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MECHANICAL CHARACTERISTICS OF COMPOSITES MADE
FROM RECYCLED HDPE OBTAINED FROM MILK BOTTLES

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M.S. degree in PACKAGING

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**MECHANICAL CHARACTERISTICS OF COMPOSITES MADE FROM
RECYCLED HDPE OBTAINED FROM MILK BOTTLES**

By

Varsha Kalyankar

A THESIS

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ABSTRACT**MECHANICAL CHARACTERISTICS OF COMPOSITES MADE FROM
RECYCLED HDPE OBTAINED FROM MILK BOTTLES****By****Varsha Kalyankar**

Plastics packaging materials are reaching a crisis situation in regulatory and environmental concerns in solid waste management. The single most abundantly used packaging containers are the blow molded high density polyethylene bottles. The potential of using the HDPE regrind from milk bottles in composites with low cost, abundantly available and renewable wood-fibers was examined.

Melt mixing of wood-fibers with HDPE in a twin screw extruder gave uniform blending. Mechanical property evaluation showed that the tensile modulus increased with increase in wood-fiber content. The tensile strength, elongation at break and impact strength decreased. The composite showed fair stability of dimensions and tensile strength at equilibrium water absorption. Addition of ethylene vinyl acetate copolymer as a low cost bonding agent improved the impact strength of the composite. No improvement in tensile strength was seen.

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NOMENCLATURE

Nomenclature used in Literature Review

Symbol

E	modulus
σ	tensile strength
V	volume fraction
σ_x	stress at fiber end
τ	shear stress
S	cross sectional area
r	radius
L	length
σ_m	stress in matrix
E_R	relative modulus
V_f	volume fraction of filler
G_f	shear modulus of filler
G_p	shear modulus of polymer
ν^p	Poisson ratio of polymer
V_m	maximum packing fraction of filler
σ_c	tensile strength of composite
σ_p	tensile strength of polymer
a	constant related to stress concentration
b	constant related to geometry of filler
V_f	volume fraction of filler
σ_1	interfacial tensile strength

INTRODUCTION

Plastics play a major role in providing what were luxury items to the common man. The contributions of plastics to modern society are far too many to be listed. The advantages offered by plastics in terms of energy savings, ease of handling, appearance etc., have made it possible for this versatile material to replace other traditional materials.

Plastics have one of the most spectacular growth histories among engineering materials. It has been estimated that by the turn of the century, U.S. resin production will reach 76 billion pounds, i.e., an increase of 58% between 1985 and 2000. (Powell, 1987). Correspondingly, plastics in the waste stream have increased steadily from about one-half million tons in 1960 to 9.6 million tons in 1984. By 2000, it is expected to reach 15.5 million tons (Franklin Associates, 1986). The highly desirable properties of plastics like light weight, ease of handling and versatility are due to the long-chain structure of polymers. The long chains are so stable that they are not usually susceptible to attack by microorganisms and degrade very slowly under normal conditions. So, even though plastics constitute only 7% of the total solid waste by weight, their non-degradability has made them highly undesirable in the waste-stream.

7% by weight of plastics in the waste stream does not seem to be a big number. However, plastics are significantly lighter than other solid waste materials in the waste stream, except paper. Plastics also resist compaction because of their resilience. Thus the contribution of plastics to the solid waste stream by volume is significantly higher

than their contribution by weight.

In general, plastic waste can be segregated into two kinds: industrial waste and post-consumer waste (Leidner, 1981). Industrial waste generally consists of just one type of fairly uncontaminated plastic. It is generally reused as regrind without any significant problems. Post-consumer waste, on the other hand, consists of a mixture of different types of plastics, is contaminated and difficult to collect. Figure 1 gives the distribution of plastics consumption by end use.

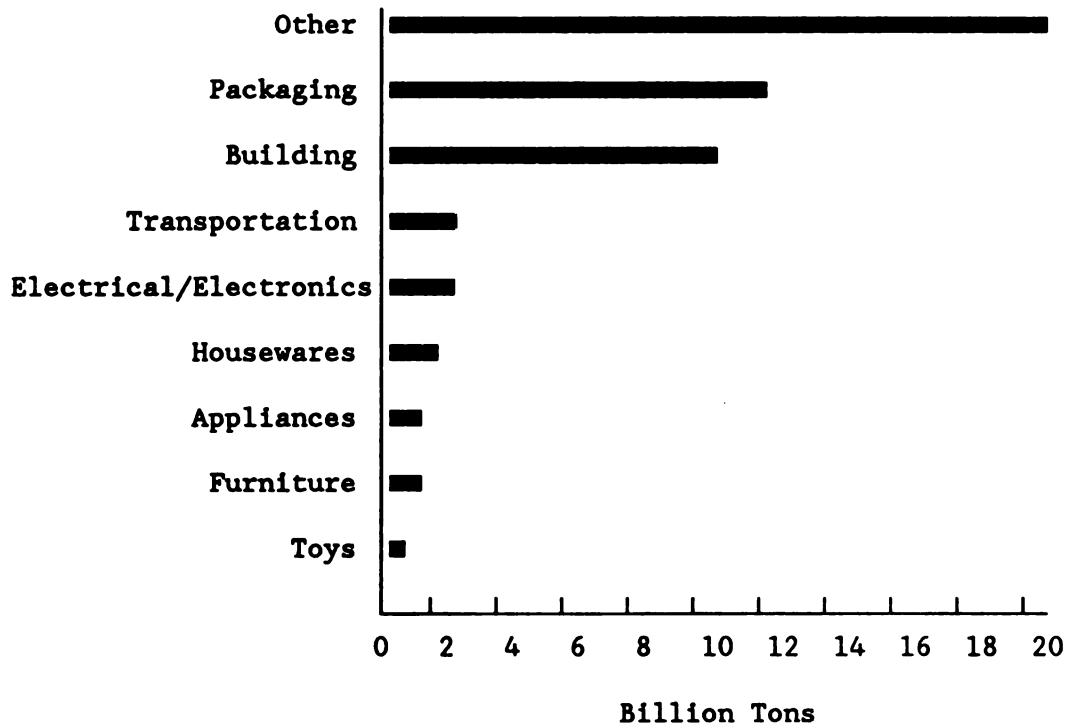


Fig 1 : U.S. Plastics Consumption in 1986. (Selke and Lai, 1987)

As can be seen, plastics find their largest application in building and construction and packaging. However, the estimated life-time of building materials is about 25 years and therefore they do not present a solid waste problem. Packaging generally enters the waste stream in less than

a year from the time of its manufacture and initial use (Selke and Lai, 1987). Packaging is one of the largest industries in the U.S. Recently, Business Communication Co. Inc, predicted the growth for plastic containers as 11% per year through 1996 (Modern Plastics, March 1987). Plastics are used in virtually every aspect of packaging: films, coatings, containers, lids, closures, and adhesives. This continuing trend towards plastics in packaging is because of its many advantages over other traditional materials. Some of the numerous desirable properties of plastics in packaging are listed below:

- versatile: can be made into flexible or rigid containers as applicable.
- break resistant
- light weight: easy to handle and savings in distribution costs
- excellent mechanical properties.
- can be foamed for cushioning.
- grease resistant. Eg. vinyls and cellulosics.
- retain odors and flavors, which is important in food packaging.
- excellent insulation properties.
- convenience. The convenience offered by plastic packages is an important characteristic in modern society.
- excellent barrier properties have been developed.

All these characteristics offered by plastics in addition to their being one of the most inexpensive materials ensure the continued growth of plastics in packaging. It is almost impossible to replace plastics with any traditional materials like glass, metals or paper in a lot of applications.

As the problem of solid waste disposal grows, environmentalists are demanding that Government should intervene in solving the problem. Packaging is the single most conspicuous component of post-consumer waste and therefore the Government has responded to the ecological concern voiced by environmentalists by passing a number of Bills in State legislatures directed towards controlling packaging. According to a recent report by the Council of Plastics and Packaging in the Environment, there are at least 256 Bills in 35 states dealing with garbage - a majority of them directed towards packaging.

One of the first bills aimed at controlling packaging as a means of waste disposal management was passed in Minnesota, usually referred to as the "Package Review Program" (Minnesota State Chapter 116F). This Act assigns the Minnesota Pollution Control Agency the authority to review new or revised packages or containers that may cause a solid waste disposal problem. Although the Act has never been implemented, it has been the basis for similar bills in other states.

Iowa recently passed a similar law and Nebraska was considering a law which assigns an agency the power to review packages for environmental and ecological problems (Wright, 1987). The New Jersey legislature is considering a bill that requires every packaging material to be at least 50% recyclable. Since paper, glass and aluminum meet this requirement, the pressure is put on plastics.

The Michigan Legislature has considered imposing a ban on non-biodegradable fast-food containers and beverage container holders. Since one of the problems with post-consumer plastic waste is the identification of the different plastics, Michigan is one of many states considering the labeling and identification of the plastic used for the

package.

According to a recent study by Business Communication Co. (Packaging Week, 1987) "plastics recycling poses a major problem for the industry and that packagers must respond to the crisis or the growth and penetration of plastics in industry will slow dramatically in the next 15 years."

Jacob Leidner in his book "Plastic Waste; Recovery of Economic Value" (1981), states four ways of handling this problem :

1. Incineration
2. Pyrolysis
3. Recycling
4. Biodegradability

Until now, most garbage was dumped into landfills. Almost all of these landfills have been closed or are reaching capacity. In reality, plastics are the ideal material to landfill as they do not degrade to give off toxic materials. Landfilling costs have also increased. in New Jersey the average tipping fee in the state has increased from \$3 per ton in 1979 to \$100 or more per ton. (Selke and Lai, 1987)

Incineration is one of the alternatives possible. Incineration is burning of solid waste. The incinerator is usually designed so that energy is recovered. This is one method of recovering some of the energy from solid waste, and is also called resource recovery. The over-all heat content of typical solid refuse is close to 5000 B.T.U. / lb. Plastics make a significant contribution to this number since the average heat content of the waste plastics is 18,000 B.T.U. / lb , the highest of any component of the refuse (Ryser, 1984).

But, there are a number of disadvantages associated with this process. The initial capital to be invested is large. Plus, the operating costs are also high. Consistent and reproducible composition of feedstock is not possible, which will cause problems. There are also concerns about the hydrochloric acid that may cause environmental problems and also damage the equipment.

Pyrolysis is the burning of material in the presence of insufficient oxygen for combustion. The products are low molecular weight entities, which vary according to the type of plastics and the pyrolysis conditions. The process is contained and so does not cause air pollution. Plants can be located in the cities, resulting in lower transportation costs. But there are some problems associated with this method of plastic disposal. Ideally, the product should be monomers. But pyrolysis usually it gives a mixture of products which does not have much value except as fuel. At the present state of technology, it is not economically feasible.

Recycling is the reuse of plastic waste for the same product as used earlier, or a different product. It gives the highest recovery value economically. According to Morrow, Amini and Adams (1987) there are three important reasons for recycling:

1. Environmental concerns.
2. Regulatory concerns.
3. Economic concerns.

Plastics are a valuable and finite resource that should not be depleted. Because they are derived almost entirely from petroleum and natural gas, they are a limited resource.

Of the two main classes of plastics, thermoplastics and thermosets, thermoplastics are more suited to recycling because of their less cross-linked structure.

Of all the plastics used in packaging, polyethylene is the one used most abundantly. According to Weiss, Pearson and Morrow (1988), 62.1% of all household containers are polyethylene. Rigid containers are easier to collect. Consumers tend to treat plastic wrapping as paper and discard it. It is easier to separate rigid containers for collection.

The primary objective of this research was to investigate the recycling potential of high density polyethylene (HDPE) milk bottles. The HDPE bottles cannot be reused for food contact. Any material to be used for food contact has to have FDA approval. The most common products made from recycled HDPE are park benches, fence posts, etc., i.e., mostly structural applications. One of the limitations of using HDPE as a structural material is its low stiffness and high creep. These can be overcome by making a composite material with fibers for reinforcement.

Short fibers are most suited for reinforcing thermoplastics. Although the reinforcing potential of short fibers is less than that of continuous fibers, the processes of fabricating a short fiber composite are simpler and less expensive. Short fiber composites can be made isotropic and are therefore easier to design with.

Since HDPE regrind is very inexpensive, the fibers should also be inexpensive. Wood-fibers, which are cheap and abundantly available were used in this study with recycled HDPE.

LITERATURE REVIEW

1. Theoretical Background

A fiber reinforced composite consists of three basic elements each of which has an impact on the properties of the composite : fiber, matrix and the fiber-matrix interface.

Fibers contribute to the increased strength and modulus of the composite. They provide the resistance to breakage and bending under applied stress or load. The matrix holds the fibers together and binds them so that they are able to act as a team. The matrix also maintains the fibers in the desired orientation and keeps them separated from one another. It transmits and distributes stress onto the individual fibers. The matrix also provides protection against fiber abrasion and harmful effects of the environment. The interface is a critical factor which determines to what extent the potential properties of the composite will be achieved and maintained during usage. The interface must have appropriate chemical and physical features in order to provide the necessary load transfer function from the matrix to the fibers. Localized stresses are usually highest at and near the interface. The interface may be the point of premature failure of the composite.

As applications of composites increase, it becomes necessary to be able to predict the properties of the composite based on the constituent matrix, fiber, fiber volume and orientation.

Ideally, the Rule of Mixtures should be able to predict the Modulus and Tensile Strength of a composite as shown:

$$E_c = V_f E_f + V_m E_m$$
$$\sigma_c = V_f \sigma_f + V_m \sigma_m$$

where E - Modulus

σ - Tensile strength

V - volume fraction

Subscripts f - fiber

m - matrix

The modulus of a continuous fiber reinforced composite can be predicted quite accurately with the above equation. However, the tensile strength predicted is higher than the actual results as other factors also influence it.

In the case of discontinuous fibers, the fiber ends also have to be considered.

Effect of fiber ends:

In the case of short fibers, an applied stress is not evenly distributed along each fiber.

Berlin et al in their book "Principles of Polymer Composites" (1986) explain the uneven distribution of stress using a fiber of cross sectional area S, radius r and length L. The tensile load transmitted to the end segment of length x by the rest of the fiber is equal to (using the mechanical equilibrium equation) the portion of the tensile load transmitted by tangential stresses developed at the interface, to the side face of the fiber and the portion of the tensile stress transmitted by normal stresses from the matrix to the fiber end face.

$$\sigma_x = \frac{1}{\pi r^2} \int_0^x \tau(x) 2\pi r \, dx + \sigma_m \pi r^2 \quad \text{eq(1)}$$

where σ_x - is the stress at the fiber end

τ - shear stress

σ_m - stress in matrix

$$\text{At the fiber end, stress } \sigma_m = \frac{\sigma_x \pi r^2}{\pi r^2}$$

ie., stress at the fiber end is equal to the stress in the matrix.

The farther away from the end, the larger will be the value of x so that the second term in equation (1) can be ignored which means that the stress transferred to the fiber increases. The stress can grow until it becomes equal to that of continuous fiber. The minimum value of x for which the stress reaches the maximum is called the critical fiber length.

When $x \geq L_c$:

$$\sigma_x S_f = E_f \epsilon_f S_f = \int_0^{x=L_c} \tau(x) 2\pi r dx$$

For plastic matrix, $\tau_x = \tau_m$ hence

$$E_f \epsilon_f S_f = \tau_m 2\pi r L_c$$

The maximum stress that can be applied to a fiber is defined as its ultimate strength :

$$(E_f \epsilon_f)_{\max} = \sigma_f^*$$

$$\text{ie } L_c = \frac{\sigma_f^* r}{2 \tau_m}$$

Substituting diameter for radius and considering both ends of the fiber:

$$\frac{L_c}{d} = \frac{\sigma_f^*}{2\tau}$$

τ depends on the processing conditions and adhesion. This relationship shows that when adhesion is strong, shorter fibers can be used to

reinforce the matrix.

