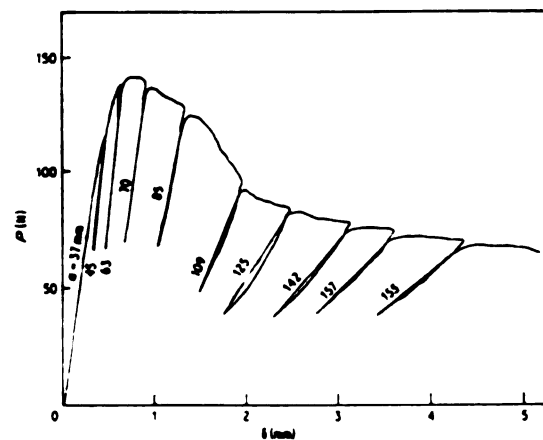
Table 2.4 Effect of Moisture Content and the Resulting Assumed Change in the Fiber Modulus of Elasticity on the Calculated Matrix Failure Strain and the Fiber Stress Across a Stable Flaw [73]

Moist. State	Fiber Modulus (GPa) *	Bond Strength (MPa) *	Matrix Failure Strain (%) **	Max. Fiber Stress Across a Stable Flaw (MPa) **
Wet	4	0.5	0.074	27
Dry	40	0.5	0.106	96
Dry	40	2.0	0.156	172

* Assumed ** Calculated; The unreinforced matrix strain is assumed to be 0.05%.



(a) Dry



(b) Wet

Figure 2.19 Typical Load (P) Against Displacement (δ) Records For Crack Propagation in Cellulose Fiber Cements[73].

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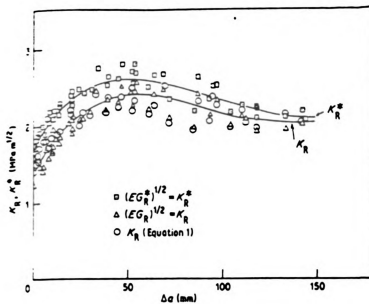
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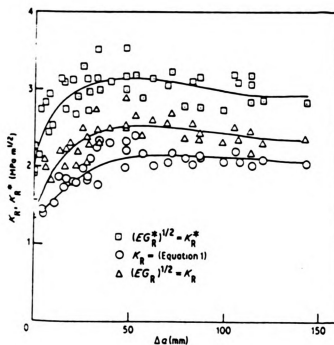
Slow crack resistance curves for the dry and wet composites are shown in the Figure 2.20. K_R values predicted from the analytical expression

$$K_R^2 = [(12 P^2 a^2)/(13BH^3)] [1 + 1.32 (H/a) + .532 (H/a)^2] \dots\dots\dots(\text{Equation 2.12})$$

are also shown in Figure 2.20. For the dry samples we must have K_R (Equation 2.12) the same as those from $K_R = (EG_R)^{1/2}$ since they both assume linear elasticity to be obeyed by the material. Also as δr is small, $K_R^* = (KG_R^*)^{1/2}$ should be approximately equal to the other two K_R calculations. These results are borne out by the similar K_R against Δa results given in Figure 2.20, even though different equations are used. For the wet samples given in the Figure 2.20, all these K_R^* curves are different. The K_R curve based on the analytical expression by Equation 2.12 gives the worst results and the one based on G_R without considering residual displacement in $K_R = (EG_R)^{1/2}$, still underestimates the true K_R^* results given by the upper most curve. It was concluded in this study that for dry samples neglecting residual displacement does not significantly alter the K_R and G_R curves. However, for wet composites the residual displacements are large and must be included in calculations of the true K_R^* and G_R^* values. Crack growth resistance curve is much higher for wet than for dry composites due to the different deformation behavior of the fiber in the two states (Figure 2.20).



(a) Dry Samples



(b) Wet Samples

Figure 2.20 Crack Growth Resistance (K) Plotted Against Crack Extension (Δa) [73].

CHAPTER 3

DETERMINATION OF INFLUENTIAL VARIABLES IN THE PROCESSING OF RECYCLED CELLULOSE FIBER-CEMENT

3.1 INTRODUCTION

Cellulose fiber reinforced cement composites are quite different in mix proportioning and processing from normal concrete or mortar. The process includes beating and refinement of fibers in a slurry, and mixing of all constituents in water. The slurry has a low solid content in order to uniformly disperse the fibers; vacuum is therefore applied to extract the excess water. The composite is finally compacted under pressure, and curing is usually reached under high-pressure steam effects for accelerated strength gain in prefabrication facilities.

In a detailed break down of this production process, eleven proportioning / processing variables were distinguished as potentially influential in determining the end product (wastepaper fiber-cement composite) qualities. The influential variables detected at this stage of research are to be optimized in the next stage of the project.

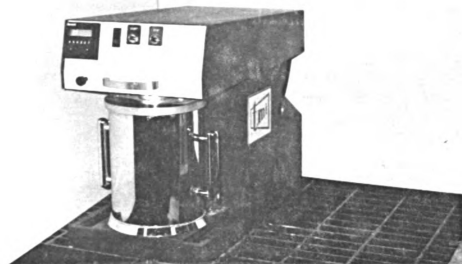
A comprehensive program was designed in order to identify those variables with statistically significant effects on the flexural performance of cellulose fiber reinforced cement composites. A 1/64 fractional factorial design of experiments was used with eleven variables. This design consisted of a total of 32 different combinations of the proportioning / processing variables, each considered at two levels. The control composite with 100% virgin cellulose fibers was also considered in this phase of study. Three flexural tests were conducted on specimens prepared from each mix, and the average values were used in statistical analyses.

3.2 VARIABLES AND EXPERIMENTAL DESIGN

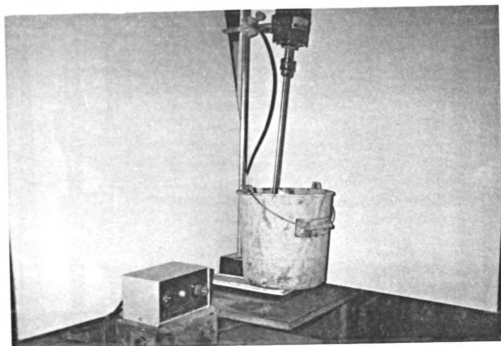
Recycled Fiber-cement composites are manufactured by: (1) Refinement (beating) of recycled wastepaper fibers to expose microfibrils on the fiber surfaces, and make the fibers discrete and compatible with cement. Beating is done using a slurry containing 5% by weight of fibers. A laboratory scale pulp disintegrator (TMI refiner) was used at a speed of 3000 rpm. The beating time ranged from 5 to 10 minutes; (2) Proportioning the fibers (recycled & virgin), sand, flocculating agent and cement; (3) Mixing the ingredients in water to produce a slurry of 20% solids; a high-speed mixer is used to achieve a uniform dispersion of recycled cellulose fibers and other mix ingredients in this slurry. Flocculating agent is the last solid constituent to be added, which improves the binding of cement particles to cellulose fibers and controls the escape of cement particles during vacuum dewatering. The mixing time needed to uniformly disperse mix ingredients is approximately 10 minutes; (4) Vacuum dewatering of the slurry to extract the excess water; the extraction of water is actually performed in two stages. First, the excess water on top of the settled slurry is removed, and then the settled slurry is put onto a vacuum box 305 mm by 305 mm (12 in by 12 in) in planer dimensions. The slurry is evenly spread onto the screen of vacuum box in layers of about 2.5 mm (0.1 in) thick, and the vacuum varied from 127 mm to 254 mm (5 in to 10 in) of mercury. It takes a build up of four to five layers during the vacuum application process in order to make the panel (about 10 mm (0.4 in) thick); (5) Compaction under pressure to produce a dense composite with even surface. A 110 KN (12 ton) hydraulic press was used for the compaction of 305 mm x 305 mm x 10 mm (12 in x 12 in x 0.4 in) panel. Compaction pressure of 0.7 to 1.4 MPa (100 to 200 psi) was used.; and (6) Curing of the composite.

Different components of this process are presented in Figure 3.1 for a laboratory scale production facility.

A total of 11 key variables (factors) defining this production process were selected to be investigated in this experimental program. These variables are: (1) recycled fiber source; (2) fiber mass fraction; (3) fiber beating level; (4) substitution level of virgin fibers with recycled ones; (5) sand/binder ratio; (6) maximum particle size of sand; (7) silica fume/binder ratio; (8) flocculating agent/binder ratio; (9) vacuum level; (10) compaction pressure; and (11) curing condition. Each factor was considered at two levels in a (1/64) fractional factorial design of experiments (Table 3.1).

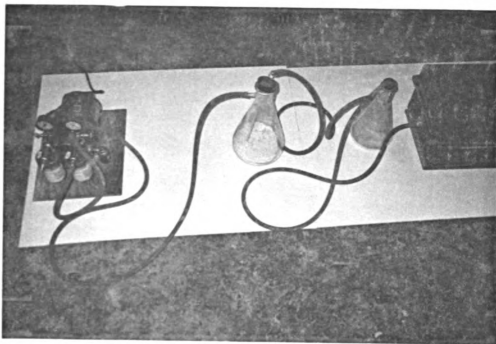


(a) Fiber Refinement

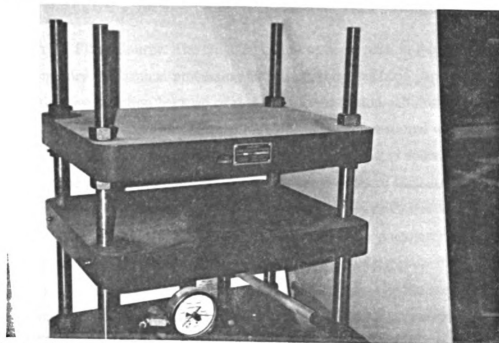


(b) Mixing

Figure 3.1 Components of the Laboratory Scale Manufacturing Process.



(c) Vacuum Dewatering



(d) Compaction

Figure 3.1 (Cont'd) Components of the Laboratory Scale Manufacturing Process.



(e) High-Pressure Steam Curing

Figure 3.1 (Cont'd) Components of the Laboratory Scale Manufacturing Process.

3.2.1 Recycled Fiber Source: The recycled cellulose fibers used in this investigation are made through dry mechanical processing of wastepaper. Magazine papers were selected for use in this investigation because of their high level of non-cellulosic materials (e.g., clay, latex, etc.); these impurities cause difficulties in the conventional wet method of deriving cellulose fibers from waste magazine paper. The wastepaper source and specifics of the dry recycling process were thought to be possible sources of variations in wastepaper fiber-cement composite qualities. A number of sources of dry processed wastepaper fibers were surveyed and finally two sources were selected which used magazine papers and different dry processing techniques. A detailed presentation of the properties of fibers obtained from these sources is presented later in this chapter. Interfibre recycled fibers are manufactured through pulverization process by different sizes equipment whereas American Fillers fibers are manufactured by hammer mill grinding process.

3.2.2 Fiber Mass Fraction. This is a key variable of the investigation. Fiber mass fraction is defined in this investigation as the mass ratio of fibers to the total solid constituents of the composite. Two fiber mass fractions, 5 and 8%, were used to study the effects on the

composite material qualities.

3.2.3 Fiber Beating Level. The beating of fibers in a water slurry exposes microfibrils on fiber surfaces. This helps improve the filtering action of fibers (to avoid cement loss during dewatering), and also enhances the mechanical bonding of fibers to cement-based matrices. Fiber beating level is measured in Canadian Standard Freeness (CSF), which decreases as fiber beating increases (Tappi 1207). The two levels of CSF presented in Table 3.1 refer to unbeaten (as received) and beaten fibers (beating was continued for 10 minutes in this case). Unbeaten fibers from source “a” and “b” had CSF values of 620 and 580, respectively. After beating the corresponding CSF values were 520 and 480, respectively.

3.2.4 Fiber Substitution Level. The next variable in Table 3.1 is fiber substitution level, which has two levels of 50% and 100%; at the 50% level, 50% of fibers (by weight) were virgin cellulose fibers and the remaining 50% were recycled fibers. At the 100% substitution level, the whole fiber content was recycled magazine fibers.

3.2.5 Sand size. Gradation of the silica sands used is illustrated in Table 3.2. Maximum particle sizes of the two silica sands were 50 μm and 600 μm (2×10^{-6} in to 24×10^{-6} in).

3.2.6 Sand Content. Silica sand was used in this study at sand/binder ratios (by weight) of 0.25 and 0.75.

3.2.7. Silica Fume. Cement substitution with silica fume at 0 and 10% was also considered (see Table 3.3 for the physical and chemical properties of the Type I Portland cement and Silica fume used in this investigation). Silica fume is a by-product from the reduction of high-purity quartz with coal in electric arc furnaces in the production of silicon and silicon alloys. The fineness and pozzolanic reactivity of silica fume make it highly effective in enhancing the density and chemical stability of the bulk of cement paste and particularly the interface zones. The consumption of calcium hydroxide (a relatively unstable cement hydration product), the reduction in the alkalinity of cement pore water, and reducing permeability of the matrix are some key mechanisms through which silica fume could positively improve the long-term stability of cellulose fiber-cement composites.

3.2.8 Flocculating Agent. Flocculating agent was used to improve the binding of cement particles to cellulose fibers and to control escape of cement particles during the vacuum dewatering process. The dosage rates of the flocculent considered in this experimental

study were 0.2% and 0.6% of binder (by weight). NALCO Chemical Company's product "NALCLEAR 9798 Pulv Flocculent" was used. It is an acrylamide acrylate polymer. Its specific gravity is 0.75, and its pH value at 0.2% is 8.2.

3.29 Vacuum Level. The vacuum level was also studied as a variable; vacuum was applied at 5 in and 10 in (127 mm and 254 mm) of mercury to the composite to extract the excess water from the slurry.

3.2.10. Compaction Pressure. The compaction pressure was another variable considered; the thin cement sheets were pressed under 0.7 Mpa and 1.4 Mpa (100 psi and 200 psi) of pressure.

3.2.11 Curing Condition. Two curing conditions were considered: moist curing ("a") and high pressure steam curing ("b"). In moist curing, the composites were stored in a moist room for 7 days followed by 21 days of air drying in the laboratory. In high-pressure curing, an autoclave was used to cure the specimens at a pressure of 0.86 MPa (125 psi) for 8 hours, with specimens subsequently stored in the laboratory environment up to the test age of 28 days.

High-pressure steam curing (autoclaving) has been developed for several reasons [76]. Firstly, it increases the rate of hydration reactions; curing in steam at 0.86 MPa (125 psi) for 8 hours is roughly equivalent to 28 days of moist curing at normal temperature. This permits a high rate of turn-over in the production of precast products. Secondly, products can be made which are superior to those of normally cured concrete; chemical resistance and dimensional stability can be improved. Lastly, there is a possibility of replacing the cement partly or wholly by waste materials, which are unreactive at ordinary temperatures but which possess cementing properties at higher temperatures. The adoption of steam curing (high-pressure or atmospheric pressure) in commercial practice is determined primarily by economic considerations. The development of strength in a high-pressure steam cured product depends at least in part, on the reaction between cement and the fine silica. The initial setting reactions, and phases and subsequent behavior of the product in the case of autoclaved cement-silica are more stable than those occurring at ordinary temperatures. The primary reaction at 85-200° C is probably always the hydration of cement to give tobermorite gel and Calcium hydroxide [76]. Addition of small amounts of ground silica contributes to suppress the formation of calcium hydroxide. So autoclaving has a number

of positive effects; namely reduction of calcium hydroxide, and formation of better crystallized calcium silicate hydrate [76].

The 1/64 fractional factorial design of experiments in this phase of the study (Table 3.1) was completely randomized. All main effects were clear of 2-factor interactions, but due to the fractional nature of design, 2-factor interactions were compounded with one another; in other words, this experimental design reveals the effects of all variables on the composite material performance, but can not provide any information on possible interactions between different variables. The resulting specimens were tested for flexural performance (strength, toughness, and initial stiffness). The flexural test set-up (Japanese Standard JCI SF4 [77] and ASTM C1186 [78]) is presented in Figure 3.3.

Table 3.1: Fractional Factorial Design of Experiments

Experiment	Run Order	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
Variables	Units																
Fiber Source	name	a	a	a	b	b	a	a	a	b	a	a	a	b	b	b	b
Fiber Mass Fraction	percent	5	5	5	8	5	8	5	8	5	5	8	8	8	8	8	5
Fiber Beating Level	CSF	b	a	a	b	b	a	a	b	b	a	b	a	b	a	a	b
Fiber Substitution Level	percent	100	50	100	100	50	50	100	50	50	50	100	100	50	100	100	100
Sand/Binder	weight ratio	.25	.25	.25	.75	.75	.75	.75	.75	.25	.75	.75	.25	.75	.25	.75	.75
Sand Maximum Size	µm	600	50	600	600	50	600	50	50	50	50	50	600	600	50	50	600
Silica Fume/Binder	weight ratio	0	0	.1	.1	.1	.1	.1	0	.1	0	.1	0	0	0	0	0
Floc. Agent/binder	weight ratio	.006	.002	.006	.006	.006	.002	.002	.002	.002	.006	.006	.002	.002	.002	.006	.002
Vacuum Level	inch of mercury	5	5	10	10	5	10	5	5	10	10	10	10	5	10	5	10
Compaction Pressure	psi	100	200	200	200	100	200	100	100	100	100	100	200	100	100	100	100
Curing Condition	name	a	b	a	a	b	a	b	a	a	a	b	a	b	b	a	b

Table 3.1 (Cont'd): Fractional Factorial Design of Experiments

Experiment	Run Order	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	control
Variables	Units																	
Fiber Source	name	a	a	b	b	b	b	b	b	b	a	a	b	b	b	a	a	ssk
Fiber Mass Fraction	percent	8	8	5	5	8	8	5	5	8	5	8	5	8	5	5	8	8
Fiber Beating Level	CSF	b	a	a	a	b	a	b	a	b	b	b	b	a	a	b	a	a
Fiber Substitution Level	percent	100	50	50	100	100	50	100	100	50	50	50	100	50	50	50	100	0
Sand/binder	weight ratio	.25	.25	.25	.75	.25	.25	.25	.25	.25	.25	.25	.75	.75	.75	.75	.75	.75
Sand Maximum Size	µm	50	600	50	600	600	50	50	600	600	600	50	50	50	600	600	600	50
Silica Fume/Binder	weight ratio	.1	.1	0	.1	.1	.1	0	.1	0	.1	0	0	.1	0	.1	0	.1
Floc. Agent/binder	weight ratio	.002	.002	.002	.002	.002	.006	.006	.006	.006	.002	.006	.002	.002	.006	.006	.006	.006
Vacuum Level	inch of mercury	5	5	5	5	5	5	5	10	10	10	10	10	10	10	5	5	10
Compaction Pressure	psi	100	100	200	100	200	200	200	100	100	200	200	200	200	200	200	200	200
Curing Conditions	name	a	b	a	a	b	a	b	b	a	b	b	a	b	b	a	b	a

Table 3.2: Sand Gradation (ASTM C-371: Percent Retained on Individual Sieves)

Sieve No. (ASTM E11)	Silica Sand	Ground Silica Sand
40	0.2	
50	0.5	
70	22.9	
100	47.1	
140	21.6	
200	7.3	0.1
270	0.4	1.9
325	-	2.4
- 325	-	96.6

Table 3.3 Properties of the Binders

Properties	Binder Type	
	Cement	Silica Fume
CaO	63.24	-
SiO ₂	21.14	96.50
Al ₂ O ₃	5.76	0.15
Fe ₂ O ₃	2.93	0.15
SO ₃	2.46	-
MgO	2.06	0.20
K ₂ O	0.79	0.04
C	-	1.40
Na ₂ O	-	0.20
Surface Area	0.16 m ² /gm	20.25 m ² /gm
Specific Gravity	3.15	2.3
Fineness (% retained in #325 sieve)	10.7	-

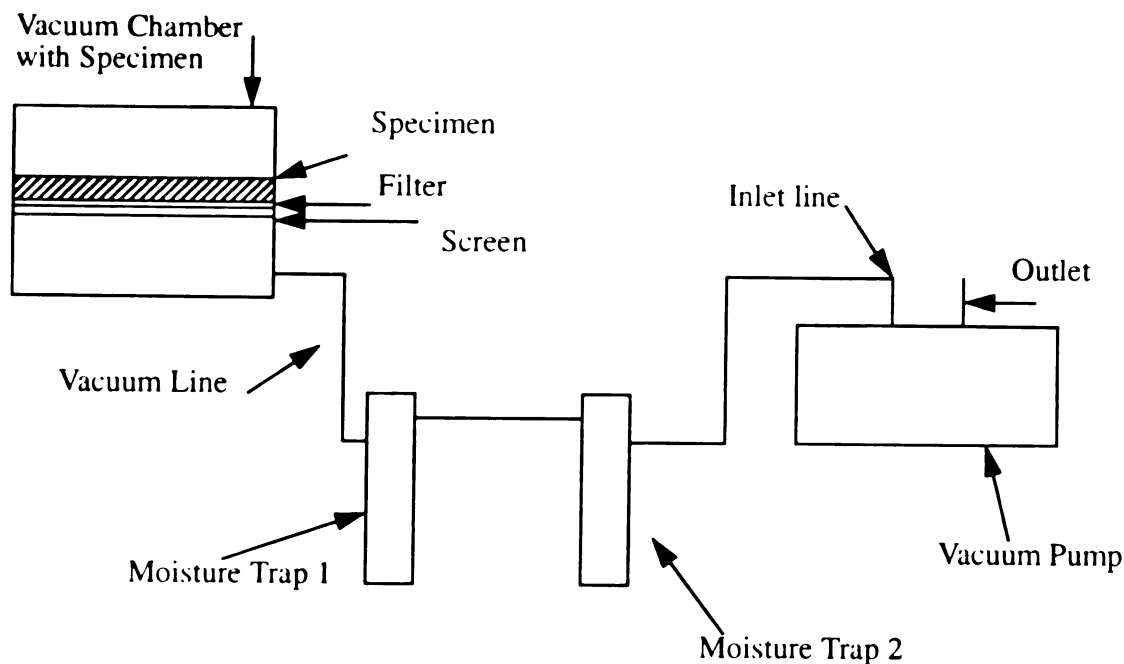


Figure 3.2 Manufacturing Process- Slurry Dewatering.

3.3 RECYLED WASTEPAPER FIBERS

Recycled fibers are derived from recycling of wastepaper (magazine) by dry mechanical processing. American Filler's recycled fibers (fiber "b" in Table 3.1) had 100% magazine paper whereas the recycled fibers from Interfibe (fiber "a" in Table 3.1) was made of wastepaper with 90% magazine paper constituent. Both used dry mechanical processing to disintegrate paper into fibers with increased surface area. These fibers are mainly cellulosic fibers and, being dry processed have a fraction of fine fragments and fine coating materials from the wastepaper (mainly glossy coated magazine paper). Cellulose fibers are derived from wood and in the process of paper making these fibers undergo some modifications. Magazine papers are mostly made from chemical pulp whereas newsprint is made mainly from ground pulp. The fibers used in magazine are a blend of softwood and hardwood to produce desirable properties at minimum cost. Besides fibers, magazine paper also constitutes 20% of its composition as coating materials (clay, processing chemicals, pigment particles, and latex) which impart desirable characteristic to magazine paper. The recycled fibers were analyzed through Scanning Electron Microscope observations, measurement of length distribution, and thermo-gravimetric compositional analysis.

3.3.1 Fiber Length Distribution

Standard equipment used for cellulose fiber length distribution (Kajani FS100 machine) was used to carry out this analysis. This equipment microscopically measures the lengths of all fibers in a sample and gives fiber length ranges in weight (see Table 3.4). The results presented in Table 3.4 confirm that the recycled fibers are shorter than the virgin softwood kraft pulp (Southern Softwood Kraft, SSK). The virgin kraft pulp (SSK) used in this investigation presents a high-quality fiber with desirable (3 mm) length and high reinforcement efficiency for cement applications. The high fine content of recycled fibers may result from the damage to fibers during recycling. The presence of shorter hardwood fibers in recycled wastepaper, and the presence of impurities in wastepaper are also responsible for the high fine content of recycled fibers. Shorter recycled fibers are expected to have lower reinforcement efficiencies; the fines in recycled fibers would act more like fillers than reinforcing fibers. The virgin cellulose fibers had an average length of 3.3 mm (0.13 in) whereas the recycled fibers from Interfibe (fiber “a” in Table 3.1) had an average length of 1.2 mm (0.05 in) and that for American Filler (fiber “b”) had an average length of 1.3 mm (0.051 in). The average values and 95% confidence intervals of the fiber lengths are presented in Figure 3.4.

Table 3.4 Recycled Fiber Length Distribution (Weight%)

Length (mm)	Virgin Cellulose	Recycled Source 1	Recycled Source 2
<0.2	4.16	12.47	1.96
0.2-1.0	16.72	44.43	49.86
1.0-2.0	20.63	27.96	29.63
2.0-3.0	16.56	12.12	15.28
3.0-4.0	12.38	2.62	2.66
4.0-5.0	10.90	0.38	0.51
5.0-6.0	7.55	-	-
6.0-7.0	4.11	-	-
> 7.0	6.95	-	-

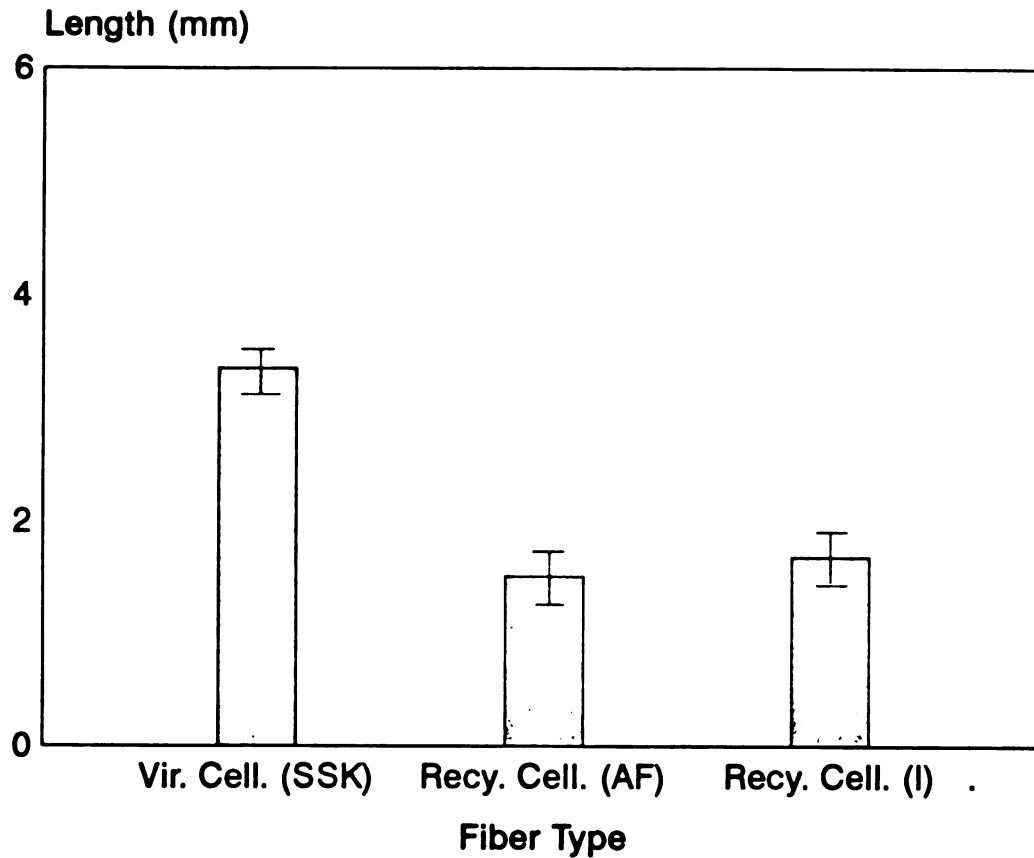


Figure 3.3 Fiber Length Distribution: Average Values and 95% Confidence Intervals.

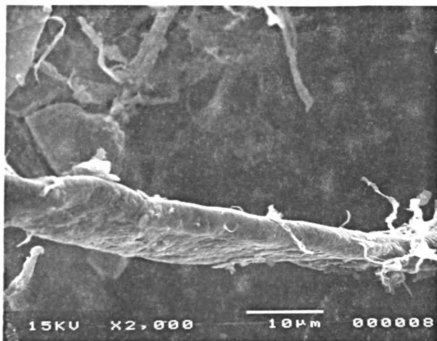
3.3.2 Morphology

The morphology of recycled and virgin fibers was studied under a Scanning Electron Microscope (SEM). The shape of the recycled fibers was observed to be quite different from that of virgin cellulose fibers. Virgin fibers are hollow and cylindrical in shape with a varying diameter. Recycled fibers appear to be flatter and twisted. The surface of the recycled cellulose fibers, as compared to the virgin counterpart, is relatively rough, with microfibrils exposed on the surfaces. Fine clay particles and fiber fragments can also be seen in the micrographs (Figure 3.5)

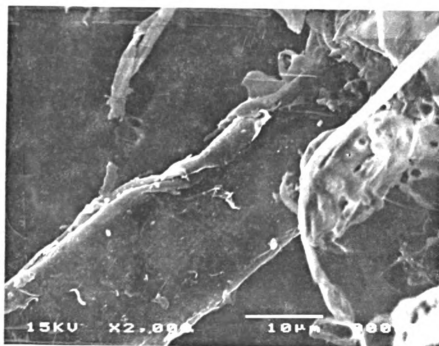
(a)

(b) R

Figur

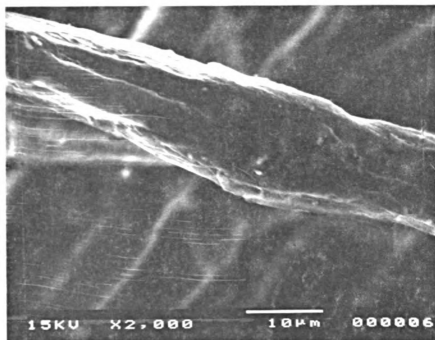


(a) Recycled Fibers (Interfibe, "a")



(b) Recycled Fibers (American Fillers, "b")

Figure 3.4 Scanning Electron Micrographs of Virgin and Recycled Cellulose Fibers.



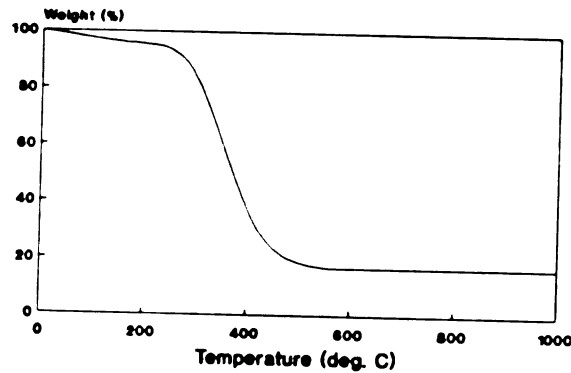
(c) Virgin Cellulose Fibers (SSK)

Figure 3.4 (Cont'd) Scanning Electron Micrographs of Virgin and Recycled Cellulose Fibers

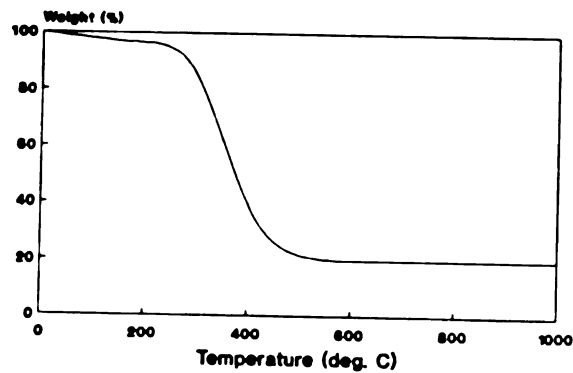
3.3.3 Compositional Analysis

In order to investigate the composition of virgin and recycled fibers, Thermo Gravimetric Analysis (TGA) was carried out. The aim was to investigate and estimate the contents of pure cellulose in these fibers. The rate of heating used was 20°C (68°F) per minute and the weight loss increasing with temperature changes was recorded. Cellulose decomposes at 350°C (660°F). In all cases a sharp weight loss is observed around this temperature. The trends in weight loss are shown in Figure 3.6. From these weight loss curves one may estimate cellulose content in recycled fibers around 67%; one may also approximate lignin content of and 13% (lignin decomposes at 450°C , 840°F). The ash is the fine clay which was used in surface texture of the magazine paper and also the latex and other materials used in the formation of paper. Virgin cellulose fibers had about 90% of cellulose content;

their lignin content was small (probably because magazine paper uses chemical pulps with low lignin content), and the ash content was also small, as expected, due to the low levels of impurities in virgin fibers

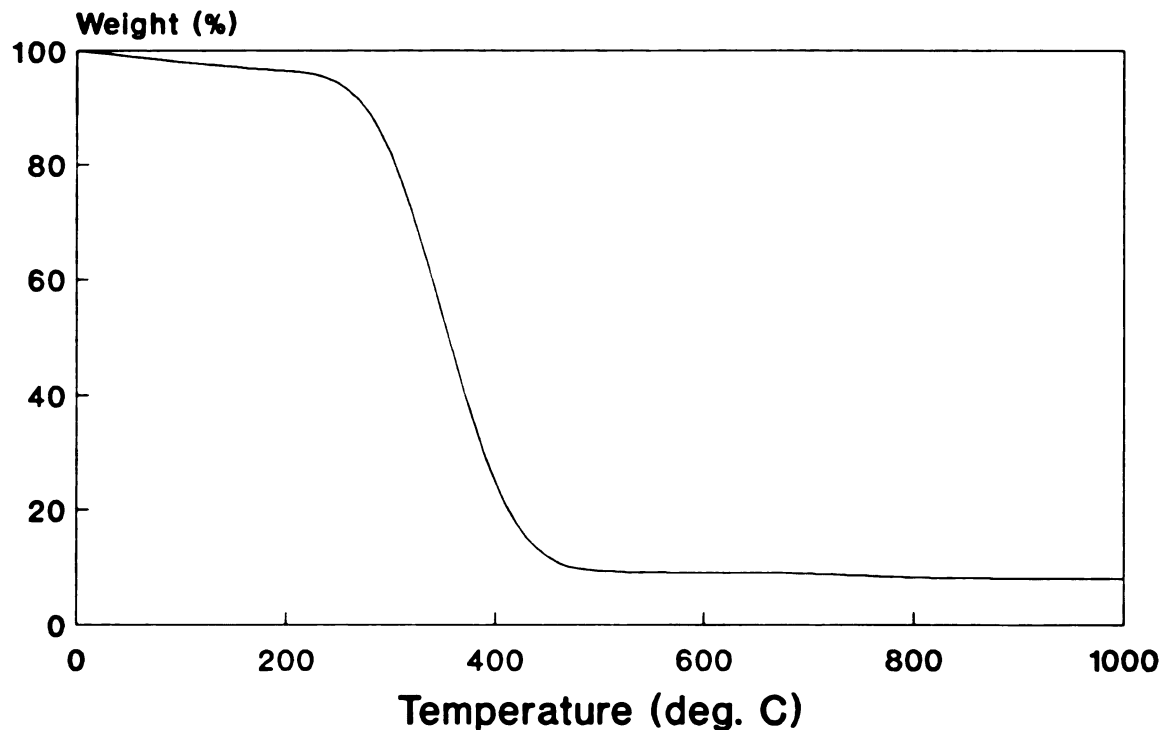


(a) Recycled Fibers (Interfibe, "a")



(b) Recycled Fibers (American Fillers, "b")

Figure 3.5 Fiber Compositional Analysis by Thermogravimetry.



(c) Virgin Cellulose Fibers

Figure 3.6 (Cont'd) Fiber Compositional Analysis by Thermogravimetry.

3.3.4 Canadian Standard Freeness and Fiber Refinement. Freeness test is an empirical process that gives an arbitrary measure of the rate at which a suspension of 3 gm (0.048 oz.) of pulp in one liter of water may be drained [79]. The result depends mainly upon the quantity of debris present, the degree of fibrillation of fibers, their flexibility and fineness. The higher the value the Canadian Standard Freeness (CSF) the lesser would be the refinement and vice versa. Virgin cellulose without refinement had a CSF value of 700, whereas the recycled fibers without refinement had a CSF value of 620 in the case of Interfibe ("a") and 580 in the case of the American Filler (b") fibers. After refinement these values dropped to 600, 520, and 480 respectively.

3.4 EXPERIMENTAL SET UP

The flexural test procedures recommended by the Japanese Concrete Institute (JCI-SF) [77] were followed for testing the thin-sheet samples. The flexural test samples had a clear span of 9 in (228 mm), width of 4.5 in (115 mm) and thickness of 0.25 in (6 mm). The test set up is shown in Fig. 3.6. A displacement-controlled flexural test was conducted at a displacement rate of 1/3000 of span length per minute. A computer-controlled data acquisition system was used to record the test data and plot the flexural load-deflection curves. These load-deflection curves can be characterized by flexural strength, flexural toughness (determined by the Japanese Concrete Institute [77] as the area under the load-deflection curve up to a flexural deflection equal to span length divided by 150), and initial flexural stiffness (defined here as the stiffness obtained through linear regression analysis of the load-deflection points for loads below 20% of maximum flexural load).

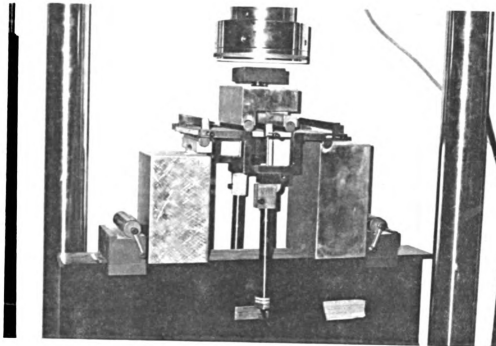


Figure 3.6. Flexural Test Setup.

3.5 TEST RESULTS AND STATISTICAL ANALYSIS

The test results obtained for the fractional factorial experimental design of Table 3.1 are shown in the Figures 3.7 -3.9. Typical flexural load-deflection curves are presented in Figure 3.10. The trends in the effects of different variables, as indicated by the fractional factorial analysis of variance of test results, are shown in Figures 3.11 -3.21. One may visually estimate the relative significance of different variables through comparing the slopes of the trend lines shown in these figures.