When the length of a fiber is less than the critical length, the length is called ineffective length. When a tensile load is applied, the matrix will flow around the fiber and there is no reinforcement.

According to Folkes (1981), for a fiber to reinforce a matrix:

1. The fibers have to be "wetted out" i.e. each fiber must be totally enclosed by the matrix.
2. The fibers should be uniformly dispersed throughout the matrix.
3. The fibers should be of sufficient length compared to the diameter to ensure an effective transfer of stress from the matrix to the fibers.

Modulus of short fiber reinforced composites

Modulus is the easiest property to predict because it is a bulk property that depends primarily on the geometry, modulus, particle size, distribution and concentration of filler. (Bigg, 1985). Bigg lists a number of models which have been developed by various people to predict the modulus of short-fiber reinforced composites:

MODEL	EQUATION
Einstein (1906)	$E_R = 1 + 2.5 V_f$
Guth (1944)	$E_R = 1 + 2.5 V_f + B\phi^2$ where $B = 14.1$ usually
Mooney (1951)	$E_R = \exp \left[\frac{K V_f}{1 - V_f/V_m} \right]$ where $K = 2.5$ usually

Kerner (1956)

$$E_R = \frac{G V_f [(7-5\nu)G_p + (8-10\nu)G_f]}{G V_f [(7-5\nu)G_p + (8-10\nu)G_f] + \frac{V_f [15(1-\nu)]}{V_f [15(1-\nu)]}}$$

Thomas (1965)

$$E_R = 1 + 2.5V_f + 10.05 V_f^2 + 2.73 \exp 16.6 V_f * 10^{-3}$$

Frankle-Acrivos (1962)

$$E_R = 1 + \frac{9}{8} \left[\frac{V_f / V_m}{1 - V_f / V_m} \right]$$

Nielsen (1970)

$$E_R = \frac{1 - AV_f}{1 - WV}$$

where $W = 1 + (1 - V_m / V_m^2) V_f$
 $A = f(\text{geometry})$

Quemada (1972)

$$E_R = 1 / (1 - 0.5KV_f)^2$$

$K = 2.5$ usually

E_R - relative modulus
 V_f - volume fraction of filler
 G_f - shear modulus of filler
 G_p - shear modulus of polymer
 ν_p - Poissons ratio of polymer
 V_m - maximum packing fraction of filler

The first few models developed (Einstein, Guth, Thomas) are for spherical particles. Quemada introduces a variable coefficient to account for interparticle interactions and differences in particle geometry. Mooney and Frankle and Archivos introduce the concept of maximum packing fraction to account for the differences in particle geometry. Nielsen's modification of Kerner's model takes into account both the maximum packing fraction of the filler, inter particle interactions, and the relative modulus of the two constituents.

Tensile Strength of short fiber reinforced composites

The tensile strength is more difficult to predict as it depends strongly on local polymer-filler interactions. According to Bigg (1985), two responses are possible :an upper bound response and a lower bound response. Good adhesion of filler with polymer will make it possible to transfer stress from matrix to filler and hence will give an upper bound response while poor or no adhesion will give a lower bound response.

A model for lower bound response was developed by Nicolais and Nicodemo (1973).

$$\sigma_c = \sigma_p (1 - a V_f^b)$$

where σ_c - tensile strength of composite.

σ_p - tensile strength of polymer.

a - constant related to stress concentration.

b - constant related to geometry of filler.

V_f - volume fraction of filler.

When there is no adhesion, $a = 1.21$. When there is some adhesion, the stress induced by the polymer is less and $a < 1.21$. The degree of adhesion influences the tensile strength more then the concentration of filler.

$b = 2/3$ when sample fails by random fracture.

$b = 1$ when sample fails by planar fracture.

The upper bound response is more difficult to predict because it depends on quantitative information regarding the adhesion between the polymer and the filler. An upper bound response indicates some degree of reinforcement. This model was proposed by Leidner and Woodhams (1974):

$$\sigma_c = (\sigma_1 + C \tau_c) V_f + \sigma_p (1 - V_f)$$

where σ_1 - interfacial tensile strength

C - constant (determined to be 0.83)

This model predicts a linear relationship between relative strength and concentration. Many systems, however, show an increase in tensile strength at lower concentrations and a decrease at higher concentrations.

An alternative model proposed accounts for the reinforcement of the polymer by improved adhesion between the polymer and filler and the competing loss in strength due to stress concentrations at the interface.

$$\sigma_c = \sigma_p (1 - aV_f^b + c V_f^d)$$

a and b are same as for lower bound response.

c and d depend on polymer-filler adhesion.

Impact strength of short fiber composites

Impact strength is influenced to a large extent by the processing conditions and therefore very difficult to predict. According to Agarwal and Broutman (1980) the Izod impact energy depends on the type of matrix. In the case of a ductile matrix (like polyethylene), the triaxial restraint of the matrix between fibers limits the elongation of the matrix, and thus addition of rigid fibers greatly reduces the toughness.

2. Use of wood-fibers in composites with polymers

A sizable amount of research with wood-fibers has been conducted in tropical countries which have an abundance of fiber producing trees. A

lot of this work has involved thermosets. Many thermoplastics also have been reinforced with wood-fibers to form composites.

Semarzabeh (1985), showed that cellulosic fibers are comparable to glass fibers as reinforcement for polyester at a fraction of the cost. Pretreatment with acrylic acid increased the chemical bonding of the wood-fibers to polyester. It improved the mechanical properties and decreased the tendency of wood-fibers to absorb water.

Per Flodin has studied wood-fiber polyester composites. Flodin and Zadorecki (1986) made composites by impregnating paper sheets with polyester. Under dry conditions, they found an increase in mechanical properties. However, when immersed in water the moisture uptake was high and a drastic reduction in strength and stiffness was observed. Flodin, Hua, Zadorecki and Powell (1987) showed that treatment with formaldehyde and di-methylomelamine, to modify the surface of cellulosic fibers, reduced water uptake by 46 - 52 %. The wet strength improved by 50% or more. They also conducted surface analysis of the wood-fibers to study adhesion under dry and wet conditions using SEM, transmission electron microscopy and electron spectroscopy.

Goettler (1983) used natural cellulosic fibers treated to provide dispersion and strong interfacial bonding (Santoweb-W fiber, provided by Monsanto Company) to reinforce air and water hose made of plasticised PVC. Performance of this hose was shown to compare favorably with commercial cord constructions. Cellulose fiber reinforced hoses provide a means for economical hose production in a single step by eliminating the need for separate reinforcing and covering operations.

Polystyrene was shown to be reinforced with wood-fibers. Sanchagrin, Sean and Kokta (1988) used Poly(methylene(polyphenyl isocyanate)) as a

coupling agent.

Bataille, Ricard and Sapicha (1987) fabricated a composite of polypropylene containing wood-fibers. With unmodified polypropylene and cellulose fibers, there was a decrease in the ultimate stress until it reached a plateau indicating that there was no real adhesion between the two components. They, however, observed an increase in the modulus, with a rather abnormal increase at 30% volume fraction content. They tried various additives to modify the surface interactions between matrix and cellulose : Silane A-174, Silane A-1100 and maleic anhydride modified polypropylene. Improvement was observed in all the mechanical properties. Their conclusion was that the oxidation of polypropylene improved the adhesion between the phases.

Polyethylene has also been used as the matrix in composites with wood-fibers. Beshay, Kokta and Daneault (1985) used chemithermo-mechanical pulp of Aspen fibers, with a silane as a coupling agent, as reinforcement for linear low density polyethylene. Compared to the resin, the composite showed an increase in Modulus by a factor of 2.6, in stress by a factor of 2.1 and energy at yield by 2.1. The wood-fiber composite compared very favorably with glass-fiber LLDPE composite. The mechanical properties of these were compared to mica, glass-fiber and wood-flour composites exposed to extreme conditions (Kokta, Daneault and Beshay, 1986). They found that the grafted Aspen fiber composites withstood the extreme conditions the best.

Michell, Vaughan and Willis (1978) fabricated a composite by hot pressing sheets of paper coated with low density polyethylene. Mechanical properties of the laminate were studied with particular attention to the effects of moisture. The flexural properties of the

laminate at low relative humidities compared favorably with those of glass filled HDPE and of paper-phenolic resin laminates. At high relative humidities, the properties of the laminate declined sharply.

Ramirez and Sanchez (1984) developed a new material from waste polyethylene, wood-fibers and mineral fillers. The fibers were in the form of a mat and a mixture of PE and sand was deposited over the mat and then compression molded. The composite panel had good flexural properties and low water absorption.

3. Structure and composition of Wood-fibers

Each natural wood-fiber is essentially a composite in itself, in which rigid cellulosic ultimate fibers are immersed in a cementing matrix of relatively unoriented amorphous short chain structures. The rigid cellulosic backbone possesses one of the highest moduli of rigidity of all known polymers (Cruz-Ramos, 1986). But the fibers that it produces are of moderately high strength because of various structural combinations needed to form the microfibrils which are in turn helically wound to form ultimate hollow fibers. These are the building blocks of the whole wood-fibers.

The fiber wall consists of a primary layer in which the cellulose microfibrils lie transversely to the fiber axis, and three secondary layers in which one or more principle orientations are present. (Michell, Vaughan and Willis, 1985). The great majority of the microfibrils in the secondary wall tend to lie in the plane of the wall, thus creating a laminar structure that is highly orthotropic in nature.

According to Gordon and Jeronimidis (1983), fibers with open lumen are responsible for the toughness of the wood. Ideally, for composites, lignified fibers are the most suited as they are stiffer. They should be delignified only so far as to separate them without damage.

Typically, wood-fibers are multilayered hollow cylinders 1-4 mm in length and 15-30 μm in diameter. Wood-fibers are composed of various amounts of cellulose, hemicellulose and lignin. Minor quantities of other materials like pectins, fat, waxes, etc., may be present.

One of the chief advantages of wood-fibers is that they are renewable. It takes approximately 3 to 7 years for a regular hard fiber producing plant to become a supply of fiber material. From a production point of view, cellulosic fibers are highly energy efficient. Only a small fraction of fossil fuel is needed to produce a wood-fiber compared to the manufacture of synthetic fiber (Cruz-Ramos, 1986).

Some of the other advantages include less machine wear, no health hazard and light weight. The composites produced are some of the lightest materials known. The fibers are more flexible than other synthetic fibers. This is an advantage during processing as the fibers bend rather than break during processing. The hollow nature of wood-fibers may impart acoustic insulation properties to a composite fabricated with wood-fibers (FAO, Research Series on Hard Fibers, 1970). The low cost of wood-fibers, 3-10 cents / lb (Katz and Milewski, 1987) is one of the most important factors which makes it very attractive specially in composites with recycled plastics as matrix.

There are some disadvantages of using wood-fibers. First, the properties of wood-fibers are dependent on a number of factors like age of the plant, climatic conditions etc. This adds to the variability in

the properties of the wood-fibers. According to Cruz-Ramos (1986) a standard deviation of 50% in the properties of the wood-fibers is not uncommon.

Another major problem is the water sensitivity of wood-fibers. Moisture content affects the dimensional stability and the properties of wood-fibers. The biodegradability and photosensitivity of the fibers also have to be considered.

The diameter of wood-fibers is larger than that of synthetic fibers which indirectly decreases the aspect ratio.

The thermal properties of wood-fibers set a limit on the processing temperatures. Most wood-fibers lose their mechanical properties at temperatures above 170⁰ C.

A number of solutions have been found for the problems associated with wood-fibers. Eg. According to Belmares, Barrera and Monjaras (1983), pretreatment with polyvinyl alcohol or polyvinyl acetate controls the water sensitivity of wood-fibers. Zinc chloride in small amounts arrests biodegradability.

MATERIALS AND METHODS

MATERIALS

The materials used to make composites for this study consist of:

1. Softwood-fibers. Spruce fibers were obtained from Masonite Corp. The fibers were air dried before using.

High Density Polyethylene regrind: Post-consumer milk bottles were cleaned and then ground into flake form in a low-line granulator model 68-913 from Polymer Machinery Corp.

Virgin High Density Polyethylene "FORTIFLEX A60-70-119" from Soltex Polymer Corp.

Ethylene Vinyl Acetate (EVA) copolymer "NPE 3304" obtained from Universal Chemical Company.

METHODS

The following ASTM standards were used to evaluate material properties:

ANSI/ASTM D638 - 77a "Test Method for Tensile Properties of Plastics"

ASTM D256 - 84 "Test Methods for Impact Resistance of Plastics and Electrical Insulating Materials"

ASTM D570 - 81 "Test Method for Water Absorption of plastics"

Samples were cut from compression molded plates. A Carver Laboratory Press model M25 ton was used for compression molding. The procedure was as follows:

The required amount of material to be molded was placed between the two

plates of the Compression molder and allowed to melt for a few minutes at 150⁰ C. Next a pressure of about 30,000 lbs was applied for 10 minutes. The press was cooled using running water for 15 minutes while the pressure was maintained.

The compression molded plates were conditioned for at least 40 hours at 23±2⁰ C and 50±5% R.H. before testing.

A. Tensile properties determination (ANSI/ASTM D638-77a)

Specimens were cut according to Specimen Type I of the Standard using a Tensilekut cutting machine. The sides were smoothed using sand paper. An Instron testing machine, model no. 1114, equipped with load cell F having a range varying from 200 lbs to 10,000 lbs was used. The loads used were 200 lbs and 500 lbs. Abrasive paper was used between the grips and the specimen to prevent slippage. A crosshead speed of 0.5 in. per minute was used.

Tensile strength, elongation at break and the tensile modulus were calculated for each specimen as outlined in the standard. Each data point reported is the average of five measurements.

B. Impact Strength Determination (ASTM D256-84)

Specimens measuring 2.5 in. x 0.5 in. were cut from compression molded plates using a band saw, as per the standard. A notching machine from TMI, Notching Cutter model TMI22-05 was used to notch the samples 0.1 in. deep according to the standard for Izod Impact Strength. TMI Impact Testing Instrument Model 43-02 was used. The pendulum weight used was 5 lbs. The Impact tester was calibrated for the pendulum weight each time. The type of break was identified as complete, hinge, partial or

non-break based on the description given in the standard.

C. Determination of water absorbed by specimen

ASTM D570-81 was followed with some variations. Specimens measuring 2.5in. x 0.5in. were cut from the compression molded plates. The edges were smoothened using a sand paper to prevent absorption from the uneven surface. The specimens were conditioned at $23 \pm 2^{\circ}\text{C}$ and $50 \pm 5\%$ R.H. for 2 weeks to allow the specimens to reach equilibrium. The specimens were immersed in water and change in weight was monitored as a function of time. The specimen was wiped with filter paper before weighing to remove surface water. Immersion was continued until equilibrium was reached.

Percent water absorbed was calculated when equilibrium was reached using the formula :

$$\text{water absorbed (\%)} = \frac{\text{gain in weight (g)}}{\text{original weight (g)}} \times 100$$

D. Determination of change in dimensions after water absorption

The exact dimensions of the specimens cut for water absorption were measured using a vernier caliper to ± 0.001 in. Dimensions were again measured when the specimens had reached equilibrium. The percent change in dimensions was calculated using the formula:

$$\text{change in dimension (\%)} = \frac{\text{change in dimension (in.)}}{\text{original dimension (in.)}} \times 100$$

E. Determination of change in tensile strength after water absorption

Specimens were cut from compression molded plates according to specimen type I as outlined in the ASTM standard D638. The specimens were immersed in water at the same time as the water absorption specimens. Tensile tests were conducted according to ANSI / ASTM D638

when equilibrium was reached in the water absorption specimens.

The tensile strength of the specimens was calculated according to ASTM D638.