Table 3.5 Flexural Performance of Recycled Wastepaper Fiber-Cement Composites

Experiment (By run order)	Flex. Str. (MPa)	Mean Flex. Str (95% Con. Int.)	Flex. Tou. N-mm	Mean Tou. (95% Con. Int.)	Init. Stif. N/mm	Mean Init Stif. (95% Con. Int.)
1	7.372 7.865 7.05	7.429 (1.609)	13.234 13.897 13.675	13.710 (0.677)	211.0 220.7 211.9	214.5 (20.98)
2	10.930 11.32 10.56	10.936 (1.489)	34.890 30.143 34.560	32.865 (9.334)	198.2 204.6 217.7	206.8 (38.82)
3	8.5 8.889 8.324	8.571 (1.133)	45.112 45.456 48.345	45.253 (0.705)	69.44 71.54 70.23	70.40 (4.158)
4	8.198 7.678 8.123	7.999 (1.101)	2.900 2.334 2.789	2.686 (1.203)	290.5 278.7 260.5	276.6 (59.22)
5	6.876 6.559 7.231	6.888 (1.317)	65.123 63.234 62.675	63.498 (5.918)	245.6 270.5 253.7	256.6 (49.97)
6	8.543 8.667 8.987	8.732 (0.898)	53.023 56.345 52.760	54.281 (7.061)	171.0 178.2 167.3	172.2 (21.74)

7	6.785 6.987 7.454	7.075 (1.345)	32.876 34.498 31.992	33.755 (3.213)	119.9 123.5 105.3	116.2 (37.61)
8	14.012 14.567 15.089	14.556 (2.111)	16.765 18.654 15.167	17.455 (4.085)	313.8 301.8 296.2	303.9 (35.15)
9	7.871 7.897 7.650	7.806 (0.532)	78.491 83.432 81.880	81.465 (10.27)	202.1 211.3 190.5	201.3 (40.85)
10	4.773 4.987 5.023	4.927 (0.529)	77.453 77.098 77.087	77.878 (4.154)	87.54 77.22 83.92	82.89 (20.53)
11	6.123 6.030 6.022	6.058 (0.219)	10.543 9.811 10.290	10.174 (1.434)	59.53 68.43 63.70	63.89 (17.46)
12	5.456 5.555 5.987	5.666 (1.106)	8.765 8.176 8.432	8.471 (1.154)	155 162.3 169.2	158.7 (20.37)
13	7.024 7.453 7.065	7.180 (0.928)	38.777 40.209 39.430	39.509 (2.808)	108.7 96.66 91.43	98.92 (34.67)
14	5.667 5.713 5.986	5.789 (0.678)	79.337 85.987 79.235	81.468 (15.34)	107.8 90.54 111.0	103.1 (43.13)
15	4.603 4.967 4.815	4.815 (0.742)	13.875 13.650 15.776	13.694 (0.64)	169.9 180.3 184.7	178.3 (29.75)
16	7.345 7.123 6.456	6.974 (1.813)	42.342 45.112 45.666	44.215 (6.36)	41.47 45.55 38.81	41.94 (13.31)
17	5.495 5.567 5.890	5.653 (0.84)	5.678 6.234 5.556	5.853 (1.292)	31.22 34.2 33.23	32.92 (6.126)
18	4.786 4.667 4.897	4.783 (0.45)	37.231 38.998 39.223	38.402 (3.976)	140.2 160.0 150.4	150.20 (38.75)

19	4.320 4.234 3.987	4.180 (0.677)	26.456 29.543 28.345	27.704 (6.373)	141.1 151.2 133.0	141.8 (35.84)
20	4.123 4.674 4.430	4.409 (1.08)	24.345 23.765 22.333	23.568 (3.492)	270.2 245.7 243.8	253.2 (57.84)
21	5.732 4.564 5.678	5.324 (2.58)	21.560 22.543 23.767	22.232 (2.286)	220.1 230.4 242.9	231.10 (44.73)
22	7.213 7.512 7.123	7.282 (0.798)	75.977 76.988 71.440	75.843 (4.768)	142.3 130.2 150.2	140.9 (39.44)
23	6.267 6.378 6.543	6.396 (0.544)	13.300 10.432 13.101	12.565 (4.322)	74.54 80.23 81.66	78.81 (14.77)
24	5.987 6.320 6.230	6.179 (0.675)	32.987 32.876 35.032	33.252 (2.188)	190.2 194.7 198.3	194.4 (15.96)
25	7.467 7.231 7.560	7.419 (0.644)	53.890 57.452 58.543	55.943 (7.223)	450.2 471.2 460.3	460.6 (41.17)
26	8.234 8.987 8.654	8.625 (1.479)	11.222 11.977 10.897	11.499 (1.629)	183.3 190.5 203.7	192.5 (40.47)
27	5.754 5.456 5.532	5.58 (0.606)	5.654 5.567 5.719	5.623 (0.19)	130.5 147.5 122.8	133.6 (49.57)
28	10.378 10.689 10.002	10.356 (1.348)	10.888 11.651 11.312	11.278 (1.496)	251.3 275.6 261.2	262.7 (47.83)
29	6.567 6.345 6.976	6.629 (1.254)	12.765 13.543 13.444	13.102 (1.568)	282.4 297.8 295.4	291.9 (32.53)
30	5.543 5.125 5.487	5.385 (0.889)	69.665 64.120 67.430	67.191 (11.05)	341.2 353.0 369.4	354.5 (55.53)

1
1
12
10
8
8
4
2
0

F

31	8.345	8.330	14.638	14.667	110.5	118.6
	8.123	(0.785)	14.678	(0.108)	128.4	(35.65)
	8.523		17.334		116.7	
32	4.234	4.307	16.876	16.404	160.5	161.3
	4.675	(1.322)	15.955	(1.806)	151.9	(38.26)
	4.012		16.290		171.4	
33 (Control)	14.564	14.792	81.987	84.402	388.1	407.1
	14.024	(3.545)	80.654	(21.08)	423	(69.18)
	15.789		90.567		410.3	

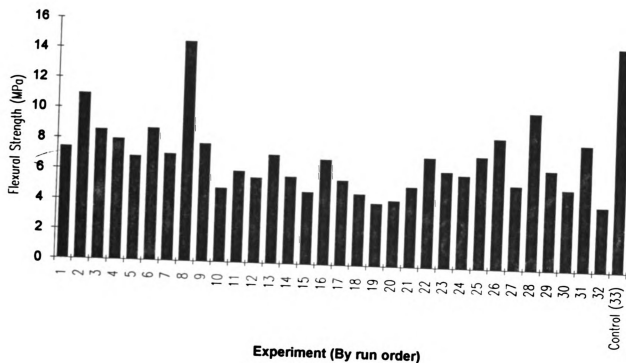


Figure 3.7. Flexural Strength Test Results.

Flexural Toughness (N mm)

Initial Stiffness (N/mm)

F

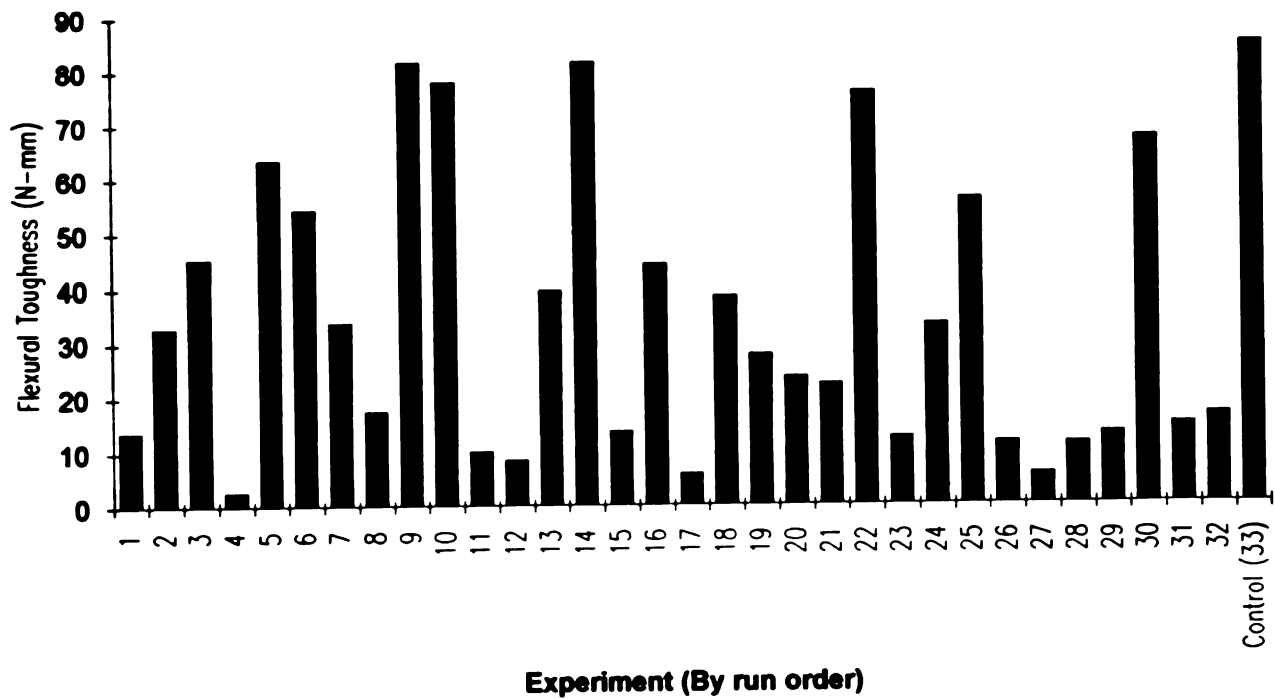


Figure 3.8 Flexural Toughness Test Results.

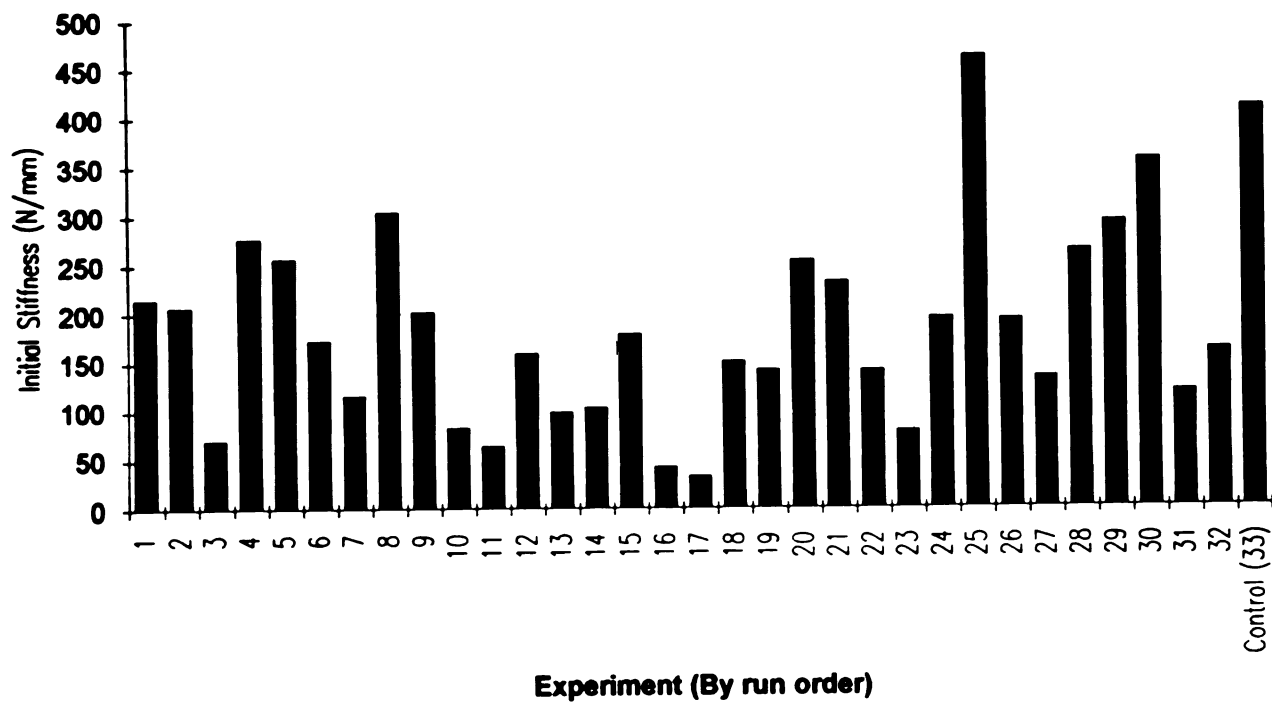


Figure 3.9 Initial Stiffness Test Results.

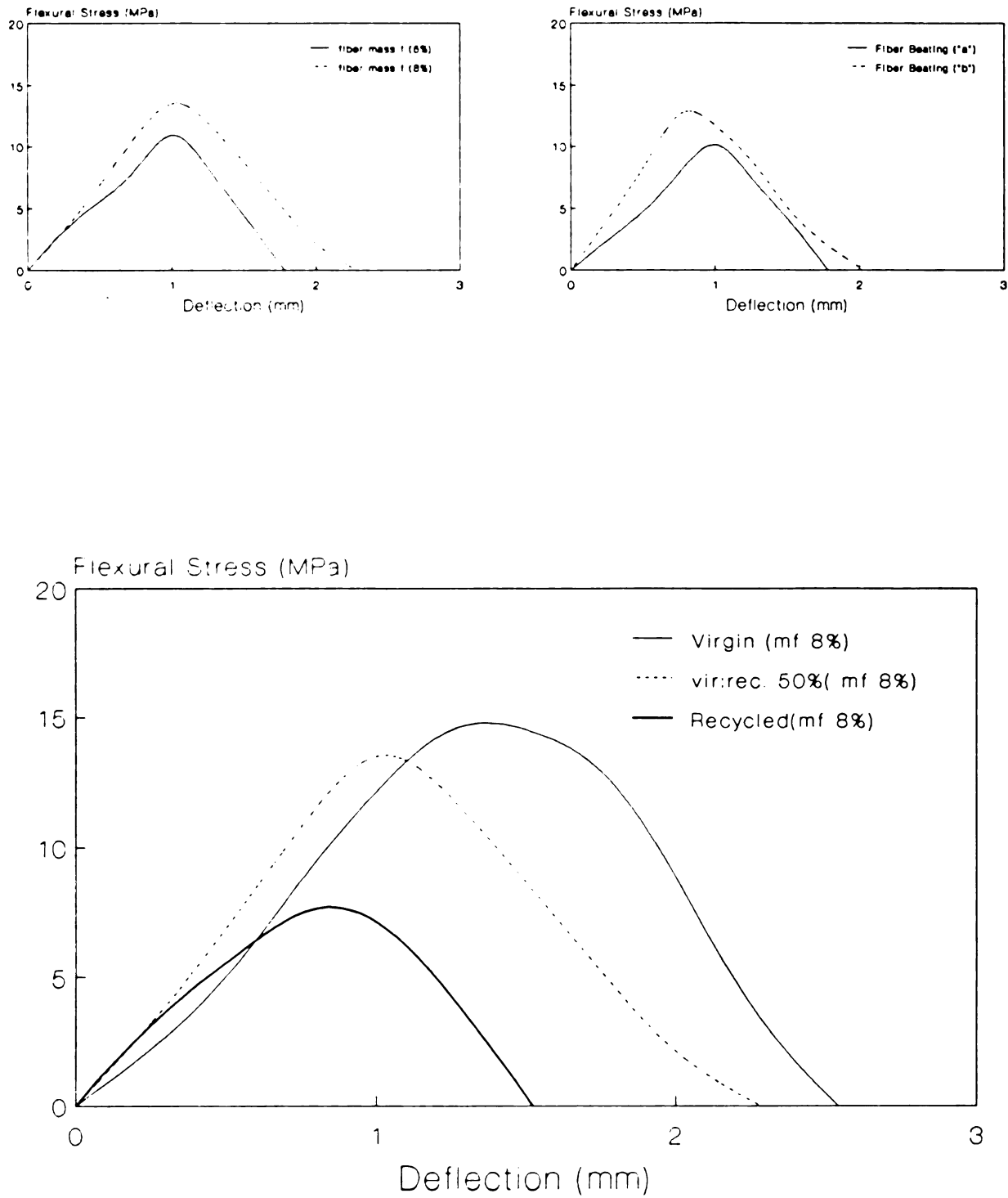


Figure 3.10 Typical Flexural Load-Deflection Curves.

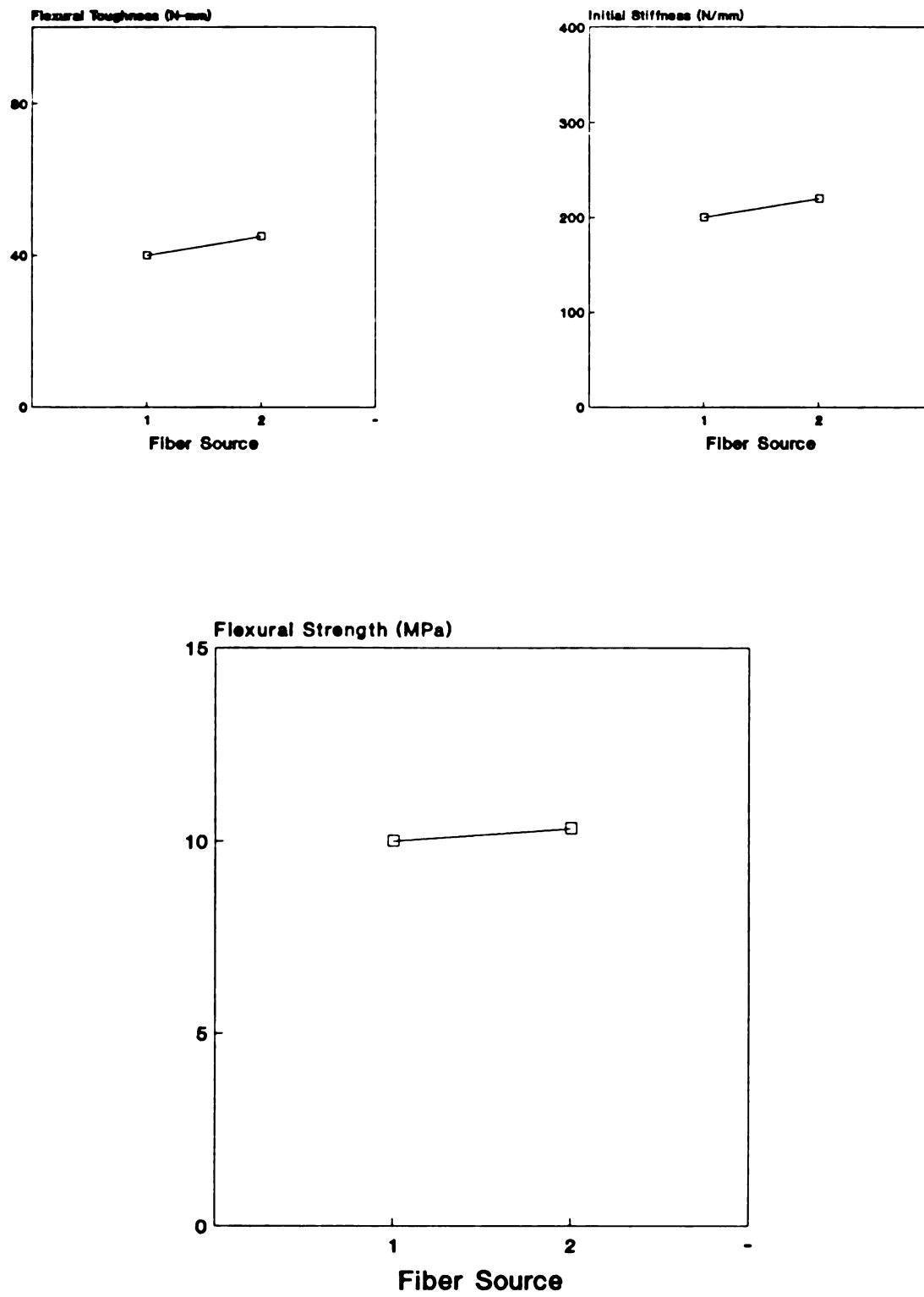


Figure 3.11 The Trends in Fiber Source Effects.

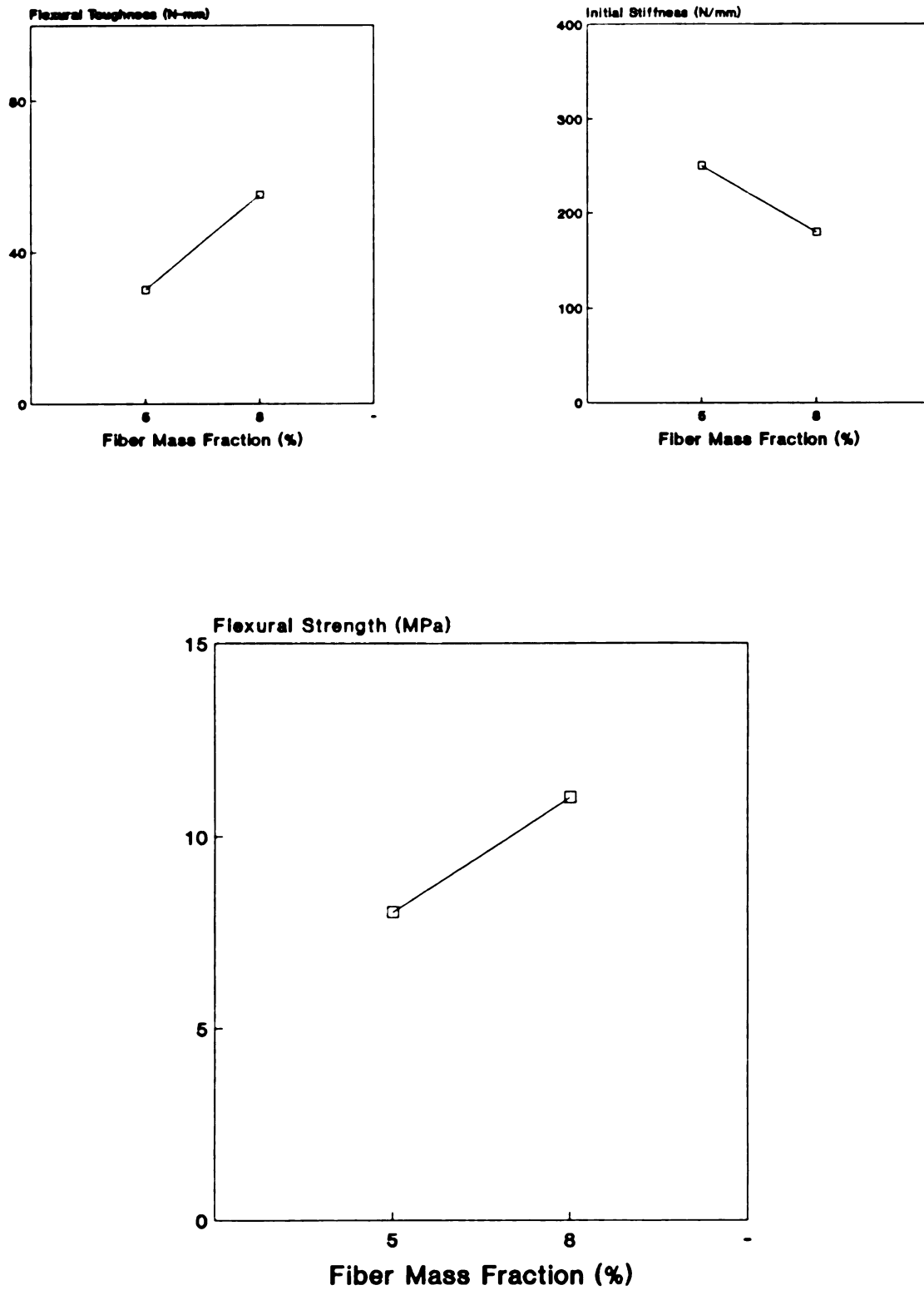


Figure 3.12 The Trends in Fiber Mass Fraction Effects.

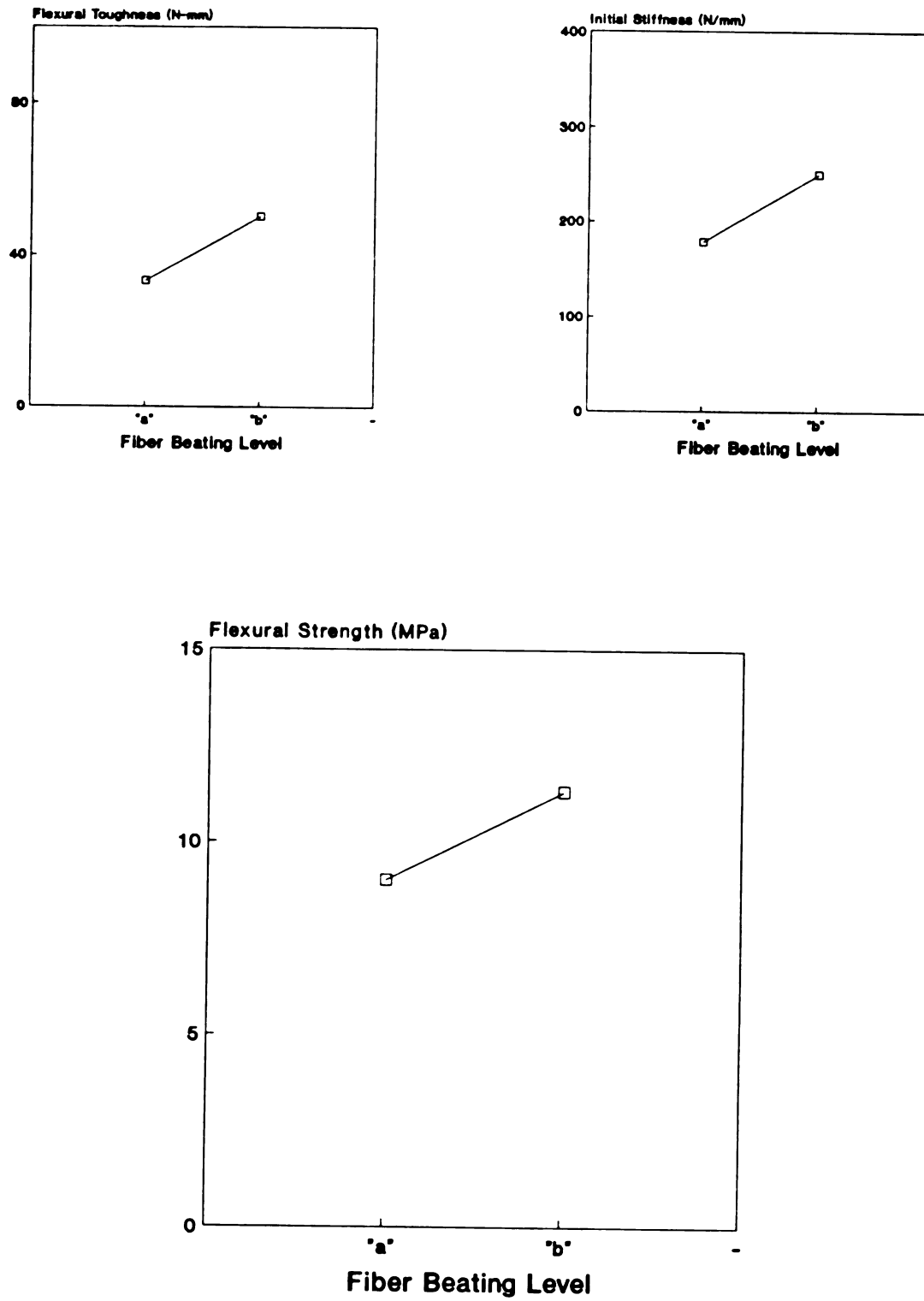


Figure 3.13 The Trends in Fiber Refinement Effects.

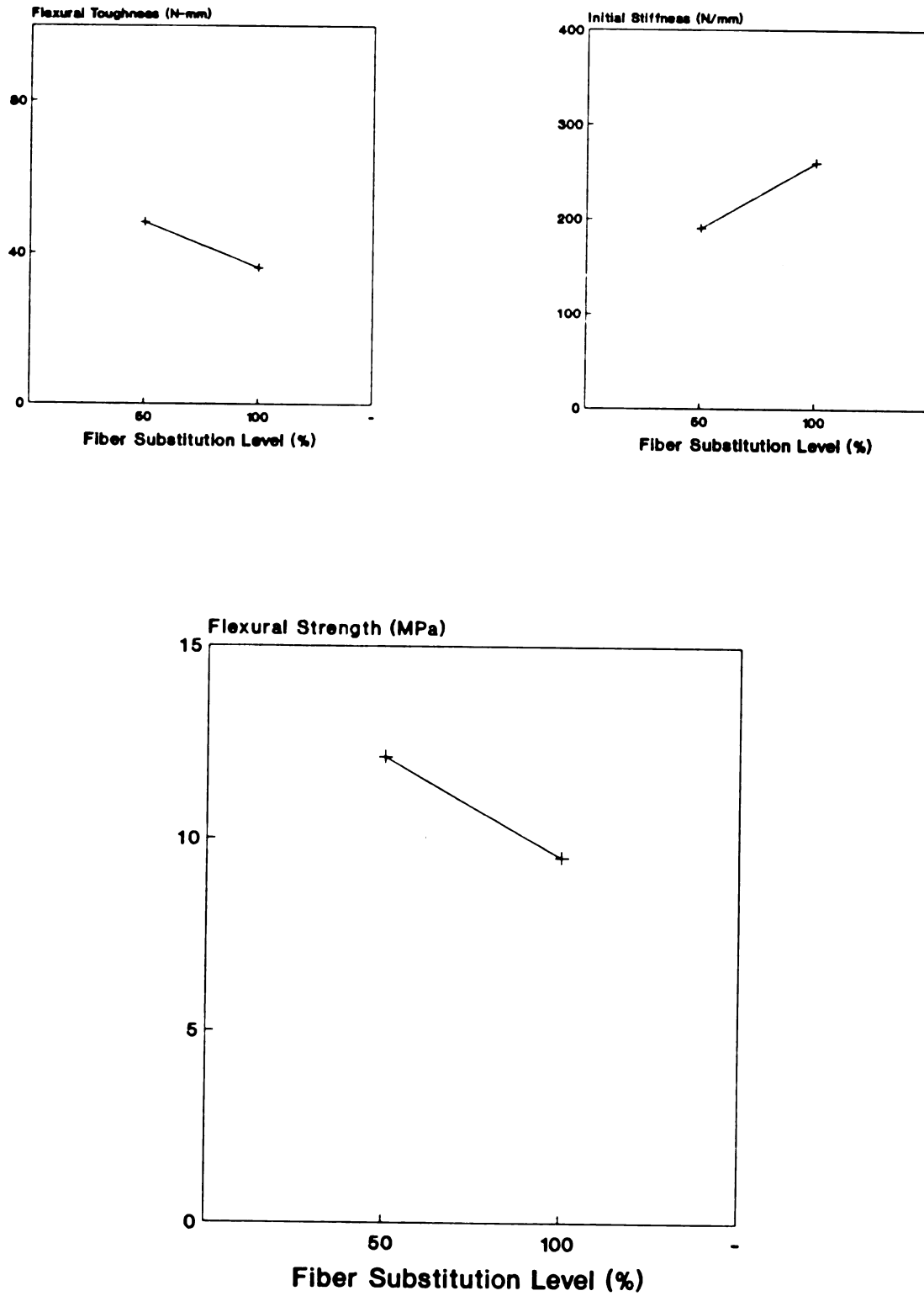


Figure 3.14 The Trends in Fiber Substitution Level Effects.

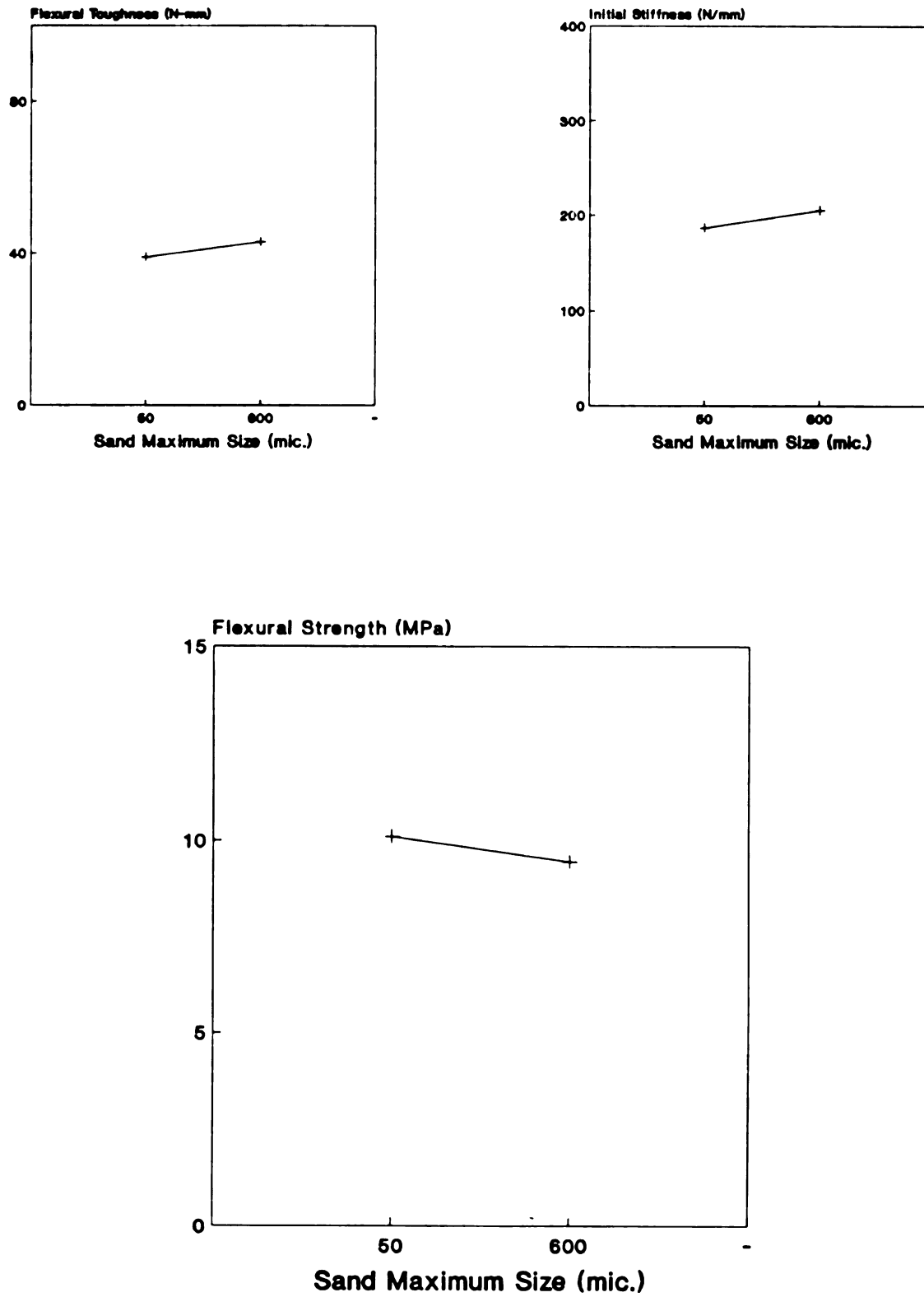


Figure 3.15 The Trends in Sand Maximum Size Effects.

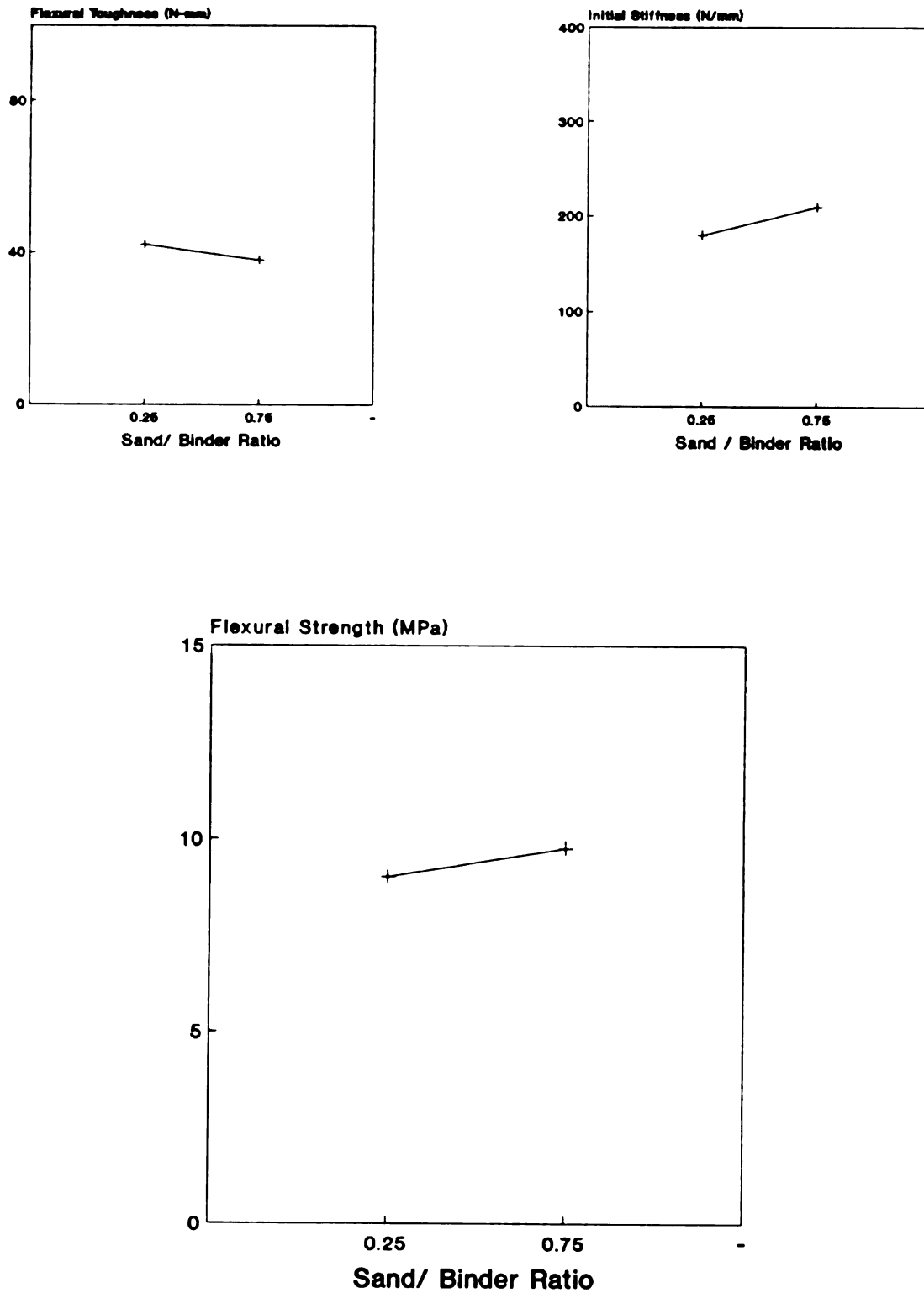


Figure 3.16 The Trends in Sand/Binder Ratio Effects.

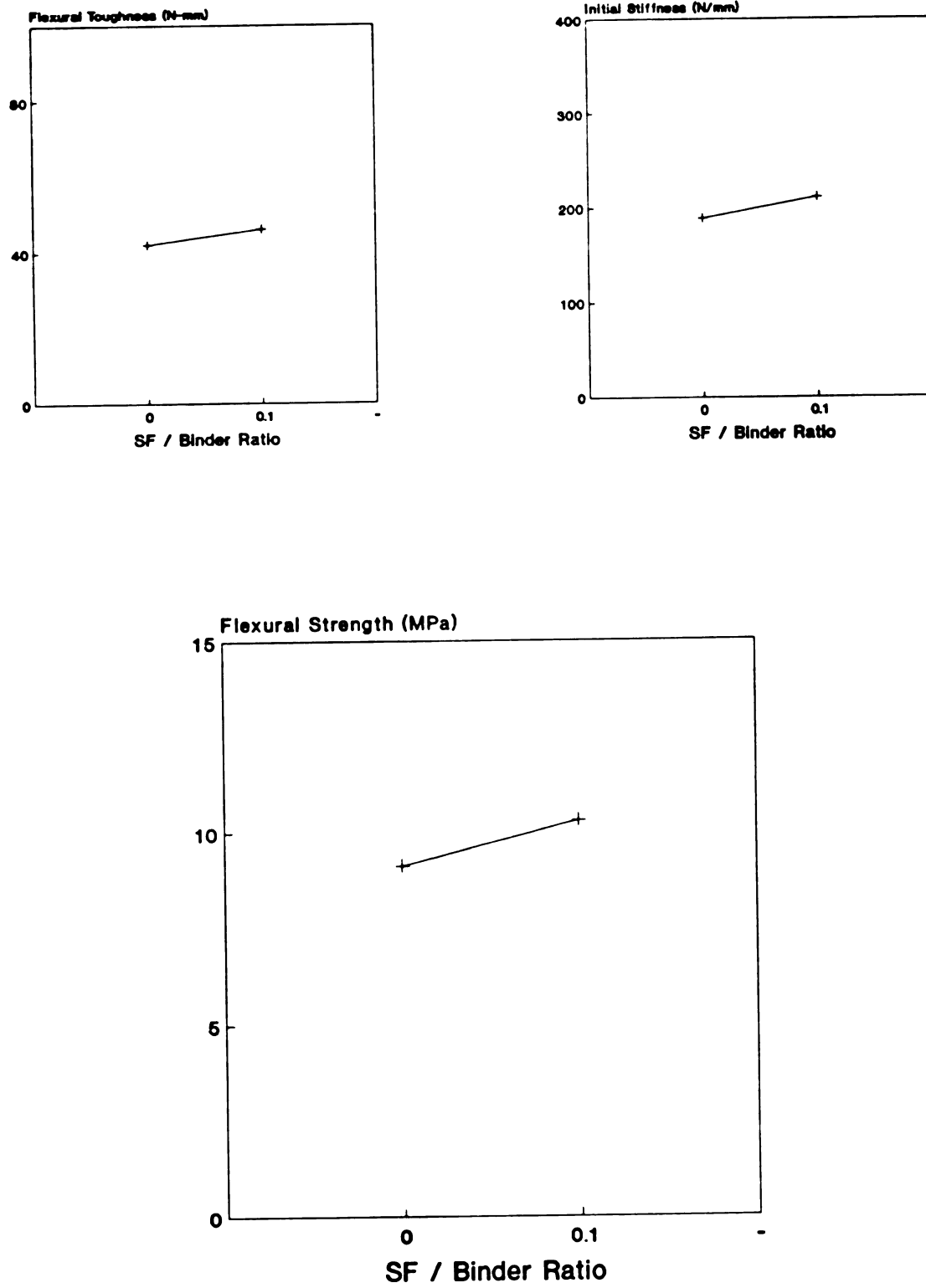


Figure 3.17 The Trends in Silica Fume/Binder Ratio Effects.