DRY-MIXING

INTRODUCTION

To mix the wood fibers with the polymer, the first method used was dry mixing. Varying percentages by weight of wood fibers and regrind were mixed in a domestic blender for ten minutes at high speed. The dry mixture was compression molded into plates. Procedure and conditions of molding used were as stated in the methods section. Specimens for testing were cut from the compression molded plates in accordance with ASTM standards.

RESULTS

Table 1: Tensile Properties of Composite Fabricated by Dry Mixing.

Weight % wood-fiber	Tensile strength		Elongation		Tensile Modulus	
	(psi)		%		(psi)	
	ave.	s.d.	ave.	s.d.	ave.	s.d.
0	4220	160	45	8	94,000	412
2	4150	104	10.5	4.4	110,000	6123
4	4127	146	10.2	2.6	120,000	4570
8	4020	177	5.4	1.13	140,000	6834

DISCUSSION

As can be seen from Figure 2, there is a decrease in tensile strength with increase in wood-fiber content. Another problem with this method was that there was inadequate mixing between the two phases. Clumps of wood-fibers could be visibly detected at various regions of

the compression molded plates. According to Sanschagrin, Sean and Kokta (1988) it is extremely difficult to get a good dispersion of long cellulosic fibers due to the strong intermolecular H-bonding system in cellulose.

The advantages of this method were:

1. It is an inexpensive and easy method. The equipment required is minimal
2. Fiber damage is minimal.
3. There is an increase in the Tensile Modulus of elasticity of the composite.

Katz and Milewski (1987) in their book "Handbook of Fillers and Reinforcement for Plastics", state that poorly packed particles will have larger, less uniform zones of matrix between particles. When stress is applied, the smaller matrix zones, which are the weakest points, bear the strain and break more readily. More uniformly packed systems can distribute the stress more evenly and thus, impart higher strength.

For the fibers to reinforce the matrix, it is necessary to have a fiber loading higher than 8%. Based on the rule of mixtures, it can be seen that increase in fiber-loading will increase the effect of the fibers on the composite properties. This is desirable since the wood-fibers have a higher tensile strength and modulus than HDPE.

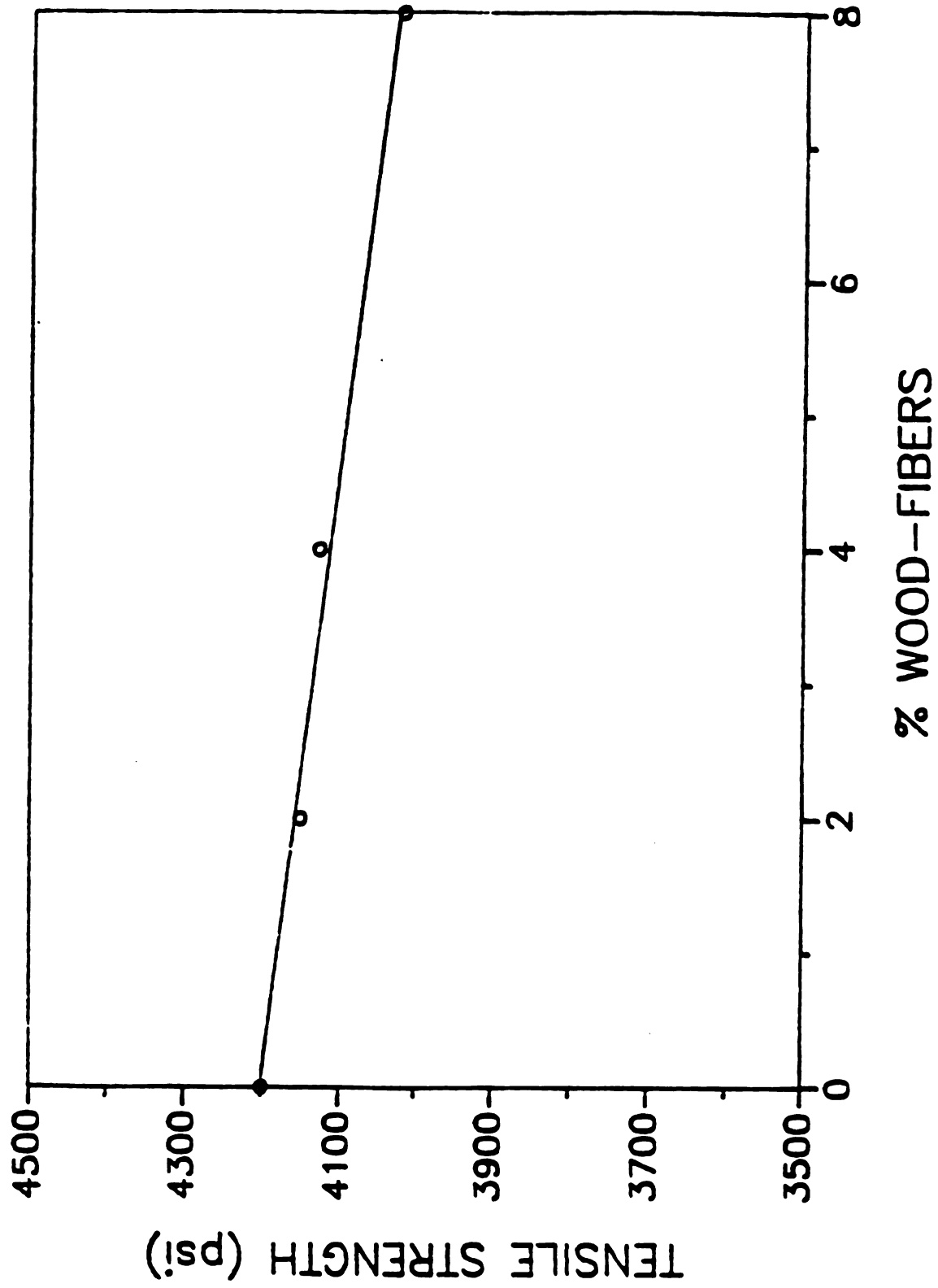


Fig. 2: Tensile Strength of Composites Fabricated by Dry Mixing.

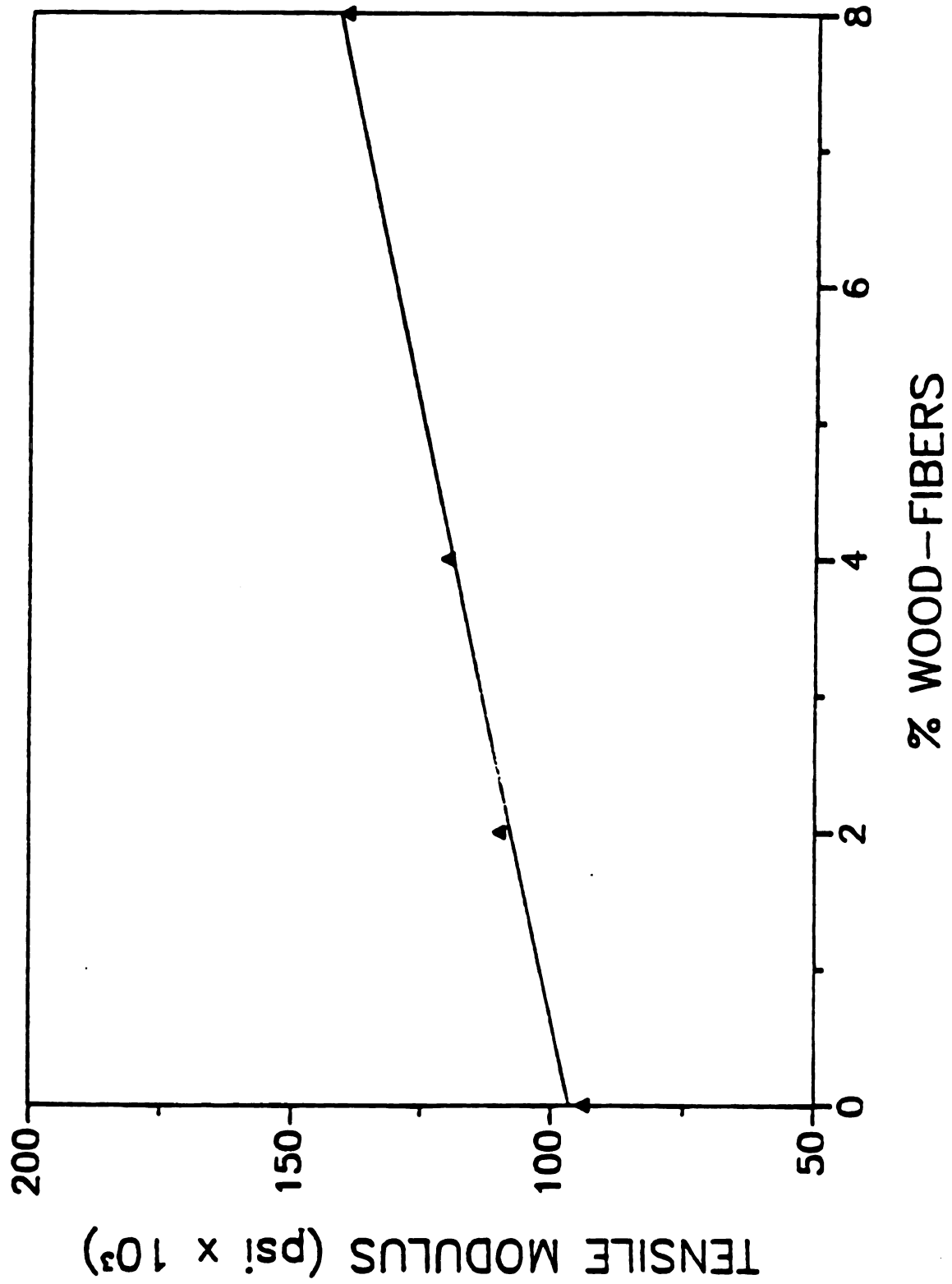


Fig. 3: Tensile Modulus of Composites Fabricated by Dry Mixing.

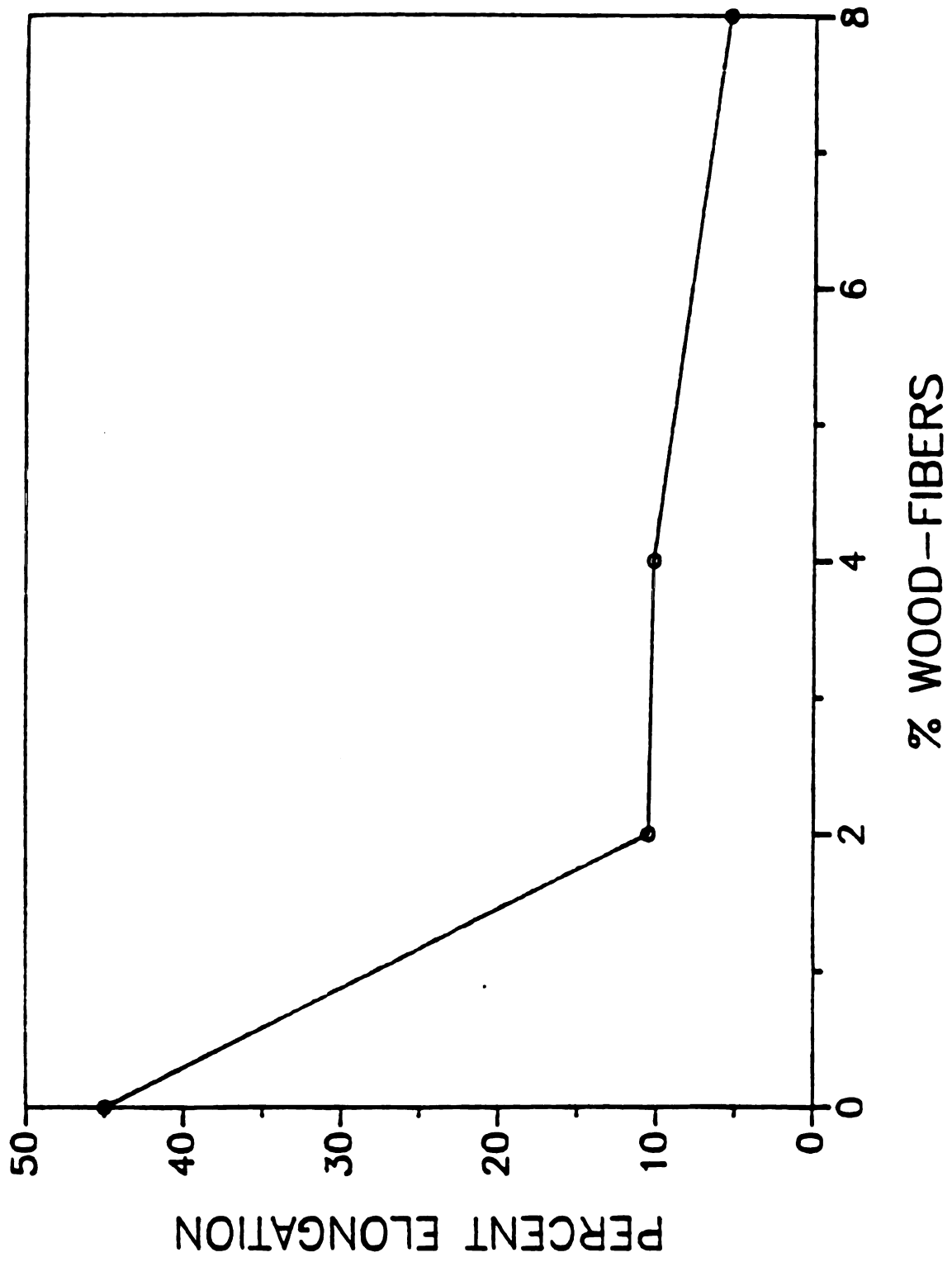


Fig. 4: Variation in Elongation of Composites Fabricated by Dry Mixing.

MELT-MIXING

INTRODUCTION

Better dispersion of fibers in the matrix can help in improving the strength of a composite by distributing the stress throughout the matrix. (Katz and Milewski, 1987). In order to achieve better mixing, a twin screw extruder, Model MPC/V-30 DE, a 38 mm, 13:1 corotating intermeshing extruder from Baker - Perkins was used to melt the polymer and mix the fibers with the molten polymer. The fibers were hand fed into the port at the second stage. The desired fiber loading was obtained by monitoring the torque produced in the extruder. The material obtained from the extruder was compression molded into plates using a Carver compression molder as described earlier and test specimens were cut.

DISCUSSION

The procedure is inexpensive, simple and easily applicable in industry.

As can be seen from Figure 5, the tensile modulus increases with increase in wood-fiber content. At 59% fiber loading, the increase is about 130% compared to HDPE regrind. Modulus is a bulk property that depends primarily on the geometry, particle size and concentration of the fibers.

HDPE regrind cannot be reused for food packaging. All materials that are used for food contact have to have FDA approval. HDPE from milk bottles absorbs a number of chemicals from milk especially butyric acid which cannot be removed from the polyethylene by simple methods like

RESULTS**Table 2 : Properties of composite fabricated by melt-mixing.**

PROPERTY	% WOOD-FIBERS				
	0	18	30	41	59
Tensile Strength (psi)	ave. 4200 s.d. 160	3637 235	3520 264	3250 268	2761 188
Elongation (%)	ave. 45 s.d. 8.0	6.16 0.47	5.1 0.14	3.84 0.79	1.4 0.19
Tensile Modulus (psix10 ³)	ave. 94 s.d. 0.41	117 2.8	133 9.1	143 5.6	218 21.0
Impact Strength (ft-lb/in)	ave. 1.76 s.d. 0.12	1.0 0.09	0.74 0.04	0.726 0.09	0.04 0
Change in weight(%)	ave. 0 s.d. 0	1.74 0.15	3.2 0.98	9.8 0.39	18.3 0.74

Table 2 (Cont'd)

PROPERTY	% WOOD-FIBERS				
	0	18	30	40	59
Change in length(%)	ave. 0	0	0	0.54	1.1
	s.d. 0	0	0	0.11	0.14
Change in width(%)	ave. 0	0	0	0.92	4.14
	s.d. 0	0	0	0.24	0.95
Change in thickness(%)	ave. 0	0.86	1.7	7.6	13
	s.d. 0	0.13	0.17	1.2	2.4
Tensile Strength after absorption (psi)	ave. 4200	3659	3360	3120	2458
	s.d. 149	204	325	141	82
Change in Tensile Strength (%)	ave. 0	0	4.5	9.8	11

washing. Recycled HDPE, at present, is used for the most part, in structural applications eg. in making park benches, fence posts etc. Though HDPE has the strength required for such applications, the tensile modulus, which is an indication of stiffness is low. Therefore, the increase in stiffness achieved by the addition of the fibers will make recycled HDPE more attractive as a structural material.