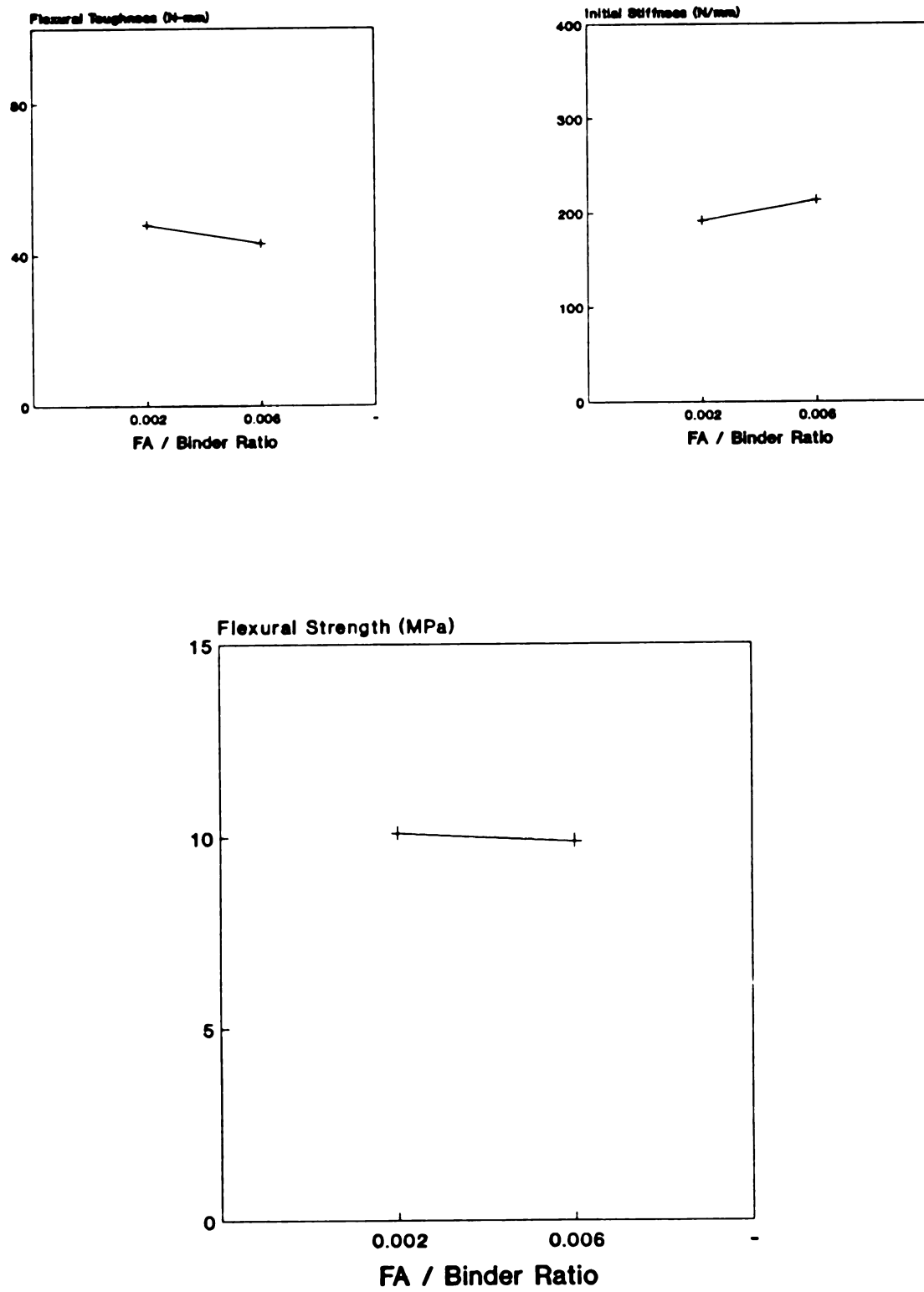


Figure 3.18 The Trends in Flocculating Agent/Binder Ratio Effects.

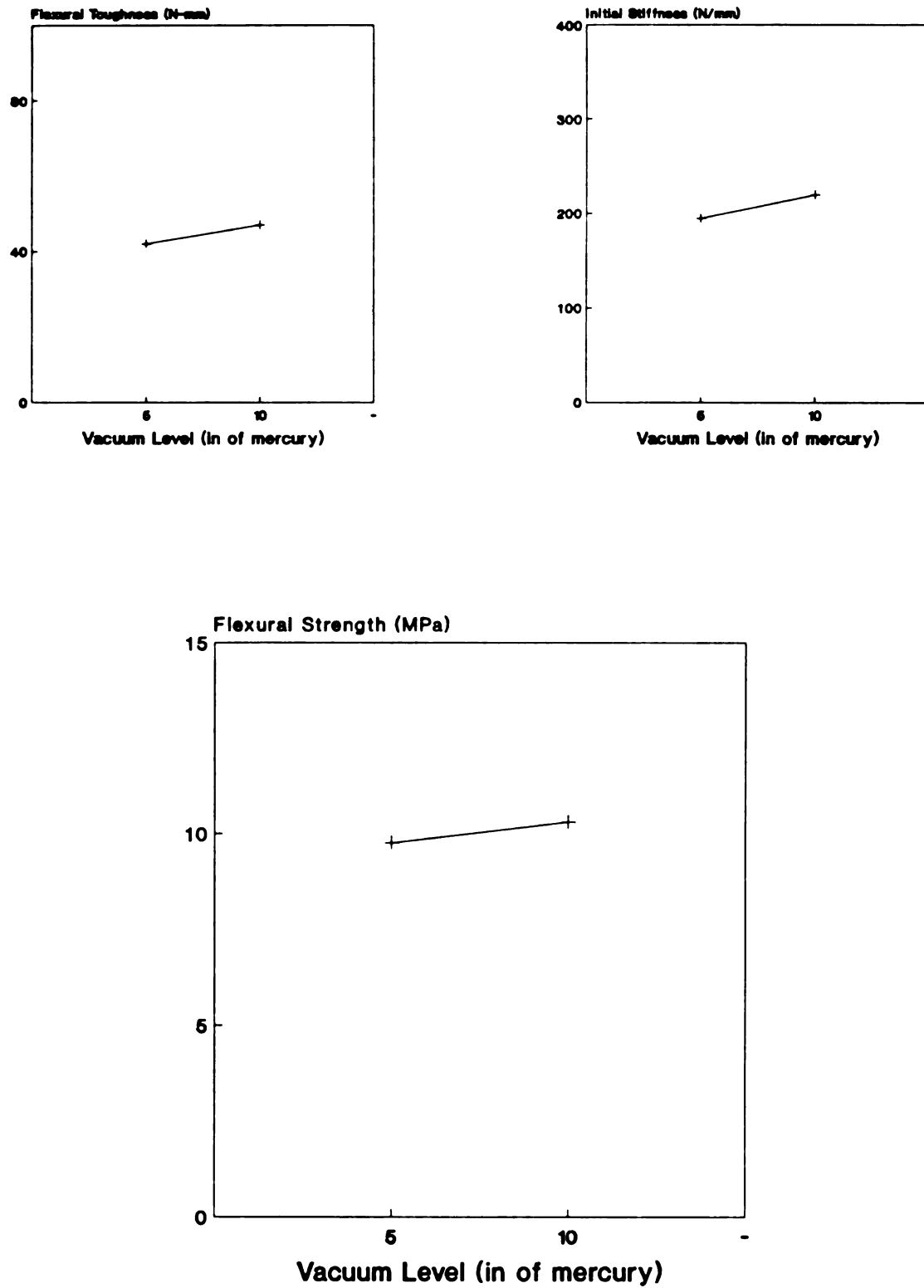


Figure 3.19 The Trends in Vacuum Level Effects.

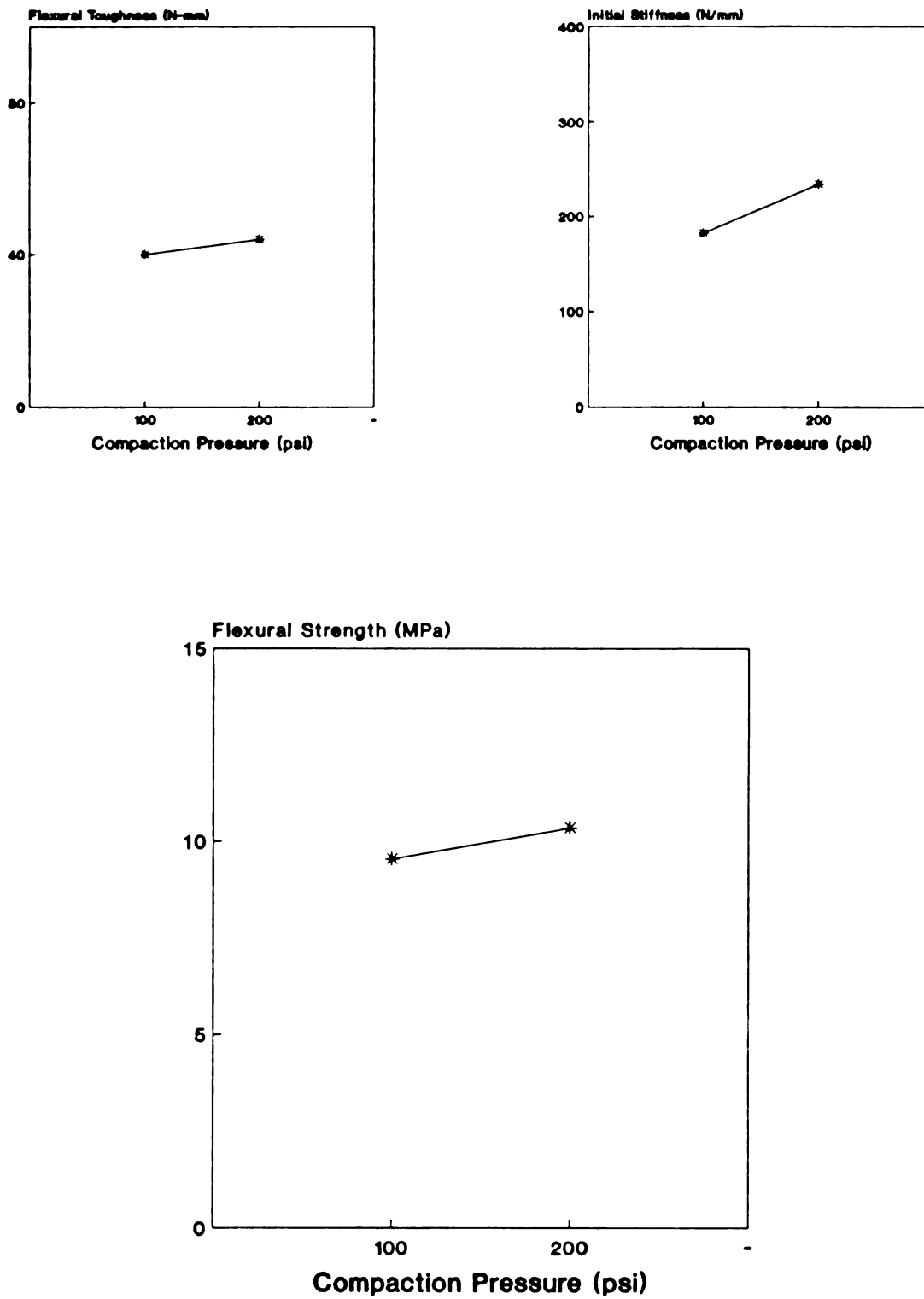


Figure 3.20 The Trends in Compaction Pressure Effects.

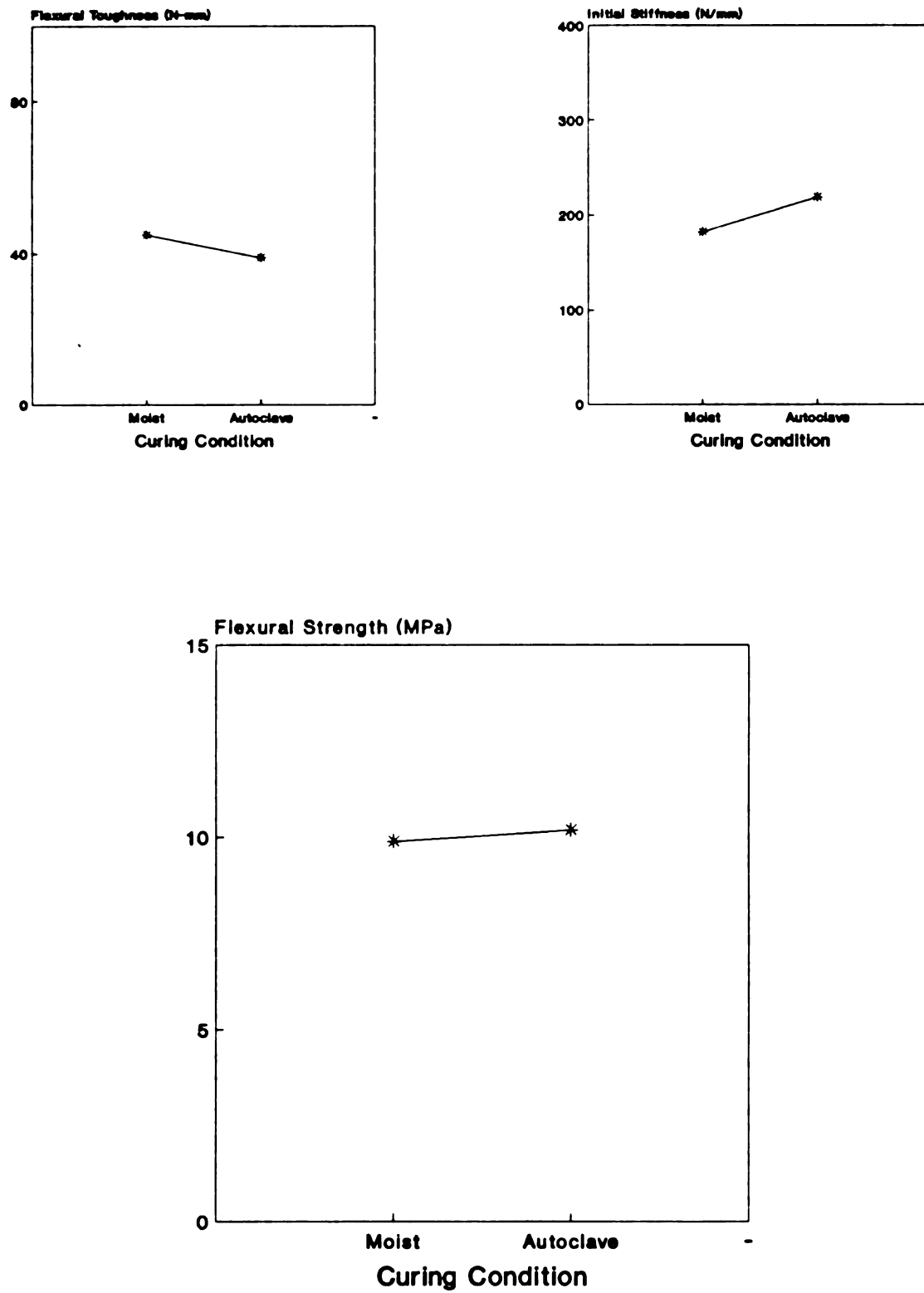


Figure 3.21 The Trends in Curing Condition Effects.

Among the proportioning / processing variables investigated in the fractional factorial design of experiments (fiber source, fiber mass fraction, fiber beating level, fiber substitution level, sand maximum size, sand/binder ratio, silica fume/binder ratio, flocculating agent/binder ratio, vacuum level, compaction pressure and curing conditions), fiber mass fraction, fiber substitution level and fiber refinement were found, through fractional factorial analysis of variance, to have statistically significant effects on the flexural performance of composite at 95% level of confidence (the power of analysis was 0.91). A comprehensive presentation of statistical analysis outcomes for flexural strength, flexural toughness and flexural stiffness is given in Table 3.6. ASTM C1186 does not specify precision limits for this type of composite; however coefficient of variation was calculated to be 3%, 7% and 9% for flexural strength, toughness and stiffness (as obtained from the analysis of variance of variables), which is within acceptable limits as reported in the literature on cellulose fiber-cement composites.

Table 3.6 Results of the Analysis of Variance (Flexural Strength, Toughness and Initial Stiffness)

	Flexural Strength				
Source	Sum-of -Squares	DF	Mean-Square	F-Ratio	P
Fiber Source	150442.702	1	150442.702	2.937	0.093
Fiber Mass F.	366238.845	1	366238.485	7.149	0.010
Fiber Beating l.	249820.824	1	249820.824	4.877	0.032
Fiber Sub. l	1292251.069	1	1292251.069	25.225	0.000
Sand/Binder	362.939	1	362.939	0.007	0.933
Sand Max. S.	57650.788	1	57650.788	1.125	0.294
Silica F/ Bin.	144313.158	1	144313.158	2.817	0.099
Floc. Ag./Bin	30731.074	1	30731.074	0.600	0.442
Vacuum L.	125095.997	1	125095.997	2.442	0.124
Compaction P.	72959.154	1	72959.154	1.424	0.238
Curing Con.	17725.932	1	17725.932	0.346	0.559
Error	2663920.899	52	51229.248		

Table 3.6 (Cont'd.) Results of the Analysis of Variance (Flexural Strength, Toughness and Initial Stiffness

	Toughness				
Source	Sum-of -Squares	DF	Mean-Square	F-Ratio	P
Fiber Source	44.020	1	44.020	0.978	0.327
Fiber Mass F.	45.166	1	45.166	1.004	0.321
Fiber Beating l.	33.483	1	33.483	0.744	0.392
Fiber Sub. l	47.193	1	47.193	1.049	0.311
Sand/Binder	50.929	1	50.929	1.132	0.292
Sand Max. S.	39.176	1	39.176	0.870	0.355
Silica F/ Bin.	49.905	1	49.905	1.109	0.297
Floc. Ag./Bin	54.118	1	54.118	1.202	0.278
Vacuum L.	46.974	1	46.974	1.044	0.312
Compaction P.	49.669	1	49.669	1.104	0.298
Curing Con.	40.520	1	40.520	0.900	0.347
Error	2340.381	52	45.007		

	Initial Stiffness				
Source	Sum-of -Squares	DF	Mean-Square	F-Ratio	P
Fiber Source	40.591	1	40.5911	1.866	0.22
Fiber Mass F.	0.893	1	0.893	0.041	0.840
Fiber Beating l.	19.840	1	19.840	0.912	0.344
Fiber Sub. l	159.915	1	159.915	7.353	0.009
Sand/Binder	20.242	1	20.242	0.931	0.339
Sand Max. S.	57.351	1	57.351	2.637	0.110
Silica F/ Bin.	1.224	1	1.224	0.056	0.813
Floc. Ag./Bin	22.788	1	22.788	1.048	0.311
Vacuum L.	11.912	1	11.912	0.548	0.463
Compaction P.	27.326	1	27.326	1.252	0.268
Curing Con.	0.397	1	0.397	0.018	0.893
Error	1130.839	52	21.747		

3.6 DISCUSSION OF RESULTS

Among the eleven proportioning / processing variables considered in this study, three (total fiber mass fraction, substitution level of virgin cellulose fibers with recycled ones, and fiber refinement condition) proved to have statistically significant effects, at 95% level of confidence, on the flexural performance of wood fiber reinforced cement composites. In order to optimize the composites, it is thus necessary to determine the optimum combination of these variables which produce composites with highest performance-to-cost ratios. In the optimization process, other variables with statistically insignificant effects on the end product qualities may be fixed. The specific levels for these fixed variables (selected based on the trends observed in fractional factorial analysis of variance shown in Figures 3.11-3.21, with due consideration given to the ease of processing and commercial production conditions) are presented below:

Fiber source: American Fillers ("b")

Sand maximum size: 50 μm (Refer to Table 3.2 for gradation)

Sand/binder ratio: 1.0

Silica fume/binder ratio: 0.1 (Refer to Table 3.3 for properties)

Flocculating agent/Binder ratio: 0.002

Vacuum level: 15 in (254 mm) of mercury

Compaction pressure: 200 psi (1.4 MPa)

Curing Condition: High pressure steam curing

3.7 SUMMARY AND CONCLUSIONS

In this first phase of the experimental investigation, total of 11 key variables (factors) defining the production process of wastepaper fiber-cement composites were selected; the main intent was to distinguish those factors with statistically significant effects on the composite material performance characteristics. These variables were: (1) recycled fiber

source; (2) fiber mass fraction; (3) fiber beating level; (4) substitution level of virgin fibers with recycled ones; (5) sand/binder ratio; (6) maximum particle size of sand; (7) silica fume/binder ratio; (8) flocculating agent/binder ratio; (9) vacuum level; (10) compaction pressure; and (11) curing condition. Each factor was considered at two levels in a (1/64) fractional factorial design of experiments. This experimental design reveals the effects of all variables on the composite material performance, but can not provide any information on the possible interactions between different variables. The resulting composite, were tested for flexural performance (strength, toughness, and initial stiffness).

The flexural test data was analyzed statistically by fractional factorial analysis of variance. Among the eleven proportioning / processing variables considered in this study, three (total fiber mass fraction, substitution level of virgin cellulose fibers with recycled ones, and fiber refinement condition) proved to have statistically significant effects, at 95% level of confidence, on the flexural performance of wood fiber reinforced cement composites. In order to optimize the composites, it is thus necessary to determine the optimum combination of these variables to produce composites with highest performance-to-cost ratios. In the optimization process, other variables with statistically insignificant effects on the end product qualities may be fixed.

The recycled wastepaper fibers were also analyzed and compared with virgin cellulose fibers. The recycled fibers were found to be smaller in length than virgin cellulose fibers. The surface of the recycled fibers was more roughened and fibrillated by the recycling process as compared to virgin cellulose fibers. Cellulose content in recycled fibers was found to be lower than virgin cellulose fibers. Recycled fibers had a significant amount (close to 20%) of fines which are expected to play a filling role, rather than reinforcing role, in cellulose fiber-cement composites.

CHAPTER 4

OPTIMIZATION OF INFLUENTIAL VARIABLES

4.1 INTRODUCTION

The three influential variables identified in the previous phase of study (fiber mass fraction, substitution level of virgin with recycled fibers, and fiber refinement level) were selected to be optimized for producing composites with highest performance-to-cost ratios. The optimization experimental design was formulated based on response surface analysis techniques. The composites were optimized considering their flexural performance (strength, toughness, and initial stiffness) and cost.

“Design-Expert” a commercial software developed for the purpose of statistical data analysis and presentation, was used for the optimization purposes. In general, this software helps the user to understand relations between several variables ($x_1, x_2, \dots$) and one or more response variables ($R_1, R_2, \dots$). “Design-Expert” can accomplish following types of goals:

(1) Response Surfaces: Response surfaces are plotted in the form of contour lines presenting the response as function of two variables, while the remaining variables are held constant.

(2) Optimization: The word optimization, as used in this dissertation refers, to the process of establishing the ranges of some variables ($x_1, x_2, \dots$) that allow some response variables ($R_1, R_2, \dots$) to meet certain specifications (or constraints).

Optimization as done by “Design Expert” gives a graphical display of the region of the explanatory variables that simultaneously satisfies the constraints (or the specification requirements). Regions are shaded (or filled in) if one or more constraints fail within the region. Blank region (non-shaded) do satisfy all constraints and they are referred to as the optimum region (see Figure 4.2).

The optimized composite identified in this phase of research were produced (at different total fiber volume fractions), and their mechanical and physical performance were compared with those of conventional composites made fully with virgin fibers.

4.2 OPTIMIZATION EXPERIMENTAL PROGRAM

The experimental program for optimization through response surface analysis (using “Design Expert”) is presented in Table 4.1 Various combinations of the three statistically influential variables are considered in this experimental program of the production of wood fiber-cement composites.

Table 4.1 Optimization Experimental Program

Experiment #	Variables		
	Fiber mass Fraction (%)	Fiber Substitution Level (%)	Fiber Refinement Level*
1.	12	75	12.5(500)
2.	8	50	20(480)
3.	16	50	20(480)
4.	8	100	5(490)
5.	16	100	20(410)
6.	12	75	12.5(500)
7.	12	75	12.5(500)
8.	16	100	5(490)
9.	12	75	12.5(500)
10.	8	50	5(570)
11.	16	50	5(570)
12.	8	100	20(410)

* min. of beating @ 3000 rpm (Canadian Standard Freeness)

For the remaining eight proportioning/processing variables (not statistically influential), fixed levels were used in the optimization experimental program. The fixed values were as follows; fiber source: American Fillers (fiber “b”), sand maximum size: 50 μm , sand/binder ratio: 1, silica fume/ binder ratio: 0.1, flocculating agent /binder ratio: 0.002, vacuum level:15 in (254 mm) of mercury, compaction pressure: 200 psi (1.4 MPa) and curing condition as high pressure steam curing.

4.3 TEST RESULTS AND ANALYSIS

Typical flexural load-deflection curves produced for the composites of Table 4.1 are presented in Figure 4.1. Flexural strength, toughness and initial stiffness test results are given in Table 4.2. Figure 4.2 presents some key analytical results produced in the optimization process.

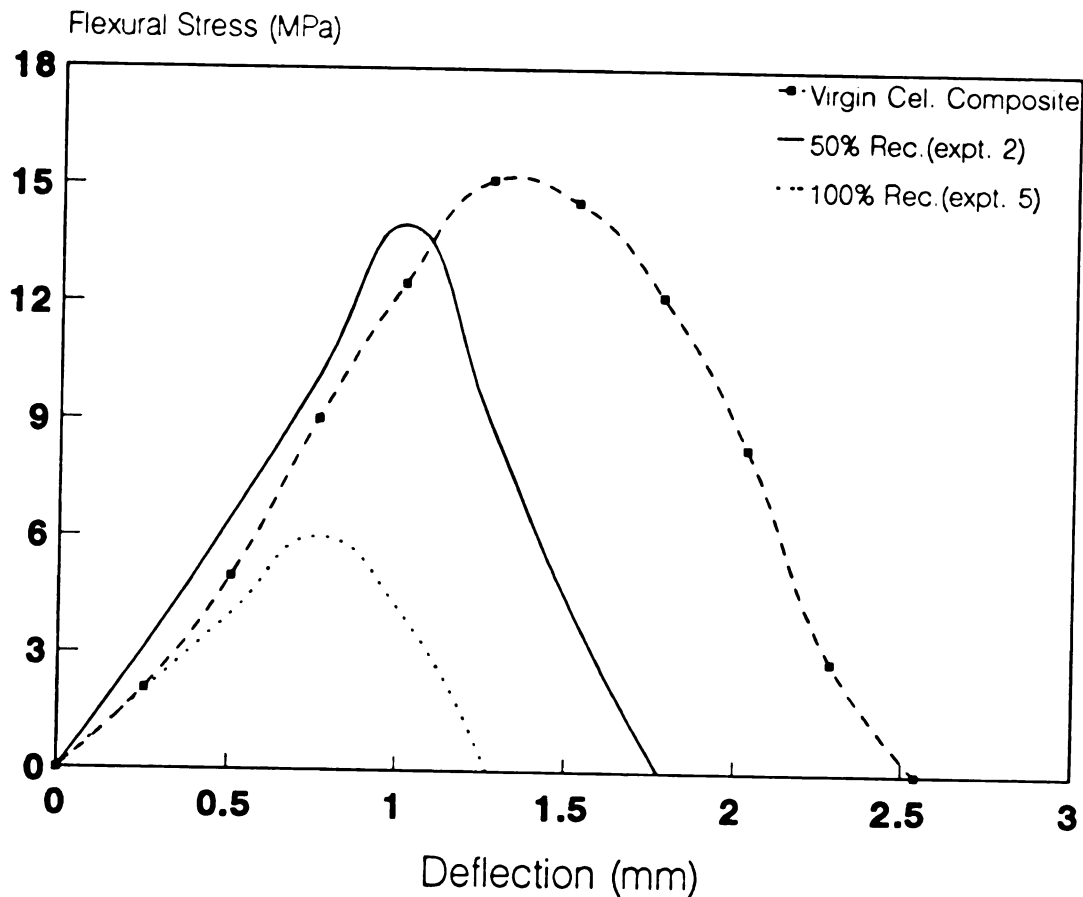
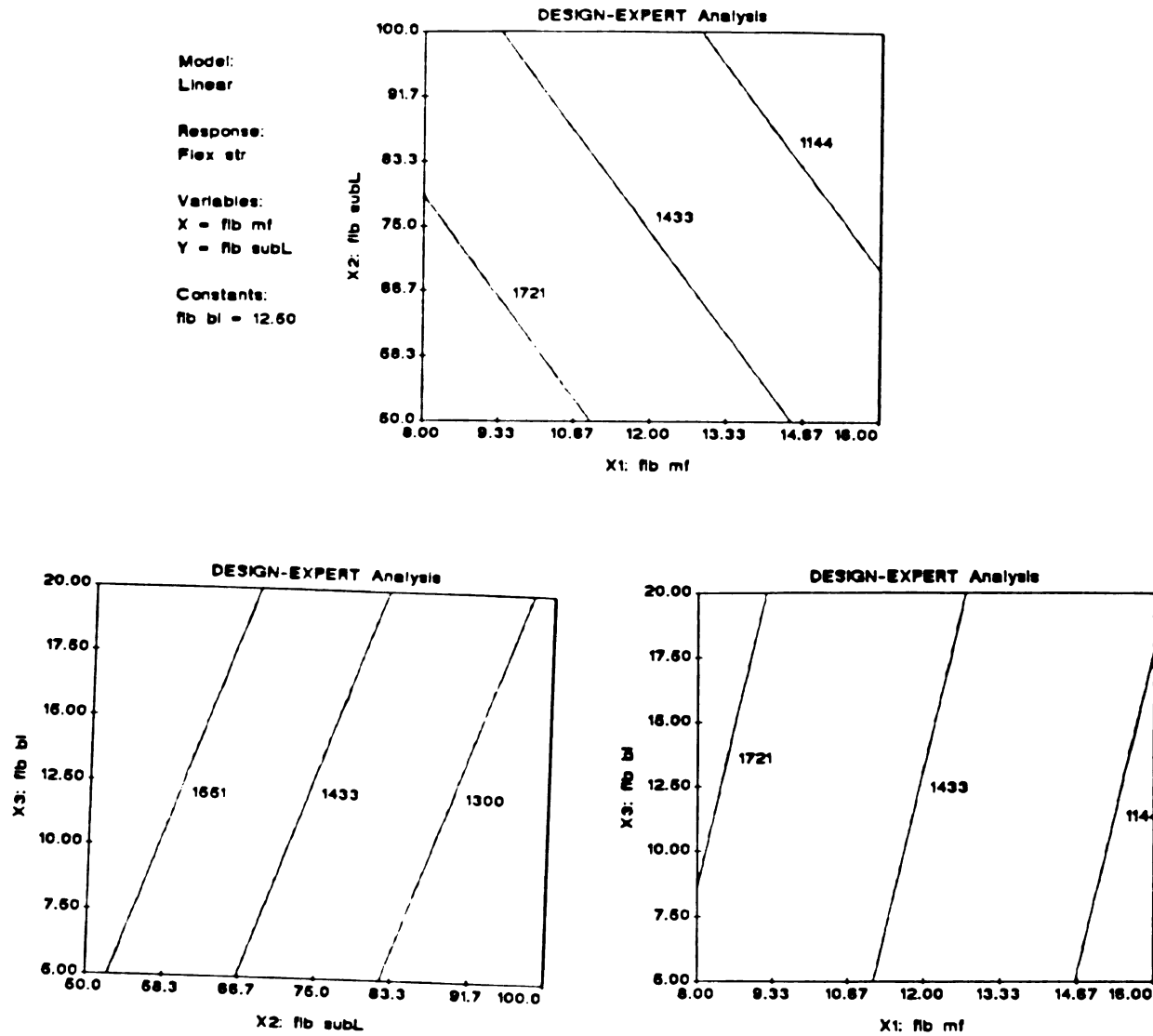


Figure 4.1 Typical Load-Deflection Curves.

Table 4.2 Flexural Performance

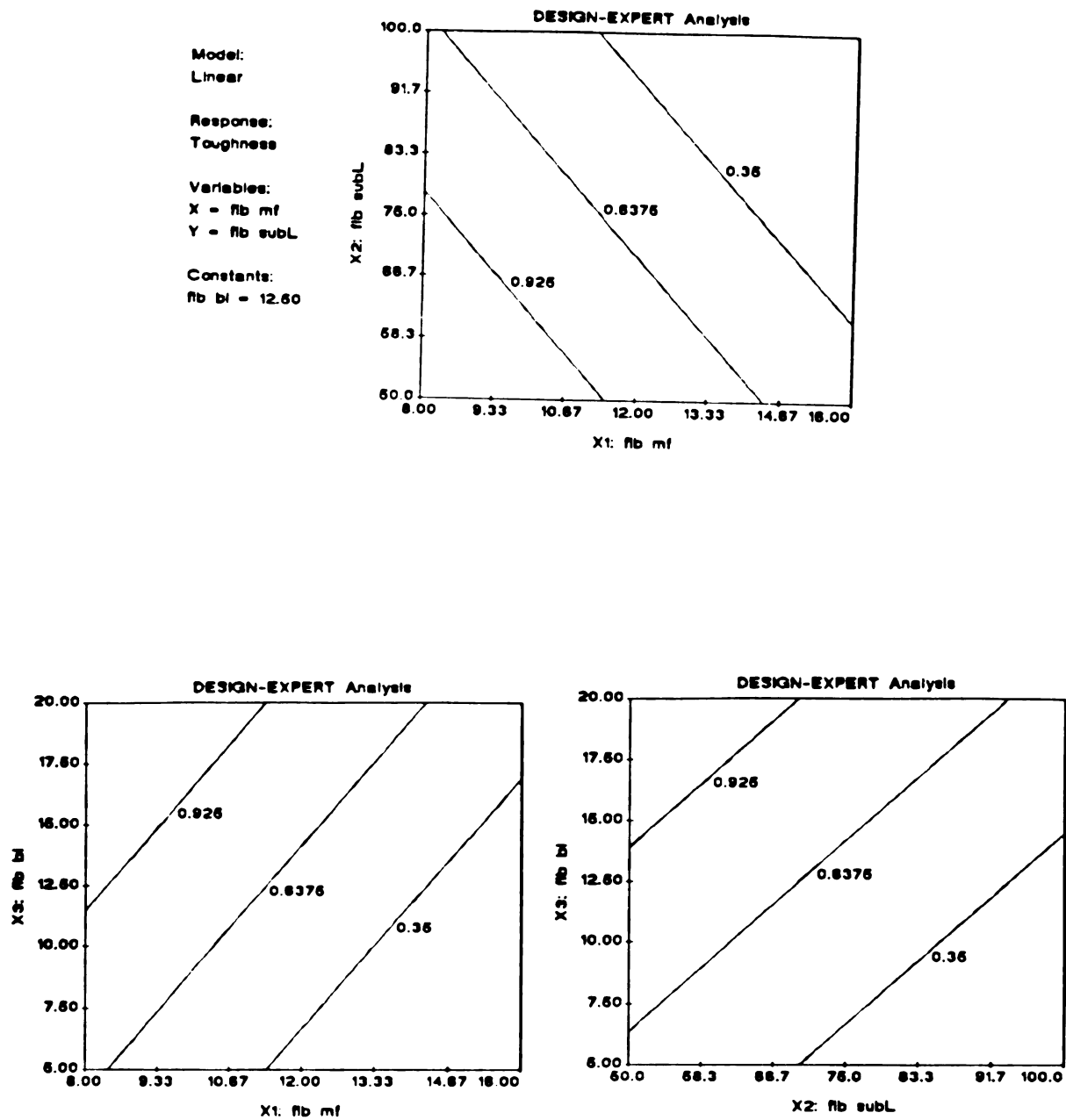
Experiment (By Run Order)	Flex. Str. (MPa)	Mean Flex. Str (95% Con. Int.)	Flex. Tou. N-mm	Mean Tou. (95% Con. Int.)	Init. Stif. N/mm	Mean Init Stif. (95% Con. Int.)
1	12.99 13.23 13.09	13.10 (0.24)	87.50 89.23 81.22	85.985 (± 16.5)	278.30 294.23 287.99	286.84 (± 31.4)
2	13.92 14.11 13.95	13.99 (0.18)	103.23 99.00 91.67	97.971 (± 22.9)	171.11 158.34 149.26	159.57 (± 43.0)
3	7.87 7.66 7.71	7.71 (0.30)	31.50 31.22 32.77	31.83 (± 3.23)	24.87 29.432 32.123	28.808 (± 14.3)
4	7.78 7.39 7.65	7.61 (0.4)	26.25 25.99 29.09	26.119 (± 0.66)	247.23 233.67 249.98	243.62 (± 34.2)
5	6.23 5.92 5.87	6.00 (0.38)	8.75 9.24 8.72	8.901 (± 1.15)	90.381 99.366 86.77	92.172 (± 25.4)
6	10.22 10.33 10.17	10.24 (0.16)	87.50 88.24 78.23	84.657 (± 21.8)	250.44 261.49 243.21	251.71 (± 36.0)
7	12.16 12.28 12.06	12.17 (0.21)	92.66 87.45 85.12	88.429 (± 15.1)	260.32 255.88 245.91	254.04 (± 28.9)
8	6.37 6.27 6.20	6.284 (0.17)	15.75 15.55 15.66	15.655 (± 0.38)	66.88 69.70 63.21	66.597 (± 12.7)
9	11.44 11.51 11.35	11.437 (0.16)	105.01 97.34 101.00	101.11 (± 15.0)	131.29 143.23 145.78	140.10 (± 30.3)
10	13.23 13.08 13.34	13.219 (0.26)	91.117 88.33 87.50	88.984 (± 7.42)	167.34 161.39 149.12	159.28 (± 36.4)

11	7.13 6.89 6.19	6.738 (0.35)	89.23 89.33 87.45	88.689 (4.04)	31.29 29.92 33.01	31.408 (6.06)
12	10.02 10.28 10.18	10.159 (0.49)	115.23 110.55 123.98	116.58 (5.49)	69.93 73.67 65.62	69.741 (15.75)



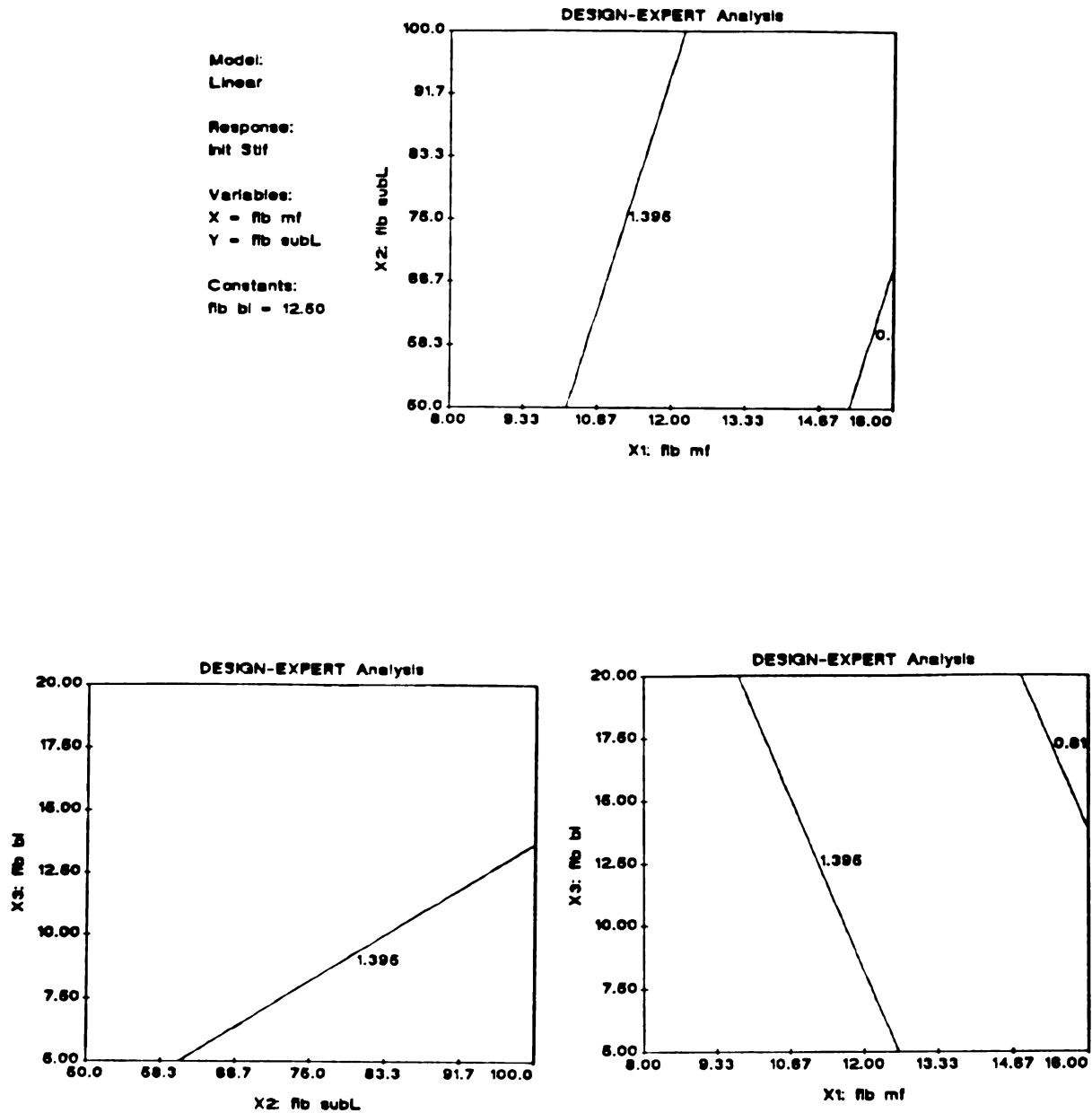
a. Flexural Strength

Figure 4.2 Optimization: Response Surface Analysis.



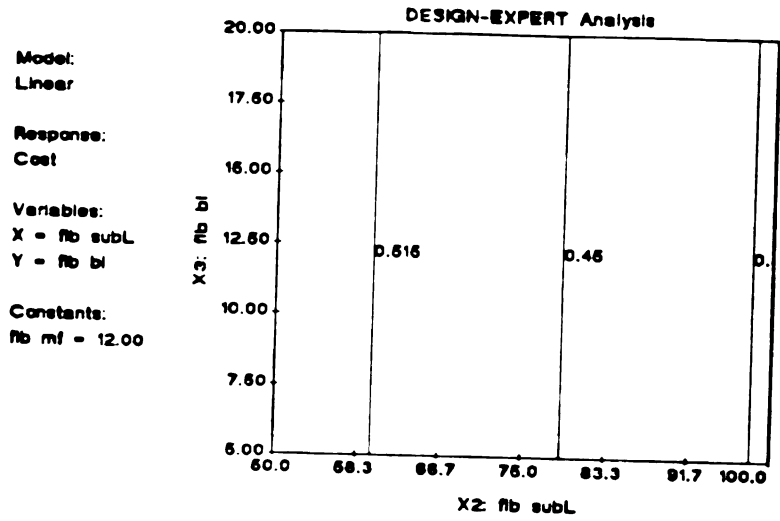
b. Flexural Toughness

Figure 4.2 (Cont'd) Optimization: Response Surface Analysis.