Tensile strength depends strongly on local polymer-filler interactions. (Bigg, 1985). Two important factors that affect the tensile strength of randomly oriented short fiber composites are the fiber length and adhesion between the fiber and the matrix.

As can be seen in Figure 6, there is a decrease in tensile strength with increase in wood-fiber content. This possibly is due to :

1. Poor adhesion between the fibers and the matrix. Cellulose, which is the main component in wood-fibers, has hydroxyl groups on the surface which gives it polarity. Polyethylene, on the other hand, has only carbon and hydrogen and is non-polar. The non-polar HDPE matrix is not compatible with the polar wood-fibers. Additives which form a bond between the two are necessary to increase the tensile strength.
2. Fiber damage during processing which causes the fiber length to decrease below the critical fiber length which is necessary for reinforcement of the matrix by the fibers. Fiber lengths were measured before and after mixing by magnifying the fibers onto a screen. HDPE in the composite was dissolved in xylene at 90°C. The lengths of 200 fibers were measured to get an average. It was found that the aspect ratio decreased from 43.5 to 28. Processing techniques which cause less fiber damage may also bring about reinforcement of the matrix.

The elongation also decreases with increase in fiber content. There

is a steep decrease in elongation when wood fibers are added. At higher fiber loadings, there is only slight decrease in elongation (Figure 7). Elongation decreases from 45% at 0% fiber loading to 6.16% at 18% fiber content. Fibers restrict the movement of the polymer chains resulting in low elongation. According to Berlin et al. (1985) weak interfacial bonding causes almost immediate separation of the matrix from the fibers causing cavitation at low elongation.

The area under the stress-strain curve is an indication of toughness. Since, for this composite, the tensile strength and elongation decrease with increase in wood-fiber content, a decrease in impact strength can be expected. The impact strength, like elongation decreases sharply when fibers are added and the decrease is lower at higher fiber loadings (Figure 8). According to Agarwal and Broutman (1974) the impact strength of a short fiber composite depends on the type of matrix. The addition of short fibers to a ductile matrix, like HDPE, restrains the flow of the matrix resulting in lowering of the impact resistance of the matrix with the addition of fibers. The results obtained (Figure 8) show a decrease in impact resistance with increase in fiber content. As can be seen in Table 2, while the impact of HDPE regrind is 1.76 ft-lb/in it decreases to 1 ft-lb/in at 18% fiber loading, 0.74, 0.726 and 0.04 at 30, 40 and 59 % fiber loadings respectively.

One of the limitations of using wood-fibers is their sensitivity to water. The hydroxyl groups in the cellulose attract water resulting in a decrease in the mechanical properties and an increase in the dimensions. As might be expected, the greatest effect of water is seen on the composite with the largest content of wood-fibers (Figure 9). Almost no

effect of water on the composite is seen till 30% wood-fiber content (Table 2) There was almost no change in the weight, dimensions or tensile properties. Above 30% fiber content, water has a greater effect on the composite. Water absorption increases sharply. The effect on the dimensions and the tensile strength also increases sharply. At 59% fiber content, the composite absorbs about 18% of water. The change in length is 1.1%, width 4.14% and thickness 13%.

As can be seen from Figure 10, there is no effect of water on the tensile strength until 40% wood-fiber content. The hydroxyl groups of water compete with those of cellulose for hydrogen bonds. This results in disruption of the cellulose structure which is responsible for the strength of the wood-fibers. Table 2 shows that the decrease at 59% fiber content is 11%. This is much less than might be expected, as the decrease when wood saturation is about 75% (Wood Handbook, 1974). The HDPE in the composite might be forming a protective layer around the cellulose so that the hydrogen bonds in cellulose are not disrupted.

Based on the results obtained, relationships can be established between the property and the components of the composite in the form of empirical equations. These equations can be used to predict the various properties of the composite containing different % of components. The equations developed are:

$$\sigma_c = 4166 - 23.38 W_f$$

$$E_c = 82.65 + 1.97 W_f$$

where σ_c = tensile strength of composite in psi

E_c = tensile modulus of composite in psi

These equations hold good only for composites containing below 60% wood-fibers. Beyond the experimental design constraints, these equations will have to be experimentally validated.

At $W_f \geq 18\%$:

$$\epsilon_c = 45 - 2.15 W_f$$

$$I_c = 1.76 - 0.042 W_f$$

At $W_f > 18\%$:

$$\epsilon_c = 8.7 - 0.117 W_f$$

$$I_c = 1.43 - 0.023 W_f$$

where ϵ_c = % elongation of composite

I_c = impact strength of composite in ft-lb/in

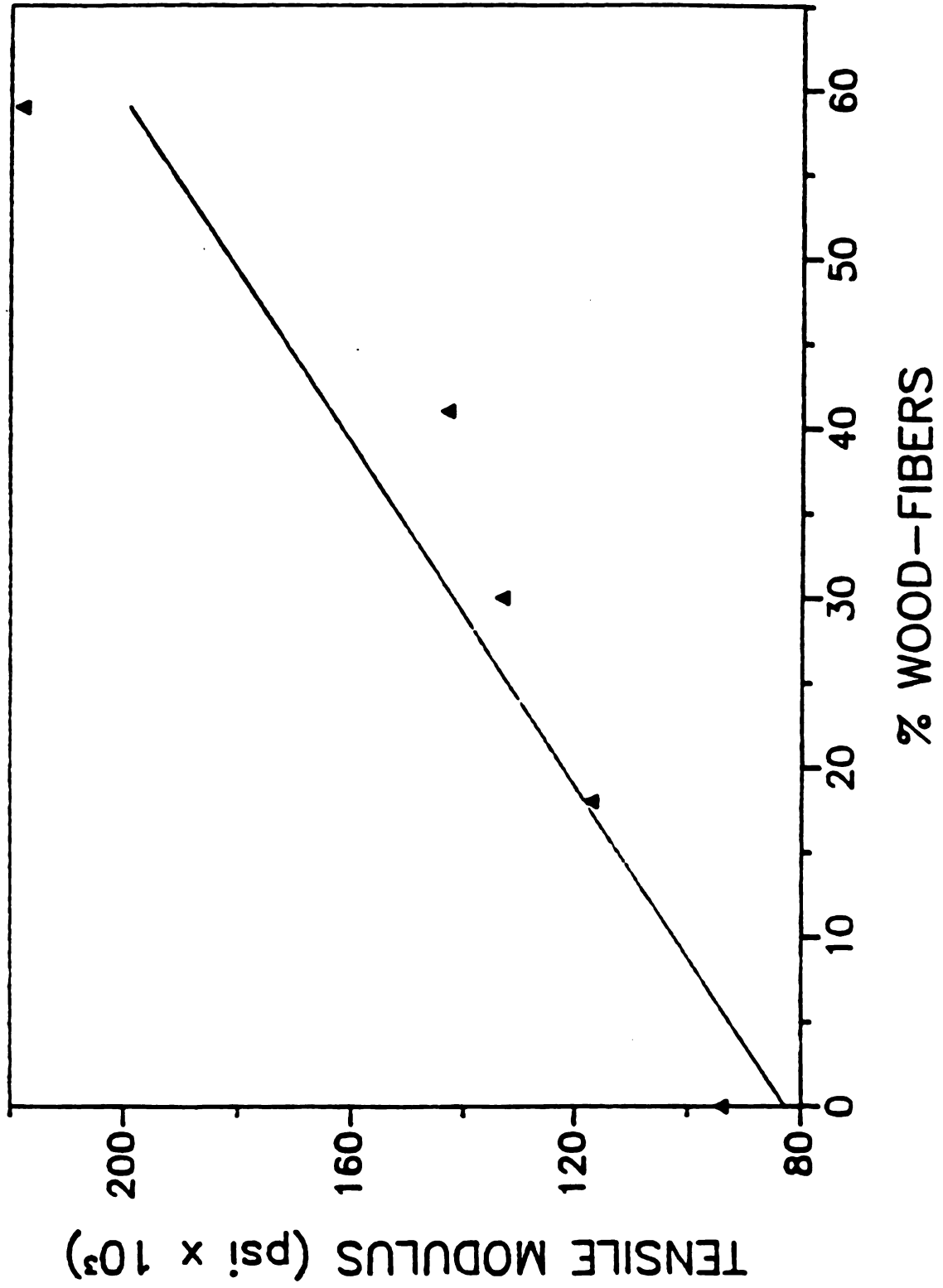


Fig. 5: Tensile Modulus of Composites Fabricated by Melt mixing.

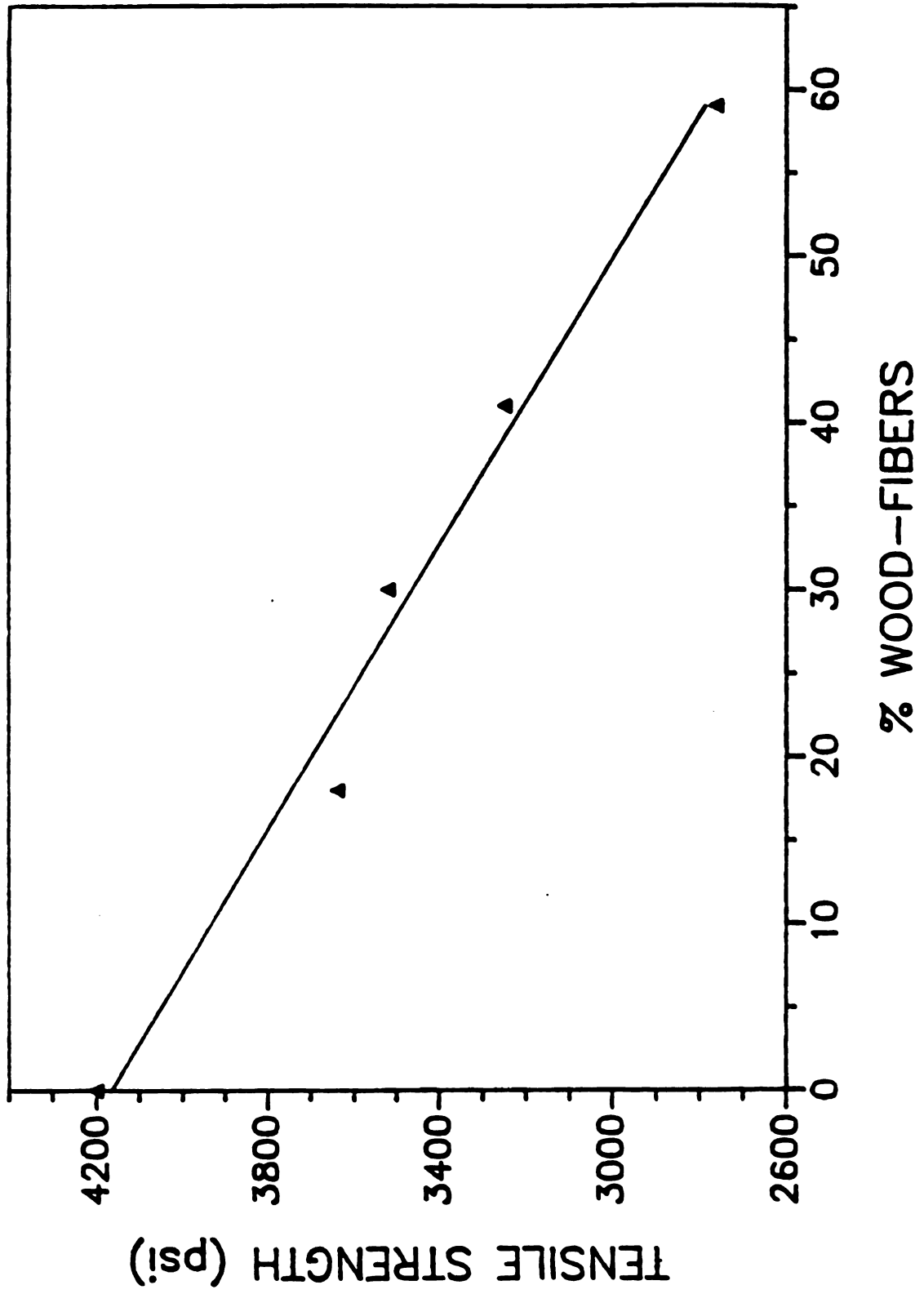


Fig. 6: Tensile Strength of Composites Fabricated by melt Mixing.

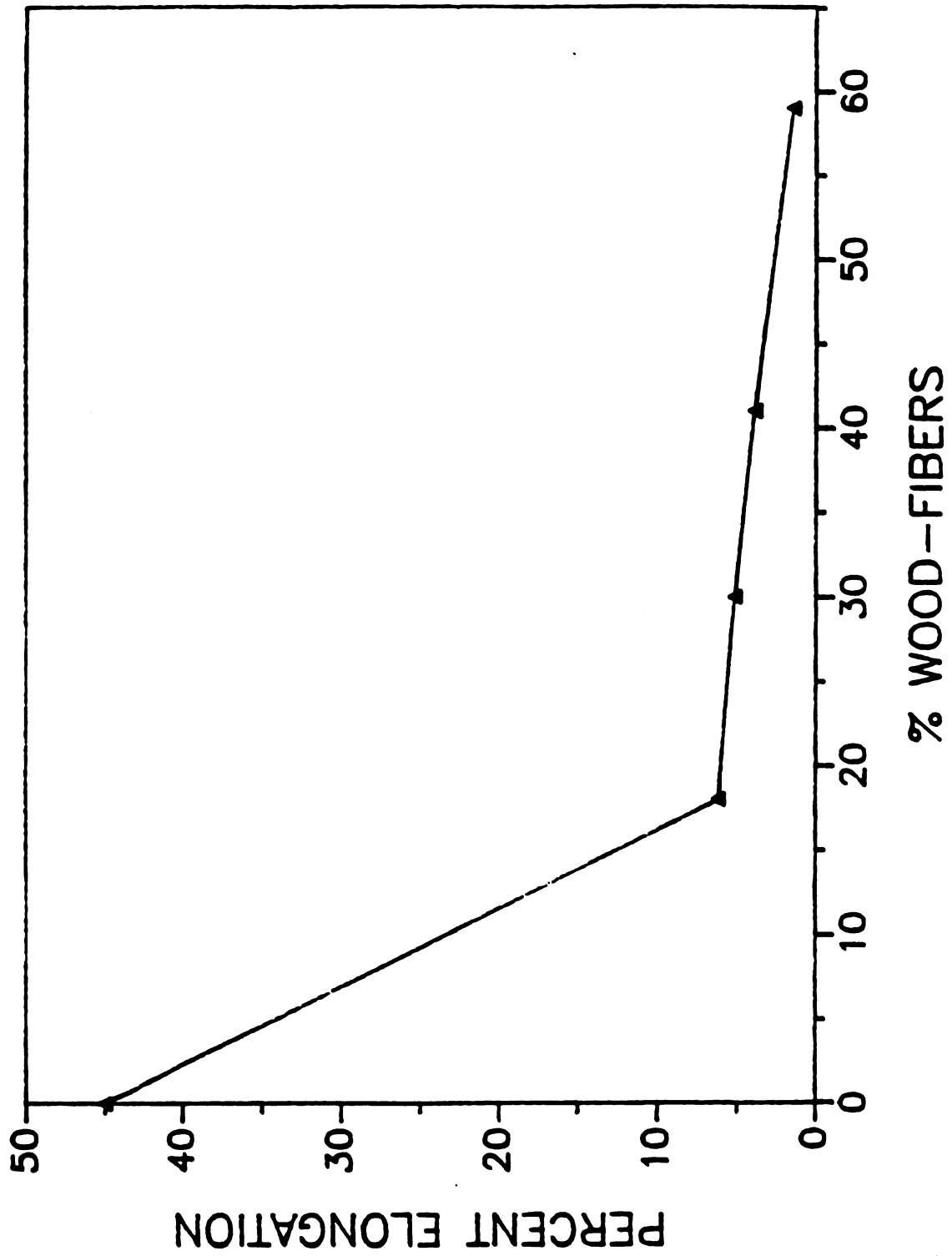


Fig. 7: Variation in Elongation of Composites Fabricated by Melt Mixing.