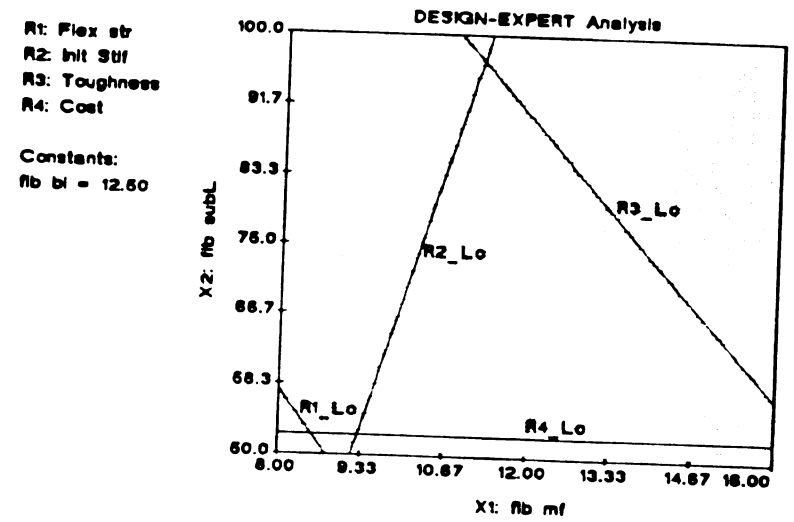
c. Initial Stiffness

Figure 4.2 (Cont'd) Optimization: Response Surface Analysis.



d. Cost

	Response	Units	Lo	Hi	Transform	Model
R1:	Flex str	psi	1900.000	.	---	Linear
R2:	Init Stif	k/in	1.500	.	---	Linear
R3:	Toughness	lb in	0.390	.	---	Linear
R4:	Cost	\$	0.540	.	---	Linear



e. Optimization

Figure 4.2 (Cont'd) Optimization: Response Surface Analysis.

The response surface contours of Figure 4.2 can be interpreted to understand effects of several variables on response. Looking at the response surfaces for flexural strength, we find that flexural strength (Figure 4.2 a) increases as the fiber substitution level is decreased from 100% to 50%; also we observe flexural strength increases as fiber mass fraction is reduced from 16% to 8%. We also observe that the flexural strength increases as the fiber beating is increased from 0 to 15 minutes of beating.

In the analysis of flexural toughness response surfaces (Figure 4.2 b), it can be observed that toughness decreases as the fiber mass fraction is reduced from 16% to 8%. Toughness is also reduced the fiber substitution is increased from 50% to 100%. Toughness increases as the beating level increases from 0 to 20 minutes of beating.

Analyzing the response surfaces contours for initial stiffness (Figure 4.2 c), stiffness is observed to increase as the fiber mass fraction is decreased from 16% to 8%. Stiffness increases also with the increase of fiber substitution level from 50% to 100%. As the beating time is increased the initial stiffness also increases.

The increase in the fiber substitution level is observed in Figure 4.2 d to reduce the cost of raw materials in the composite.

Optimization plots were then generated (see Figure 4.2 e) for achieving minimum strength of flexural strength (1900 psi, 13.2 MPa), flexural toughness of (0.39 lb in, 44 N-mm) and initial stiffness of (1.5 K/in, 175 N/mm) at the lowest possible cost. Figure 4.2 e shows the discarded (shaded) regions and the optimum (clear) region for optimum performance.

The optimum levels of the statistically influential variables derived from the above process are as follows:

Fiber Mass Fraction: 8%

Substitution Level of Virgin with Recycled Fibers: 50%

Fiber Refinement Level: Canadian Standard Freeness of 540 (12.5 minutes of beating @3000 rpm)

The fixed levels of other proportioning / processing variables in the optimized composite were as follows:

Sand/Binder Ratio =1; Silica Fume/Binder Ratio = 0.1; Vacuum Level = 15 in (254 mm)

of mercury; Compaction Pressure = 200 psi (1.4 MPa); Curing Condition = High Pressure Steam Curing.

The flexural performance of the optimized composite is compared in Figure 4.3 and Table 4.3 with that of the control composite made fully with virgin softwood kraft cellulose fibers. The optimized recycled composite is observed to produce flexural performance characteristics comparable to those obtained when the composite is made fully with high-quality virgin fibers (softwood kraft pulp). Analysis of variance of flexural strength, toughness and stiffness test results (Table 4.3) indicated that, at 95% level of confidence, the optimized recycled and virgin composites has statistically comparable flexural strength, and the difference in flexural toughness and stiffness was statistically significant. The recycled composites had an average flexural strength which was only 1.6% less than that of virgin composites.

Table 4.3 Results of the Analysis of Variance (Flexural Strength, Toughness and Initial Stiffness)

Flexural Strength					
Source	Sum-of -Squares	DF	Mean-Square	F-Ratio	P
Type of Composite	0.061	1	0.061	1.837	0.191
Error	0.635	18	366238.485	0.033	
Toughness					
Type of Composite	636.079	1	636.079	26.331	0.00
Error	434.825	18	24.157		
Initial Stiffness					
type of Composite	1306.051	1	1306.051	97.559	0.00
Error	240.97	18	13.387		

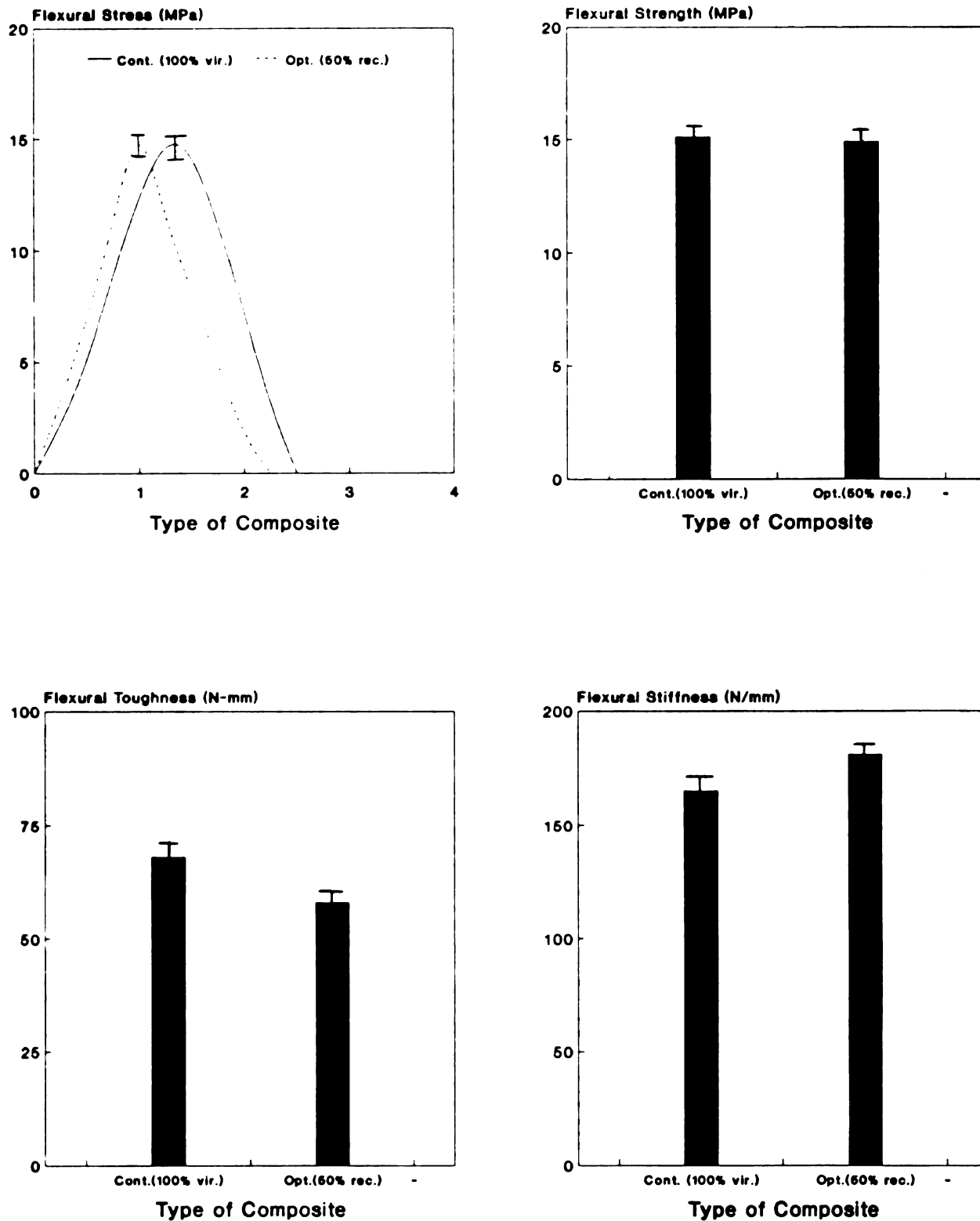


Figure 4.3 Flexural Performance of the Optimized Recycled Composite Vs. Virgin Composite.

4.4 EVALUATION OF THE OPTIMIZED COMPOSITE

Density, water absorption and dimensional stability tests were carried out on the optimized composites following ASTM C1186 [78] procedures. Density is defined in this investigation as the mass per unit volume of the composite expressed in gm/cm^3 . Water absorption is the increase in mass of the test specimen expressed as a percentage of its dry mass after immersion in water for a specified period of time as prescribed in ASTM C1185 and ASTM C1186. Dimensional stability is measured in terms of the linear variation in length of test specimen, with change in humidity from 90% to 30% as per ASTM C1185. An environmental chamber (see Figure 4.4) was used to produce the humidity and temperature conditions required for different tests.

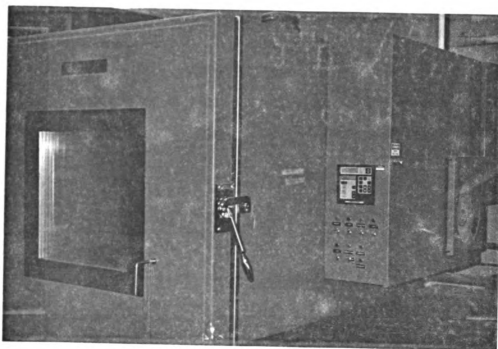


Figure 4.4 Environmental Chamber For Conditioning of Test Specimens.

Optimized composites were produced at different fiber made fraction of 4%, 8%, and 12%, noting that 8% is the optimum fiber content. These composites were evaluated (versus the control composite) based on flexural performance, density, water absorption, dimensional stability and moisture content (ASTMC1186).

4.4.1 FLEXURAL PERFORMANCE

The flexural load-deflection curves for the optimized composites with different total fiber contents are compared with that of control composite made fully with virgin softwood kraft pulp (see Figure 4.5). The recycled composites all had 50% virgin fibers replaced with recycled magazine paper fibers.

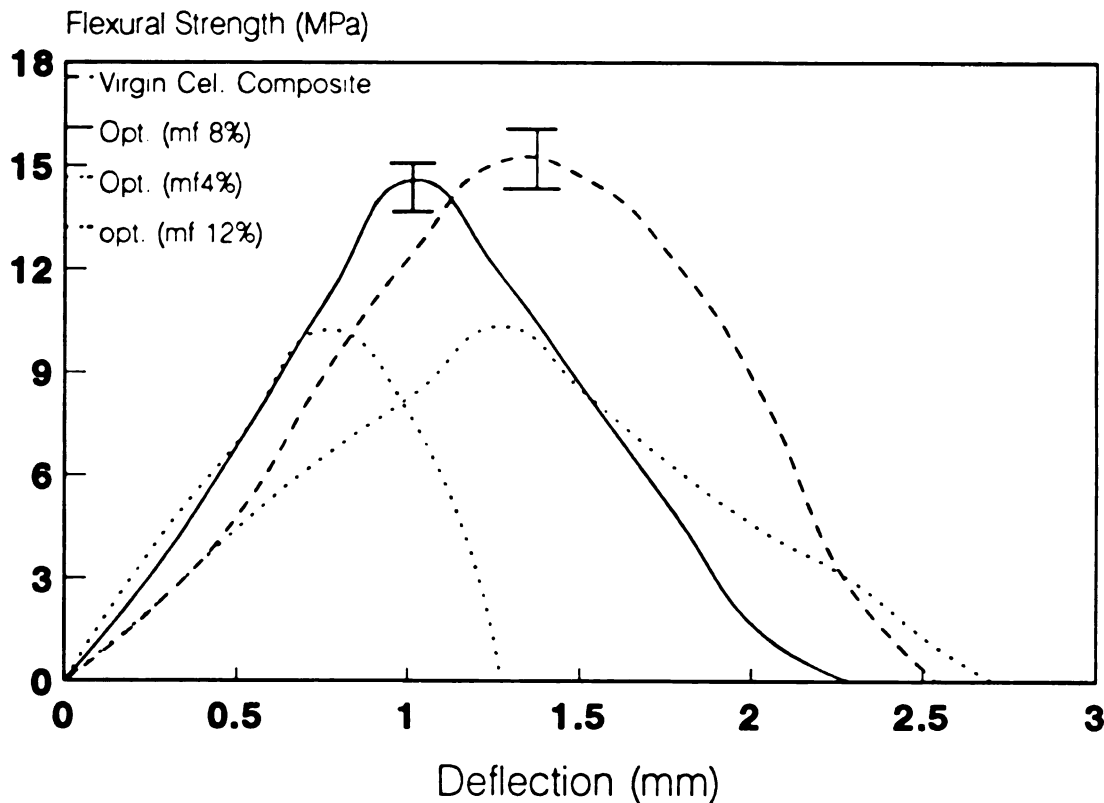


Figure 4.5 Flexural Load-Deflection Curves.

Figures 4.6 through 4.8 compare the flexural strength, toughness, and initial stiffness of optimized composite vs. those of the control composites. While flexural strength and toughness at 8% fiber mass fraction is observed to drop with the substitution of 50% of virgin fibers with recycled ones, the initial flexural stiffness is observed to increase with the use of recycled fibers. This may be illustrated by the fact that the fine fraction of recycled fibers acts more as fillers than reinforcing fibers in the composite. The higher reinforcement efficiency of virgin fibers reflect in higher flexural strength and toughness qualities of the composite, while the filler action of fines in recycled fibers leads to a denser structure of the matrix which reflects in a higher initial stiffness. It is important, however, to note that the differences between the qualities, obtained with virgin and optimized recycled fibers (at 8% fiber mass fraction), are relatively small. Figures 4.6 through 4.8 also indicate that the increase in fiber content from the optimum levels of 8% to 12% has adverse effects on flexural strength and stiffness, but tends to improve toughness characteristics.

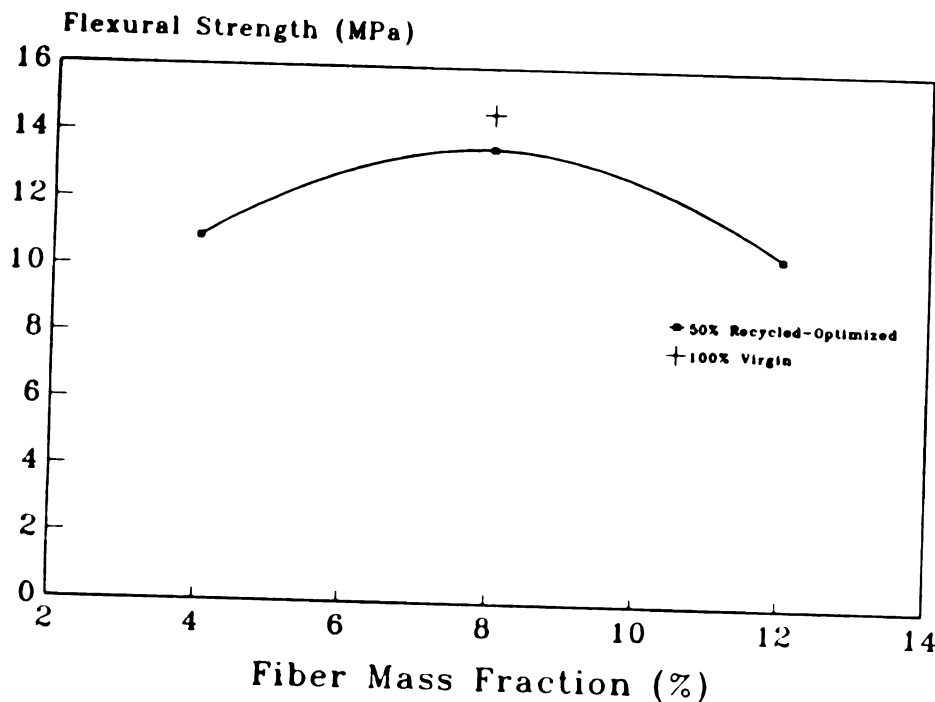


Figure 4.6 Flexural Strength Vs. Fiber Mass Fraction.

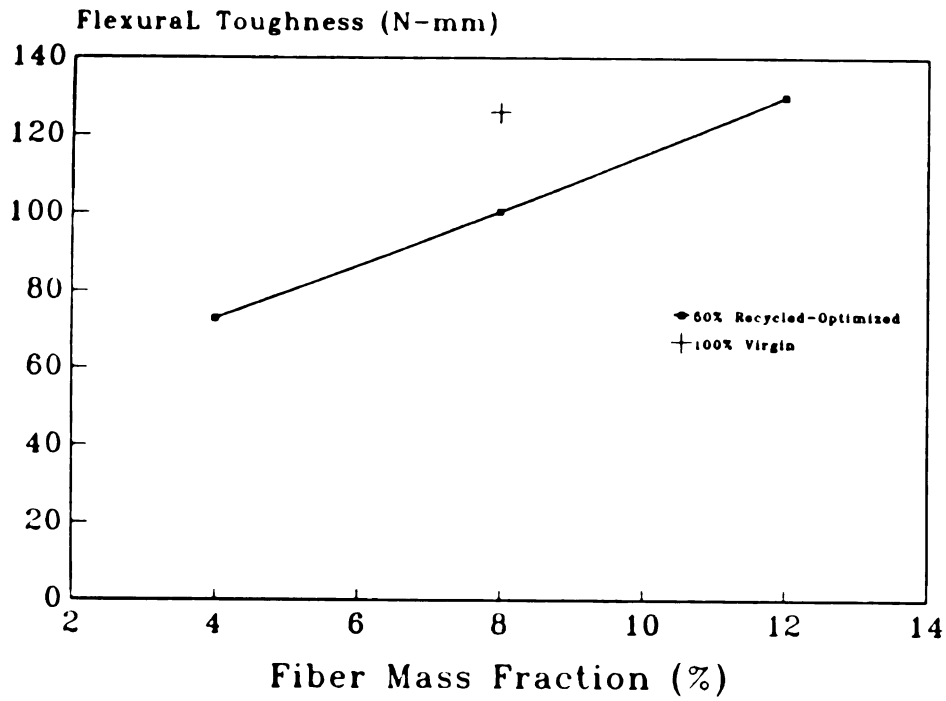


Figure 4.7 Flexural Toughness Vs. Fiber Mass Fraction.

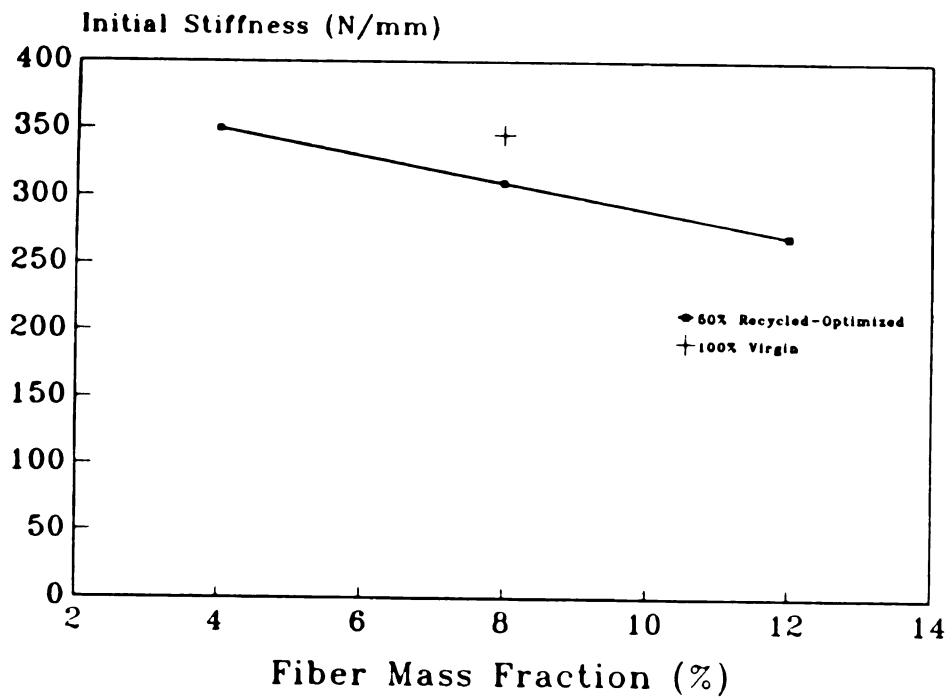


Figure 4.8 Initial Stiffness Vs. Fiber Mass Fraction.

4.4.2 DENSITY

The measured density of recycled and virgin fiber-cement composites are presented in Figure 4.9. At 8% fiber mass fraction, substitution of half of the virgin fibers with recycled fibers is observed to increase the density of the composite. This further confirms the filling action of the fine fraction of recycled fibers which leads to a denser structure of composite. The increase in the total fiber mass fraction is observed in Figure 4.9 to consistently reduce the density of the composite material.

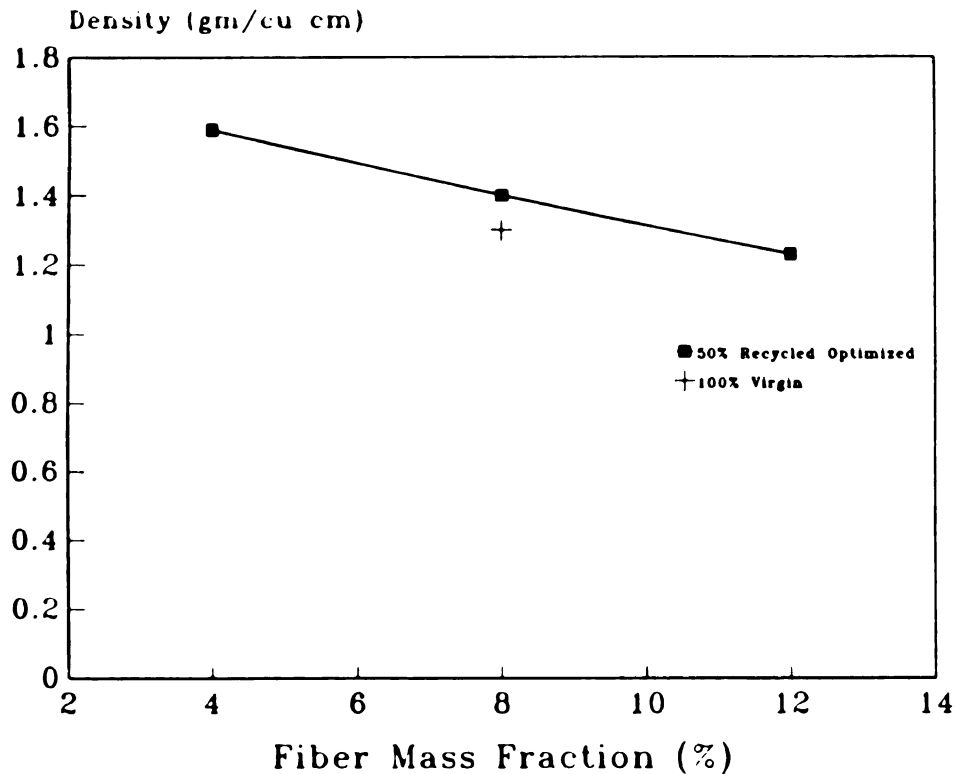


Figure 4.9 Density Vs. Fiber Mass Fraction.

4.4.3 WATER ABSORPTION

Optimized composites with recycled fibers are observed in Figure 4.10 to show reduced water absorption when compared with control composites made fully with virgin fibers. The denser structure resulting from the filling action of the fines in recycled fibers could be responsible for this phenomenon. Figure 4.11 shows strong correlation between water absorption and density of recycled composites with different total fiber mass fraction (correlation coefficient 0.81).

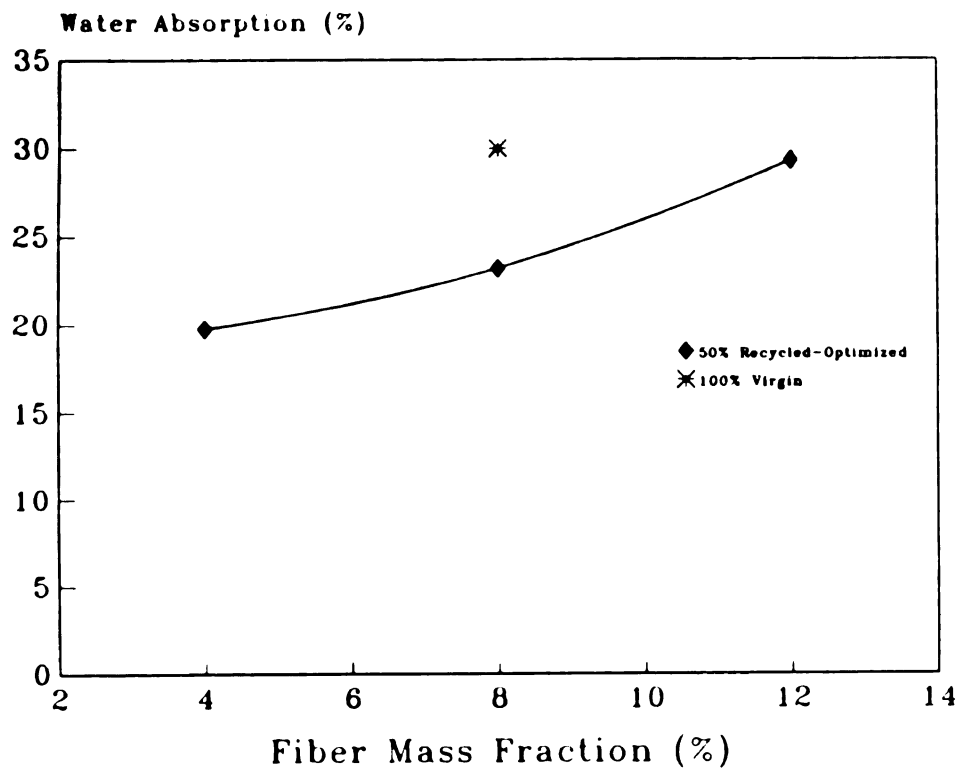


Figure 4.10 Water Absorption Vs. Fiber Content.

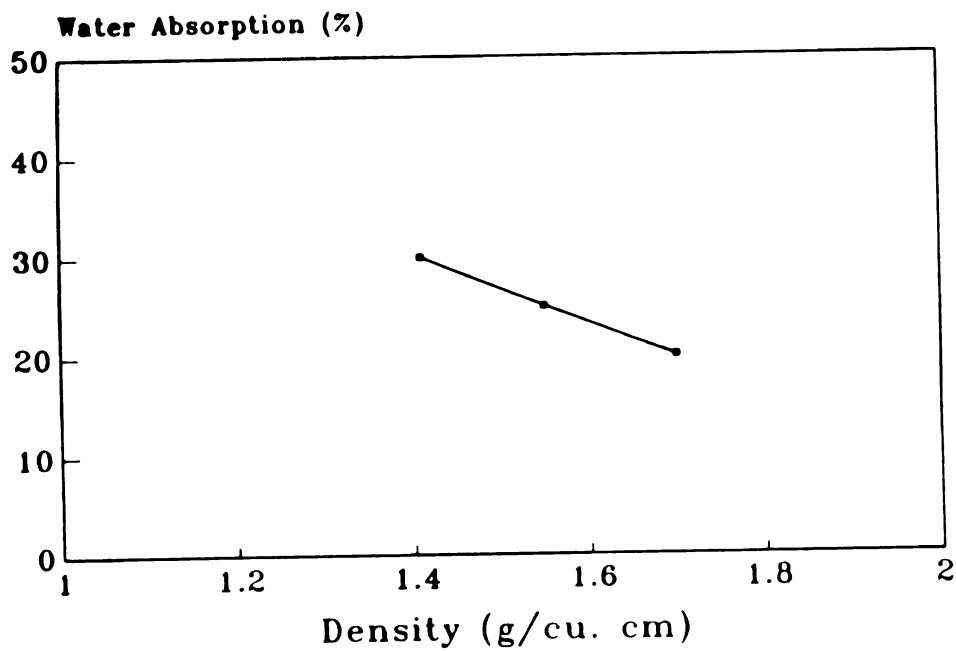


Figure 4.11 Correlation Between Density and Water Absorption in Recycled Composite.

4.4.4 DIMENSIONAL STABILITY

It is important for the thin-sheet cement products to have an acceptable dimensional stability at varying moisture content. Dimensional stability is measured (ASTM C1186) in terms of moisture (dimensional) movements expressed as the percentage changes in length as relative humidity is increased from 30% to 90%.

The dimensional (moisture) movement of the optimized recycled composite is compared with that of the control composite (both at 8% total fiber mass fraction) in Figure 4.12. The optimized composite is observed to possess a better dimensional stability than the virgin fiber-cement composite. The denser structure of recycled composite and its reduced water absorption at least partly describe the desirable aspects of the recycled composite performance.

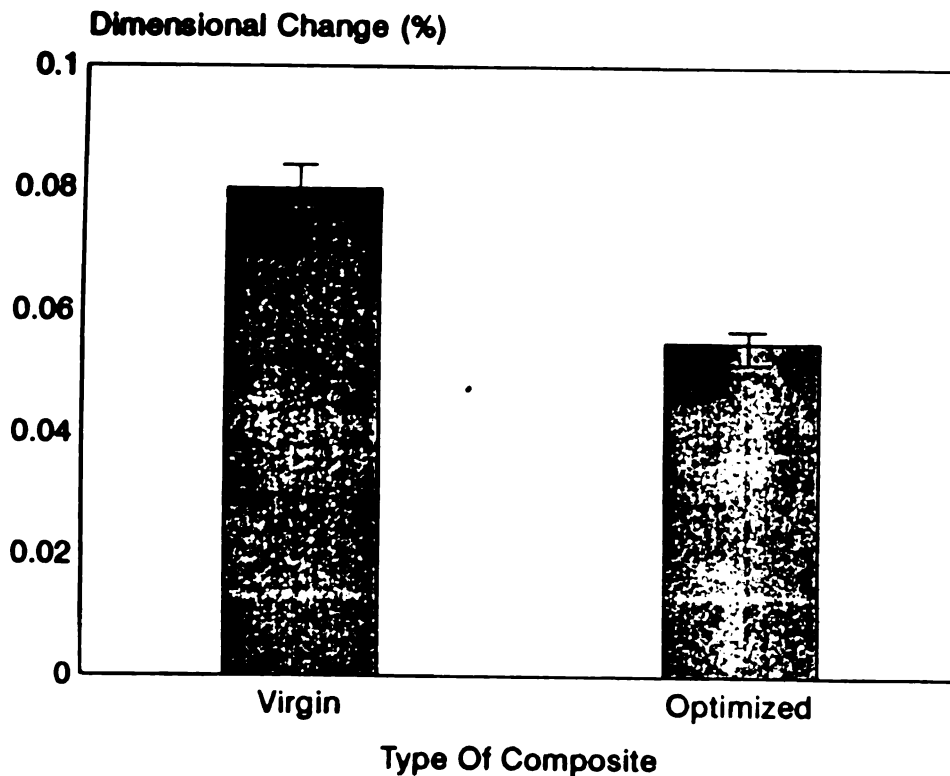


Figure 4.12 Dimensional Stability Test Results.

4.7 MOISTURE CONTENT

The moisture contents (at 50% Relative Humidity in the environment) of recycled composites with different mass fraction are compared in Figure 4.13 with that of the control composites. The increase in total fiber content of recycled composite is observed in Figure 4.13 to increase the composite moisture content. This could be illustrated by the increase in porosity of matrices incorporating higher fiber contents, and also by the affinity of wood fiber for moisture. Recycled composites, with a dense structure resulting from the filling action of fine constituents in recycled fibers, are observed to have lower moisture contents than virgin composites.

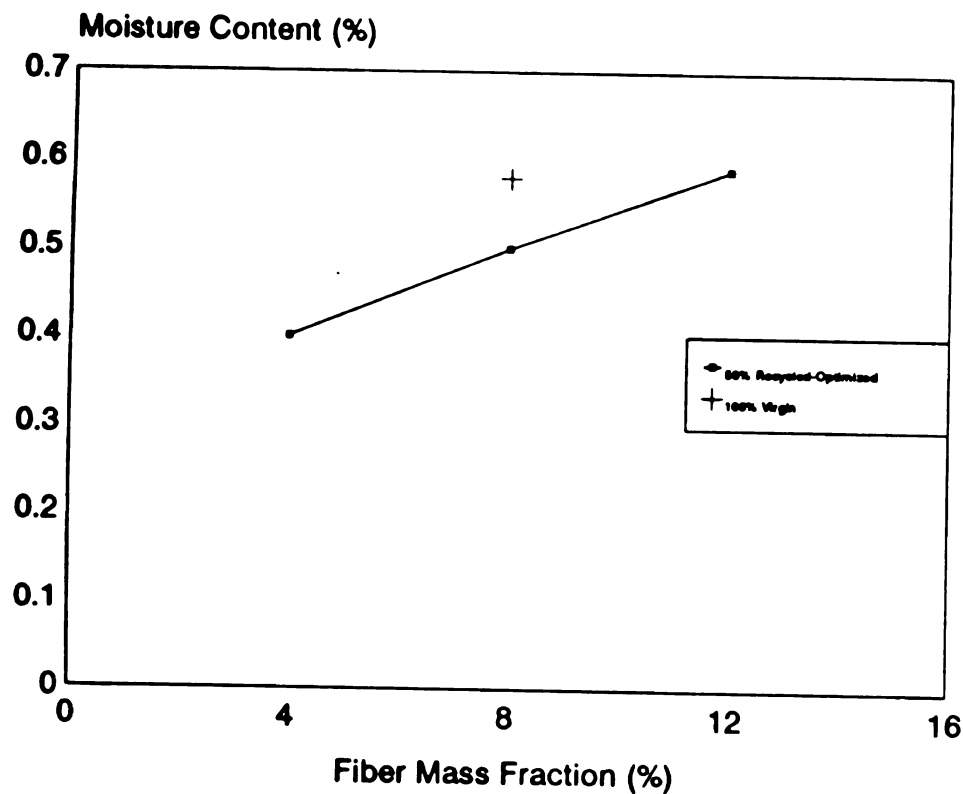


Figure 4.13 Moisture Content Vs. Fiber Mass Fraction.

4.5 TECHNICAL EVALUATION OF RECYCLED COMPOSITES

ASTM specifications and two leading commercial products provided the criteria for technical evaluation of the optimized recycled wastepaper fiber-cement composites. Table 4.4 presents a comparison of some key technical qualities of the recycled composite produced in this research versus ASTM limits and also those of commercial products.

Table 4.4 Technical Evaluation

Properties	ASTM C1185	Commercial Product 1 *	Commercial Product 2 **	Optimized Composite
Flexural Strength (psi)	Grade I: 580 Grade II: 1450 Grade III: 2320	2000-3000	2000	1900-2100
Density (gm/cm ³)	does not specify	1.21	1.35	1.4
Dimensional Stability (%)	does not specify	0.06	0.05	0.06

* manufactured by James Hardie; ** manufactured by Eternit

Table 4.3 indicates that the optimized composite meets the minimum requirements for flexural strength of ASTM Grades I and II thin-sheet cement products. ASTM C1186-91 specifies two types ("A" and "B") of flat sheets, according to their intended applications. Type "A" sheets are intended for exterior applications, where they may be subjected to the direct action of sun, rain, or snow. Type "B" sheets are intended for interior applications, and for exterior applications, where they will not be subjected to the direction action of sun, rain, or snow. The sheets are further classified into four grades according to their flexural strengths. ASTM C1186 does not specify limits on density or dimensional stability. When compared with commercial products, the recycled composites are observed in Table 4.4 to possess acceptable density and dimensional stability.

The recycled composites produced in this research seem to present technically viable and economically/ environmentally superior alternatives to conventional wood fiber reinforced

thin-sheet cement products.

4.6 SUMMARY AND CONCLUSIONS

The influential variables in the processing of recycled wood fiber-cement composites were optimized based on response surface analysis techniques. The variables optimized here were: total fiber mass fraction, level of substitution of virgin fibers with recycled fibers, and the beating (refinement) level of fibers. Optimization was based on flexural strength, initial stiffness and toughness of the composites. Due consideration was also given in the optimization process to the cost of raw materials. The optimized composites were then technically evaluated versus virgin composites, ASTM specifications, and commercial products. The conclusions derived are summarized below.

- (1) Analysis of results indicated that optimum composites are obtained using 8% fiber mass fraction, 50% substitution level of virgin with recycled fibers, and refinement (beating) of fibers to a Canadian Standard Freeness (CSF) of 540.
- (2) The optimized recycled wood fiber-cement composites were shown to possess flexural strength, density and dimensional stability characteristics satisfying ASTM specifications and comparable to those of commercially available virgin wood fiber reinforced thin-sheet cement products.
- (3) The optimized recycled composites produced flexural strength, stiffness and toughness characteristics comparable to those of virgin composites. Compared to virgin wood fiber-cement composites, the optimized recycled composites possessed somewhat lower flexural strength and toughness but higher initial flexural stiffness. The difference in flexural toughness and toughness were statistically significant. Recycled composites also showed reduced moisture (dimensional) movements, lower water absorption and moisture content, and higher density when compared with virgin wood fiber-cement composites.
- (4) The fine content of recycled fibers seem to play more of a filling role than a reinforcing role. Hence, recycled composites present a denser microstructure which reflects in higher stiffness, lower water absorption and moisture content and reduced dimensional (moisture) movements of recycled composites. Reduced reinforcing action of fines in recycled fibers, however, reflects in somewhat reduced flexural strength and toughness of recycled composites when compared with virgin composites.

CHAPTER 5

DURABILITY AND MOISTURE SENSITIVITY

5.1 INTRODUCTION

The service life of construction materials is expected to be several decades, and therefore there is a need to evaluate the long-term performance of new construction materials in the environment they will be exposed to. Potential problems with the durability of wood in cement-based matrices further underline the critical need for durability studies of any wood-cement composite. Furthermore, the sensitivity of wood to moisture makes it necessary to assess the composite material performance at variable moisture conditions.

The main thrust of this phase of research was to assess and (if necessary) improve the long-term durability and moisture-sensitivity of the optimized wastepaper fiber-cement composites developed in this investigation. For the assessment of long-term durability, accelerated ageing tests in laboratory were adopted in order to simulate long-term field exposure conditions.

With the development of new fibers, and their application in composites in different climates, a need often arises to devise tests for a particular material and application, and such tests are seldom detailed in the standards or specifications of the various agencies. In developing accelerated ageing tests, two stages must be considered. First, the potential ageing mechanisms should be identified in order to choose an appropriate means of accelerating them, using variables such as temperature, radiation or moisture effects. Second, the duration or number of cycles in the accelerated test should be translated into time in natural weathering conditions. The correlation between the time in accelerated and natural ageing is not a unique function, since it depends on the climatic conditions in different

zones, and even within the same zone there may be differences in the micro-climate, for instance, the direction in which the component faces.

The accelerated ageing tests adopted in this study cover the ASTM C1186 [78] methods as well as those selected to pronounce certain aspects of the weathering effects on wood fiber-cement composites. The ageing effects on engineering properties and the microstructure of composites were investigated. Microstructural studies utilized the Scanning Electron Microscopy, thermogravimetry and X-ray diffraction techniques. Appropriate measures were adopted and evaluated for controlling the ageing and moisture effects on wastepaper fiber-cement composites.

5.2 EXPERIMENTAL METHODS

The effects of moisture and accelerated ageing on the flexural performance and microstructural characteristics of the optimized wastepaper fiber-cement composites and control composites (made fully with virgin softwood kraft fibers) were investigated. This section introduces the moisture sensitivity, accelerated ageing and microstructural evaluation procedures used in this investigation.

5.2. 1 Moisture Sensitivity

In order to assess moisture effects on the composite material performance, flexural test specimens were conditioned as follows and then subjected to flexural loading [78]:

Air drying: Place the test specimens, for 4 days in a controlled atmosphere of 73 ° F (23 °C) and 50% relative humidity and in such a manner that all faces are adequately ventilated.

Oven-drying: Dry out the test specimen in an oven at 216 °C (102 °C) until the difference between two consecutive weighing, at intervals not less than two hours, does not exceed 0.1% by mass.

Saturation: Immerse specimens to be tested in wet conditions in water at a temperature of 73 °F (23 ° C) for a period of 48 hours. Test the specimen immediately upon removal from water.

Previous studies on virgin wood fiber-cement composites indicate that moisture effects on

the fibers and their bond to cementitious matrices fully govern the moisture effects on composite material properties. There are, however, some effects of moisture on cement-based matrices (though overshadowed by moisture effects on the fibers and interface zones) which are described in the following.

Rapid drying of cement based materials may induce tensile cracks due to non-uniform drying (and hence differences in drying shrinkage) of the specimen. These cracks do not have much effect on compressive strength but will lower the flexural and tensile strengths [70,71]. If drying takes place very slowly, the internal stresses can be redistributed and alleviated by creep, an increase in strength may result from drying.

Wetting of cement-based materials may lead to losses in compressive strength as a result of the dilation of cement gel by adsorbed water and also breaking of Si-O-Si bonds, which lead to reduction of the cohesion between solid particles. Conversely, when the wedge-action of water upon drying ceases, an apparent increase in strength of the specimen is recorded. Resoaking of oven dried specimens in water reduces their strength to the value of continuously wet-cured specimens, provided they have been hydrated to the same degree. The variation in strength due to drying is thus a reversible phenomenon.

5.2.2 Accelerated Ageing

The accelerated ageing conditions used in this investigation are adopted to pronounce the physical and chemical causes of deterioration in wood fiber reinforced cement composites. These methods and mechanisms through which they accelerate the ageing process are described in this section.

Repeated Wetting and Drying

Repeated wetting and drying cycles simulating repeated rain-heat conditions in natural weathering promote some key chemical and physical mechanisms of deterioration in wood fiber-cement composites. These conditions accelerate any potential attack by the alkaline pore water of cement-based matrices on certain wood fiber constituents; they also promote migration (through dissolution and re-precipitation) of some cement hydration products from the matrix into the fiber cores and their interface zones. These microstruc-

tural changes would reflect the engineering qualities of aged composites.

The repeated wetting-drying test adopted in this investigation (see Figure 5.1 for test set up) follows the ASTM C1185 procedure. A total of 25 cycles of wetting/drying were used. In each cycle, specimens were moistened by spraying water for three hours at 30°C (86 °F), and then dried for three hours at 60°C (140 °F).

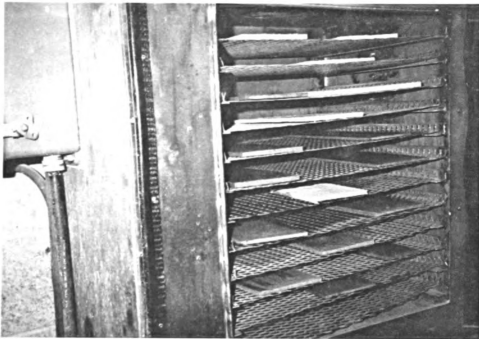


Figure 5.1 Wetting/Drying Experimental Set Up.

Freeze/thaw

This test investigates the possible degradation of cement-based materials exposed to repeated freeze- thaw cycles. Freezing of water in the cement paste capillary pores, due to the volume increase of water upon turning to ice, would cause internal pressures which lead to cracking and deterioration of concrete. A total of 50 cycles are applied as required by ASTM C1185. Each cycle lasts 4 hours, and consists of cooling the specimen to -20°C (-4 °F) over a period of one hour, holding the specimen at -20°C (-4 °F) for one hour,

thawing it to 20° C (68 °F) over a period of one hour, and maintaining the specimen for one hour at 20°C (68 °F) before proceeding to freezing. The experimental set up is shown in Figure 5.2.

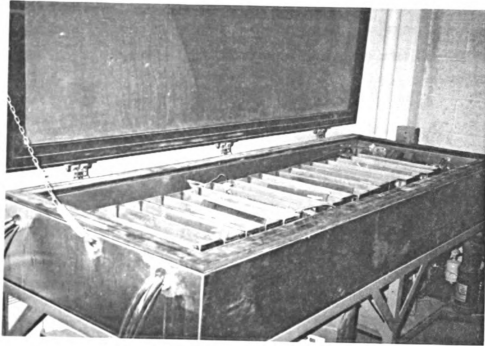


Figure 5.2 Freeze-Thaw Test Apparatus.

Wetting/Drying and Carbonation

Carbonation plays a key role in natural ageing of cellulose fiber-cement composites. Dissolution of calcium hydroxide in pore water and its precipitation within wood fiber cores and at the interface zones would be accompanied with carbonation which turns calcium hydroxide into calcium carbonate and pronounces the weathering effects. Each cycle in this accelerated ageing test consists of 8 hours of saturation under water, heating in oven for one hour at 80° C (176 ° F), carbonation for a period of 5 hours in a rich carbon dioxide environments, heating for 9 hours in oven, followed by cooling for one hour at room temperature

A constant flow carbon dioxide incubator was used to produce the rich carbon dioxide en-

vironment. The carbon dioxide (supply) cylinder regulator was adjusted to provide 0.14 MPa (20 psi) to the unit. The carbon dioxide flowmeter was adjusted to 0.4 liters per minute by the adjusting valve on front of the chamber. The air flowmeter was adjusted to 4.0 liters per minute to obtain 10% CO₂ concentration (rich carbon dioxide environment) in the chamber. The carbonation chamber used in this study is shown in Figure 5.3.

The extent of carbonation was determined using thermogravimetric analysis and x-ray diffraction. Density of the optimized composite was also determined to investigate any physical changes accompanied with chemical changes before and after the wetting-drying cycles and carbonation.

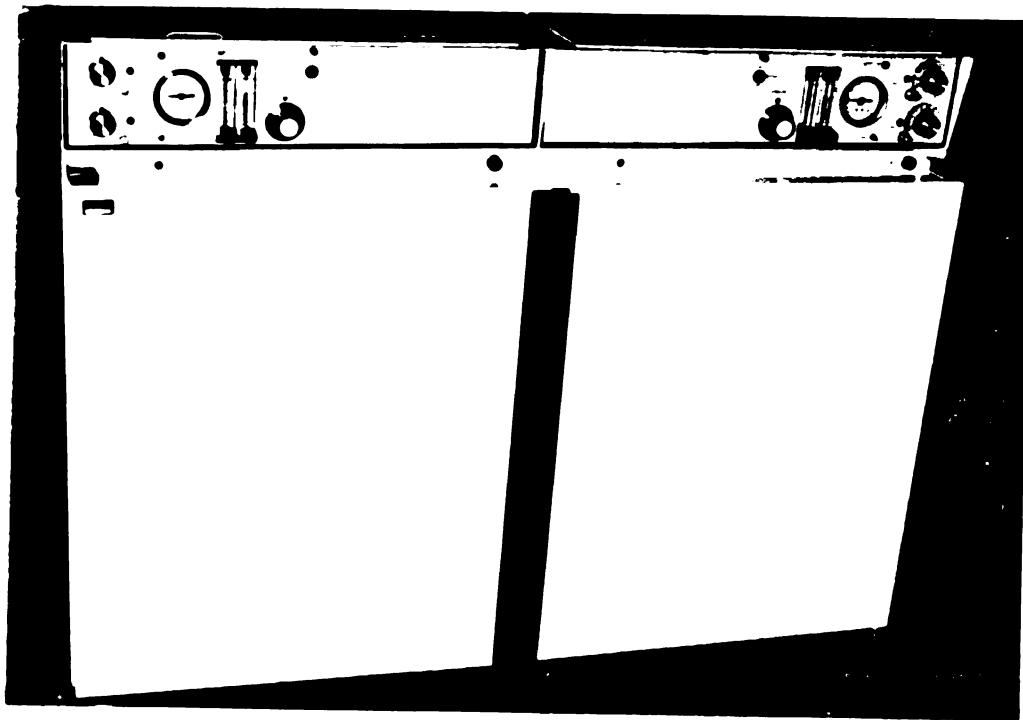


Figure 5.3 Carbonation Chamber Producing Rich Carbon Dioxide Environment.

Hot Water Bath

Any deleterious chemical reactions (e.g. alkali attack of pore water on some wood fiber constituents) taking place under natural ageing would be accelerated upon immersion in

hot water. ASTM C1185 specifications were followed; the temperature of water was 60° C (140 °F), and the immersion period was 55 days.

Ten replicated panel specimens (12 x 4.5 x 0.25 in) were soaked in hot water. Flexural tests were performed according to the Japanese Specification JCI-SF [77]. The test results on aged specimens were compared with those of saturated unaged specimens. The hot water bath used in this investigation is shown in Figure 5.4

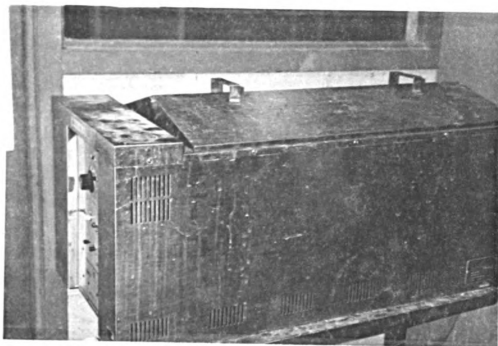


Figure 5.4 Hot Water Bath.

Permeability

Water permeation through capillary pores into cement-based materials is fundamental to their long-term durability characteristics. In hydrated cement paste, the size and continuity of the pores at any point during the hydration process would control the coefficient of permeability. A direct water permeability test set up was designed (see Figure 5.5). The water permeated was measured and the coefficient of permeability was determined using Darcy's expression.

The test specimens used for this test was cylindrical with 100 mm (4 in) diameter and 6 mm (0.25 in) thickness. The saturated test specimen is placed in between plexiglass rings and clamped by four threaded bars. The specimen perimeter surface is glued in order to ensure all permeation is across the thickness of the specimen. Top portion of the ring is filled with care to ensure all air bubbles are eliminated during filling and a head of water about 300 mm (12 in) is formed, in the upper pipet. The drop in head is noted after 24 hours. The observations are continued until a steady state flow is achieved, and permeability coefficient is then calculated.

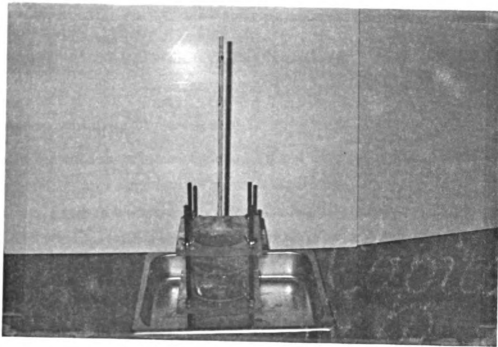


Figure 5.5 Direct Water Permeability Test Set Up.

5.2.3 Microstructural Investigations

Changes in the microstructure of composites upon ageing were investigated using the Scanning Electron Microscopy and thermogravimetry techniques. X-ray diffraction was also used to confirm the results of thermogravimetric analysis. These microstructural test procedures are briefly described in this section.

Scanning Electron Microscopy: In order to investigate the ageing mechanism and the

corresponding microstructural changes, fractured surfaces of the composites were observed under SEM. The focus was to observe changes in fiber morphology and failure modes, fiber-matrix interface zones and the matrix upon ageing.

Thermogravimetry: Thermogravimetric analysis was used to determine compositional changes (in calcium hydroxide and calcium carbonate content) in wastepaper fiber-cement composites under various accelerated ageing conditions.

X-Ray Diffraction: In order to supplement and support the thermogravimetric observations, x-ray diffraction was used to determine the compositional changes (in calcium hydroxide and calcium carbonate content) upon ageing.

5.3 MOISTURE AND AGEING EFFECTS ON ENGINEERING PROPERTIES

5.3.1 Moisture Sensitivity

The flexural load-deflection behavior of recycled and virgin wood fiber-cement composites at different moisture conditions are shown in Figure 5.6a. It appears that the increase in moisture to saturated level adversely affects the flexural strength and stiffness of both virgin and recycled composites but causes an increase in flexural toughness (see Table 5.1 and Figures 5.6 b through 5.6 d).

Results of the analysis of variance of test data is presented in Table 5.2. One way analysis of variance of flexural strength, stiffness and toughness test results at different moisture contents confirmed the moisture sensitivity of recycled and virgin composites.

There was a significant difference in flexural strength for various conditions of moisture content in virgin and recycled composite. However, air-dried and oven dried recycled composites proved to be statistically equivalent in flexural strength but different in flexural toughness and stiffness at 95% confidence level (Using multiple comparison by contrast). Two-way analysis of variance of results was also conducted for different composites (recycled versus virgin) and moisture conditions. The fiber-moisture interaction proved to be statistically significant at 95% level of confidence, indicating that moisture effects on recycled composites differ from those on virgin composites. Damaging effects of saturation are less pronounced in the case of recycled fibers. Upon saturation recycled composites,

when compared with air-dried condition, has a drop of 37% in flexural strength and 16% in initial stiffness, while the corresponding drops in virgin composites were 47% and 28%, respectively. Both recycled and virgin composites showed an increase of 32% in flexural toughness upon saturation.

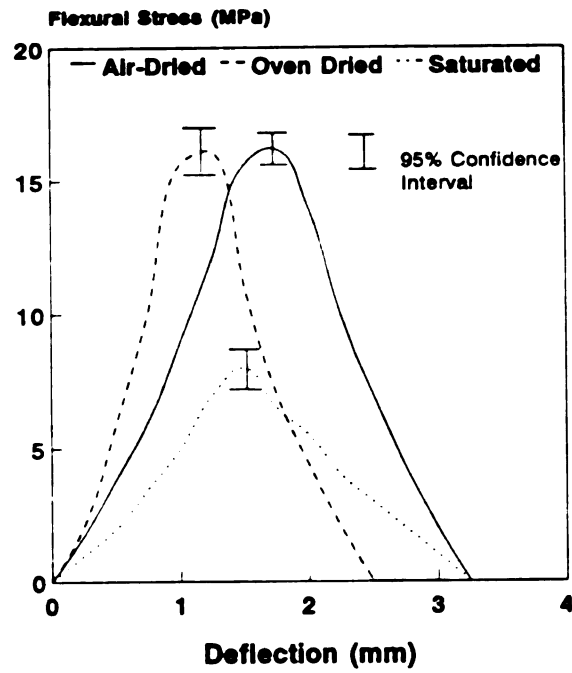
Table 5.1 Flexural Performance of Recycled Wastepaper Fiber-Cement Composite at Different Moisture Conditions

Experiment	Flex. Str. (MPa)	Mean Flex. Str (95% Con. Int.)	Flex. Tou. N-mm	Mean Tou. (95% Con. Int.)	Init. Stif. N/mm	Mean Init Stif. (95% Con. Int.)
Control Saturated	8.2	8.06 (± 28)	91.234	90.72 (± 11.4)	119.90	118.6 (± 19.9)
	7.993		89.887		121.29	
	8.114		87.81		130.32	
	8.003		92.431		115.87	
	8.117		91.098		117.32	
	8.007		85.125		110.43	
	7.996		90.543		115.55	
	8.01		89.67		117.98	
	8.12		95.098		119.56	
	8.09		94.333		118.54	
Control Air Dried	15.321	15.09 (± 63)	70.39	67.88 (± 8)	171.17	164.35 (± 18.1)
	15.003		69.342		170.77	
	14.894		66.455		166.33	
	14.998		63.22		167.45	
	14.888		67.87		163.38	
	15.226		66.43		160.23	
	15.115		68.23		159.98	
	15.301		69.09		157.78	
	15.227		69.02		160.98	
	15.02		68.78		165.43	

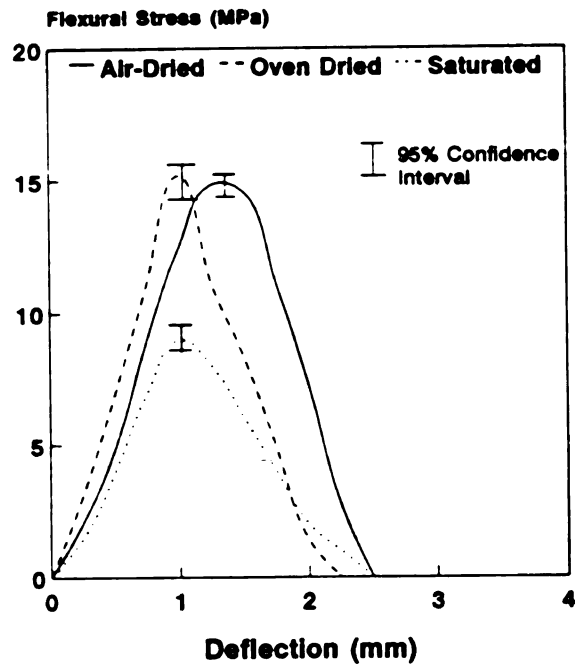
Control Oven dried	15.72	15.38 (± 0.88)	53.234	51.99 (± 7.84)	189.32	151.66 (± 10.2)
	15.12		54.123		180.87	
	15.439		50.669		183.66	
	15.445		52.144		182.87	
	15.056		50.1		179.95	
	15.554		48.21		183.32	
	15.221		48.321		185.79	
	15.666		53.872		184.45	
	15.221		50.989		183.24	
	15.432		51.996		185	
Recycled Saturated	9.554	9.392 (± 0.5)	80.003	77.512 (± 7.0)	155.32	151.66 (± 8.7)
	9.112		78.211		154.22	
	9.443		77.439		153.87	
	9.234		77.55		150.37	
	9.501		80.891		152.12	
	9.456		76.8		153.87	
	9.342		75.124		149.96	
	9.37		76		150.27	
	9.531		75.898		150.44	
	9.493		77.205		148.99	
Recycled air dried	15	14.91 (± 0.34)	59.999	58.65 (4.72)	183.98	180.51 (7.52)
	14.891		60.21		182.34	
	14.995		58.367		182.34	
	14.992		59.381		180.54	
	14.89		58.002		180.65	
	14.95		57.10		180.22	
	14.96		57.98		178.43	
	14.765		56.993		177.87	
	14.779		58.554		178.87	
	14.914		60.013		179.87	
Recycled Oven dried	15.31	15.03 (± 0.42)	54.11	53.23 (± 5.75)	234.46	228.53 (± 21.5)
	15.029		54.123		230.00	
	15.002		54.987		221.98	
	15.01		53.1		225.55	
	15.08		54.876		228.11	
	14.997		53.223		218.89	
	15.026		54.775		232.32	
	14.96		52.092		236.33	
	14.992		51.796		230.34	
	14.9		51.285		227.34	

Table 5.2 Results of the Analysis of Variance (Flexural Strength, Toughness and Initial Stiffness)

[illegible]



Virgin

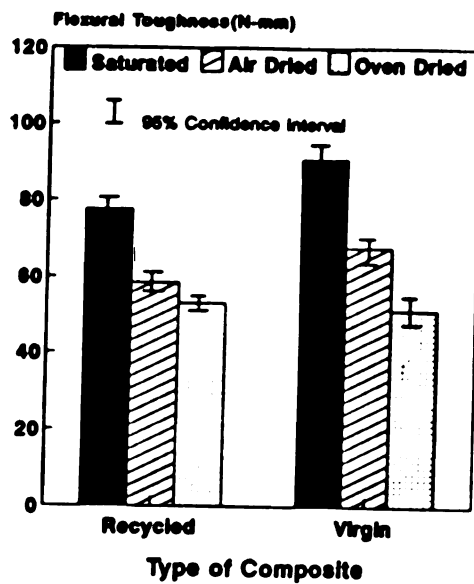
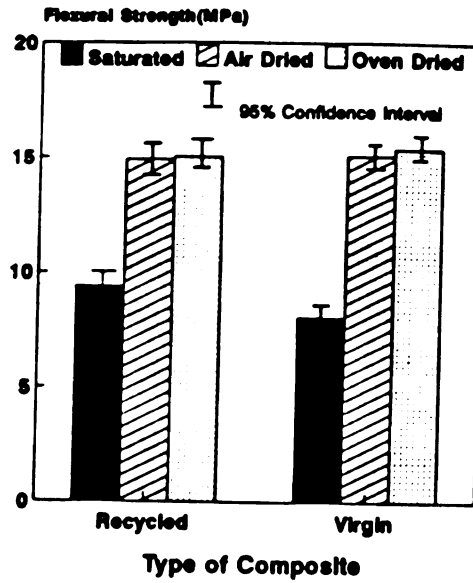


Recycled

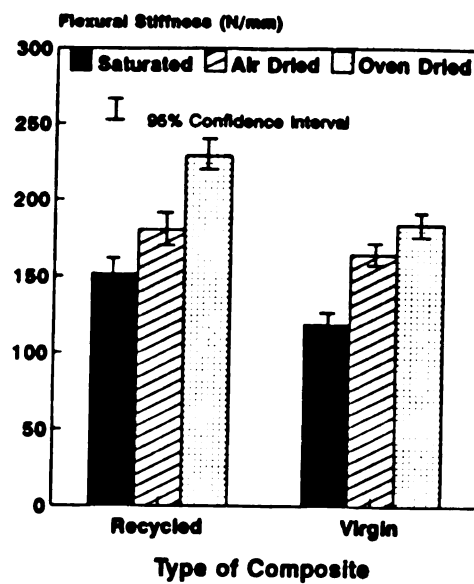
(a) Load-Deflection Behavior

Figure 5.6 Moisture Effects on Flexural Behavior of Recycled and Virgin Wood Fiber-Cement Composites.

(b) Flexural Strength



c Toughness



d Initial Stiffness

Figure 5.6 (Cont'd.) Moisture Effects on Flexural Behavior of Recycled and Virgin Wood Fiber-Cement Composites.

5.3.2 Repeated Wetting and Drying

Figure 5.7a presents the effects of repeated wetting-drying cycles on the flexural load-deflection behavior of recycled and virgin cellulose fiber-cement composites. The effects of repeated wet-dry cycles on flexural strength, stiffness and toughness of recycled and virgin composites are presented in Table 5.3 and Figures 5.2 b-d. In both cases of virgin and recycled composites it is observed that repeated wetting and drying leads to increase in flexural strength and initial stiffness whereas the toughness is reduced substantially and failure occurs in a brittle mode.

Results of the statistical analysis of the test data Table 5.3 are presented in Table 5.4. Repeated wetting-drying cycles had statistically significant effects on all flexural properties (strength, toughness and stiffness) of recycled composites. Two-way analysis of variance (with two factors of recycled versus virgin fibers and aged versus unaged composites) revealed that there was a statistically significant difference, at 95% level of confidence, between ageing effects on recycled and virgin composites as far as flexural strength and toughness are concerned; the initial stiffness of virgin and recycled composites, however, effected similarly by ageing under repeated wetting-drying cycles. Damaging effects of repeated wet-dry cycles on recycled composites (which lost 32% of toughness upon ageing was less than that on virgin composites (which lost 45% of toughness upon ageing).

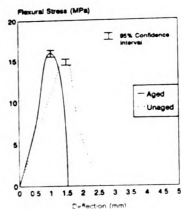
Table 5.3 Repeated Wetting-Drying Effects on the Flexural Performance of Recycled and Virgin Fiber-Cement Composites

Experiment	Flex. Str. (MPa)	Mean Flex. Str (95% Conf. Int.)	Flex. Tou. N-mm	Mean Tou. (95% Conf. Int.)	Init. Stif. N/mm	Mean Init Stif. (95% Conf. Int.)
Control	15.70	15.71	39.99	37.32	228.3	229.21
Aged	15.66	(± 0.5)	40.11	(± 7.39)	235.4	(± 19.6)
	15.55		35.76		227.9	
	15.60		36.77		228.2	
	15.67		37.87		220.0	
	15.67		37.99		226.5	
	15.63		35.87		233.5	
	15.87		36.44		235.1	
	15.98		40.10		223.9	
	15.76		35.32		232.8	

Recycled Aged	15.101 14.992 15.002 14.987 14.987 14.994 14.998 15.105 15.112 15.006	15.04 (± 0.22)	38.11 39.02 37.97 40.23 43.23 41.23 40.23 39.19 41.22 41.98	40.241 (± 6.6)	249.09 240.98 240.65 240.98 245.67 243.98 249.87 248.88 249.92 248.99	245.9 (± 15.4)
Control Unaged	15.1 15.02 15.43 15.1 14.99 14.98 15.11 15.09 14.96 15.11	15.09 (± 0.51)	67 66.39 63.12 69.34 70.74 69.43 66.98 66.12 67.38 66.55	67.3 (± 8.3)	164.4 154.3 171.6 167.4 152.2 162.4 159.9 157.7 162.9 165.7	161.9 (± 23.3)
Recycled Unaged	14.9 14.88 14.77 14.92 14.89 14.81 14.75 14.92 14.81 14.86	14.85 (± 0.23)	58 57.23 59.98 60.11 58.23 59.12 58.87 59.98 60.34 59.22	59.11 (± 4.0)	180.2 178.2 180.9 189.2 187.7 185.9 186.7 186.6 187.9 177.7	184.1 (± 17.1)

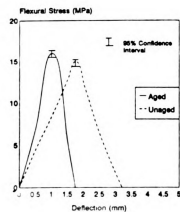
Table 5.4 Results of Analysis of Variance (flexural strength, toughness and stiffness)

Source	Sum-of Sq.	DF	Mean-Sq.	F-Ratio	P
One Way Analysis of Variance					
Flexural Strength					
Ageing(wd)	0.072	1	0.072	11.59	0.009
Error	0.05	8	0.006		
Toughness					
Ageing(wd)	973.18	1	973.18	659.58	0.00
Error	11.8	8	1.475		
Initial Stiffness					
Ageing(wd)	9778.1	1	9778.1	890.1	0.00
Error	87.88	8	10.98		
Two way Analysis of Variance					
Flexural Strength					
Fiber	1.10	1	1.10	91.92	0.00
Ageing(wd)	0.77	1	0.77	64.55	0.00
Fiber *Age.	0.20	1	0.20	17.17	0.00
Error	0.19	16	0.01		
Toughness					
Fiber	44.01	1	44.01	10.68	0.01
Ageing(wd)	3141.27	1	3141.27	762.25	0.00
Fiber*Age	170.47	1	170.47	0.00	
Error	65.93	16	4.12		
Initial Stiffness					
Fiber	959.39	1	959.39	64.02	0.00
Ageing(wd)	20147.2	1	20147.2	1344.52	0.00
Fiber*Age.	0.51	1	0.51	0.03	0.85
Error	239.756	16	14.98		

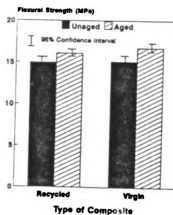


Recycled

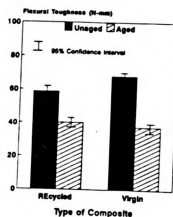
a Load Deflection



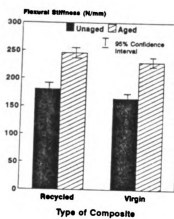
Virgin



b Flexural Strength



c Toughness



d Initial Stiffness

Figure 5.7. Effect of Repeated Wetting and Drying Cycles on Flexural Behavior.

5.3.3 Repeated Freeze-Thaw

The recycled and virgin cellulose fiber-cement composites were tested for flexural performance before and after exposure to freeze-thaw cycles. The effects of repeated freeze-thaw cycles on flexural load-deflection behavior are presented in Figure 5.10a. Table 5.5 and Figures 5.8 b-d show the freeze-thaw effects on flexural strength, stiffness and toughness of virgin and recycled composites. No delamination was observed in the specimens subjected to repeated freeze-thaw cycles; the aged specimens were intact and marginally stiffer than unaged optimized composites.

Results of the analysis of variance of test data are presented in Table 5.6. It was concluded that freeze thaw ageing did not have statistically significant effect on flexural strength and toughness of recycled composite; however, the effect on initial stiffness was statistically significant at 95% level of confidence. Two way analysis of variance (taking into account the virgin composite) revealed that freeze-thaw cycles affect recycled composites in a way different from virgin composites (similar effects were observed on flexural strength and toughness irrespective of the composite type). While recycled composites showed a 1% increase in stiffness under repeated freeze-thaw cycles, virgin composites showed a drop of 9% in stiffness under this ageing condition.

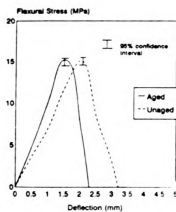
Table 5.5 Repeated Freeze-Thaw Effects on the Flexural Performance of Recycled and Virgin Fiber-Cement Composites.

Experiment	Flex. Str. (MPa)	Mean Flex. Str (95% Conf.Int.I	Flex. Tou. N-mm	Mean Tou. (95% Conf. Int.)	Init. Stif. N/mm	Mean Init Stif. (95% Conf. Int.)
Control Aged	15.00 14.99 15.11 15.01 14.95 14.99 14.93 14.82 14.99 14.91	14.97 (± 0.29)	67.334 67.34 68.21 68.43 65.98 66.32 66.87 65.54 66.93 67.87	66.88 (± 3.27)	170.32 170.11 168.84 167.32 166.43 167.79 169.32 168.76 169 168.87	168.68 (± 4.71)

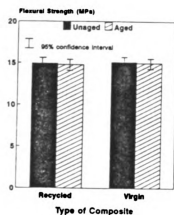
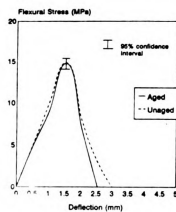
Control Unaged	15.1 15.02 15.43 15.1 14.99 14.98 15.11 15.09 14.96 15.11	15.09 (± 1.1)	67 66.39 63.12 69.34 70.74 69.43 66.98 66.12 67.38 66.55	67.3 (± 3.2)	164.4 154.3 171.6 167.4 152.2 162.4 159.9 157.7 162.9 165.7	161.9 (± 2.9)
Recycled Aged	14.80 14.79 14.7 14.79 14.78 14.80 14.79 14.79 14.83 14.81	14.79 (± 0.06)	59.08 59.10 58.09 57.09 56.12 57.88 58.5 58.39 59.07 58.34	58.177 (± 3.74)	187.56 186.65 187.54 187.43 185.55 185.39 187.6 186.53 188.54 187.54	187.04 (± 3.89)
Recycled Unaged	14.9 14.88 14.77 14.92 14.89 14.81 14.75 14.92 14.81 14.86	14.85 (± 0.23)	58 57.23 59.98 60.11 58.23 59.12 58.87 59.98 60.34 59.22	59.11 (± 4.0)	180.2 178.2 180.9 189.2 187.7 185.9 186.7 186.6 187.9 177.7	184.1 (± 17.1)

Table 5.6 Results of Analysis of Variance (flexural strength, toughness and stiffness)

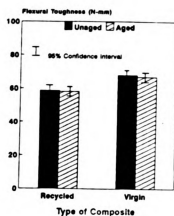
Source	Sum-of Sq.	DF	Mean-Sq.	F-Ratio	P
One Way Analysis of Variance					
Flexural Strength					
Ageing(FT)	0.01	1	0.01	10.5	0.012
Error	0.008	8	0.001		
Toughness					
Ageing(FT)	2.246	1	2.246	1.13	0.317
Error	15.788	8	1.974		
Initial Stiffness					
Ageing(FT)	8.31	1	8.31	7.66	0.02
Error	8.68	8	1.08		
Two way Analysis of Variance					
Flexural Strength					
Fiber	0.18	1	0.18	56.32	0.00
Ageing(FT)	0.01	1	0.01	2.81	0.11
Fiber *Age.	0.00	1	0.00	0.028	0.87
Error	0.051	16	0.003		
Toughness					
Fiber	308.03	1	308.03	278.7	0.00
Ageing(FT)	3.72	1	3.72	3.38	0.08
Fiber* Age	0.06	1	0.06	0.054	0.819
Error	17.62	16	1.101		
Initial Stiffness					
Fiber	2296.3	1	2296.3	1053.89	0.00
Ageing(FT)	119.786	1	119.78	54.97	0.00
Fiber* Age.	30.71	1	30.71	14.09	0.002
Error	34.863	16	2.179		



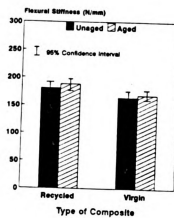
a Load Deflection



b Flexural Strength



c Toughness



d Initial Stiffness

Figure 5.8 Effects of Repeated Freeze-thaw Cycles on Flexural Behavior.

5.3.4 Repeated Wetting-Drying and Carbonation

This accelerated condition seems to best simulate natural weathering effects on cellulose fiber-cement composites [32]. The effect of repeated wetting-drying and carbonation on flexural load-deflection behavior is presented in Figure 5.9a. This ageing process is observed to increase flexural strength and initial stiffness while causing embrittlement of the composite.

The test data of Table 5.7 was analyzed statistically (Table 5.8). Wetting-drying and carbonation ageing had (in one way analysis of variance) statistically significant effect on flexural strength, toughness and stiffness. In addition, the fiber*ageing interactions were statistically significant at 95% level of confidence for all the three flexural responses (strength, toughness and stiffness), indicating that this ageing process affects recycled and virgin composites differently. The damaging effects of repeated wetting-drying and carbonation on recycled composites (47% loss) was less pronounced than for virgin composites (54% drop).

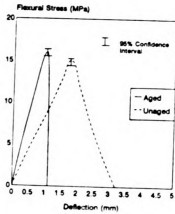
Table 5.7 Effects of Repeated Wetting-Drying and Carbonation on the Flexural Performance of Recycled and Virgin Fiber-Cement Composites

Experiment	Flex. Str. (MPa)	Mean Flex. Str (95% Conf. Int.)	Flex. Tou. (N-mm)	Mean Tou. (95% Conf. Int)	Init. Stif. (N/mm)	Mean Init Stif. (95% Conf. Int.).
Control	16.11	16.13	32.12	31.05	273.23	270.40
Aged	16.20	(± 0.93)	27.45	(± 8.12)	260.56	(± 23.2)
	16.19		28.54		280.54	
	16.20		31.99		268.67	
	16.15		29.09		267.87	
	16.2		33.34		270.98	
	16.25		32.87		278.78	
	16.54		31.22		265.54	
	15.77		30.00		267.31	
	15.712		33.87		270.54	

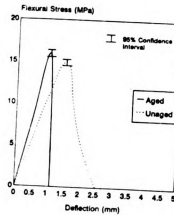
Recycled. Aged	15.62 15.55 15.76 15.55 15.75 15.66 15.61 15.63 15.71 15.67	15.65 (± 0.29)	33.19 32.76 29.67 34.76 32.98 31.87 32.87 28.00 27.98 29.11	31.322 (± 9.48)	270.12 265.87 275.98 272.65 269.98 267.77 263.87 275.65 276.76 270.41	270.91 (± 17.1)
Control Unaged	15.1 15.02 15.43 15.1 14.99 14.98 15.11 15.09 14.96 15.11	15.09 (± 0.51)	67 66.39 63.12 69.34 70.74 69.43 66.98 66.12 67.38 66.55	67.3 (± 8.3)	164.4 154.3 171.6 167.4 152.2 162.4 159.9 157.7 162.9 165.7	161.9 (± 23.3)
Recycled Unaged	14.9 14.88 14.77 14.92 14.89 14.81 14.75 14.92 14.81 14.86	14.85 (± 0.23)	58 57.23 59.98 60.11 58.23 59.12 58.87 59.98 60.34 59.22	59.11 (± 4.0)	180.2 178.2 180.9 189.2 187.7 185.9 186.7 186.6 187.9 177.7	184.1 (± 17.1)

Table 5.8 Results of Analysis of Variance (flexural strength, toughness and stiffness)

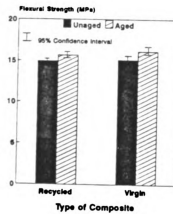
Source	Sum-of Sq.	DF	Mean-Sq.	F-Ratio	P
One Way Analysis of Variance					
Flexural Strength					
Age.(WDC)	2.134	1	2.134	380.13	0.00
Error	0.045	8	0.006		
Toughness					
Age.(WDC)	2018.19	1	2018.19	524.99	0.00
Error	30.75	8	3.84		
Initial Stiffness					
Age.(WDC)	19380	1	19380	615	0.00
Error	252.08	8	31.51		
Two way Analysis of Variance					
Flexural Strength					
Fiber	0.702	1	0.702	229.72	0.00
Age.(WDC)	4.5	1	4.5	1476	0.00
Fiber *Age.	0.131	1	0.131	42.75	0.00
Error	0.049	16	0.003		
Toughness					
Fiber	53.694	1	53.694	13.158	0.002
Age.(WDC)	5048	1	5048	1237	0
Fiber*Age	78.28	1	78.28	19.18	0.00
Error	65.28	16	4.081		
Initial Stiffness					
Fiber	265.64	1	265.64	10.08	0.006
Age.(WDC)	47821	1	47821	1815	0.00
Fiber*Age.	336.1	1	336.1	12.76	0.003
Error	421.375	16	26.336		



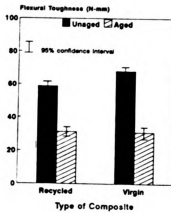
Virgin



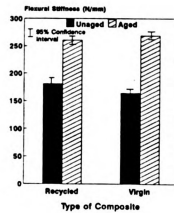
Recycled



b Flexural Strength



c Toughness



d Initial Stiffness

Figure 5.9 Effects of Repeated Wetting-Drying and Carbonation on Flexural Behavior.

5.3.5 Extended Immersion in Hot Water Bath

Long-term immersion in hot water bath had relatively small effects on the flexural performance of virgin and recycled fiber-cement composites; this ageing process resulted in some embrittlement of the flexural behavior (Figure 5.10 and Table 5.9).

Results of the analysis of variance of test data are presented in Table 5.10. The effect of long-term immersion in hot water on flexural strength, stiffness and toughness of recycled composites was not statistically significant at 95% level of confidence. Two-way analysis of variance of results (virgin and recycled fibers) concluded that only flexural stiffness of virgin and recycled composites was affected differently by this ageing process. While hot water immersion slightly reduced the initial stiffness of recycled composites (by 3%) it caused a small increase of 3% in the stiffness of virgin composites.

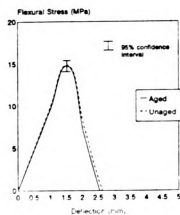
Table 5.9 Effects of Hot Water Immersion on the Flexural Performance of Recycled and Virgin Fiber-Cement Composites

Experiment	Flex. Str. (MPa)	Mean Flex. Str (95% Conf. Int.)I	Flex. Tou. (N-mm)	Mean Tou. (95% Con. Int.)	Init. Stif. (N/mm)	Mean Init Stif. (95% Con. Int.)
Control	15.07	15.09	61.13	63.43	165.43	168.3
100% virgin	14.98	(± 0.28)	62.45	(± 6.93)	166.76	(± 12.3)
Aged	15.06		67.32		170.54	
	15.12		63.33		162.34	
	15.11		62.98		171.11	
	15.09		63.54		169.87	
	15.1		65.45		168.43	
	15.09		63.22		165.67	
	15.16		61.98		172.09	
	15.23		62.87		170.72	

Control Unaged	15.1	15.09 (± 0.51)	67	67.3 (± 8.3)	164.4	161.9 (± 23.3)
	15.02		66.39		154.3	
	15.43		63.12		171.6	
	15.1		69.34		167.4	
	14.99		70.74		152.2	
	14.98		69.43		162.4	
	15.11		66.98		159.9	
	15.09		66.12		157.7	
	14.96		67.38		162.9	
	15.11		66.55		165.7	
Recycled Aged	14.80	14.81 (± 0.15)	63.32	59.59 (± 14)	184.32	178.93 (± 13.57)
	14.77		54.43		183.33	
	14.79		55.54		178.98	
	14.80		57.12		180.56	
	14.82		55.23		175.43	
	14.79		61.09		172.87	
	14.81		62.23		176.87	
	14.80		63.32		178.45	
	14.92		62.54		177.99	
	14.78		62.21		180.54	
Recycled Unaged	14.9	14.85 (± 0.23)	58	59.11 (± 4.0)	180.2	184.1 (± 17.1)
	14.88		57.23		178.2	
	14.77		59.98		180.9	
	14.92		60.11		189.2	
	14.89		58.23		187.7	
	14.81		59.12		185.9	
	14.75		58.87		186.7	
	14.92		59.98		186.6	
	14.81		60.34		187.9	
	14.86		59.22		177.7	

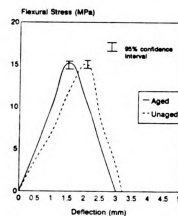
Table 5.10 Results of Analysis of Variance (flexural strength, toughness and stiffness)

Source	Sum-of Sq.	DF	Mean-Sq.	F-Ratio	P
One Way Analysis of Variance					
Flexural Strength					
Age.(HW)	0.00	1	0.00	0.097	0.764
Error	0.021	8	0.003		
Toughness					
Age.(HW)	1.421	1	1.421	0.108	0.751
Error	105.51	8	13.189		
Initial Stiffness					
Age.(HW)	1.183	1	1.183	0.11	0.748
Error	85.712	8	10.714		
Two way Analysis of Variance					
Flexural Strength					
Fiber	0.27	1	0.27	112.746	0.00
Age.(HW)	0.00	1	0.00	0.042	0.84
Fiber *Age.	0.00	1	0.00	0.117	0.736
Error	0.038	16	0.002		
Toughness					
Fiber	171.4	1	171.4	27.15	0.00
Age.(HW)	34.03	1	34.03	5.39	0.034
Fiber*Age	0.286	1	0.286	0.045	0.834
Error	100.99	12	6.312		
Initial Stiffness					
Fiber	1540.89	1	1540.89	91.45	0.00
Age.(HW)	86.32	1	86.32	5.12	0.038
Fiber*Age.	128.37	1	128.37	7.619	0.014
Error	269.579	16	16.849		



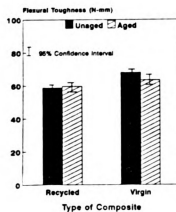
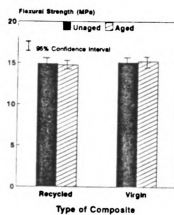
Recycled

a Load Deflection

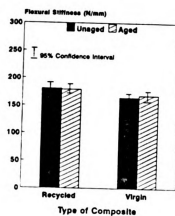


Virgin

(b) flexural Strength



c Toughness



d Initial Stiffness

Figure 5.10 Effects of Hot Water Bath Immersion on Flexural Behavior.