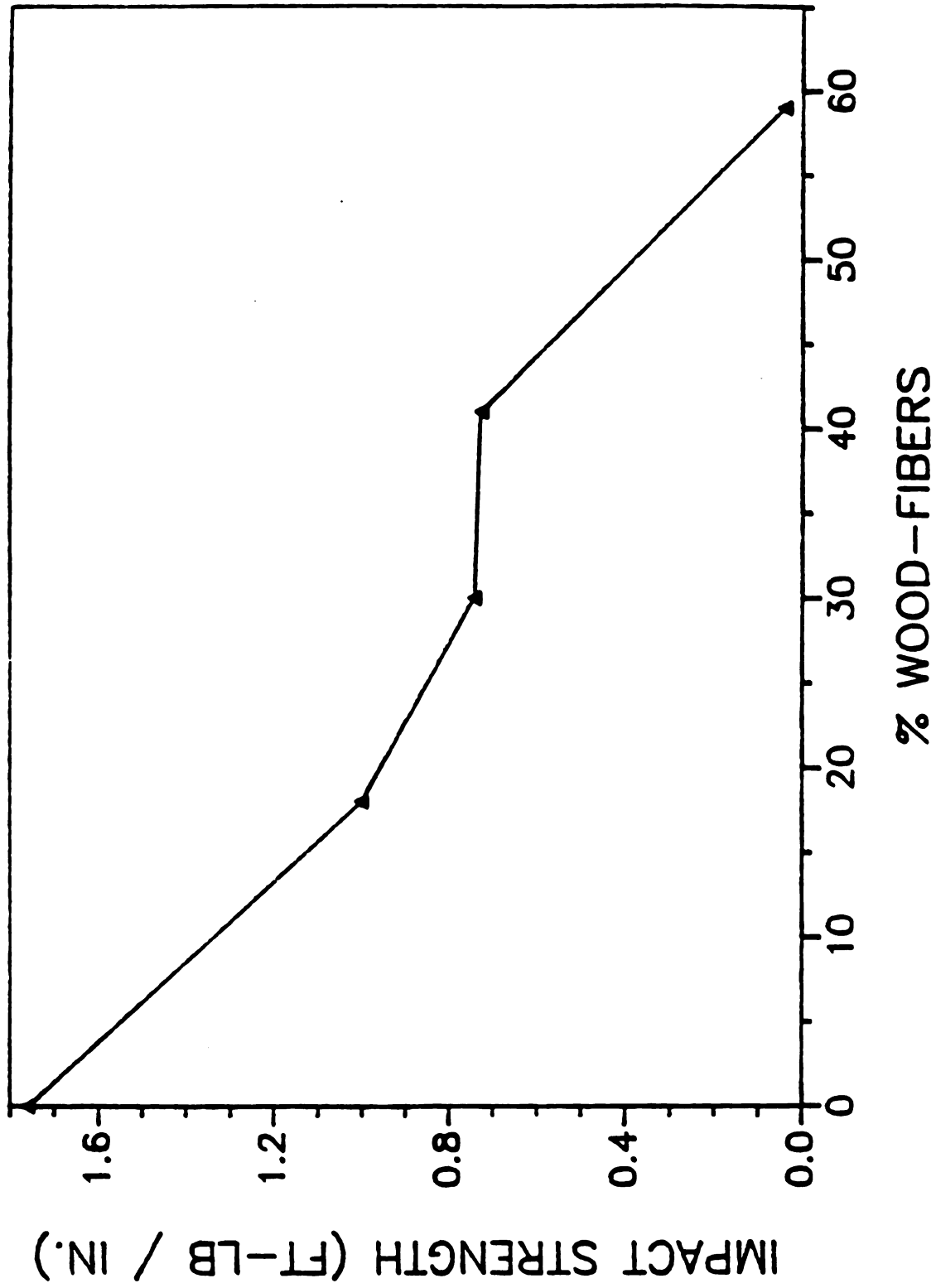


Fig. 8: Impact Strength of Composites Fabricated by Melt Mixing.

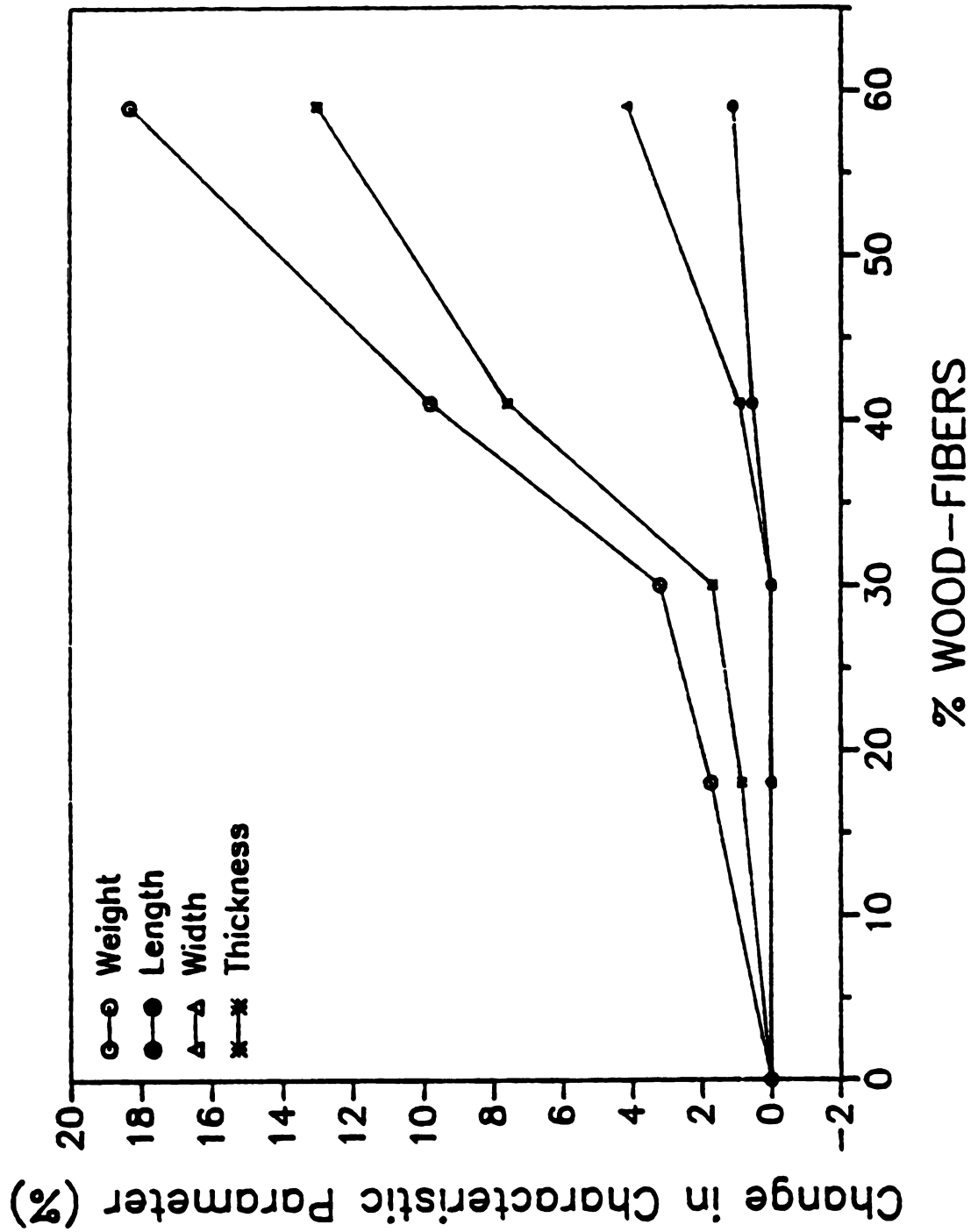


Fig. 9: Effect of Water Absorption at Equilibrium on Characteristic Parameters of Composite Specimens at various Wood-fiber Content

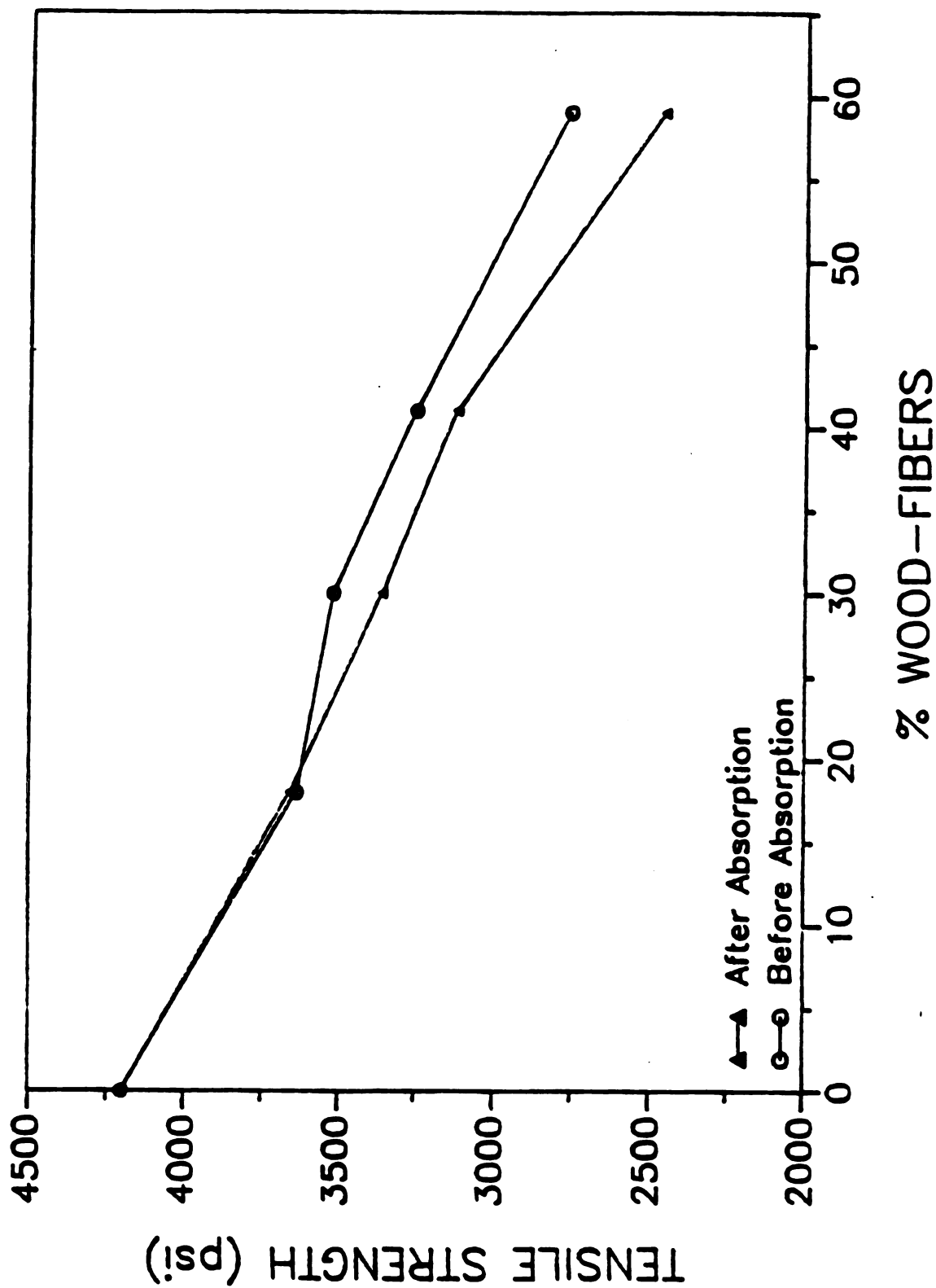


Fig. 10: Effect of Water Absorption on Tensile Strength of Composites of Various Wood-fiber Contents.

COMPARISON OF VIRGIN AND REGRIND HDPE AS MATRIX FOR THE COMPOSITE

INTRODUCTION

Plastics are generally perceived as non-recyclable. The common belief is that the properties of the plastics including that mechanical properties, degrade while processing. While it is true that plastic packaging materials cannot be reused for food contact, many other uses can be found.

HDPE regrind was compared with virgin HDPE as matrix for the wood-fiber HDPE composite. The melt mixing technique, using a twin screw extruder as outlined earlier, was followed to fabricate the composite.

RESULTS

Table 3: Comparison of virgin and regrind HDPE as matrix.

% wood-fibers	Tensile Strength (psi)		Tensile Modulus (psi x 10 ⁻³)	
	Regrind	Virgin	Regrind	Virgin
18	3637	3500	117	116
30	3520	3350	133	123
40	3460	3200	143	133.5
59	2761	2500	218	195

DISCUSSION

Table 3 compares virgin and regrind HDPE as matrix for the wood-fiber composite. As can be seen from Figures 10 and 11, the tensile strength and tensile modulus are higher for the composite with regrind

HDPE as matrix at all wood-fiber contents.

Chate Pattanakul in his thesis "Characteristic changes in Recycled HDPE from Milk bottles" concluded that the tensile strength and tensile modulus of virgin and recycled HDPE do not show any significant difference.

Schnabel (1981), defines polymer degradation as changes in physical properties caused by chemical reactions involving bond scission in the backbone of the macromolecule, which will lead to a reduction in molecular weight. According to him, polymer degradation can be initiated by six mechanisms: thermal, mechanical, chemical, biological, radiation and photooxidation.

Thermal degradation is caused by heat. In polyethylene, thermal degradation can take place during processing by chain-scission or depolymerization ie., 'unzipping' of the polymer chains.

In the case of photo degradation, the energy required to break the bonds is supplied by light. Polyethylene has two kinds of bonds:

C - C which requires 420 kJ / mole

C - H which requires 340 kJ / mole.

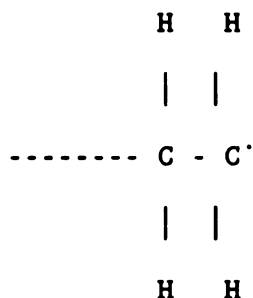
UV radiation which can supply between 300 - 390 kJ / mole can cause C-H bond breakage. For UV radiation to be effective in bond breakage, energy absorbers are necessary. Post-consumer HDPE contains a number of contaminants which can act as energy absorbers.

Mechanical stresses induced while blow-molding and processing the milk bottles can cause polymer chain degradation.

The HDPE bottles may absorb various chemicals from milk or the environment which can initiate HDPE degradation.

Biological degradation of HDPE is also possible. Micro-organisms usually act by attacking chain ends. Since HDPE has long chains, with few chain ends, the effect of micro-organisms may not be noticeable. The presence of chemicals from milk which are food for micro-organisms may aid in biological degradation.

Whatever the initiation process, it will lead, in most cases to the formation of free radicals:



The free radicals formed abstract electrons from other molecules leading to the propagation of more free radicals.

Some of the free radicals can combine with each other leading to cross-linking and cyclization. Sadmohaghegh and Scott (1980), studied the effect of reprocessing on LDPE. They concluded that allylic radicals were formed in the polymer by mechanochemical and oxidative reactions that led to cross-linking.

According to Mitterhofer (1980), chain scission and cross-linking occurred simultaneously during processing of HDPE.

During the processing of HDPE, three changes in the molecular structure are possible which will have an effect on the mechanical properties:

1. reduction of polymer chain length by polymer degradation.
2. cross-linking
3. cyclization.

The degradation of the polymer chains during processing is probably balanced by cross-linking and cyclization of polymer chains so that the tensile properties remain essentially unchanged.

The use of regrind HDPE as matrix offers two advantages :

1. HDPE regrind will be cheaper than virgin HDPE so that the final cost of the composite will be reduced.
2. use of HDPE regrind will help in reducing the plastic waste disposal problem.

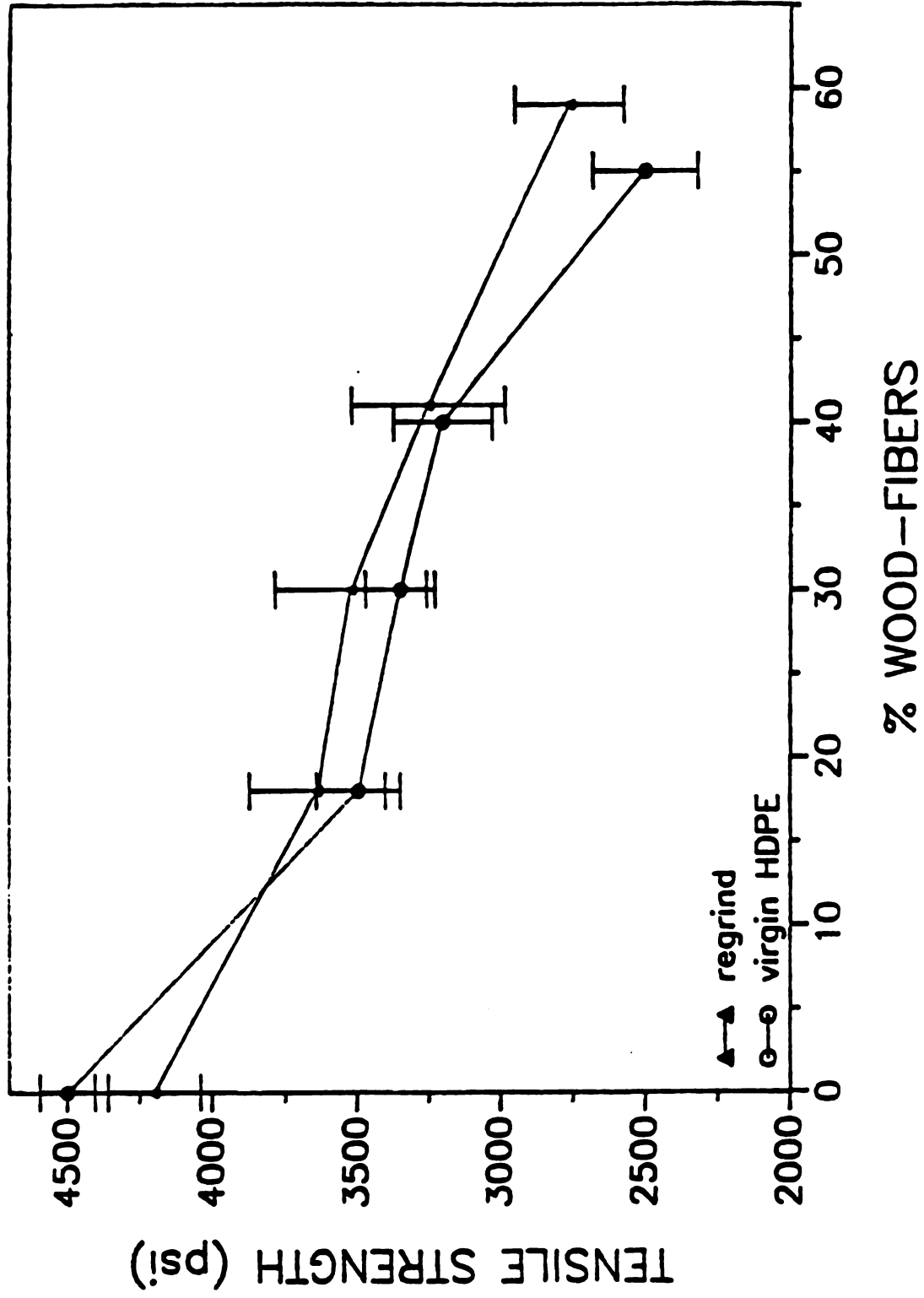


Fig. 11: Comparison of Virgin and Regrind HDPE as Matrix for the Composite; Tensile Strength.

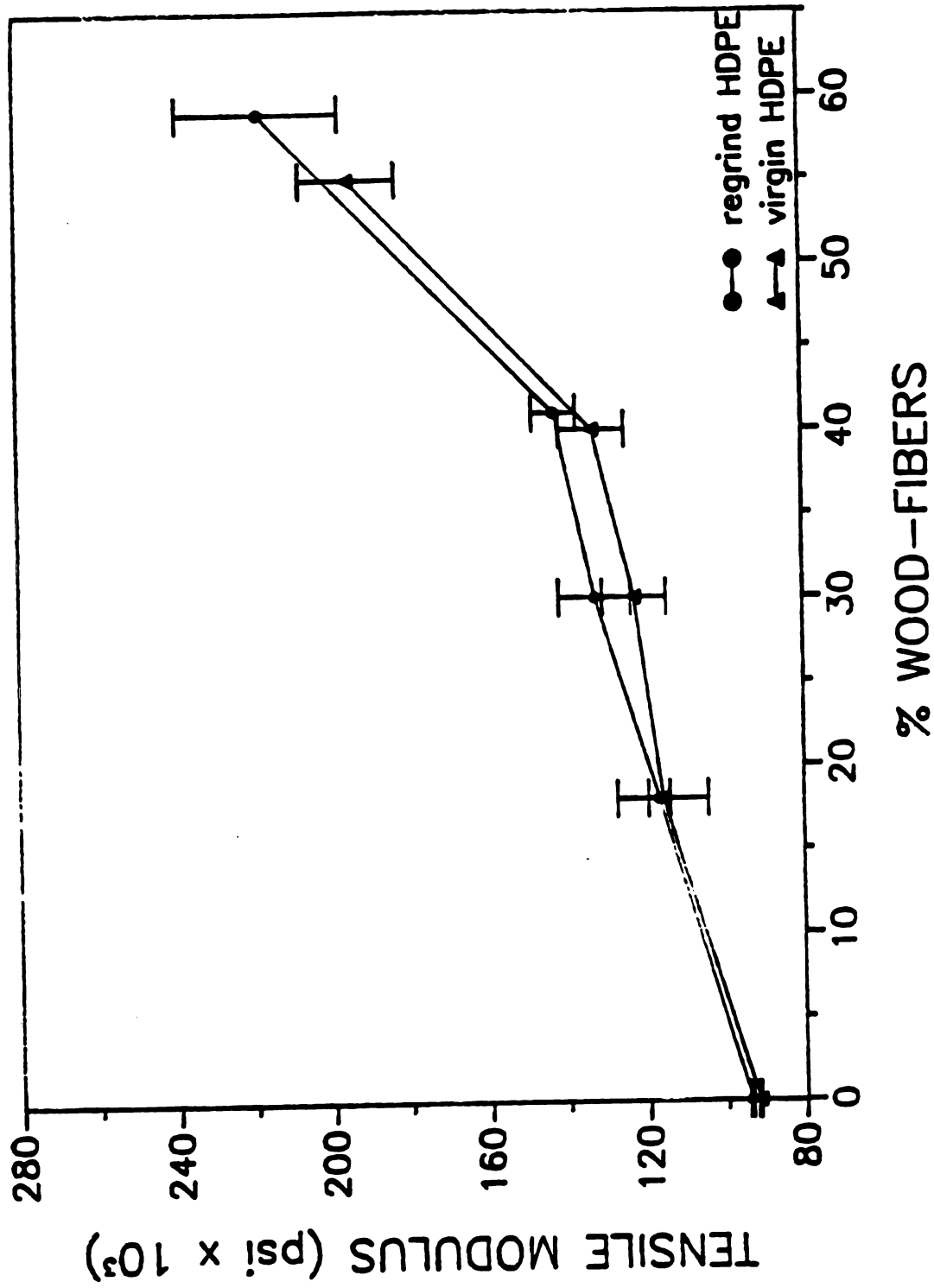


Fig. 12: Comparison of Virgin and Regrind HDPE as Matrix for the Composite; Tensile Modulus.

EFFECT OF ADDITION OF EVA

INTRODUCTION

Tensile strength of the composite can be improved by improving the adhesion between the non-polar HDPE and the polar wood-fibers. Coupling agents with a polar group at one end and a non-polar group at the other end of the molecule will act as a connecting 'bridge' between the two components. Many methods have been tried by different researchers to enhance the adhesion. Bataille et.al. (1987) used various silanes as coupling agents to fabricate a composite of polypropylene with cellulose fibers. Kotha et.al. (1988) added methylene polyphenyl isocyanate as a coupling agent to polystyrene - cellulose fibers. According to them the polar isocyanate groups were compatible with the hydroxyl groups in the cellulose fibers and the methylene groups with the polystyrene.

The coupling agents that have been shown to be effective, are expensive. Pregrafting also is an expensive technique. To use expensive coupling agents in a composite of HDPE regrind and wood-fibers would defeat the purpose of fabricating a low cost structural material.

A material which is cheaper than any of the coupling agents tried and has the required polar and non-polar groups available for bonding, is Ethylene vinyl acetate copolymer (EVA). The polar acetate groups should be compatible with the hydroxyl groups on the cellulose and the ethylene with HDPE so that a 'bridge' is formed between HDPE and wood-fibers.

Varying percentages of EVA were mixed with HDPE regrind and the composites fabricated by the melt-mixing technique.

RESULTS**Table 4 : Properties of composites of EVA and wood-fibers.**

Composition of composite (weight %)	Tensile Strength (psi)		Tensile Modulus (psi)		Elongation (%)		Impact Strength (ft-lb/in.)	
	ave.	s.d.	ave.	s.d.	ave.	s.d.	ave.	s.d.
EVA-fibers								
100 - 0	675	15	1020	0	>200	-	non-break	
79 - 21	780	66	5430	320	>200	-	non-break	
72 - 28	955	41	8800	565	36.5	3.3	2.54	0.15
62 - 38	1030	48	11520	1073	26	4	1.98	0.3

Table 5 : Effect of addition of EVA on composite.

Composition of composite (weight %)	Tensile Strength (psi)		Tensile Modulus (psi)		Elongation (%)		Impact Strength (ft-lb/in.)	
	ave.	s.d.	ave.	s.d.	ave.	s.d.	ave.	s.d.
H - W - E								
27-27-45	1145	35.8	28386	4574	30.8	8.8	1.8	0.19
24-44-32	1630	119	68912	4417	6.5	1.8	1.64	0.09
53-15-32	2167	202	59650	7511	14	4.3	1.52	0.1
10-40-50	1060	117	24700	3899	12	2.4	non-break	
39-22-39	1375	117	25560	1904	37	18	3.12	0.53
23-33-44	960	29	19963	1245	18.6	6	1.98	0.18
20-20-60	762	44.6	15488	2770	> 200		3.66	0.23
36-36-28	2018	241	75412	13100	8.28	1.1	1.04	0.05
10-43-47	908	121	19896	3093	13.8	4.8	2.0	0.07
10-49-41	1064	66.9	24100	0	12	6.2	1.86	0.13
62-32-06	3005	164	133680	9383	4.54	0.57	0.7	0
55-33-12	2774	101	129050	25642	4.7	0.57	0.62	0.04
49-33-18	2790	232	132197	9136	5	0.5	0.72	0.044
40-54-06	2012	160	184490	21058	1.8	0.5	0.42	0.044
36-52-12	2212	167	120330	10595	1.98	0.32	0.5	0
32-50-18	3147	372	135632	10296	2.4	0.55	0.52	0.04
95-00-05	4060	83	92715	262	64	10	3.56	0.24
80-00-20	3654	24	90140	1040	90	10.4	9.22	0.45
60-00-40	3216	49	39268	3982	72.4	11.9	non-break	
20-00-80	1160	42	2344	420	> 200		non-break	

where H, W, E are the weight percents of HDPE, wood-fibers and EVA respectively in the composite.

DISCUSSION

Table 4 shows the effect of addition of wood-fibers to EVA. The tensile properties increase with increase in fiber content. The tensile strength of EVA is 675 psi. At 38% wood-fiber content it increases to 1030 psi, which is an increase of about 50 %. The tensile modulus increases more than 10 times. The increase in tensile strength could be due to the compatability of cellulose with vinyl acetate.

The possible bond formation could be as shown in figure 13.

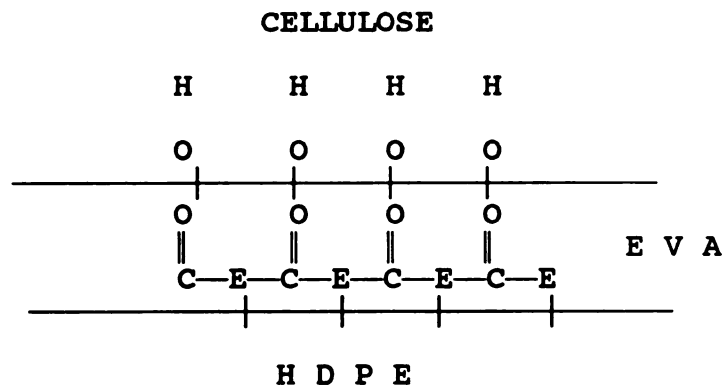


Figure 13 : Possible 'bridge' formation by EVA between HDPE and wood-fibers.

O-H represents the hydroxyl group in cellulose. C=O represents the carboxyl group in the acetate and E the ethylene in EVA.

The polar carboxyl groups of EVA bond with the hydroxyl groups of cellulose while the ethylene in EVA bonds with HDPE. Formation of a bond results in better stress transfer from matrix to fibers. There will be less voids formed around the fibers so that cracks are not easily initiated.

EVA has a lower melt temperature. At 150°C, which is the processing temperature, EVA will be less viscous than HDPE at the same temperature. This may result in less shear during processing so that the damage to the fibers is lowered, thus increasing the effective length of the fibers over which stress transfer can take place. The bond formation and longer fiber length after fabrication of composite must be resulting in reinforcement of EVA by the wood-fibers.

Table 5 gives the properties of wood fiber - HDPE composites with varying percentages of EVA added as a bonding agent.

Table 6 lists the combinations that gave the best properties.

Table 6: Composition of the composites that had the best property.

Property	Content by weight
	H - W - E
Tensile Strength (3147 psi)	32 - 50 - 18
Tensile Modulus (184490 psi)	40 - 54 - 06
Elongation (1.8 %)	40 - 54 - 06
Impact Resistance (non-break)	10 - 40 - 50

As can be seen from Table 6, the composite containing 18% EVA gave the highest tensile Strength. It is higher than the composite containing 33% wood-fibers and 18% EVA. This may be the optimum content of EVA required for reinforcement to occur. But, the tensile strength is still lower than that of HDPE. Though EVA has the potential for bond formation

between wood-fibers and HDPE, it may not be realised at the conditions and the method used.