5.3.6 Comparison of Different Ageing Effects

In order to compare the effects of different accelerated ageing conditions on the flexural performance of recycled and virgin fiber-cement composites (see Figure 5.11), two-way analysis of variance of ratios of aged to unaged flexural strength, stiffness and toughness test results were conducted. The two factors in these analyses were: composite type (recycled and virgin), and accelerated ageing condition (wet-dry, wet-dry and carbonation, freeze-thaw, hot water immersion). Results of the analysis of variance are presented in Table 5.11. the global analysis of variance suggested that, at 95% level of confidence, all the ageing effects have comparable effects on flexural strength but there are statistically significant differences between the effects of different accelerated ageing conditions on flexural stiffness and toughness. There was no statistically significant difference between virgin and recycled composites as far as ageing effects on flexural strength and stiffness are concerned; the two composites, however, behaved differently in ageing effects on flexural toughness.

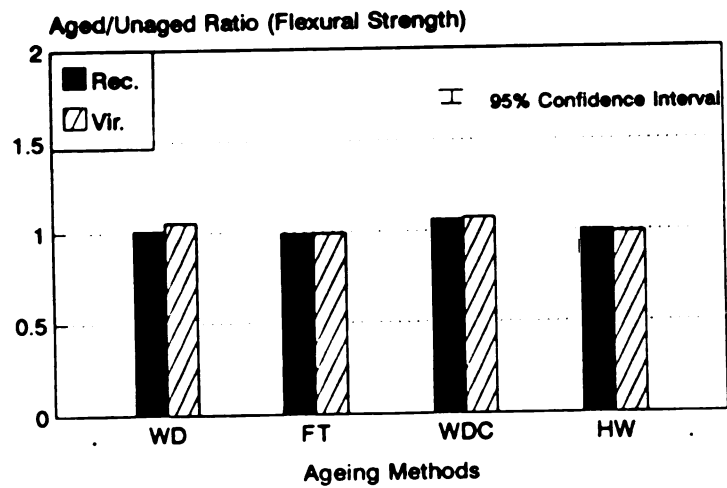
Multiple comparison of results indicated, at 95% level of confidence, that each of the accelerated conditions have distinctly different effect on flexural toughness and stiffness, except for the hot water immersion and freeze-thaw condition which had statistically comparable effects on flexural stiffness.

As far as the overall ageing effects on flexural performance is concerned, repeated wetting-drying and carbonation cycles produced the most pronounced effects whereas hot water immersion caused the least effects. The trends in the response of virgin and recycled composites to ageing were generally comparable.

Table 5.11 Results of Analysis of Variance of Different Ageing Methods**(see Appendix III for notations)**

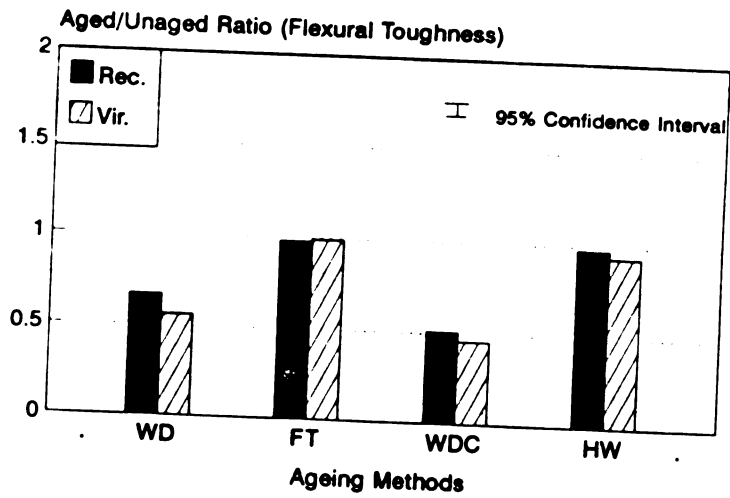
Source	Sum-of Sq.	DF	Mean-Sq.	F-Ratio	P
One Way Analysis of Variance					
Flexural Strength					
Age. meth-ods	0.009	3	0.003	2.189	0.167
Error	0.011	8	0.001		
WD VS WDC	0.002	1	0.002	1.494	0.256
Error	0.011	8	0.011		
WDC VS HW	0.002	1	0.002	1.235	0.299
Error	0.011	8	0.001		
Toughness					
Age. meth-ods	0.48	3	0.16	548.52	0.00
Error	0.002	8	0.00		
WD Vs FT	0.144	1	0.144	494.22	0.00
Error	0.002	8	0.00		
WD WDC	0.035	1	0.035	120.914	0.00
Error	0.002	8	0.00		
WD Vs HW	0.141	1	0.141	483.657	0.00

Error	0.002	8	0.00		
Stiffness					
Age. Meth-ods	0.534	3	0.178	296.83	0.00
Error	0.005	8	0.001		
WDC Vs HW	0.375	1	0.375	625	0.00
Error	8	0.001			
FT Vs HW	0.00	1	0.00	0.25	0.63
Error	0.005	8	0.001		

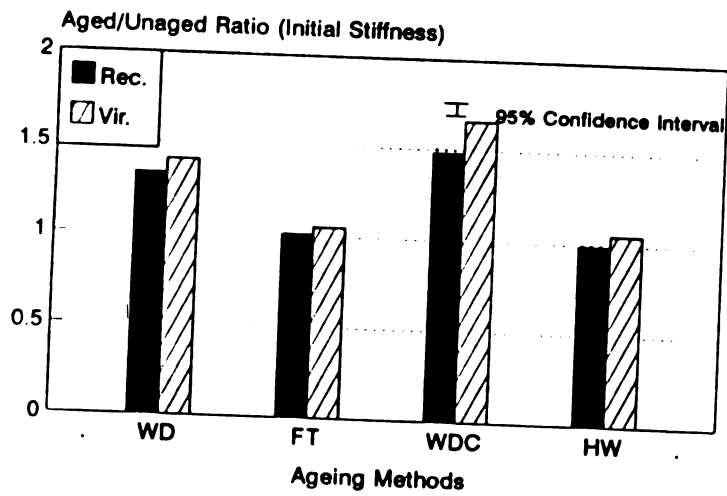


Flexural Strength

Figure 5.11 Comparison of Different Ageing Methods



Toughness



Stiffness

Figure 5.11 (Cont'd.) Comparison of Different Ageing Methods

5.3.7 Permeability

The permeability coefficient was measured in three replications for the optimized recycled and virgin cellulose fiber-cement composites, and also for the cement-based matrix with no fibers all in unaged and aged (after repeated wetting-drying and carbonation) conditions. The permeability test results are presented in Table 5.12 and Figure 5.12. The recycled composite is observed to have a lower permeability coefficient (24×10^{-11} cm/sec, 9×10^{-11} in/sec) than virgin composites (36×10^{-11} cm/sec, 14×10^{-11} in/sec) in unaged condition. This further confirms the better consolidation of recycled composites (where the fines in fiber play a filler role and, unlike fibers, facilitate consolidation) reflected in the higher density and lower water absorption of recycled composites (when compared with virgin ones). Fibrous composites, however, had permeability coefficient higher than the plain cement-based matrix (6×10^{-11} cm/sec, 2×10^{-11} in/sec).

Table 5.12 Permeability Coefficient of Virgin and Recycled Composites

Type of Composite	Permeability Coefficient (10^{-11} cm/sec)	Mean (95% Confidence Interval)
Control (Unaged)	36.6,38.7,35.3	36.86 (± 6.72)
Recycled (Unaged)	22.0,24.5,25.0	23.83 (± 6.30)
Control (Aged)	33.0,26.7,40.0	33.23 (6.65)
Recycled (Aged)	19.2,18.7,19	18.96 (± 0.98)

One way analysis of variance was carried out on permeability test results (Table 5.13). Permeability coefficients of different composites found to be statistically different at 95% level of confidence; recycled composites had a lower permeability than virgin composites. The effect of ageing on reducing the permeability coefficient was also statistically significant. This observation could be attributed to the densification effects of ageing of the fiber cores and interfaces.

Two-way analysis of variance with composite type (virgin versus recycled) and age (unaged versus aged) the two factors indicated that while recycled and control composite had

different permeability at 99% level of confidence, ageing effects on permeability was significant at 95% level of confidence. The recycled and virgin composites were also observed to perform similarly as far as the ageing effects on permeability is concerned.

Table 5.13 Results of the Analysis of Variance of Permeability Coefficients

Source	Sum-of-Sq.	DF	Mean-Sq.	F-Ratio	P
Effect of Type (recycled, control)					
type of Composite	1463.48	2	731.74	366.48	0.00
Error	11.98	6	1.997		
Effect of Ageing					
type of composite	1907.9	5	381.5	45.2	0.00
Error	12	101.24	12	8.43	
Two way analysis of variance					
Type	557.849	1	557.849	44.7	0.00
Age	54.53	1	54.53	4.37	0.07
Type*Age	1.19	1	1.19	0.09	0.765
Error	99.82	8	12.47		

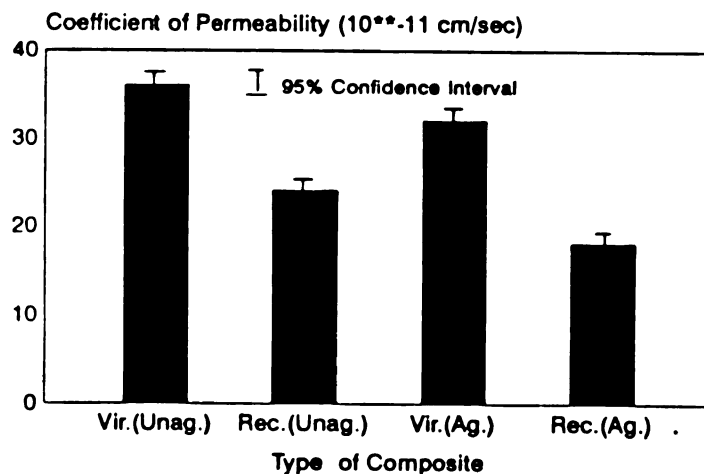


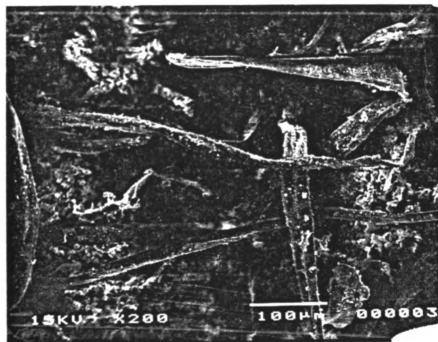
Figure 5.12 Water Permeability Coefficient Test Results.

5.4 MICROSTRUCTURAL AND COMPOSITIONAL CHANGES UNDER MOISTURE AND AGEING EFFECTS

5.4.1 SEM Observations:

The Scanning Electron Micrographs of the fracture surfaces of unaged and aged composites are presented in Figure 5.12 a through 5.12 e. For the unaged composite the dominant mode of failure is observed to be fiber pull out (see Figure 5.12a). In the case of specimens subjected to repeated cycles of freeze-thaw, the fracture surface is observed to have a combination of fiber pull out and fiber fracture (see Figure 5.12b). In the case of hot water immersed composites (Figure 12c) also the mode of failure is observed as fiber pull out accompanied with fiber fracture. For the repeated wetting-drying test the mode of failure at the fracture surface is dominated by fiber fracture (see Figure 5.12d). Also, for repeated wetting-drying and carbonation fiber rupture is observed to dominate the failure mode (see Figure 5.12e).

In addition, the appearance of fibers and their interface is also affected by ageing effects. In the case of wetting-drying and wetting drying and carbonation, a dense fiber matrix interface is observed. the fibers in case of wetting-drying with carbonation appear to be filled with some hydration and carbonation products. In the case of hot water bath and freeze-thaw ageing tests the densification of the interface and filling of fiber core is not pronounced. It appears that the ageing process is most pronounced under repeated wetting-drying and carbonation.



a. Unaged

Figure 5.13 Scanning Electron Micrographs of Fracture Surfaces Under Various Accelerated ageing Conditions.

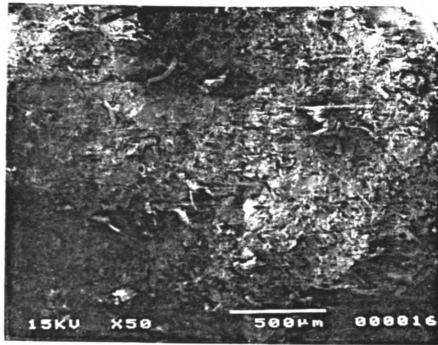


b. After Repeated wetting-Drying Cycles

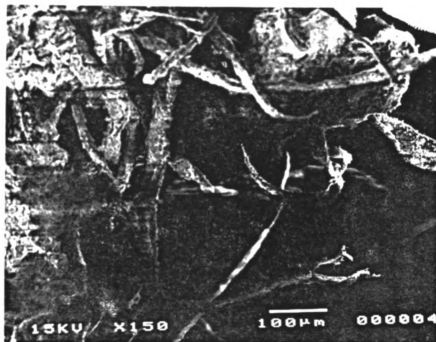


c. After Repeated Freeze-Thaw Cycles

Figure 5.13 Scanning Electron Micrographs of Fracture Surfaces Under Various Accelerated ageing Conditions.



d. After Wetting-Drying and Carbonation



e. After Hot Water Bath

Figure 5.13 (Cont'd) Scanning Electron Micrographs of Fracture Surfaces Under Various Accelerated ageing Conditions.

5.4.2 Thermogravimetric Analysis

Thermogravimetry is a technique in which the mass of a substance is monitored as a function of temperature or time as the sample specimen is subjected to a controlled temperature history [80]. This relationship was used in this investigation to determine the relative quantities of calcium hydroxide and calcium carbonate in the aged and unaged composite, which are expected to correlate to the ageing process of wood fiber-cement composites. Mixing of cement with water results in a complex hydration process. The hardening process over a long period is principally associated with the hydration of the silicate phases. Although the reaction rates of these phases are very different, the final products are the same. In a mature paste, approximately 70% by volume is taken by a colloidal silicate hydrate (CSH). Calcium hydroxide (CH) comprises about 20% of volume [81].

TGA weight loss curves of fiber-cement composites can be interpreted for compositional analysis. Dehydration occurs over the range 105 to 440° C (221 °F to 824 °F), followed by dehydroxylation effecting calcium hydroxide in the range 440 to 580° C (824 ° F to 1076 ° F) with calcium carbonate dissociation occurring in the region 580 to 1000° C (1076 ° F to 1832 °F). Amounts of calcium hydroxide and calcium carbonate can thus be computed based on weight changes at these temperature changes. Free calcium hydroxide can be calculated as follows [81].

$$\text{Free calcium hydroxide} = 4.11 * (Ldx) + 1.68 * (Ldc)$$

where

Ldx: % weight loss within 440 °C to 580 ° C (824 ° F to 1076 ° F)

Ldc: % weight loss within 580 °C to 1000 °C (1076 ° F to 1832 °F)

The calcium carbonate content can be calculated as follows:

$$\text{Calcium carbonate} = \text{Weight loss from } 580^{\circ}\text{C to } 1000^{\circ}\text{C (1076}^{\circ}\text{ F to } 1832^{\circ}\text{ F)}.$$

Typical weight loss curves are presented in Figure 5.18; the calcium hydroxide and calcium carbonate contents may be calculated from these curves.

The amounts of free calcium hydroxide and calcium carbonate for unaged and various

aged composites are given in Table 5.14.

Table 5.14: Thermogravimetric Compositional Analysis

Type of Ageing Treatment	CH (Weight%)	Mean (95% Con. Int.)	CC (Weight%)	Mean (95% Con. Int.)
Unaged	22.21, 22.9, 21.9	22.29 (± 2.33)	3.12, 3.05, 3.37	3.18 (± 0.53)
Aged (WD)	17.7, 18.23, 17.8	17.94 (± 1.05)	7.94, 8.04, 7.78	7.92 (± 0.42)
Aged (WDC)	18.9, 18.45, 18.11	18.49 (± 1.55)	8.38, 8.12, 8.47	8.32 (± 0.58)
Aged (FT)	21.03, 20.96, 21.1	21.04 (± 0.35)	4.33, 4.23, 4.41	4.32 (± 0.28)
Aged (HW)	17.03, 16.67, 16.9	16.89 (± 0.76)	5.47, 5.39, 5.54	5.46 (± 0.24)

CC: Calcium Carbonate; CH Calcium Hydroxide

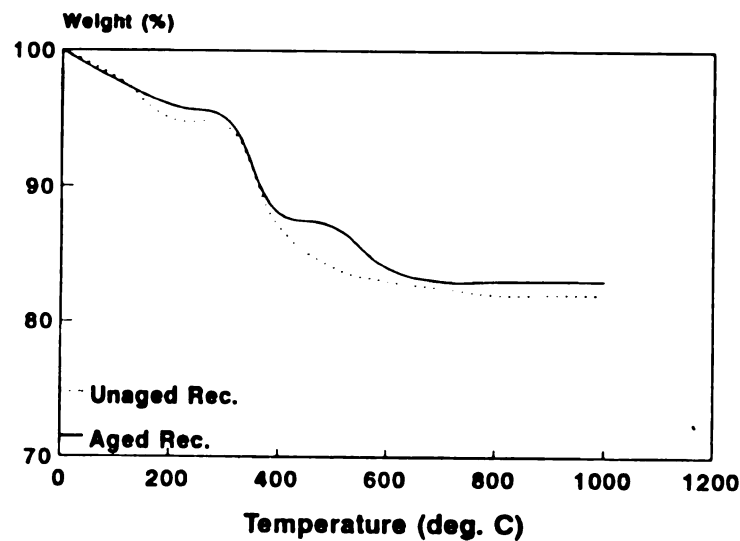


Figure 5.14 Thermogravimetric Analysis: Typical Weight Loss Curves.

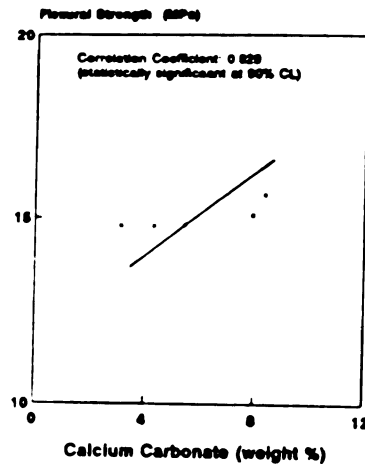
Different ageing processes are observed in Table 5.14 to cause a drop in calcium hydroxide content and an increase in the amount of calcium carbonate. The increase in calcium carbonate under repeated wetting-drying and wetting-drying with carbonation is particularly pronounced. The results suggest that recycled fiber reinforced cement composites tend to carbonate under weathering effects. The dominant mode of failure in the unaged composite was fiber pull-out when the calcium carbonate content was low. After accelerated ageing, the mode of failure was brittle with fiber rupture dominating the behavior, and the content of calcium carbonate was also increased. It is suggested that the increase in strength and rigidity of the petrified fibers, and the increase in their bond strength due to matrix densification and to elimination of shrinkage debonding at the interface zones (these phenomena involve dissolution, migration and are pronounced under carbonation effects), can account for the increase in strength and stiffness of the composite.

In order to establish the correlation of calcium carbonate and calcium hydroxide contents with engineering properties of composites, some correlation analyses were conducted the results of which are presented in Figure 5.15. These analyses were conducted in order to confirm the key role calcium hydroxide and formation of calcium carbonate play in the ageing of composites. Both unaged and aged (under different accelerated tests) composites were used in the development of Figure 5.15.

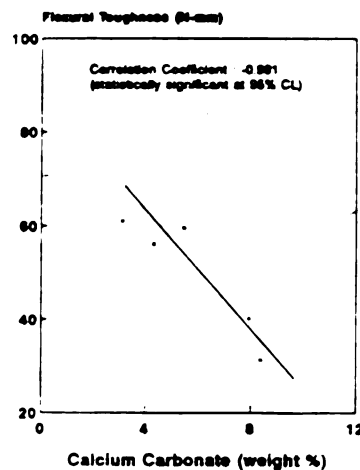
Figure 5.15 a shows a positive correlation between calcium carbonate and flexural strength; however, this correlation is not significant at 95% confidence level. But the correlation of calcium carbonate content with flexural toughness is strongly negative (Figure 5.15 b) and is statistically significant at 95% confidence level (correlation coefficient: -0.93) Calcium carbonate content shows strong positive correlation with flexural stiffness (Figure 5.15 c) at 95% confidence level (correlation coefficient 0.914). This tendency of increased stiffness with increased calcium carbonate content was observed consistently in aged composites.

The correlations of calcium hydroxide content with engineering properties (strength, stiffness and toughness) were not statistically significant (Figure 5.15 d-f). A strong positive correlation was observed between calcium carbonate content and density (correlation coefficient: 0.941) which was statistically significant at 99% confidence level (see Figure 5.15 g). Correlation of calcium hydroxide content with density is presented in Figure 5.15 h.

Calcium carbonate content seems to have a strong correlation than calcium hydroxide content with engineering properties. This may be attributed to the fact that ageing causes dominantly a migration of calcium hydroxide (and only a small change is its content as a result of carbonation) while it actually forms calcium carbonate (which reflects in increased calcium carbonate content).

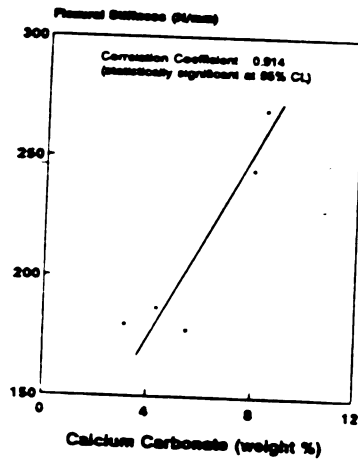


a. Correlation of Calcium Carbonate Content with Flexural Strength

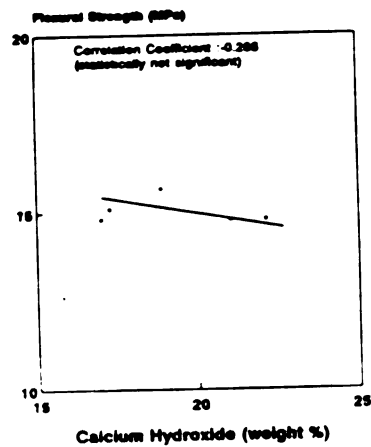


b. Correlation of Calcium Carbonate Content with Flexural Toughness

Figure 5.15 Correlation of Composition with Engineering Properties.

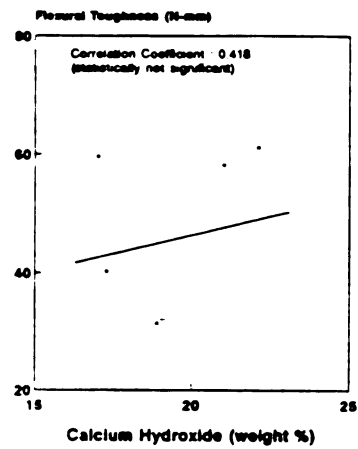


c. Correlation of Calcium Carbonate Content with Flexural Stiffness

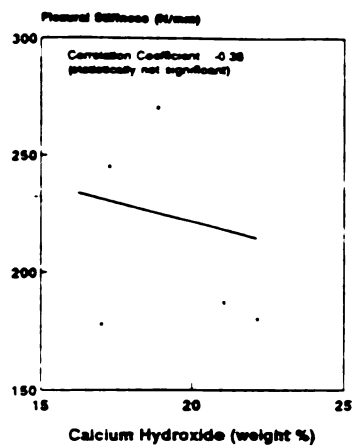


d. Correlation of Calcium Hydroxide Content with Flexural Strength

Figure 5.15 (Cont'd.) Correlation of Composition with Engineering Properties.

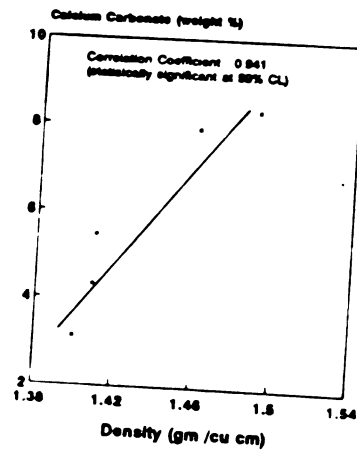


e. Correlation of Calcium Hydroxide Content with Flexural toughness

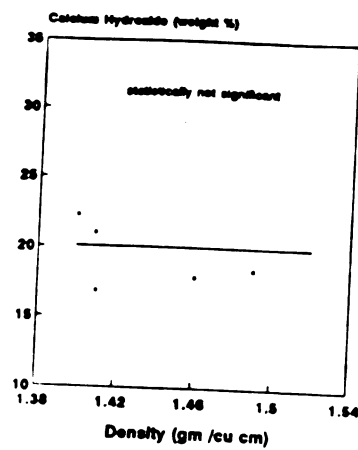


f. Correlation of Calcium Hydroxide Content with Flexural Stiffness

Figure 5.15 (Cont'd) Correlation of Composition with Ageing Properties.



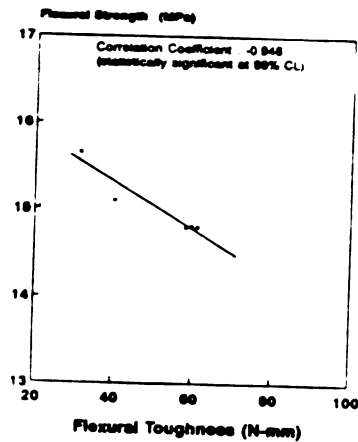
g. Correlation of Calcium Carbonate Content with Density



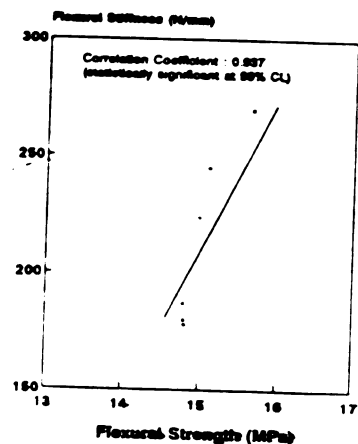
h. Correlation of Calcium Hydroxide Content with Density

Figure 5.15 (Cont'd) Correlation of Composition with Engineering Properties.

The process of ageing seems to increase density, flexural stiffness and strength while reducing toughness and permeability. The fact that these changes in engineering properties all have their roots in structural changes of composite upon ageing is reflected in strong (negative or positive) correlations between these qualities (see Figure 5.16). It should be noted that unaged and various aged composites were used to develop Figure 5.16.

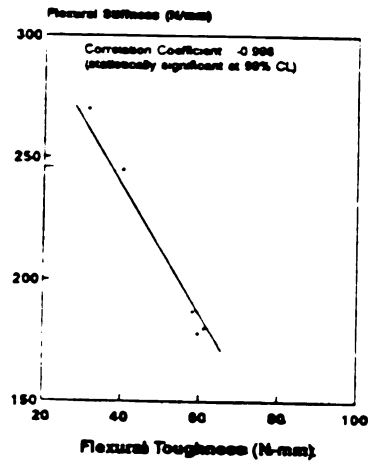


a Correlation of Flexural Strength with Flexural Toughness

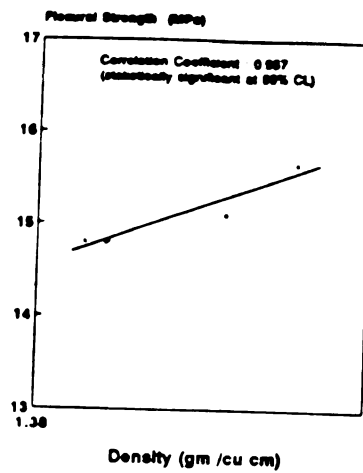


b Correlation of Flexural Strength with Flexural Stiffness

Figure 5.16 Correlation Between Various Engineering Properties

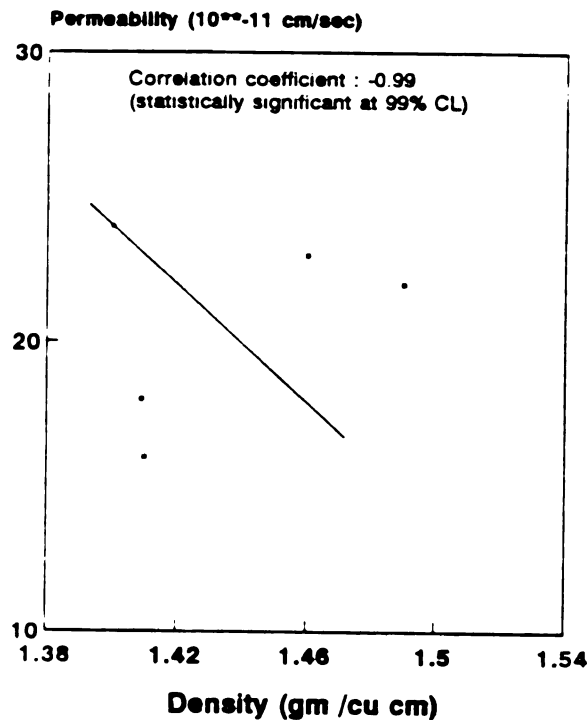


c. Correlation of Flexural Toughness with Flexural Stiffness



d. Correlation of Density with Flexural Strength

Figure 5.16 (Cont'd) Correlation Between Various Engineering Properties



e. Correlation of Density with Permeability

Figure 5.16 Correlation Between Various Engineering Properties

5.4.3 X-ray Diffraction (XRD). XRD technique offers a convenient way to determine the mineralogical analysis of crystalline solids. If a crystalline mineral is exposed to X-rays of a particular wavelengths, the layers of atoms diffract the rays and produce a pattern of peaks which is characteristic of the mineral [83]. The horizontal scale (diffraction angle) of a typical XRD pattern gives the crystal lattice spacing, and the vertical scale (peak height) gives the intensity of the diffracted ray. When the specimen being X-rayed contains more than one mineral, the intensity of characteristic peaks from the individual minerals are proportional to their amount. For the recycled wastepaper fiber-cement composites, X-ray diffraction patterns (Figure 5.17) were studied for composition of calcium hydroxide and calcium carbonate in order to confirm the results of thermogravimetry analysis. The x-ray diffraction pattern (typically shown in Figure 5.17) support that calcium hydroxide decreases and calcium carbonate increases with ageing, which seems to be the compositional change reflecting the mechanism of ageing in wastepaper fiber-cement composites.

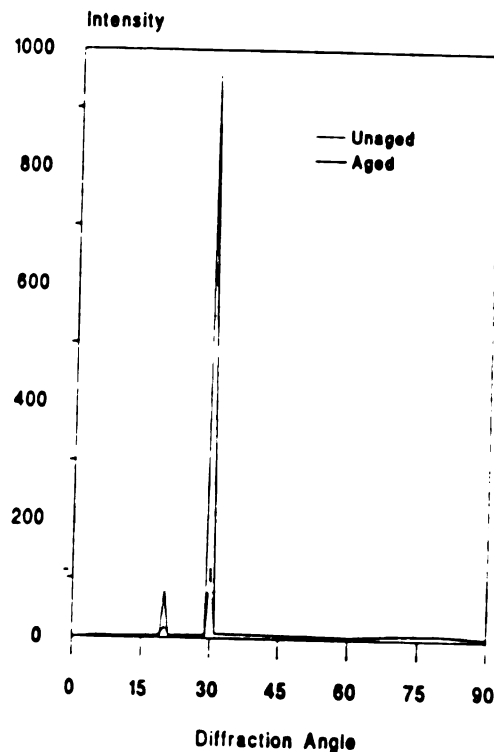


Figure 5.17 X-Ray Diffraction Pattern.

5.5 AGEING MECHANISMS

The following modes of composite failure were observed in aged and unaged composites:

- (1) Ductile: in which the fiber pulled out of the matrix to a considerable extent and the matrix around the fibers was relatively porous. This mode was typical of the unaged composite (Figure 5.12a).
- (2) Semi-ductile: in which some fibers broke while others pulled out close to the fractured surface, revealing the hollow nature of the fiber. Fiber-matrix separation or debonding at the interface could be seen, but the matrix at the interface was quite dense. This mode was typical of composites subjected to accelerated ageing in hot water bath or under repeated cycles of freeze/thaw (Figures 5.12c and 5.12d).
- (3) Brittle petrified.: in which the fibers broke at the fractured surface, revealing circular cross sections filled with dense reaction products. The matrix was as dense as in the brittle-hollow case, but no interfacial separation or debonding could be seen. This mode was

typical of composites exposed to ageing in conditions which promoted carbonation. This mode was observed in wetting/drying with carbonation and to some extent in wetting/drying accelerated ageing tests (Figure 5.12d and 5.12b). The content of calcium carbonate in composites were higher for those underwent wetting-drying and carbonation than composites exposed to wetting-drying.

In unaged composite failure was characterized by pull-out of the fibers. This resulted in a ductile failure mode. In the aged composites, the prevailing mode of failure was a brittle one, with most fibers breaking at or close to the fractured surface. The densening of the matrix around the fibers and within fiber cores, which was observed after some accelerated ageing treatments, could be a factor contributing to the reduction in toughness. These phenomena tend to reduce the flexibility and deformation capacity of fibers and increase the chance of their rupture prior to pull-out. A change of this kind can be accompanied by a reduction in strength, or an increase in strength, depending on the nature of the fibers and the effect of the ageing conditions on the properties of the fibers themselves. Petrification of fibers and increased bonding are physical phenomena that tend to increase the strength of the composite materials; the final strength of the composite would, however, be increased if these physical phenomena overshadow any attack of fibers upon ageing in the alkaline environment. It was observed that after accelerated ageing under hot water immersion or repeated freeze-thaw cycles the composite became brittle, and the mode of failure of the fibers was brittle-hollow. After accelerated ageing under repeated wetting-drying and carbonation the composite showed a reduction in toughness but its strength and elastic modulus increased. In this case, the more typical mode of fiber failure was brittle-petrified, and brittle-hollow failure was less frequently observed. Thus there appears to be a correlation between the changes in the properties of the composite after ageing and the fiber failure mode: semi-ductile failure occurred in composite which did not undergo wetting-drying (and carbonation), say under hot water immersion or repeated freeze-thaw cycles, whereas brittle-petrified failure mode was more pronounced in the composite which underwent wetting-drying (repeated wetting-drying, repeated wetting-drying and carbonation) which also gained strength. In any case; the fact that flexural strength was either on start or increased under different accelerated ageing effects indicates that structural changes in the composite which tend to favor strength increase overshadowed any attack on ligno-cellulosic fibers in the alkaline environment of cement.

5.6 IMPROVEMENT OF DURABILITY

The petrification of fibers upon ageing (as observed in accelerated wetting-drying with carbonation tests) was identified as the main concern for the longevity of composites. Four alternative methods were evaluated in this investigation in order to physically or chemically control the process of ageing and petrification in wastepaper fiber-cement composites. These methods are described below.

5.6.1 Use of Polymer Dispersion

Efforts were made to modify the cement-based matrix using styrene butadiene polymer dispersion. The polymer particles are spherical and very small (0.01 to 1 μm in diameter), and are held in suspension in water by surface active agents [83].

Voids are responsible for low strength as well as poor durability of cement-based composites in severe environments. Eliminating voids by filling them with polymer should improve the characteristics of the material. Polymers have low viscosity, high boiling point and low cost. Polymer modification has been used to improve the water-tightness, durability and adhesion capacity of concrete materials. They block the capillary pores and improve fiber-matrix bond.

In our study the latex polymer was added to the slurry. Once the slurry was subjected to vacuum dewatering, however, the dewatering process was disrupted in the presence of the polymer dispersion. The screens used to prevent the loss of solids from the fresh composite during dewatering were blocked by the fine latex particles and the excess water could not be extracted. The use of polymer dispersions in slurry-dewatered wood fiber-cement composites was thus unsuccessful.

5.6.2 Higher Silica Fume Substitution

Silica fume is a by-product from the reduction of high-purity quartz with coal in electric arc furnaces in the production of silicon and silicon alloys. The fineness and pozzolanic reactivity of silica fume make it highly effective in enhancing the density and chemical stability of the bulk of cement paste and particularly at the interface zones. The consumption

of calcium hydroxide (a cement hydration product), the reduction of the alkalinity of cement pore water, and reduced permeability of the matrix are some key mechanisms through which silica fume could positively improve the long-term stability of cellulose fiber-cement composites. Potential advantages of using silica-fume contents in cellulose fiber-cement composites are discussed below.

(1) Pozzolanic reaction of silica fume with the calcium hydroxide produced by the hydration of cement leads to the formation of products which are more stable than calcium hydroxide; this may reduce the tendencies toward petrification which is at least partly responsible for the embrittlement of composites upon ageing. The presence of silica fume also helps further enhance the chemical stability of the matrix when high-pressure steam curing is used.

(2) Fineness as well as the pozzolanic activity of silica fume produce a dense microstructure at the fiber-matrix interface zones, which is expected to reduce moisture-sensitivity of the composite.

(3) Reduced permeability and porosity of the matrix in the presence of silica fume is expected to protect fibers and thus interface zones against moisture and ageing effects.

(4) Reduced alkalinity of the cement pore water in the presence of silica fume helps control any alkali attack on the fibers.

In this investigation, 30% of cement by weight was substituted with silica fume (in control mixtures presented so far only 10% of cement was substituted with silica fume). Other proportioning and processing variables were kept constant at the optimum levels used in the control composites (e.g. 50% of cellulose fiber was replaced with recycled magazine paper).

The flexural load deflection curves of the high-silica fume recycled composite (30% silica fume content), the conventional recycled composite and the control composite (100% virgin fibers) in unaged, aged (subjected to repeated wetting-drying and carbonation) and aged saturated conditions are presented in Figure 5.18 a. The corresponding flexural strength, stiffness and toughness test results are presented in Table 5.15 and Figures 5.18 b-d.

Results of the analysis of variance of the data in Table 5.15 are presented in Table 5.16. Ageing (repeated wetting-drying) and carbonation had statistically significant effects on

flexural strength and toughness at 99% level of confidence, but not on flexural stiffness. Two-way analysis of variance with composite type (low silica fume versus high-silica fume) and age (unaged versus aged) as the two factors indicated that ageing effects on low-silica fume and high-silica fume recycled composite were statistically different at 99% level of confidence, further confirming the effectiveness of high-silica fume contents in controlling the ageing mechanisms.

A thermogravimetric analysis of the aged composite was carried out for the high silica fume composite. The calcium carbonate content was observed to drop significantly when compared to the aged low silica fume (optimized) recycled composite considered earlier.

The results presented above indicate that 30% replacement of cement with silica fume in recycled fiber-cement composites is highly effective in controlling the ageing mechanisms and moisture effects; this presents a practical, economical and efficient approach for enhancing the durability and moisture resistance of wastepaper fiber-cement composites.

Two-way analysis of variance with silica fume content (high versus low) and moisture conditions (aged versus aged-saturated) as factors indicated that while saturation has statistically significant effects on flexural performance at 99% level of confidence, the saturation effects on the flexural performance at 99% level of confidence, the saturation effects on the flexural strength and stiffness (but not toughness) of aged high-silica fume composites were less pronounced than the corresponding effects on aged low-silica fume composites.

Table 5.15 Flexural Performance of Recycled Wastepaper Fiber-Cement Composite After Higher Silica Fume Substitution.