Multiple regression analysis was used to develop prediction equations for the various properties studied.

$$\sigma = -46781 + 0.1217 [H] + 0.005 [W] + 0.696 [E]$$

$$E_c = 8.9 ([H] E_H)^{0.154} ([W] E_W)^{0.053} ([E] E_E)^{-0.119}$$

$$\epsilon_c = 6.09 ([H] \epsilon_H)^{0.279} ([W] \epsilon_W)^{0.7} ([E] \epsilon_E)^{-0.07}$$

$$I_c = -136 + 0.798 [H] + 81 [W] + 0.092 [E]$$

where σ_c - tensile strength of composite

E_c - tensile modulus of composite

ϵ_c - elongation of composite

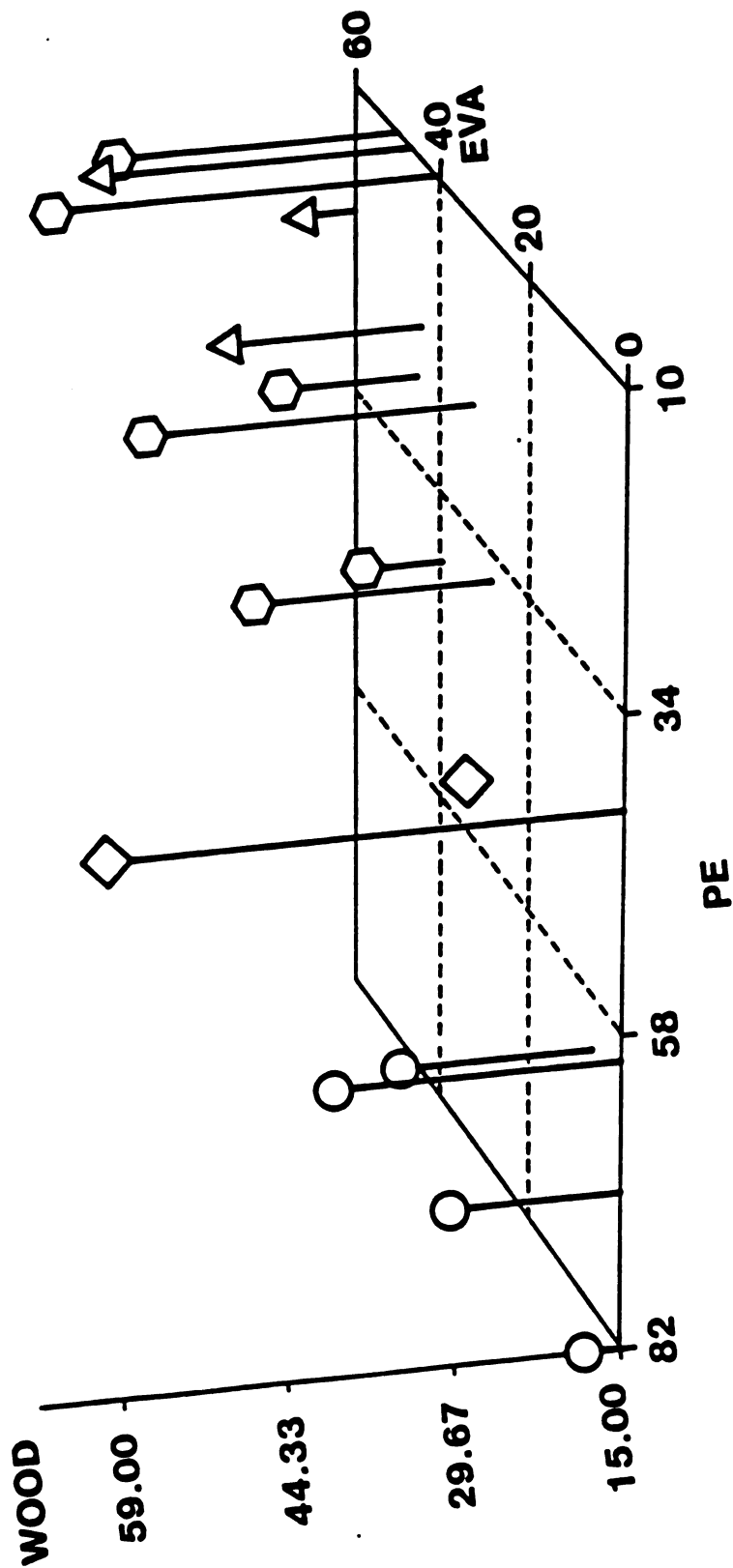
I_c - impact strength of composite

[H] - content of HDPE in composite

[W] - content of wood-fibers of composite

[E] - content of EVA in the composite

TENSILE STRENGTH

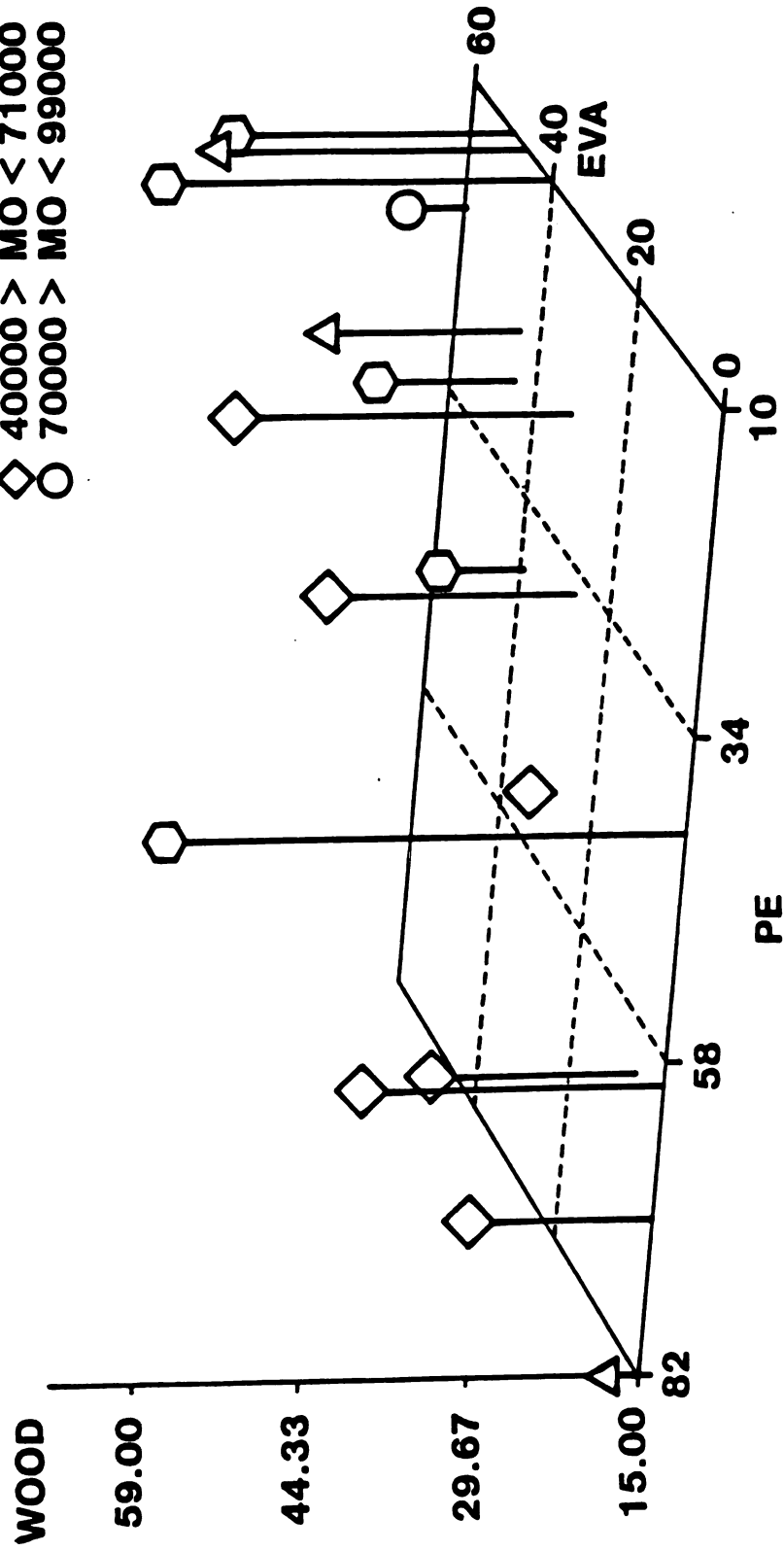


TS: TENSILE STRENGTH

Fig. 14: Effect of addition of EVA on Tensile Strength of Composite.

MODULUS OF ELASTICITY

- △ 5000 > MO < 21000
- ◊ 20000 > MO < 41000
- ◊ 40000 > MO < 71000
- 70000 > MO < 99000

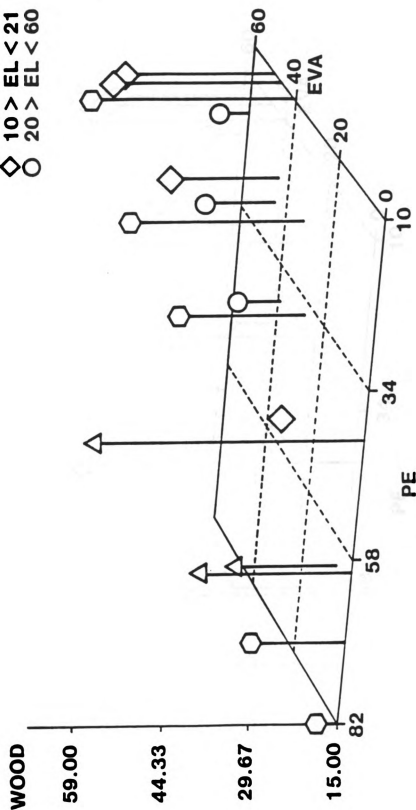


MO : MODULUS OF ELASTICITY

Fig. 15: Effect of addition of EVA on Tensile Modulus of Composite.

PERCENT ELONGATION

- Δ 0 > EL < 6
 $\diamond$ 5 > EL < 11
 $\circ$ 10 > EL < 21
 $\circ$ 20 > EL < 60

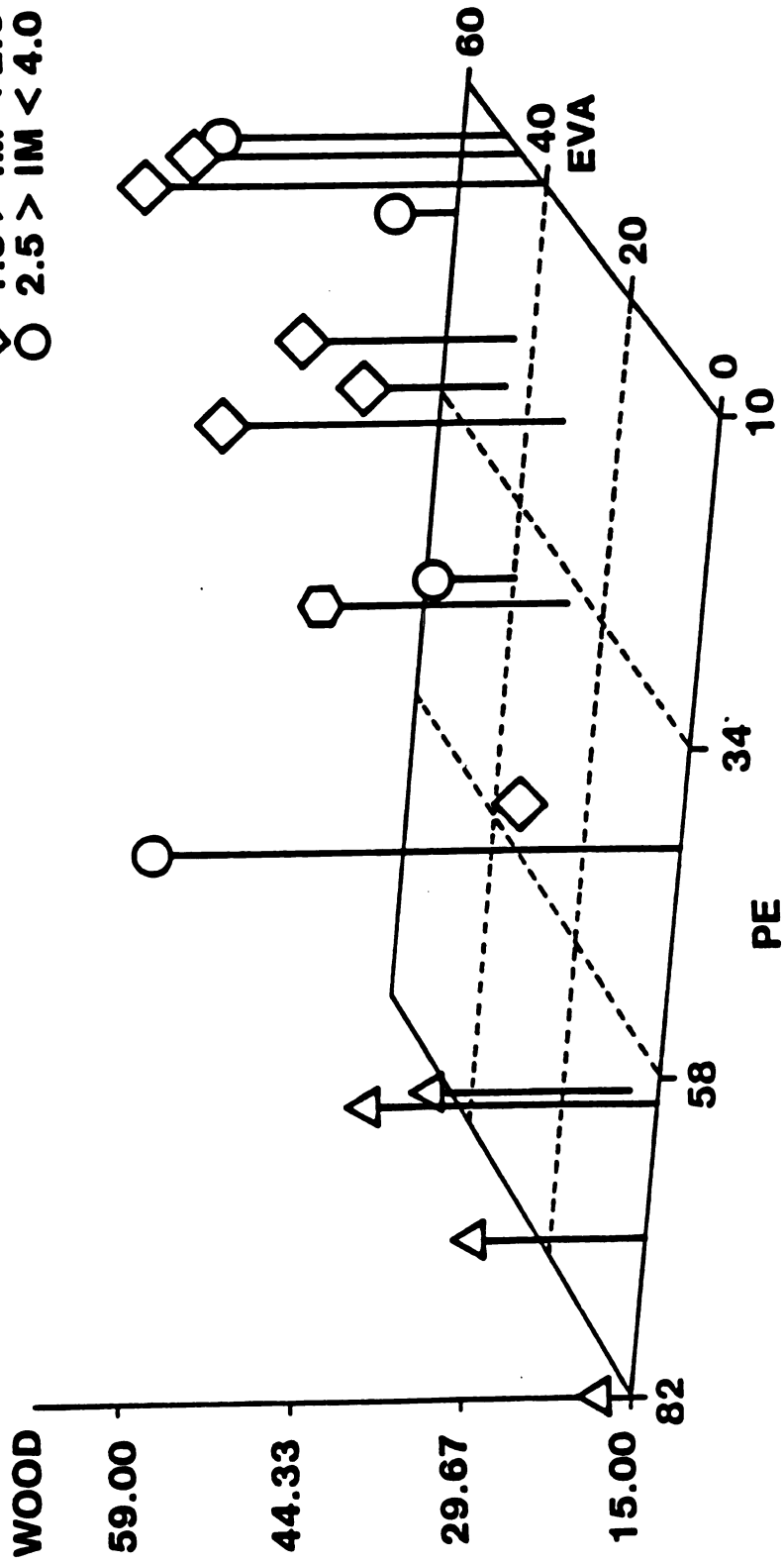


EL: PERCENT ELONGATION

Fig. 16: Effect of addition of EVA on Elongation of Composite.

IMPACT STRENGTH

- △ 0 > IM < 1.0
- ◻ 0.9 > IM < 1.6
- ◇ 1.5 > IM < 2.6
- 2.5 > IM < 4.0



IM : IMPACT STRENGTH

Fig. 17: Effect of addition of EVA on Impact Strength of Composite.

CONCLUSION

I could make the following conclusions based on my research:

1. Melt-mixing was a feasible method for fabricating the HDPE - wood fiber composites. Good mixing and even distribution of the fibers in the matrix was obtained using a twin screw extruder.
2. Addition of wood-fibers increased the tensile modulus. The increase in stiffness of HDPE will be useful in low cost structural applications.
3. Water absorption and its effects on the dimensions and properties of the composite indicate that this composite could be considered to replace wood in some applications.
4. Composites with recycled HDPE as matrix has mechanical properties comparable with the mechanical properties of composites with virgin HDPE as matrix.
5. Addition of EVA as a bonding agent imparts good impact resistance to the composite.
6. Though EVA has the potential to act as a low cost bonding agent between HDPE and wood-fibers, it is not realised at the conditions and the method used to fabricate the composite.
7. The material could be used in low cost structural applications where strength requirements are not high.

APPENDIX A

DATA FOR EACH AVERAGE REPORTED

APPENDIX - A

TABLE A1: Tensile properties of composite fabricated by dry mixing.