Experiment	Flex. Str. (MPa)	Mean Flex. Str (95% Con. Int.)	Flex. Tou. (N-mm)	Mean Tou. (95% Con. Int.)	Init. Stif. (N/mm)	Mean Init Stif. (95% Con. Int.)
High-Silica Fume Recycled Unaged	15.28 15.32 15.41 15.321 15.67 15.55 15.43 15.55 15.54 15.67	15.475 (± 0.56)	79.98 80.23 81.43 81.09 76.34 85.43 83.32 89.11 81.22 80.19	81.83 (± 13.5)	170.0 169.8 176.5 160 159.6 156 161.6 180.4 167 165.65	166.48 (± 30.1)

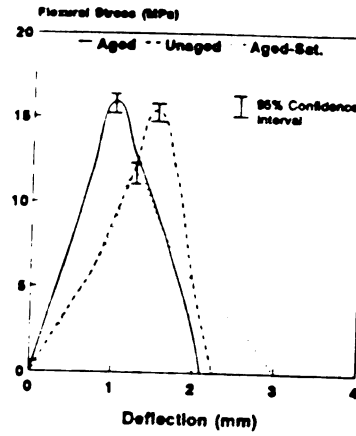
High-Silica Fume Recycled Aged	16.1	15.963 (± 0.5)	72.234	71.721 (± 6.1)	180.87	170.85 (± 39.9)
	16.23		73.657		176.5	
	15.99		73.11		190.6	
	15.876		71.009		160.6	
	15.834		76.543		174.0	
	15.811		75.211		175.0	
	15.892		76.543		163.8	
	15.912		70.006		165.0	
	15.997		71.721		162.1	
	15.712		73.33		159.9	
High-Silica Fume Recycled Aged and Saturated	11.98	11.68 (± 1.4)	68.543	60.991 (± 23.5)	160.23	156.44 (± 39.1)
	11.96		60.765		164.09	
	12.11		59.328		158.54	
	11.89		58.123		165.0	
	11.22		62.985		176.0	
	11.01		61.432		147.5	
	11.43		57.543		145.1	
	11.88		59.876		149.0	
	11.55		60.887		149.9	
	11.76		62.311		152.2	
Recycled (10% Silica Fume) Unaged	14.9	14.85 (± 0.23)	58	59.11 (± 4.0)	180.2	184.1 (± 17.1)
	14.88		57.23		178.2	
	14.77		59.98		180.9	
	14.92		60.11		189.2	
	14.89		58.23		187.7	
	14.81		59.12		185.9	
	14.75		58.87		186.7	
	14.92		59.98		186.6	
	14.81		60.34		187.9	
	14.86		59.22		177.7	
Recycled (10% Silica Fume) Aged	15.62	15.65 (± 0.29)	33.19	31.322 (± 9.48)	270.12	270.91 (± 17.1)
	15.55		32.76		265.87	
	15.76		29.67		275.98	
	15.55		34.76		272.65	
	15.75		32.98		269.98	
	15.66		31.87		267.77	
	15.61		32.87		263.87	
	15.63		28.00		275.65	
	15.71		27.98		276.76	
	15.67		29.11		270.41	

Recycled (10% Silica Fume) Aged and satu- rated	10.2	10.22 (± 0.73)	55.3	54.41 (± 9.31)	206	208.66 (± 33.2)
	10.5		50.12		210	
	10.11		57.65		211.	
	10.23		54.4		195.4	
	10.37		51.08		213	
	10.01		53		204.5	
	9.99		55.2		225	
	10.43		56.69		216.8	
	10.18		55.8		204.8	
	10.33		54.91		200.1	
Control Unaged	15.1	15.09 (± 0.51)	67	67.3 (± 8.3)	164.4	161.9 (± 23.3)
	15.02		66.39		154.3	
	15.43		63.12		171.6	
	15.1		69.34		167.4	
	14.99		70.74		152.2	
	14.98		69.43		162.4	
	15.11		66.98		159.9	
	15.09		66.12		157.7	
	14.96		67.38		162.9	
	15.11		66.55		165.7	
Control Aged	16.11	16.13 (± 0.93)	32.12	31.05 (± 8.12)	273.23	270.40 (± 23.2)
	16.20		27.45		260.56	
	16.19		28.54		280.54	
	16.20		31.99		268.67	
	16.15		29.09		267.87	
	16.2		33.34		270.98	
	16.25		32.87		278.78	
	16.54		31.22		265.54	
	15.77		30.00		267.31	
	15.712		33.87		270.54	
Control Aged and Satu- rated	10.43	10.703 (± 0.29)	50.234	49.002 (± 5.56)	160.77	160.79 (± 18.2)
	11.12		49.543		165.87	
	10.54		45.876		138.54	
	10.32		51.987		128.43	
	10.77		55.654		170.34	
	10.34		39.998		144.51	
	11.009		51.987		187.65	
	10.987		42.123		166.43	
	10.883		57.876		177.76	
	10.626		50.002		167.54	

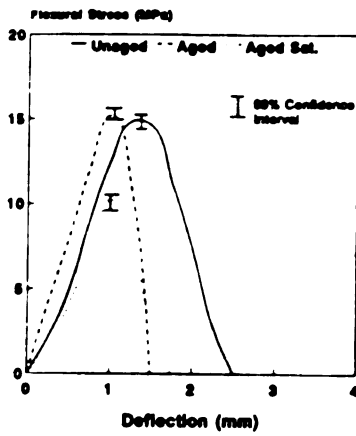
Table 5.16 Results of Analysis of Variance (flexural strength, toughness and stiffness)

Source	Sum-of Sq.	DF	Mean-Sq.	F-Ratio	P
One Way Analysis of Variance					
Flexural Strength					
Age.	0.529	1	0.529	30.446	0.001
Error	0.139	8	0.017		
Toughness					
Age.	163.21	1	163.21	29.8	0.001
Error	53.8	8	5.47		
Initial Stiffness					
Age.	116.07	1	116.07	2.73	0.137
Error	340.13	8	42.51		
Two way Analysis of Variance					
Flexural Strength					
Silica F.	1.049	1	1.049	109.42	0.00
Age.	2.549	1	2.549	265.93	0.00
SF *Age.	0.125	1	0.125	13.02	0.002
Error	0.153	16	0.01		
Toughness					
SF	4339	1	4339	972.04	0.00
Age.	1741.7	1	1741.7	390.15	0.00
SF*Age	435.8	1	435.8	97.62	0.00
Error	71.42	16	4.464		
Initial Stiffness					
SF	4178.218	1	4178.2	84.132	0.00
Age.	7150.43	1	7150.43	143.98	0.00
SF*Age.	12117	1	12117	244	0.00
Error	794.6	16	49.66		

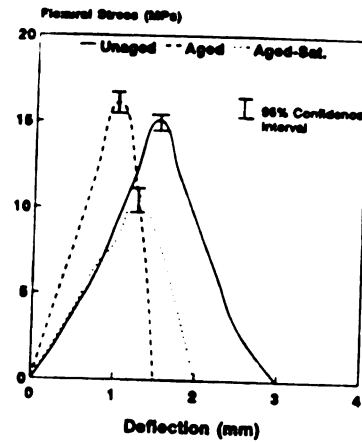
Source	Sum-of Sq.	DF	Mean-Sq.	F-Ratio	P
One Way Analysis of Variance (Aged Vs aged Sat.)					
Flexural Strength					
Sat. Age.	33.74	1	33.74	469.08	0.00
Error	0.576	8	0.072		
Toughness					
Sat. Age.	894.916	1	894.916	63.379	0.00
Error	112.96	8	14.12		
Initial Stiffness					
Sat. Age.	1086.05	1	1086.05	15.745	0.004
Error	551.83	8	68.97		
Two way Analysis of Variance (aged versus aged sat. and silica fume content)					
Flexural Strength					
Silica F.	7.07	1	7.077	155.17	0.00
Sat. Age.	7.80	1	7.8	171.2	0.00
SF *Sat. Age.	1.438	1	1.438	31.53	0.00
Error	0.73	16	0.046		
Toughness					
SF	4373	1	4373	585	0.00
Sat. Age.	812	1	812	108.767	0.00
SF*ASat. ge	21.37	1	21.37	2.86	0.11
Error	119.45	16	7.466		
Initial Stiffness					
SF	28146	1	28146	2254	0.00
Sat. Age.(WDC)	16229	1	16229	1299	0.00
SF*Sat. Age	3707	1	3707	297	0.00
Error	199.74	16	12.48		



High Silica Fume



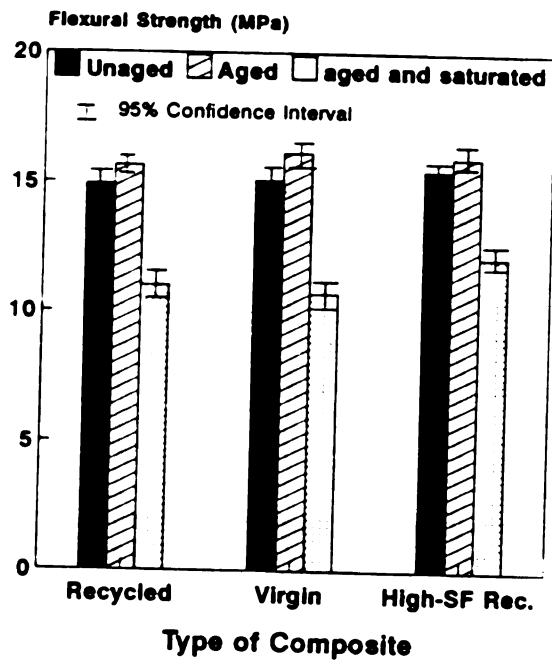
Recycled



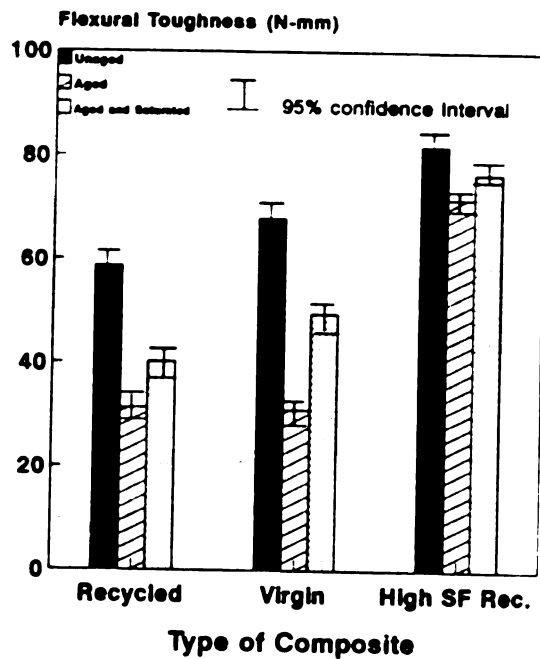
Virgin

a Load Deflection

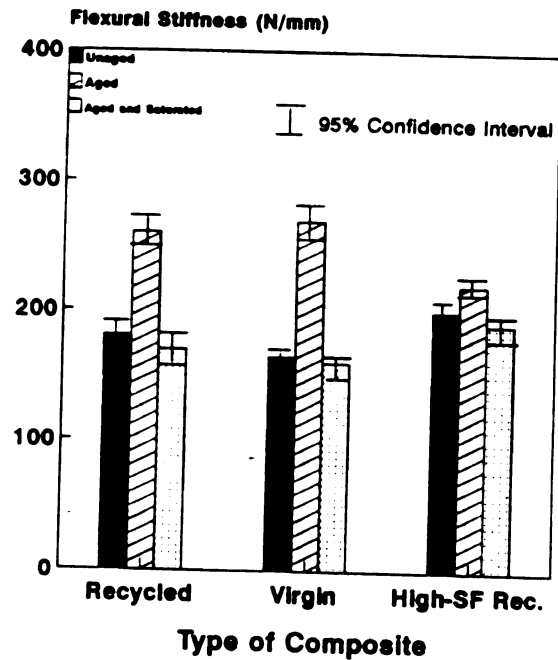
Figure 5.18 Effects of High Silica Fume Contents on Flexural Performance, Durability and Moisture Sensitivity.



b Flexural Strength



c Toughness



d Initial Stiffness

Figure 5.18 (Cont'd) Effects of High Silica Fume Contents on Flexural Performance, Durability and Moisture Sensitivity.

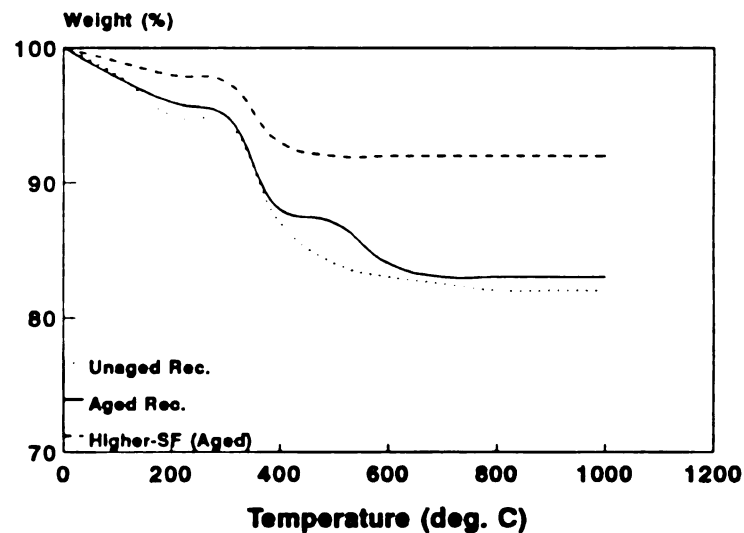


Figure 5.19 TGA Results Silica Fume Modified

5.6.3 Using Carbonation in Processing

Carbonation of the fresh composite at appropriate moisture content was hypothesized to stabilize the composite by controlled conversion of calcium hydroxide into calcium carbonate. While this concept seems to warrant further investigation, preliminary efforts to establish a moisture content in fresh composite at which carbonation can effectively take place were not successful.

5.6.4 Using Special (low calcium hydroxide) Cement

Calcium hydroxide is the cement hydration product which seems to be responsible for the petrification and thus embrittlement of wood fiber-cement composites upon ageing. Adverse effects of calcium hydroxide on ageing are also observed in glass fiber reinforced

concrete. In order to control ageing effects, the glass fiber-cement industry, in cooperation with cement industry, has developed cements the hydration of which produces minimal calcium hydroxide. The special cement considered in this investigation was from Molloy Company [84]. The cement in general has 8 parts of ordinary Portland cement, 3 parts are calcium sulphoaluminate and one part of a synthetic powder. The exact chemical composition is not revealed by the company. It is called a low calcium hydroxide cement. It has been used successfully in glass fiber reinforced concrete products. Its cost is five to six times that of ordinary Portland cement.

The flexural load-deflection curves for unaged, aged (under repeated wetting-drying and carbonation) and aged-saturated composites made with special cement and recycled fibers, regular cement and recycled fibers, and virgin fibers and regular cement are presented in Figure 5.20 a. The corresponding flexural strength, stiffness and toughness test results are presented in Table 5.17 and Figures 5.20 b-d.

Analysis of variance was carried out on the test data of Table 5.17 (see Table 5.18). The ageing effects on the flexural toughness and stiffness (but not strength) of composites made with special cement were statistically significant at 95% level of confidence. Two-way analysis of variance suggested, at 99% level of confidence, that ageing effects on flexural stiffness and toughness were less pronounced when special cement was used to replace regular Portland cement.

One -way analysis of variance (aged versus aged-saturated recycled composites with special cement) followed by two-way analysis of variance with composite type (special cement versus regular cement) and saturation condition (aged versus aged-saturated) indicated, at 99% level of confidence, that saturation influences the flexural strength, stiffness and toughness of recycled composites with special cement; while moisture effects on flexural strength were comparable in composites with special cement and regular cement, the corresponding effects on toughness and stiffness were influenced by the type of cement. With special cement moisture effects were less pronounced when compared with regular cement.

Thermogravimetric compositional analysis (Figure 5.21 and Table 5.19) indicate that the special cement reduces the calcium carbonate content of aged composites. This can be attributed to the reduced calcium hydroxide content of the special cement hydration prod-

ucts.

A comparison of the calcium hydroxide and calcium carbonate contents of aged composites with regular cement, high-silica fume binder and special cement is presented in Table 5.19. Analysis of variance of the results presented in Table 5.19 (see Table 5.20) suggested that ageing effects on calcium carbonate content were significant at 99% level of confidence, and different composites (high-silica fume, special cement, and optimum with low silica fume content) produced different calcium carbonate contents upon ageing. The least calcium carbonate content (and thus conceivably the most stable performance under ageing effects) was obtained in the high-silica fume composite.

Table 5.17 Flexural Performance of Virgin and Recycled Wastepaper Fiber-Cement Composite Using Special and Regular Cement

Experiment	Flex. Str. (MPa)	Mean Flex. Str (95% CI)	Flex. Tou. (N-mm)	Mean Tou. (95% CI)	Init. Stif. (N/mm)	Mean Init Stif. (95% CI)
Recycled Using Special Cement	14.88	14.78 (± 1.34)	60.556	61.32 (± 7.88)	160.65	159.61 (± 31.0)
	14.78		61.987		161	
	14.98		62.334		155.87	
	15.1		59.113		167.43	
Unaged	14.854		60.543		150.54	
	14.657		61.321		170.33	
	14.765		60.876		152.98	
	15.32		58.123		148.25	
	14.23		63.129		170.43	
	14.24		65.222		158.65	
Recycled Using Spec. Cem.	15.1	15.215 (± 0.94)	55.665	54.772 (8.52)	190.54	188.67 (± 25.1)
	14.887		56.321		185.34	
	15.54		55.449		187.34	
	15.342		58.21		177.45	
Aged	15.119		53.123		195.33	
	14.995		54.567		199.22	
	14.987		52.32		190.44	
	15.236		51.987		193.33	
	15.339		57.32		183.25	
	15.612		52.765		184.44	

Recycled Using Spec. Cem. Aged and Saturated	11	11.07	65.76	65.32	170.43	171.97
	10.99	(± 1.13)	65.44	(± 10.9)	175	(± 27.1)
	11.02		64.98		180.65	
	10.98		69.43		169.32	
	11.06		68.23		161	
	10.57		61.22		171	
	11.11		61.87		168.8	
	11.08		68.54		185.44	
	11.15		64.32		167.2	
	11.76		63.33		170.9	
Recycled Using Regular cement Aged	15.62	15.65	33.19	31.322	270.12	270.91
	15.55	(± 0.29)	32.76	(± 9.48)	265.87	(± 17.1)
	15.76		29.67		275.98	
	15.55		34.76		272.65	
	15.75		32.98		269.98	
	15.66		31.87		267.77	
	15.61		32.87		263.87	
	15.63		28.00		275.65	
	15.71		27.98		276.76	
	15.67		29.11		270.41	
Recycled Using Regular cement Aged and Satu- rated	10.2	10.22	55.3	54.41	206	208.66
	10.5	(± 0.73)	50.12	(± 9.31)	210	(± 33.2)
	10.11		57.65		211.	
	10.23		54.4		195.4	
	10.37		51.08		213	
	10.01		53		204.5	
	9.99		55.2		225	
	10.43		56.69		216.8	
	10.18		55.8		204.8	
	10.33		54.91		200.1	
Recycled Using Regular cement Unaged	14.9	14.85	58	59.11	180.2	184.1
	14.88	(± 0.23)	57.23	(± 4.0)	178.2	(± 17.1)
	14.77		59.98		180.9	
	14.92		60.11		189.2	
	14.89		58.23		187.7	
	14.81		59.12		185.9	
	14.75		58.87		186.7	
	14.92		59.98		186.6	
	14.81		60.34		187.9	
	14.86		59.22		177.7	

Control Unaged	15.1 15.02 15.43 15.1 14.99 14.98 15.11 15.09 14.96 15.11	15.09 (± 0.51)	67 66.39 63.12 69.34 70.74 69.43 66.98 66.12 67.38 66.55	67.3 (± 8.3)	164.4 154.3 171.6 167.4 152.2 162.4 159.9 157.7 162.9 165.7	161.9 (± 23.3)
Control Aged	16.11 16.20 16.19 16.20 16.15 16.2 16.25 16.54 15.77 15.712	16.13 (± 0.93)	32.12 27.45 28.54 31.99 29.09 33.34 32.87 31.22 30.00 33.87	31.05 (± 8.12)	273.23 260.56 280.54 268.67 267.87 270.98 278.78 265.54 267.31 270.54	270.40 (± 23.3)
Control Aged and Satu- rated	10.43 11.12 10.54 10.32 10.77 10.34 11.009 10.987 10.883 10.626	10.703 (± 0.29)	50.234 49.543 45.876 51.987 55.654 39.998 51.987 42.123 57.876 50.002	49.002 (± 5.56)	160.77 165.87 138.54 128.43 170.34 144.51 187.65 166.43 177.76 167.54	160.79 (± 18.2)

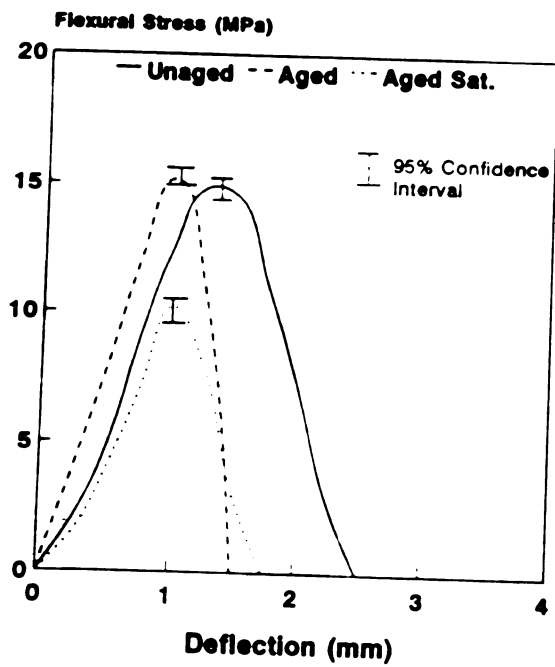
Table 5.18 Results of Analysis of Variance (flexural strength, toughness and stiffness)

Source	Sum-of Sq.	DF	Mean-Sq.	F-Ratio	P
One Way Analysis of Variance					
Flexural Strength					
Age.	0.161	1	0.161	3.72	0.09
Error	0.345	8	0.043		
Toughness					
Age.	144.4	1	144.4	32.08	0.00
Error	36	8	4.5		
Initial Stiffness					
Age.	2000.52	1	2000.52	84.05	0.00
Error	190.4	8	23.8		
Two way Analysis of Variance					
Flexural Strength					
Spec. Cem.	232.49	1	232.49	0.946	0.345
Age.	207.56	1	207.56	0.844	0.372
SC *Age.	264.192	1	264.19	1.075	0.315
Error	3933.8	16	245.86		
Toughness					
SF	731.8	1	731.8	276.25	0.00
Age.	1295.7	1	1295.7	489.1	0.00
SC*Age	684.21	1	684.2	258.29	0.00
Error	42.38	16	2.649		
Initial Stiffness					
SC	13520	1	13520	515.5	0.00
Age.	17169	1	17160	654.71	0.00
SC*Age.	4620	1	4620	176.19	0.00
Error	419.6	16	26.22		

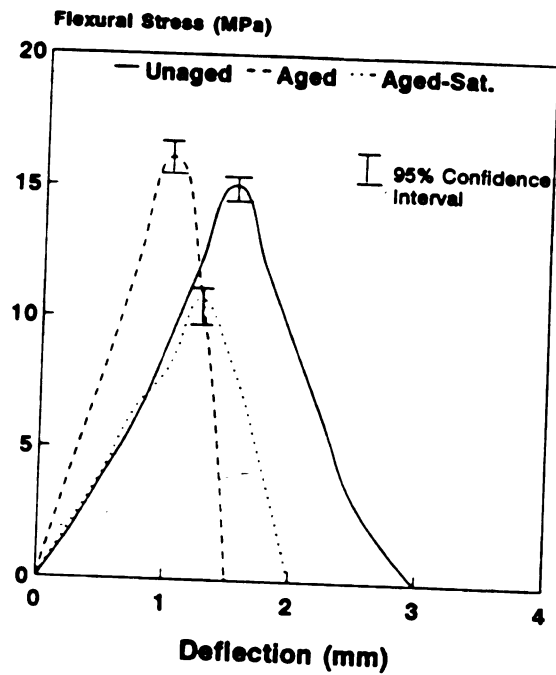
Source	Sum-of Sq.	DF	Mean-Sq.	F-Ratio	P
One Way Analysis of Variance (aged vs. sat. aged)					
Flexural Strength					
Sat. Age.	1.832	1	1.832	33.207	0.00
Error	0.44	8	0.055		
Toughness					
Sat. Age.	1570	1	1570	210.9	0.00
Error	59.552	8	7.444		
Initial Stiffness					
Sat. Age.	2992	1	2992	72.11	0.00
Error	332	8	41.5		
Two way Analysis of Variance (aged vs aged sat., special cement vs. regular cement)					
Flexural Strength					
Spec. Cem.	8.32	1	8.32	93.107	0.00
Sat. Age.	0.8	1	0.8	8.952	0.009
SC *Sat. Age.	0.003	1	0.003	0.038	0.848
Error	1.43	16	0.089		
Toughness					
SC	1277.29	1	1277	48.68	0.00
Sat. Age.	2060	1	2060	78.53	0.00
SC*ASat. ge	463.55	1	463.55	17.66	0.001
Error	393.55	15	26.237		
Initial Stiffness					
SC	5985	1	5985	28.54	0.00
Sat. Age	1377.8	1	1377.8	6.57	0.021
SC*Sat. Age.	1805	1	1805	8.607	0.01
Error	3355	16	209.72		



Special Cement



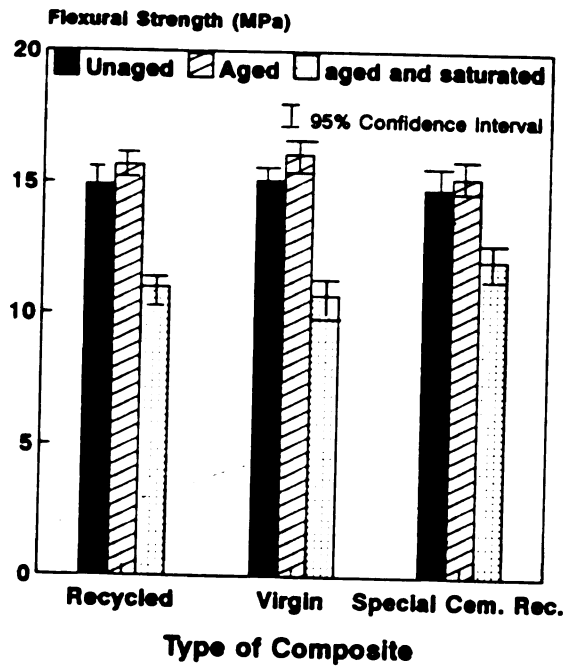
Recycled



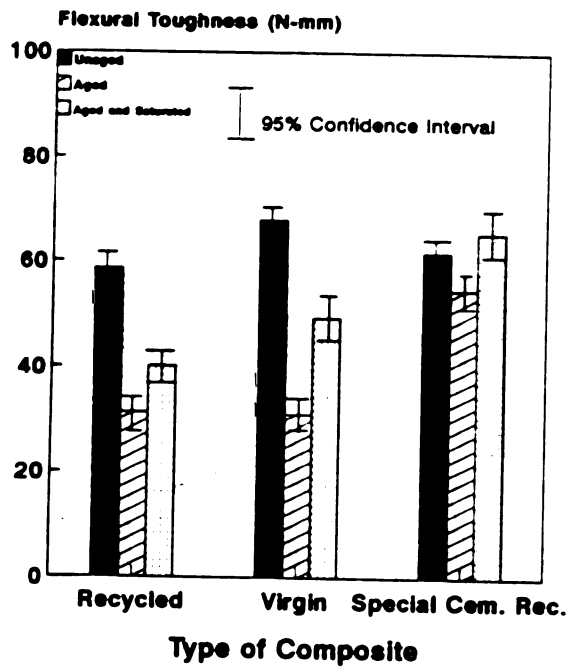
Virgin

a Load Deflection

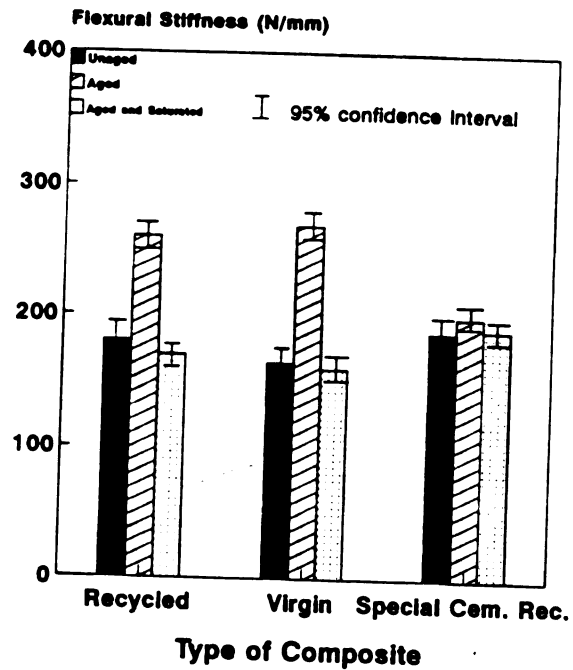
Figure 5.20 Effects of Using Special Cement on Flexural Performance, Durability and Moisture Sensitivity



b Flexural Strength



c Toughness



d Initial Stiffness

Figure 5.20 (Cont'd.) Effects of Using Special Cement on Flexural Performance, Durability and Moisture Sensitivity

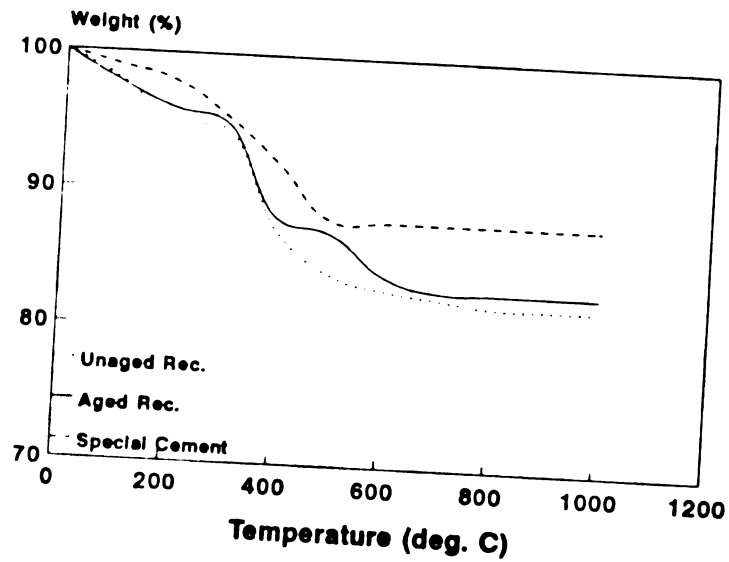


Figure 5.21 TGA Special Cement Composite

Table 5.19 Thermogravimetric Compositional Analysis

Type of Refinement	CH (Weight%)	Mean (95% Con. Int.)	CC (Weight%)	Mean (95% Con. Int.)
Unaged	22.21, 22.9, 21.9	22.29 (± 2.33)	3.12, 3.05, 3.37	3.18 (± 0.53)
Aged	17.7, 18.23, 17.8	17.94 (± 1.05)	7.94, 8.04, 7.78	7.92 (± 0.42)
SF Aged	4.68, 4.65, 4.50	4.61 (± 0.37)	1.3, 1.26, 1.32	1.29 (± 0.09)
SC Aged	9.91, 9.16, 10.21	9.76 (± 2.12)	2.65, 2.32, 2.1	2.35 (± 0.88)

Table 5.20 Results of the Analysis of Variance (TGA)

Source	Sum-of Sq.	DF	Mean-Sq.	F-Ratio	P
One Way Analysis of Variance					
Calcium Carbonate Content					
Ageing	60.02	4	15.00	812.916	0.00
Error	0.185	10	0.018		
Type of Composite	26.58	1	26.58	1440.23	0.00
Error	0.185	10	0.018		

5.6.5 Comparison of Special Cement with High Silica Fume Contents

The flexural performance of aged-saturated composites with special cement and with high-silica fume binder were composed in this section. Analysis of variance followed by comparison of means (see Table 5.21 for special cement versus high-silica fume composites) indicated that, at 95% level of confidence, silica fume and special cement produced higher strengths in aged saturated condition than optimized (low silica fume with regular cements) recycled composite; high silica fume composites were found to be superior to special cement composites as far as aged-saturated flexural strength, stiffness and toughness are concerned.

Flexural strength of the silica fume was statistically significantly superior at 95% level of confidence. On ageing the increase(3%) in flexural strength was observed in both composites.

Two-way analyses of variance were also conducted to study the effects of ageing on high-silica fume and special cement composites. Upon ageing both composites exhibited comparable (about 5%) and statistically significant, at 95% level of confidence, increases in flexural strength. Ageing also has statistically significant effects on toughness, and high-silica fume and special cement composites showed comparable drops (about 12%) in toughness upon ageing (when compared with 40% drop in low silica fume composites with regular cement and the increase in stiffness in high-silica fume composite upon ageing (about 5%) was less than that in special cement composite (about 18%).

Table 5.21 Results of Analysis of Variance

Source	Sum-of Sq.	DF	Mean-Sq.	F-Ratio	P
One Way Analysis of Variance					
Flexural Strength					
cem vs sf	4.598	1	4.598	76.643	0.00
Error	0.96	16	0.06		
Toughness					
cem vs sf	4902.2	1	4902.2	1479.6	0.00
Error	53.0	16	3.31		
Stiffness					
cem vs sf	48381.3	1	48381	131.4	0.00
Error	5889	16	368		

5.7 SUMMARY AND CONCLUSIONS

The effects of moisture and accelerated ageing on the flexural performance and microstructural characteristics of the optimized wastepaper fiber-cement composites and control composites (made fully with virgin softwood kraft fibers) were investigated. Microstructural studies utilized the Scanning Electron Microscopy, thermogravimetry and X-ray diffraction techniques. Appropriate measures were adopted and evaluated for controlling the ageing and moisture effects on wastepaper fiber-cement composites. It was concluded that:

(1) The increase in moisture content of virgin and recycled composites reduced flexural strength and stiffness, and increased toughness of the composites. One way analysis of variance of flexural strength, stiffness and toughness test results at different moisture contents confirmed the moisture sensitivity of recycled and virgin composites.

Among the moisture conditions considered (oven-dried, air-dried and saturated), saturation produced a distinct flexural behavior. Air-dried and oven dried recycled composites

proved to be statistically equivalent in flexural strength but different in flexural toughness and stiffness at 95% confidence level (using multiple comparison by contrast). Two-way analysis of variance of results was also conducted for different composites (recycled versus virgin) and moisture conditions. The fiber-moisture interaction proved to be statistically significant at 95% level of confidence, indicating that moisture effects on recycled composites differ from those on virgin composites. Damaging effects of saturation are less pronounced in the case of recycled fibers. Upon saturation, recycled composites exhibited a drop of 37% in flexural strength and 16% in initial stiffness when compared with air-dried composites; the corresponding drops in virgin composites were 47% and 28%, respectively. Both recycled and virgin composites showed an increase of 32% in flexural toughness upon saturation.

(2) Repeated wetting-drying and particularly wetting-drying and carbonation cycles caused an increase in flexural stiffness and strength of virgin and recycled composites but led to reduced toughness and embrittlement of the materials.

Repeated wetting-drying cycles had statistically significant effects on all flexural properties (strength, toughness and stiffness) of recycled composites. Two-way analysis of variance (with two factors of recycled versus virgin fibers and aged versus unaged composites) revealed that there was a statistically significant difference, at 95% level of confidence, between ageing effects on recycled and virgin composites as far as flexural strength and toughness are concerned; the initial stiffness of virgin and recycled composites, however, was affected similarly by ageing under repeated wetting-drying cycles. Damaging effects of repeated wet-dry cycles on recycled composites (which lost 32% of toughness upon ageing) was less than that on virgin composites (which lost 45% of toughness upon ageing).

(3) Statistical analyses indicated, at 95% level of confidence, that the addition of carbonation to wet-dry cycles leads to pronounced effects of ageing on composites

(4) Repeated freeze thaw cycles did not have statistically significant effect on flexural strength and toughness of recycled composite; however, the effects on increasing the initial stiffness was statistically significant at 95% level of confidence. Two way analysis of variance (taking into account the virgin composite) revealed that freeze-thaw cycles affect the initial stiffness recycled composites in a way different from virgin composites (similar

effects were observed on flexural strength and toughness irrespective of the composite type). While recycled composites showed a 1% increase in stiffness under repeated freeze-thaw cycles, virgin composites showed a drop of 9% in stiffness under this ageing condition.

(5) The effects of long-term immersion in hot water on flexural strength, stiffness and toughness of recycled composites were not statistically significant at 95% level of confidence. Two-way analysis of variance of results (virgin versus recycled fibers) concluded that only flexural stiffness of virgin and recycled composites was affected differently by this ageing process. While hot water immersion slightly reduced the initial stiffness of recycled composites (by 3%) it caused a small increase of 3% in the stiffness of virgin composites.

(6) Comparing the effects of different accelerated ageing tests based on statistical analyses, it appears that wetting-drying and carbonation is the most effective method to bring about changes in physical and mechanical properties of the wood fiber-cement composites. In order to compare the effects of different accelerated ageing conditions on the flexural performance of recycled and virgin fiber-cement composites, two-way analysis of variance of ratios of aged to unaged flexural strength, stiffness and toughness test results were conducted. The two factors in these analyses were: composite type (recycled and virgin), and accelerated ageing condition (wet-dry, wet-dry and carbonation, freeze-thaw, hot water immersion). Results of the analysis of variance indicated that, at 95% level of confidence, all the ageing conditions have comparable effects on flexural strength but there are statistically significant differences between the effects of different accelerated ageing conditions on flexural stiffness and toughness. There was no statistically significant difference between virgin and recycled composites as far as ageing effects on flexural strength and stiffness are concerned; the two composites, however, behaved differently in ageing effects on flexural toughness.

Multiple comparison of results indicated that, at 95% level of confidence, each of the accelerated conditions have distinctly different effects on flexural toughness and stiffness, except for the hot water immersion and freeze-thaw condition which had statistically comparable effects on flexural stiffness. As far as the overall ageing effects on flexural performance is concerned, repeated wetting-drying and carbonation cycles produced the most pronounced effects whereas hot water immersion caused the least effects. The trends in the

response of virgin and recycled composites to ageing were generally comparable.

(7) Ageing effects on the morphology of fibers and failure mode as observed in SEM (comparing different accelerated ageing conditions) were studied and it was concluded that fibers were not unaffected by the freeze-thaw and hot water immersion ageing conditions. In the case of wetting-drying and carbonation, however, tendencies towards filling of fibers, densification of interfaces and dominance of fiber rupture in failure mode were observed. For the unaged composite, the dominant mode of failure was observed to be fiber pull out. In the case of specimens subjected to repeated cycles of freeze-thaw, the fracture surface was observed to show a combination of fiber pull out and fiber fracture. In the case of hot water immersed composites also the mode of failure was observed to be fiber pull out accompanied with fiber fracture. For the repeated wetting-drying ageing condition the mode of failure at the fracture surface was dominated by fiber fracture. Also, for repeated wetting-drying and carbonation fiber rupture was observed to dominate the failure mode.

In addition, the appearance of fibers and their interfaces was also affected by the ageing processes. In the case of wetting-drying and wetting drying with carbonation, a dense fiber matrix interface was observed. Fibers in case of wetting-drying with carbonation appeared to be filled with hydration and carbonation products. In the case of hot water immersion and freeze-thaw ageing conditions the densification of the interface and filling of fiber core was not pronounced. It appears that the ageing process is most pronounced under repeated wetting-drying and carbonation condition.

(8) Results of thermogravimetric analysis suggested that compositional changes occur in wood fiber-cement composites upon ageing. The calcium carbonate content increases and calcium hydroxide content decreases under accelerated ageing conditions. These trends were confirmed through X-ray diffraction analysis. Wetting-Drying and carbonation followed by wetting-drying were observed to result in most pronounced compositional changes. These changes partly explain the brittle behavior of the composite after wetting-drying and carbonation. Under ageing effects there seems to be a tendency in the calcium hydroxide constituent of cement hydration products to dissolve in cement pore water and this process is accompanied with the carbonation of calcium hydroxide which produces calcium carbonate. The "petrified" fibers with strong bonding to matrix tend to be strong but brittle. It may be hypothesized that the increase in strength and rigidity of the petrified

fibers, and the increase in their bond strength due to the densification and also elimination of shrinkage debonding at the interface zones account for the increase in strength and stiffness of the composite. Petrification and well-bonded fibers, however, tend to fracture prior to pulling out of the matrix; this eliminates the energy absorption associated with fiber pull out and thus causes embrittlement of the composite.

(9) A statistically significant negative correlation was observed between calcium carbonate content and toughness, in unaged and aged composites, the positive correlation between calcium carbonate content and stiffness were also statistically significant. A statistically significant positive correlation was also observed between density and calcium carbonate content. These correlations confirmed the key role the formation of calcium carbonate plays in the ageing of the composites. The correlations between calcium hydroxide content and various engineering properties of unaged and aged composites were not statistically significant. This further underlines the significant effects of carbonation in the process of petrification of fibers upon ageing.

The correlations of calcium hydroxide content with engineering properties (strength, stiffness and toughness) were not statistically significant. A strong positive correlation was observed between calcium carbonate content and density (correlation coefficient: 0.941) which was statistically significant at 99% confidence level. Calcium carbonate content seemed to have a strong correlation than calcium hydroxide content with engineering properties. This may be attributed to the fact that ageing causes dominantly a migration of calcium hydroxide (and only a small change in its content as a result of carbonation) while it actually forms calcium carbonate (which reflects in increased calcium carbonate content).

(10) Since formation of calcium carbonate through carbonation of calcium hydroxide as well as the transport of calcium hydroxide to fiber cores and interfaces play critical roles in ageing effects on the composites, reduction of calcium hydroxide seems to provide for a more stable composite. Replacement of relatively high levels of cement with silica fume presents an approach to the reduction of calcium hydroxide content. Silica fume also helps reduce the permeability of composites and the alkalinity of pore water. While the optimized recycled composites has 10% silica fume content, the refined high-silica fume composite considered had 30% silica fume content.

Repeated wetting-drying and carbonation still had statistically significant effects, at 95% level of confidence on flexural strength and toughness (but not stiffness) of high-silica fume composites. Statistical analyses including results for low silica fume composites, however, indicated that ageing effects on high-silica fume composites were much less pronounced; the calcium carbonate content of aged high-silica fume recycled composites was also less than that of low silica fume composites. While saturation of aged high-silica fume composites led to statistically significant effects on flexural performance, statistical analyses indicated, at 99% level of confidence, that the damaging effects of moisture on flexural strength and stiffness were less pronounced in aged high-silica fume recycled composites when compared with corresponding low-silica fume composites.

In short, 30% replacement of cement with silica fume in recycled fiber-cement composites was found to be highly effective in controlling the ageing mechanisms and moisture effects; this approach presents a practical, economical and efficient approach for enhancing the durability and moisture resistance of wastepaper fiber-cement composites.

(11) In an alternative approach to reduce calcium hydroxide content of cement hydration products a special cement was considered (consisting of 8 parts of Portland cement, 3 parts of calcium sulphoaluminate, and one part of synthetic powder) the hydration of which does not produce much calcium hydroxide.

While repeated wetting-drying and carbonation effects on the flexural toughness and stiffness (but not strength) of composites made with special cement were still statistically significant at 95% level of confidence, it was concluded at 99% level of confidence, that ageing effects on flexural stiffness and toughness were less pronounced when special cement was used to replace regular Portland cement in recycled composites.