Wood-fiber content	Tensile strength (psi)	Elongation (%)	Tensile Modulus (psi)
0%	4150	35	94,300
	4200	42	93,700
	4150	40	94,575
	4500	55	93,675
	4100	60	93,750
ave.	4220	45	94,000
s.d.	160	8	412
2%	4265	18	110,000
	4210	7.5	110,000
	4010	11	115,000
	4075	8	115,000
	4190	8	100,000
ave.	4150	10.5	110,000
s.d.	104	4.4	6123
4%	4255	14	125,000
	3970	7.8	125,000
	3980	10	116,500
	4275	11.3	116,500
	4158	8	117,000
ave.	4127	10.2	120,000
s.d.	146	2.6	4570
8%	4253	4	147,000
	3875	6	147,000
	3858	6.5	132,000
	4160	6.2	136,000
	3958	4.4	137,000
ave.	4020	5.4	140,000
s.d.	177	1.13	6834

TABLE A2: Properties of composite fabricated by melt mixing.

1. HDPE regrind composite containing 18% wood-fibers.

Property	1	2	3	4	5	ave.	s.d.
Tensile Strength (psi)	3630	3628	3515	3685	3392	3637	235
Tensile Modulus (psi)	112980	115800	120350	118575	117225	117000	2799
Elongation (%)	6.2	6.2	5.75	5.75	6.9	6.16	0.47
Impact Resistance (ft-lb/in)	1.14	1.06	0.93	0.97	0.91	1.00	.096
Change in weight (%)	1.6	1.6	1.7	1.9	1.9	1.74	0.15
Change in length (%)	0	0	0	0	0	0	0
Change in width (%)	0	0	0	0	0	0	0
Change in thickness (%)	1	0.8	0.8	1	0.7	0.86	0.13
Change in tensile strength (%)	0	0	0	0	0	0	0

Table A2 (cont'd)

2. HDPE regrind composite containing 30% fibers.

Property	1	2	3	4	5	ave.	s.d.
Tensile Strength (psi)	3530	3865	3550	3120	3535	3520	264
Tensile Modulus (psi)	128240	142900	120975	132600	141300	133200	9139
Elongation (%)	5.25	5	5	5	5.25	5.1	0.14
Impact Resistance (ft-lb/in)	0.78	0.78	0.74	0.70	0.70	0.74	0.04
Change in weight (%)	4.2	1.9	2.5	3.3	4.0	3.2	0.98
Change in length (%)	0	0	0	0	0	0	0
Change in width (%)	0	0	0	0	0	0	0
Change in thickness (%)	1.9	0.47	1.6	1.5	1.7	1.7	0.17
Change in tensile strength (%)	0	0	0	0	0	0	0

Table A2 (cont'd)

3. HDPE regrind composite containing 40% wood-fibers.

Property	1	2	3	4	5	ave.	s.d.
Tensile Strength (psi)	3130	3682	3311	3390	3780	3460	268
Tensile Modulus (psi)	143040	135000	142560	150800	143830	143050	5606
Elongation (%)	3	5	4	3.2	4	3.84	0.79
Impact Resistance (ft-lb/in)	0.66	0.78	0.63	0.86	0.70	0.726	0.09
Change in weight (%)	9.5	10.4	9.7	9.4	9.8	9.8	0.39
Change in length (%)	0.7	0.5	0.5	0.4	0.6	0.54	0.11
Change in width (%)	0.7	0.8	1	0.13	0.81	0.92	0.24
Change in thickness (%)	8.5	7.9	5.4	8.3	8	7.6	1.2
Change in tensile strength (%)	9.2	10.0	9.5	10.4	9.9	9.8	0.46

Table A2 (cont'd)

4. HDPE regrind composite containing 59% wood-fibers.

Property	1	2	3	4	5	ave.	s.d.
Tensile Strength (psi)	2500	2950	2630	2880	2845	2761	188
Tensile Modulus (psi)	246400	189000	210400	215500	228600	217980	21353
Elongation (%)	1.25	1.7	1.5	1.25	1.5	1.4	0.19
Impact Resistance (ft-lb/in)	0.04	0.04	0.04	0.04	0.04	0.04	0
Change in weight (%)	18	19	17.2	18.5	18.9	18.3	0.74
Change in length (%)	1.3	1.0	1.0	1.2	1.0	1.1	0.14
Change in tensile strength (%)	10.4	11.3	11.8	10.1	11.4	11	0.46

TABLE A3: Tensile Properties of composites with Virgin HDPE as matrix.

Wood-fiber content	Tensile Strength (psi)	Tensile Modulus (psi)
18%	3430	134670
	3425	108670
	3580	104670
	3468	115995
	3575	115995
ave.	3495	116000
s.d.	46.5	11518
30%	3330	122500
	3325	129500
	3380	109500
	3355	129500
	3350	124000
ave.	3348	123000
s.d.	21.96	8185
40%	3306	139745
	2970	140700
	3400	120060
	3260	131920
	3085	135075
ave.	3200	133500
s.d.	173.8	8313
55%	2550	185000
	2465	215000
	2485	185000
	2395	192000
	2605	198000
ave.	2500	195000
s.d.	80.6	12430

Table A4: Properties of Composites of EVA and Wood-fibers

Composition of composite (weight %)	Tensile Strength (psi)	Tensile Modulus (psi)	Elongation (%)	Impact Strength (ft-lb/in)
EVA - Fibers				
100 - 0	675	1020	>200	non-break
	650	1020	>200	non-break
	675	1020	>200	non-break
	685	1020	>200	non-break
	690	1020	>200	non-break
average	675	1020	>200	non-break
s.d.	15	0	-	-
79 - 21	720	5600	>200	non-break
	800	5000	>200	non-break
	880	5860	>200	non-break
	720	5350	>200	non-break
	780	5350	>200	non-break
average	780	5430	>200	non-break
s.d.	66	320	-	-

Table A4 (cont'd)

Composition of composite (weight %)	Tensile Strength (psi)	Tensile Modulus (psi)	Elongation (%)	Impact Strength (ft-lb/in)
EVA - Fibers				
72 - 28	920	8800	65	2.6
	900	8000	37	2.6
	1000	9600	37	2.7
	960	8800	32	2.5
	980	8800	40	2.3
average	955	8800	36.5	2.54
s.d.	41	565	3.3	0.15
62 - 38	1000	10400	32	2.2
	1080	12000	25	1.8
	960	10400	27	1.8
	1060	12000	25	2.4
	1040	12800	21	1.7
average	1030	11520	26	1.98
s.d.	48	1073	4	0.3

Table A5: Effect of addition of EVA on properties of Composite.

Composition of composite	Tensile Strength (psi)	Tensile Modulus (psi)	Elongation (%)	Impact Strenght (ft-lb/in)
H - W - E				
27-27-45	1140	30400	22	1.9
	1120	32000	24	1.9
	1120	25600	34	2.0
	1207	32200	30	1.6
	1141	21733	44	1.6
24-44-32	1750	66680	6	1.7
	1680	64000	9	1.5
	1440	73600	7.5	1.6
	1600	73600	4	1.7
	1680	66680	6	1.7
53-15-32	2250	66680	10	1.5
	2120	67800	18	1.5
	1834	50010	10	1.4
	2310	57750	19	1.5
	2320	56000	13	1.7

Table A5 (cont'd)

10-40-50	1182	30300	12	non-break
	1110	23125	14	non-break
	870	19720	8	non-break
	1045	26100	14	non-break
	1090	24240	12	non-break

39-22-39	1440	25600	57	2.6
	1250	26672	17	2.7
	1539	27360	10	3.9
	1360	22400	32	3.0
	1288	25760	22	3.4

23-33-44	974	19800	18	2.2
	966	21465	28	2.1
	989	20935	19	1.8
	912	18415	11	1.8
	960	19200	17	2.0

20-20-60	836	19600	>200	3.3
	720	12800	>200	3.6
	760	16000	>200	3.8
	735	13040	>200	3.9
	760	16000	>200	3.7

Table A5 (cont'd)

36-36-28	2000	96000	7.2	1.1
	2050	76530	8.2	1.0
	1760	76530	10	1.0
	2400	64000	8.7	1.0
	1880	64000	7.3	1.1

10-43-47	1040	24000	17	1.9
	1000	19200	19	2.0
	774	17200	7	2.0
	790	22130	11	2.1
	935	16950	15	2.0

10-49-41	1080	24100	17	1.7
	1000	24100	20	2.0
	1160	24100	10	2.0
	1080	24100	6	1.8
	1000	24100	7	1.8

62-32-06	3130	121740	4.9	.7
	3125	142857	4	.7
	2727	127270	4	.7
	3036	142860	5.3	.7
	3005	133680	4.5	.7

Table A5 (cont'd)

55-33-12	2880	152540	4	.7
	2870	156520	4.5	.6
	2667	100000	4.5	.6
	2680	107145	5.5	.6
	2774	129050	5	.6

49-33-18	2609	139130	4.5	.7
	3180	127275	4.5	.7
	2632	140350	5.5	.7
	2710	118645	5	.8
	2810	135585	5.5	.7

40-54-06	2180	218180	1.6	.4
	2035	186440	2.6	.4
	2085	166670	1.8	.4
	1750	166670	1.2	.5
	2010	184490	1.8	.4

36-52-12	2145	125000	1.7	.5
	2087	104350	2.0	.5
	2120	118645	2.5	.5
	2500	133330	2.0	.5
	2210	120330	1.7	.5

Table A5 (cont'd)

32-50-18	3420	119660	2.0	.5
	3480	139130	3.0	.6
	3150	148150	2.0	.5
	2540	135590	3.0	.5
	3147	135630	2.0	.5

95-00-05	4000	92500	55	3.9
	4150	93000	60	3.4
	3995	92575	55	3.3
	4150	93000	75	3.7
	4000	92500	75	3.5

80-00-20	3636	90900	80	9.5
	3636	90900	92	9.4
	3636	90900	92	8.6
	3680	89000	105	9.7
	3680	89000	80	8.9

60-00-40	3180	36360	55	non-break
	3180	36360	85	non-break
	3270	36360	82	non-break
	3270	43630	70	non-break
	3180	43630	70	non-break

Table A5 (cont'd)

20-00-80	1100	2000	>200	non-break
	1150	2000	>200	non-break
	1200	2220	>200	non-break
	1200	2500	>200	non-break
	1150	3000	>200	non-break

APPENDIX B

CONVERSION OF WEIGHT CONTENT TO VOLUME FRACTION

APPENDIX - B

CONVERTING WEIGHT FRACTION TO VOLUME FRACTION

Fiber content of a composite is usually given as volume fraction rather than weight fraction. To convert weight content of fibers to volume fraction, the following method was used:

A Density Gradient Column was used to determine the density of the composites containing known weights of wood-fibers. The column was made of a mixture of toluene and carbon tetra-chloride. Calibrated glass beads were used to determine the density at different heights in the column (Table B1). A calibration graph of density of glass beads vs. height was plotted (fig. B1).

Specimens measuring 0.2 in. by 0.2 in. were dropped into the column and the height to which they sank was noted (Table B2). Based on the calibration graph, the density of the composite can be determined.

From this the volume fraction of wood-fiber content of composite could be determined based on the equation:

$$\phi_z = 1 - \frac{(1 - W_z)\rho_c}{\rho_p}$$

where ϕ_z = volume fraction of fibers on composite.

W_z = weight fraction of fibers in composite.

ρ_c = density of composite.

ρ_p = density of polymer (0.96 g/cc).

This information was used to plot a graph of weight % fibers vs. volume fraction of fibers in composite (Fig. B3).

TABLE B1: Height to which beads of different Density sank.

Density of beads (g/cc)	Height
1.1	8
1.31	18
1.34	22
1.4	56

TABLE B2: Height to which composites of different fiber loading sank.

Weight % fibers	Height	Density of composite(g/cc)
18	76	0.96
30	65	1.039
40	55	1.09
59	38	1.198

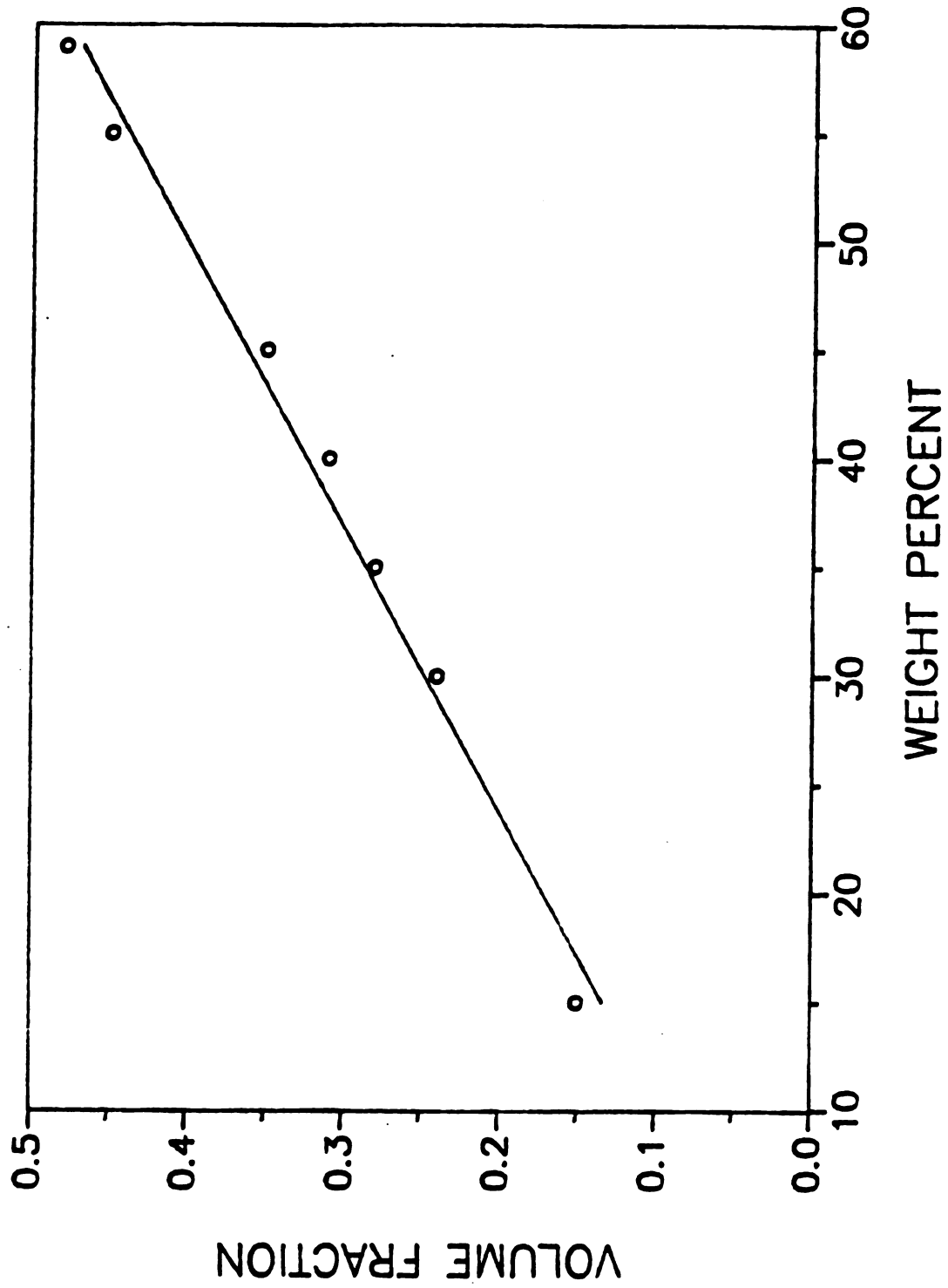


Fig. B1: Relationship between weight content and volume fraction of the wood-fiber HDPE composite

APPENDIX C

FIBER LENGTH BEFORE AND AFTER PROCESSING

APPENDIX-C

TABLE C1: Fiber length distribution before and after processing.

Length (cm.)	# before processing	# after processing
0.04	2	7
0.06	5	25
0.08	9	25
0.10	13	15
0.12	10	7
0.14	18	9
0.16	18	5
0.18	11	3
0.20	3	2
0.22	7	2
0.24	4	-

Average width : 0.0015 μ

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