One -way analysis of variance (aged versus aged-saturated recycled composites with special cement) followed by two-way analysis of variance with composite type (special cement versus regular cement) and saturation condition (aged versus aged-saturated) indicated, at 99% level of confidence, that while moisture effects on flexural strength were comparable in composites with special cement and regular cement, the corresponding effects on toughness and stiffness were influenced by the type of cement. With special cement, moisture effects were less pronounced when compared with regular cement.

Thermogravimetric compositional analysis indicated that the special cement reduces the

calcium carbonate content of aged composites. This can be attributed to the reduced calcium hydroxide content of the special cement hydration products.

(12) The effectiveness of special cement and silica fume in recycled composites was compared. It was found that silica fume is superior in performance when compared with special cement. analysis of variance of the results confirmed that different composites (high-silica fume, special cement, and optimum with low silica fume content) produced different calcium carbonate contents upon ageing. The least calcium carbonate content (and thus conceivably the most stable performance under ageing effects) was obtained in the high-silica fume composite.

(13) Effects of polymer dispersion and carbonation as partly of the process were unsuccessfully investigated for improving the durability and moisture sensitivity of the wood fiber-cement composites.

CHAPTER 6

COST ANALYSIS

Recycled wastepaper utilization as reinforcement in thin fiber-cement sheets is a potential market area for beneficial use of wastepaper. It is not only a technically feasible and environmentally beneficial alternative, but also an economically attractive option for the thin cement products industry. This section evaluates the economic advantages of recycling wastepaper fibers in cement composites for siding applications.

6.1 Cost of Recycled Wastepaper Fiber-Cement Composites

Cost of various constituents of cellulose fiber-cement composites are presented below.

Recycled Wastepaper Fibers \$0.32 / lb

Virgin Cellulose Fibers \$0.66 / lb

Portland Cement Type I \$0.07 / lb

Silica Fume \$0.16 / lb

Ground silica sand \$0.02 / lb

Flocculating agent \$2.00 /lb

Assumed cost of labor and equipment: \$0.07 per sq.ft. of panel

For the optimum mix composition of recycled fiber-cement composites considered in this investigation, the cost of 6 mm (1/4 in) panel with the above material costs can be estimated at \$0.37 per square ft.

6.2 Comparative Cost Analysis

The market was surveyed for alternative materials available for the production of siding panels. The mean costs of alternative siding panels (finished products) in the market are compared in Table 6.1 with that of the optimized recycled fiber-cement composites.

Table 6.1 Cost Comparison with Alternate Siding Materials in Market

Type of Siding	Cost/sq. ft. (\$)
aluminium siding	0.88
Vinyl Siding	0.55
Solid Wood	0.75
Plywood	0.58
Virgin Cellulose-Cement (Hardie)	0.56
Wastepaper fiber-cement	0.37

The cost comparison of Table 6.1 reflects the economic benefits of replacing virgin cellulose fibers with wastepaper fibers in thin cement products. Recycled fiber-cement panels with desirable technical characteristics present an economically superior alternative to various siding materials such as vinyl, plywood, solid wood, virgin cellulose-cements, and aluminium.

6.3 Life Cycle Costs

One major concern in cost analysis for construction materials is the life-cycle costs which cover, besides the initial expenses, the maintenance costs. In short, life-cycle costs include all costs anticipated over the life of the product. Table 6.2 presents a comparative life-cycle cost analysis of wastepaper fiber cement composites and other siding materials. Desirable durability characteristics and low maintenance expenses of wastepaper fiber-cement boards make their life-cycle cost even more attractive than their initial cost.

Table 6.2 Life Cycle Cost Analysis

Type of Siding	Initial Cost \$	Design Life Years	Maintenance Cost/yr. \$	Annual cost (\$ per unit area sq ft.)
Aluminium	0.88	50	-	0.0176
Vinyl	0.55	50	-	0.011
Solid Wood	0.75	50	\$0.005	0.02
Plywood	0.58	50	\$0.005	0.0166
Hardie (Virgin Cellulose-Cement)	0.56	50	-	0.011
Wastepaper fiber-cement	0.37	50	-	0.007

Comparative studies of loss in product value (Table 6.3) and net product value after 50 years (Table 6.4) further confirm the economic superiority of wastepaper fiber-cement composites when compared with alternative siding materials.

Table 6.3. Loss in Product Value in Design Life

Type of Siding	Product Value (%)		
	0-5 Years Value	6-14 Years Loss in value	15-50 years value
Aluminium	100	10% / year	10%
Vinyl	100	10% /year	10%
Solid Wood	95	10%	5%
Plywood	95	10%	5%
Hardie (Virgin Cellulose-Cement)	100	2.2%/year	losing @ 2.2%
Wastepaper fiber-cement	100	2.2% /year	losing @ 2.2%

Table 6.4 Net Product Value After 50 years

Type of Siding	Initial Cost *100 / sq.ft.	Inflation	Depr./yr. 1-5 years	Depr./yr/ 6-14 years	Depr. 15-50 years	Net Value After 50 years
Aluminium	88	3%	-	10%	10%	3.88
Vinyl	55	3%	-	10%	10%	2.43
Solid Wood	75	3%	1%	5%	5%	20.82
Ply wood	58	3%	1%	5%	5%	16.1
JH(vir. cell)	56	3%	-	2.2%	2.2%	92.9
WPFC	37	3%	-	2.2%	2.2%	61.39

A market survey revealed that aluminium siding was the most popular siding in the seventies and had almost 90% of market share. Presently vinyl has become the dominating siding material. Some concerns were expressed during the market survey in relation to aluminium and vinyl sidings. Aluminium sidings fade away with time. Aluminium and vinyl sidings are sheets with low load resistance when compared with fiber-cement or wood-based sidings. It is also important to note that wood sidings have to be periodically maintained throughout their life. Wood sidings also have a relatively low fire resistance; cellulose fiber cement composites, on the other hand, possess desirable fire resistance.

The savings associated with the use of wastepaper fibers in cement products are not only in the product manufacture; a comprehensive economical study, can not neglect the avoided landfill costs (estimated at \$50 per ton) associated with diverting market-limited wastepaper from landfills.

The world of building materials is very dynamic. Changes in siding materials from solid wood to aluminium and then to vinyl reflect the fact that technological progress, economical factors and environmental issues had to change the siding materials dominating at dif-

ferent times. There are good economic and environmental reasons to support successful implementation of technology developed in this research for the utilization of wastepaper fibers in thin cement products.

6.4 SUMMARY AND CONCLUSIONS

A comprehensive cost analysis was conducted on the developed high-silica fume recycled composite versus alternative siding materials. It was concluded that:

- (1) Recycled fiber-cement composites present technically desirable qualities together with lowest initial and life-cycle costs when compared with alternative siding materials.
- (2) Recycled wastepaper fiber-cement composites present a number of positive qualities which make them further competitive. Their fire resistance is superior to wood or vinyl sidings. They do not fade away like aluminium sidings with time. Aluminium and vinyl sidings are sheets with low load resistance when compared with fiber-cement or wood-based sidings.
- (3) The environmental benefits and avoided landfill costs associated with the use of the developed recycled composites further add their marketability.

CHAPTER 7

SUMMARY AND CONCLUSIONS

Recycling in construction presents the potentials for high-volume use of waste materials in products with long service life, while avoiding costly separation and purification steps. This research focussed on the use of fibers obtained through dry processing of market-limited magazine paper (with relatively high non-cellulosic constituents) as reinforcement in thin-sheet cement products. Virgin cellulose fiber-cement composites have found applications in sidings, and soffits, tile backerboard, roof tiles, fencing and a variety of commercial fields where durability and fire resistance of thin panels are of concern. This research evaluated the technical feasibility and economical viability of partly replacing virgin cellulose fibers with wastepaper fibers in thin cement products produced by the slurry-dewatering technique.

The research was conducted in four phases concerned with: (1) identification of the key proportioning/processing variables in the production of wastepaper fiber-cement composites by the slurry-dewatering methods; (2) optimization of the influential variables and determination of the physical and mechanical properties of the optimized recycled wastepaper fiber-cement composites; (3) assessment and improvement of the long-term durability and moisture sensitivity of the optimized recycled wastepaper fiber-cement composites; and (4) assessment of the cost-competitiveness of the optimized recycled fiber-cement composites versus alternative siding materials.

Comprehensive sets of replicated experimental data were generated in this study and were analyzed statistically using the analysis of variance and response surface analysis techniques in order to derive statistically reliable conclusions. The observations and trends were further investigated by micro-structural studies including scanning electron microscopy, thermogravimetric analysis and x-ray diffraction techniques.

DETERMINATION OF INFLUENTIAL VARIABLES IN THE PROCESSING OF RECYCLED CELLULOSE FIBER-CEMENT

In the first phase of the experimental investigation, total of 11 key variables (factors) defining the production process of wastepaper fiber-cement composites were selected; the main intent was to distinguish those factors with statistically significant effects on the composite material performance characteristics. These variables were: (1) recycled fiber source; (2) fiber mass fraction; (3) fiber beating level; (4) substitution level of virgin fibers with recycled ones; (5) sand/binder ratio; (6) maximum particle size of sand; (7) silica fume/binder ratio; (8) flocculating agent/binder ratio; (9) vacuum level; (10) compaction pressure; and (11) curing condition. Each factor was considered at two levels in a (1/64) fractional factorial design of experiments. This experimental design reveals the effects of all variables on the composite material performance, but can not provide any information on the possible interactions between different variables. The resulting composite, were tested for flexural performance (strength, toughness, and initial stiffness).

The flexural test data was analyzed statistically by fractional factorial analysis of variance. Among the eleven proportioning / processing variables considered in this study, three (total fiber mass fraction, substitution level of virgin cellulose fibers with recycled ones, and fiber refinement condition) proved to have statistically significant effects, at 95% level of confidence, on the flexural performance of wood fiber reinforced cement composites. In order to optimize the composites, it is thus necessary to determine the optimum combination of these variables to produce composites with highest performance-to-cost ratios. In the optimization process, other variables with statistically insignificant effects on the end product qualities may be fixed.

The recycled wastepaper fibers were also analyzed and compared with virgin cellulose fibers. The recycled fibers were found to be smaller in length than virgin cellulose fibers. The surface of the recycled fibers was more roughened and fibrillated by the recycling process as compared to virgin cellulose fibers. Cellulose content in recycled fibers was found to be lower than virgin cellulose fibers. Recycled fibers had a significant amount (close to 20%) of fines which are expected to play a filling role, rather than reinforcing role, in cellulose fiber-cement composites.

OPTIMIZATION OF INFLUENTIAL VARIABLES

The influential variables in the processing of recycled wood fiber-cement composites were optimized based on response surface analysis techniques. The variables optimized here were: total fiber mass fraction, level of substitution of virgin fibers with recycled fibers, and the beating (refinement) level of fibers. Optimization was based on flexural strength, initial stiffness and toughness of the composites. Due consideration was also given in the optimization process to the cost of raw materials. The optimized composites were then technically evaluated versus virgin composites, ASTM specifications, and commercial products. The conclusions derived are summarized below.

- (1) Analysis of results indicated that optimum composites are obtained using 8% fiber mass fraction, 50% substitution level of virgin with recycled fibers, and refinement (beating) of fibers to a Canadian Standard Freeness (CSF) of 540.
- (2) The optimized recycled wood fiber-cement composites were shown to possess flexural strength, density and dimensional stability characteristics satisfying ASTM specifications and comparable to those of commercially available virgin wood fiber reinforced thin-sheet cement products.
- (3) The optimized recycled composites produced flexural strength, stiffness and toughness characteristics comparable to those of virgin composites. Compared to virgin wood fiber-cement composites, the optimized recycled composites possessed somewhat lower flexural strength and toughness but higher initial flexural stiffness. The difference in flexural toughness and toughness were statistically significant. Recycled composites also showed reduced moisture (dimensional) movements, lower water absorption and moisture content, and higher density when compared with virgin wood fiber-cement composites.
- (4) The fine content of recycled fibers seem to play more of a filling role than a reinforcing role. Hence, recycled composites present a denser microstructure which reflects in higher stiffness, lower water absorption and moisture content and reduced dimensional (moisture) movements of recycled composites. Reduced reinforcing action of fines in recycled fibers, however, reflects in somewhat reduced flexural strength and toughness of recycled composites when compared with virgin composites.

DURABILITY AND MOISTURE-SENSITIVITY

The effects of moisture and accelerated ageing on the flexural performance and microstructural characteristics of the optimized wastepaper fiber-cement composites and control composites (made fully with virgin softwood kraft fibers) were investigated. Microstructural studies utilized the Scanning Electron Microscopy, thermogravimetry and X-ray diffraction techniques. Appropriate measures were adopted and evaluated for controlling the ageing and moisture effects on wastepaper fiber-cement composites. It was concluded that:

(1) The increase in moisture content of virgin and recycled composites reduced flexural strength and stiffness, and increased toughness of the composites. One way analysis of variance of flexural strength, stiffness and toughness test results at different moisture contents confirmed the moisture sensitivity of recycled and virgin composites.

Among the moisture conditions considered (oven-dried, air-dried and saturated), saturation produced a distinct flexural behavior. Air-dried and oven dried recycled composites proved to be statistically equivalent in flexural strength but different in flexural toughness and stiffness at 95% confidence level (using multiple comparison by contrast). Two-way analysis of variance of results was also conducted for different composites (recycled versus virgin) and moisture conditions. The fiber-moisture interaction proved to be statistically significant at 95% level of confidence, indicating that moisture effects on recycled composites differ from those on virgin composites. Damaging effects of saturation are less pronounced in the case of recycled fibers. Upon saturation, recycled composites exhibited a drop of 37% in flexural strength and 16% in initial stiffness when compared with air-dried composites; the corresponding drops in virgin composites were 47% and 28%, respectively. Both recycled and virgin composites showed an increase of 32% in flexural toughness upon saturation.

(2) Repeated wetting-drying and particularly wetting-drying and carbonation cycles caused an increase in flexural stiffness and strength of virgin and recycled composites but led to reduced toughness and embrittlement of the materials.

Repeated wetting-drying cycles had statistically significant effects on all flexural properties (strength, toughness and stiffness) of recycled composites. Two-way analysis of vari-

ance (with two factors of recycled versus virgin fibers and aged versus unaged composites) revealed that there was a statistically significant difference, at 95% level of confidence, between ageing effects on recycled and virgin composites as far as flexural strength and toughness are concerned; the initial stiffness of virgin and recycled composites, however, was affected similarly by ageing under repeated wetting-drying cycles. Damaging effects of repeated wet-dry cycles on recycled composites (which lost 32% of toughness upon ageing) was less than that on virgin composites (which lost 45% of toughness upon ageing).

(3) Statistical analyses indicated, at 95% level of confidence, that the addition of carbonation to wet-dry cycles leads to pronounced effects of ageing on composites

(4) Repeated freeze thaw cycles did not have statistically significant effect on flexural strength and toughness of recycled composite; however, the effects on increasing the initial stiffness was statistically significant at 95% level of confidence. Two way analysis of variance (taking into account the virgin composite) revealed that freeze-thaw cycles affect the initial stiffness recycled composites in a way different from virgin composites (similar effects were observed on flexural strength and toughness irrespective of the composite type). While recycled composites showed a 1% increase in stiffness under repeated freeze-thaw cycles, virgin composites showed a drop of 9% in stiffness under this ageing condition.

(5) The effects of long-term immersion in hot water on flexural strength, stiffness and toughness of recycled composites were not statistically significant at 95% level of confidence. Two-way analysis of variance of results (virgin versus recycled fibers) concluded that only flexural stiffness of virgin and recycled composites was affected differently by this ageing process. While hot water immersion slightly reduced the initial stiffness of recycled composites (by 3%) it caused a small increase of 3% in the stiffness of virgin composites.

(6) Comparing the effects of different accelerated ageing tests based on statistical analyses, it appears that wetting-drying and carbonation is the most effective method to bring about changes in physical and mechanical properties of the wood fiber-cement composites. In order to compare the effects of different accelerated ageing conditions on the flexural performance of recycled and virgin fiber-cement composites, two-way analysis of

variance of ratios of aged to unaged flexural strength, stiffness and toughness test results were conducted. The two factors in these analyses were: composite type (recycled and virgin), and accelerated ageing condition (wet-dry, wet-dry and carbonation, freeze-thaw, hot water immersion). Results of the analysis of variance indicated that, at 95% level of confidence, all the ageing conditions have comparable effects on flexural strength but there are statistically significant differences between the effects of different accelerated ageing conditions on flexural stiffness and toughness. There was no statistically significant difference between virgin and recycled composites as far as ageing effects on flexural strength and stiffness are concerned; the two composites, however, behaved differently in ageing effects on flexural toughness.

Multiple comparison of results indicated that, at 95% level of confidence, each of the accelerated conditions have distinctly different effects on flexural toughness and stiffness, except for the hot water immersion and freeze-thaw condition which had statistically comparable effects on flexural stiffness. As far as the overall ageing effects on flexural performance is concerned, repeated wetting-drying and carbonation cycles produced the most pronounced effects whereas hot water immersion caused the least effects. The trends in the response of virgin and recycled composites to ageing were generally comparable.

(7) Ageing effects on the morphology of fibers and failure mode as observed in SEM (comparing different accelerated ageing conditions) were studied and it was concluded that fibers were not unaffected by the freeze-thaw and hot water immersion ageing conditions. In the case of wetting-drying and carbonation, however, tendencies towards filling of fibers, densification of interfaces and dominance of fiber rupture in failure mode were observed. For the unaged composite, the dominant mode of failure was observed to be fiber pull out. In the case of specimens subjected to repeated cycles of freeze-thaw, the fracture surface was observed to show a combination of fiber pull out and fiber fracture. In the case of hot water immersed composites also the mode of failure was observed to be fiber pull out accompanied with fiber fracture. For the repeated wetting-drying ageing condition the mode of failure at the fracture surface was dominated by fiber fracture. Also, for repeated wetting-drying and carbonation fiber rupture was observed to dominate the failure mode.

In addition, the appearance of fibers and their interfaces was also affected by the ageing processes. In the case of wetting-drying and wetting drying with carbonation, a dense fiber

matrix interface was observed. Fibers in case of wetting-drying with carbonation appeared to be filled with hydration and carbonation products. In the case of hot water immersion and freeze-thaw ageing conditions the densification of the interface and filling of fiber core was not pronounced. It appears that the ageing process is most pronounced under repeated wetting-drying and carbonation condition.

(8) Results of thermogravimetric analysis suggested that compositional changes occur in wood fiber-cement composites upon ageing. The calcium carbonate content increases and calcium hydroxide content decreases under accelerated ageing conditions. These trends were confirmed through X-ray diffraction analysis. Wetting-Drying and carbonation followed by wetting-drying were observed to result in most pronounced compositional changes. These changes partly explain the brittle behavior of the composite after wetting-drying and carbonation. Under ageing effects there seems to be a tendency in the calcium hydroxide constituent of cement hydration products to dissolve in cement pore water and this process is accompanied with the carbonation of calcium hydroxide which produces calcium carbonate. The "petrified" fibers with strong bonding to matrix tend to be strong but brittle. It may be hypothesized that the increase in strength and rigidity of the petrified fibers, and the increase in their bond strength due to the densification and also elimination of shrinkage debonding at the interface zones account for the increase in strength and stiffness of the composite. Petrification and well-bonded fibers, however, tend to fracture prior to pulling out of the matrix; this eliminates the energy absorption associated with fiber pull out and thus causes embrittlement of the composite.

(9) A statistically significant negative correlation was observed between calcium carbonate content and toughness, in unaged and aged composites, the positive correlation between calcium carbonate content and stiffness were also statistically significant. A statistically significant positive correlation was also observed between density and calcium carbonate content. These correlations confirmed the key role the formation of calcium carbonate plays in the ageing of the composites. The correlations between calcium hydroxide content and various engineering properties of unaged and aged composites were not statistically significant. This further underlines the significant effects of carbonation in the process of petrification of fibers upon ageing.

The correlations of calcium hydroxide content with engineering properties (strength, stiffness and toughness) were not statistically significant. A strong positive correlation was

observed between calcium carbonate content and density (correlation coefficient: 0.941) which was statistically significant at 99% confidence level. Calcium carbonate content seemed to have a strong correlation than calcium hydroxide content with engineering properties. This may be attributed to the fact that ageing causes dominantly a migration of calcium hydroxide (and only a small change is its content as a result of carbonation) while it actually forms calcium carbonate (which reflects in increased calcium carbonate content).

(10) Since formation of calcium carbonate through carbonation of calcium hydroxide as well as the transport of calcium hydroxide to fiber cores and interfaces play critical roles in ageing effects on the composites, reduction of calcium hydroxide seems to provide for a more stable composite. Replacement of relatively high levels of cement with silica fume presents an approach to the reduction of calcium hydroxide content. Silica fume also helps reduce the permeability of composites and the alkalinity of pore water. While the optimized recycled composites has 10% silica fume content, the refined high-silica fume composite considered had 30% silica fume content.

Repeated wetting-drying and carbonation still had statistically significant effects, at 95% level of confidence on flexural strength and toughness (but not stiffness) of high-silica fume composites. Statistical analyses including results for low silica fume composites, however, indicated that ageing effects on high-silica fume composites were much less pronounced; the calcium carbonate content of aged high-silica fume recycled composites was also less than that of low silica fume composites. While saturation of aged high-silica fume composites led to statistically significant effects on flexural performance, statistical analyses indicated, at 99% level of confidence, that the damaging effects of moisture on flexural strength and stiffness were less pronounced in aged high-silica fume recycled composites when compared with corresponding low-silica fume composites.

In short, 30% replacement of cement with silica fume in recycled fiber-cement composites was found to be highly effective in controlling the ageing mechanisms and moisture effects; this approach presents a practical, economical and efficient approach for enhancing the durability and moisture resistance of wastepaper fiber-cement composites.

(11) In an alternative approach to reduce calcium hydroxide content of cement hydration products a special cement was considered (consisting of 8 parts of Portland cement, 3

parts of calcium sulphoaluminate, and one part of synthetic powder) the hydration of which does not produce much calcium hydroxide.

While repeated wetting-drying and carbonation effects on the flexural toughness and stiffness (but not strength) of composites made with special cement were still statistically significant at 95% level of confidence, it was concluded at 99% level of confidence, that ageing effects on flexural stiffness and toughness were less pronounced when special cement was used to replace regular Portland cement in recycled composites.

One -way analysis of variance (aged versus aged-saturated recycled composites with special cement) followed by two-way analysis of variance with composite type (special cement versus regular cement) and saturation condition (aged versus aged-saturated) indicated, at 99% level of confidence, that while moisture effects on flexural strength were comparable in composites with special cement and regular cement, the corresponding effects on toughness and stiffness were influenced by the type of cement. With special cement, moisture effects were less pronounced when compared with regular cement.

Thermogravimetric compositional analysis indicated that the special cement reduces the calcium carbonate content of aged composites. This can be attributed to the reduced calcium hydroxide content of the special cement hydration products.

(12) The effectiveness of special cement and silica fume in recycled composites was compared. It was found that silica fume is superior in performance when compared with special cement. analysis of variance of the results confirmed that different composites (high-silica fume, special cement, and optimum with low silica fume content) produced different calcium carbonate contents upon ageing. The least calcium carbonate content (and thus conceivably the most stable performance under ageing effects) was obtained in the high-silica fume composite.

(13) Effects of polymer dispersion and carbonation as partly of the process were unsuccessfully investigated for improving the durability and moisture sensitivity of the wood fiber-cement composites.

COST ANALYSIS

A comprehensive cost analysis was conducted on the developed high-silica fume recycled composite versus alternative siding materials. It was concluded that:

- (1) Recycled fiber-cement composites present technically desirable qualities together with lowest initial and life-cycle costs when compared with alternative siding materials.**
- (2) Recycled wastepaper fiber-cement composites present a number of positive qualities which make them further competitive. Their fire resistance is superior to wood or vinyl sidings. They do not fade away like aluminium sidings with time. Aluminium and vinyl sidings are sheets with low load resistance when compared with fiber-cement or wood-based sidings.**
- (3) The environmental benefits and avoided landfill costs associated with the use of the developed recycled composites further add their marketability.**

APPENDIX I

THEORETICAL CONSIDERATIONS

I.1 INTRODUCTION

Theoretical illustrations of the mechanisms through which fibers enhance the mechanical properties of concrete are generally based on two concepts [85]. According to the first one (spacing concept) fibers enhance the concrete performance mainly through limiting the size and preventing the propagation of the internal flaws in concrete. The other one (composite material concept) suggests that fibers contribute to the concrete load and deformation capacities through mobilizing their pull-out resistance. More fundamental theoretical approaches to fiber-cement composites based on fracture mechanics concepts.

A brief review of the spacing concept as applied to fibers in cementitious matrices is presented here. The virgin cellulose fibers and recycled wastepaper fibers are compared based on spatial distribution. The theoretical spacing calculated is compared with the actual SEM observations. A brief introduction to the application of fracture mechanics to fiber-cement composites is also presented.

I.2 Spacing Concept As Applied To Thin Fiber-Cement Sheets

Concrete and mortar have an inherent internally flawed structure. The strength of such a material can be increased by increasing the fracture toughness, decreasing the size of the flaws, and decreasing the stress intensity factor at the tip of the internal cracks. These objectives, according to the spacing concept, can be achieved using more closely spaced fibers in the matrix. The fiber spacing in this approach is a statistical description of the distance between centroids of fibers. A popular expression for fiber spacing is derived below [70].

If the fibers were aligned and uniformly spaced without overlapping (Figure I.1a), then the average spacing of the fiber centroids (S_c) would be:

$$S_c = (V/N)^{1/3} \dots\dots\dots \text{Equation I.1}$$

where V = total volume of fiber reinforced composite

N = total number of fibers

$$= V_f/V_1$$

V_f = volume fraction of fibers

V_1 = volume of single fiber

For unit V

$$S_c = (1/N)^{1/3}$$

$$= (V_1/V_f)^{1/3}$$

In actual conditions, however, fibers are not aligned, and they also overlap. One would assume that the ratio of the average of the projected length in one direction to the total length (η_θ) is a proper correction for the random orientation of fibers (Figure I.1b) [58].

η_θ = Average of projected length/ Total length

$$= 0.41$$

Hence, the effective volume fraction of fibers is only 41% that of the actual volume fraction, and the average fiber spacing after correction for random orientation of fibers (S_{ce}) becomes:

$$S_{ce} = (V/0.41N)^{1/3} \dots\dots\dots \text{Equation I.3}$$

$$= (V_1/0.41 V_f)^{1/3}$$

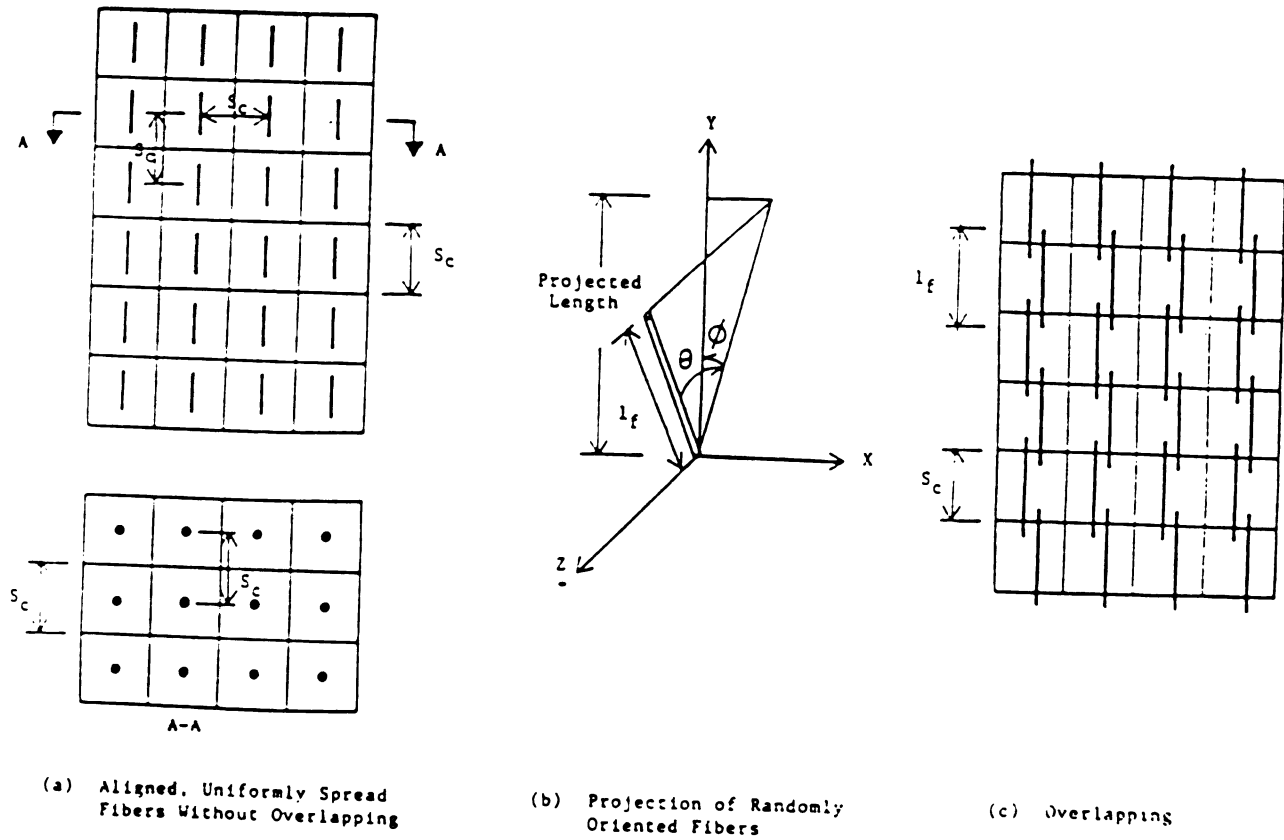


Figure I.1 Spacing of Fibers.

Overlapping of fibers is another factor that modifies the simple spacing expressions derived above. This factor is especially important for fibers with lengths greater than the average fiber spacing ($l_f > S$). Such fibers tend to overlap with other fibers, thus increasing the number of fibers crossing any section by a factor l_f/S :

$$N_e = N (l_f/S)$$

By substituting N in Eq. I.3 with N_e , one gets [56]:

$$S = (V / (0.41 * N * (l_f/S)))^{1/3}$$

Hence,

$$S = (V / (0.41 * N * l_f))^{1/2} \dots \dots \dots \text{Equation I.4}$$

where: N_e = effective number of fibers,

S = average spacing of fiber centroids in the composite reinforced with short, randomly dispersed fibers.

As the above measure of fiber spacing (S) decreases, the flaw size and the stress intensity factor at the tip of internal cracks decrease and the fracture toughness increases. Hence, the first crack and ultimate flexural strengths of fiber reinforced concrete are expected to be decreasing functions of fiber spacing. According to the spacing concept, there is a unique relationship between tensile strength of fiber reinforced concrete and fiber spacing irrespective of fiber geometry, interfacial bond properties and tensile strength. This function has been derived empirically by number of researchers, as typically shown in Figure I.2 [56, 79, 86].

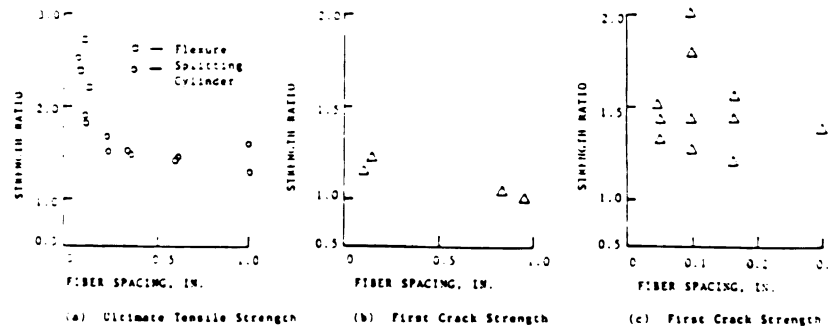


Figure I.2 Empirical Relationship Between the Tensile Strength of Steel Fiber Reinforced Concrete and the Spacing of the Fibers [89]

L3 Virgin Cellulose Vs. Recycled Wastepaper Fibers

Virgin cellulose fibers are on the average 3 mm (0.12 in) long with a negligible amount of fine fragments as shown in Table 3.4 of Chapter 3. Recycled cellulose fibers are shorter in length (average length of 1 mm, 0.04 in, for the dry-processed wastepaper fibers considered in this investigation) due to the recycling process but are larger in number. This results in a situation of fiber reinforcement conditions shown in Figure I.3.

The shorter recycled fibers are more closely spaced than the longer virgin fibers.

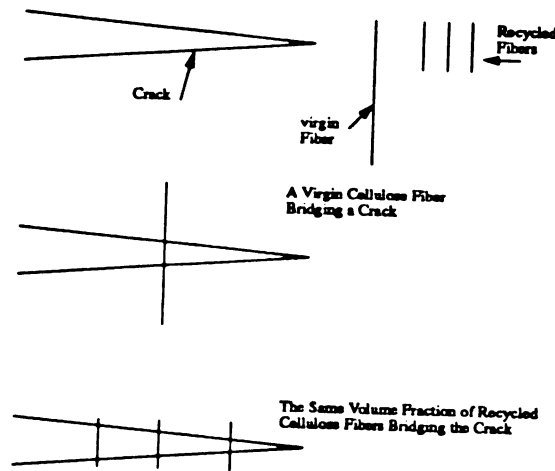


Figure I.3 Virgin Vs. Recycled Cellulose Fibers Arresting Cracks.

The fiber-cement production technique tends to orient fibers in the plane of the thin-sheet cement product. The two dimensional orientation of fibers requires the modification of Equation I.4 as follows

$$S = (V / (0.63 * N * l_f))^{1/2} \dots \dots \dots \text{Equation I.5}$$

On average there are three times more recycled fibers ($N = V_f / V_1$) when compared with virgin fibers for the same fiber volume fraction (see Figure I.3). With an average diameter of 20 μm , the average spacing of recycled and virgin fibers were 0.07 mm and 0.21 mm (0.003 in to 0.008 in) respectively, as obtained from Equation I.5.

SEM observations generally show that the inter fiber spacing varies from about 0.05 mm to 0.3 mm (0.002 in to 0.012 in).

Obviously the trends observed in experiments on virgin and recycled fiber-cement com-

posites can not be illustrated with the spacing concept. Broken recycled fibers containing fines are more closely spaced than virgin cellulose fibers; this closer spacing, however, does not lead to increased strength of the composites with recycled fibers. This could be illustrated as follows: (1) the recycling process not only shortens the fibers but also damage their tensile strength and overall qualities; (2) fines in recycled fibers do not have the slenderness needed to act as fibers and they play more of filling role; (3) the fibers used in magazine paper even in the original form may not have been as high-quality as the virgin kraft cellulose fibers used in this investigation; and (4) the spacing factor which disregards the effects of fiber-to-matrix bond and the tensile strength of fibers can not present a comprehensive approach for predicting the strength characteristics of wood fiber-cement composites.

1.4 Fracture Mechanics

Fiber reinforced cementitious matrices form a class of composites with the following distinct characteristics: the ultimate tensile strain of the matrix is much lower than the ultimate tensile strain of the fiber, and the bond strength at the fiber-matrix interface is relatively small; this implies that with increasing tensile stresses on the composite, the matrix will crack at some level of stress long before the fibers reach their failure strength. Furthermore, the composite response after matrix cracking will greatly depend on the interaction of fibers with cracks and the pullout resistance of fibers [87].

The classical fracture mechanics concept of brittle crack propagation in materials has been applied only relatively recently to cementitious matrices such as paste, mortar and concrete. In 1963 Romualdi and Batson[57] proposed a model to explain how closely spaced discontinuous stiff fibers can increase the stress causing rapid crack propagation. They essentially applied to fiber reinforced concrete, the classical Griffith approach as modified by Irwin and described by

$$\sigma_c = K_{Ic} / (\alpha \pi a)^{1/2} \dots \dots \dots \text{Equation 1.6}$$

where

σ_c = stress on onset of rapid crack propagation

K_{Ic} = critical plane strain fracture toughness (opening mode I)

α = parameter depending on the specimen and crack geometry, loading rate, crack tip sharpness, etc., and

a = half-length of a penny-shaped internal crack or length of external crack.

The expression proposed by Romualdi and Batson [57] for fiber reinforced concrete is given by

$$\sigma_c = K/(s)^{1/2} \dots \dots \dots \text{Equation I.7}$$

where

K = constant related to the fracture toughness and

s = average fiber spacing

Applying of this equation to virgin cellulose fiber-cement composites gives

$$\sigma_c = K/(s)^{1/2}$$

$$= K/(.21)^{1/2}$$

$$= 0.91K$$

For virgin cellulose fibers the stress to start crack propagation is 0.577 of critical stress intensity factor related constant.

For recycled cellulose fibers

$$\sigma_c = K/(s)^{1/2}$$

$$= K/(0.07)^{1/2}$$

$$= 3.78K$$

So in case of recycled cellulose fibers, this approach indicates that a higher value of stress is required to start rapid crack propagation. Again, the disregard for the bond strength and fiber tensile strength effects in this approach leads to the erroneous conclusion and that shorter fibers are more effective in increasing the tensile strength of composites than that

of longer virgin cellulose fibers.

The effect of fiber debonding in the classical fracture mechanics relation has been tackled by Naaman [87]. It was shown, that due to the process of debonding in fiber reinforced concrete, a pseudo-plastic zone of radius R (Figure I.4) develops at the crack tip. The pseudo-plastic zone represents an area where the matrix is cracked but where the fibers bridging the cracked surfaces are still debonding, thus providing a pullout resistance. This increases the assumed critical internal flaw size for the composite by $2R$. Consequently, they proposed the following relation which applies in the three-dimensional case of a tensile prism

$$\sigma_c = K_{Ic} / (4 * (d + R) / \pi)^{1/2}$$

where

K_{Ic} = fracture toughness at critical propagation (opening Mode I)

d = crack length; or diameter of largest weak area defined as that area having no or little amount of fiber crossings by probability analysis, and

R = length of radius of pseudo-plastic zone from crack tip.

So the spacing concept and fracture concept applications were not successful because the composite did not satisfy the assumptions made in the both approaches.

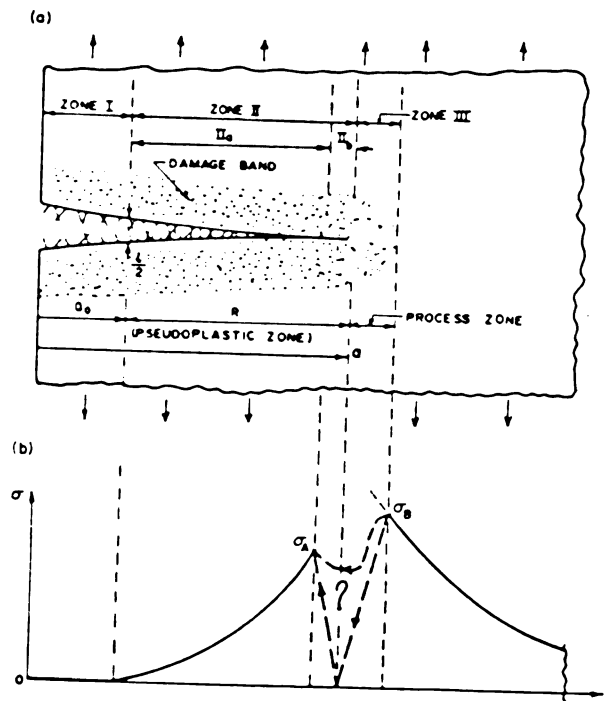


Figure I.4 Assumed Critical Crack Model Controlling Fracture of Fiber-Reinforced Concrete Composite.

APPENDIX II

NOTATIONS AND SYMBOLS

The following symbols were used in this study:

AD Air Dried

OD Oven Dried

Sat Saturated

WD Wetting-Drying

WDC Wetting-Drying and Carbonation

FT Freeze-thaw

HW hot water

SF Silica fume

CSF Canadian Standard Freeness

JCI-SF Japanese Concrete Institute Standards for Test Methods of Fiber Reinforced Concrete

RH Relative Humidity

SEM Scanning Electron Micrographs

FA Flocculating Agent

SSK Southern Softwood Kraft

SD Standard Deviation

TAPPI Technical Association of the Pulp and Paper Industry

TGA Thermogravimetric Analysis

EPA Environmental Protection Agency (USA)

psi Pounds per square inch

lb in Pound inch

K/in Kips per inch

mm millimeter

N Newton

CD Cross Direction

MD Machine Direction

σ stress

δ strain

Δ displacement

I 95% Confidence Interval (CI)

SC Special cement

APPENDIX III

STANDARD SPECIFICATIONS

The following standard specifications were used in this study:

1. ASTM C 1185 Standard Test Methods for Sampling and Testing Non-Asbestos Fiber-Cement Flat Sheet, Roofing and Siding Shingles, and Clapboards (American Society of Testing Materials)
2. ASTM C1186-91, "Standard Specification for Flat Non-Asbestos Fiber-Cement Sheets"
3. JCI SF 4 Method of Tests for Flexural Strength and Flexural Toughness of Fiber-Reinforced Concrete (Japan Concrete Institute)
4. TAPPI207 Technical Solubility of Pulp (Technical Association of the Pulp and Paper Industry)
5. ASTM C670-91a, "Standard Practice for Preparing Precision and Bias Statement for Test Methods for Construction Materials," (American Society of Testing Materials)

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