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THE EFFECT OF SURFACE SULFONATION OF HIGH DENSITY POLYETHYLENE (HDPE) ON THE MECHANICAL PROPERTIES OF HDPE/WOOD FIBER COMPOSITES

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By

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ABSTRACT

THE EFFECT OF SURFACE SULFONATION OF HIGH DENSITY POLYETHYLENE (HDPE) ON THE MECHANICAL PROPERTIES OF HDPE/WOOD FIBER COMPOSITES

By

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The ability of surface sulfonation of high density polyethylene (HDPE) resins to enhance interfacial interaction between the HDPE and aspen hardwood fibers, with a concomitant increase in mechanical properties of the resultant composites, was evaluated. Both pelletized and powdered HDPE resin were sulfonated for different lengths of reaction time. The HDPE resins were compounded with 40% weight of the wood fibers in a twin-screw extruder. Maleic anhydride modified polypropylene (MAPP) was also investigated as a coupling agent for the composites. Composite samples were evaluated for tensile, flexural, and impact properties. The effect of a longer reaction time and an increased surface area was shown to result in increased levels of sulfonation. The results showed that sulfonation, at the achieved levels, had little or no effect on enhancing the mechanical properties of the HDPE/wood fiber composites. The inclusion of MAPP resulted in an increased interfacial adhesion between HDPE and wood fibers. The powdered HDPE showed an increased compatibility with wood fibers.

To my parents, Yoshiya and Teruko Haraguchi

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INTRODUCTION

Government and industry alike have been seeking methods of disposal, that will deal with the problem of plastics in the solid waste disposal system. In the past, polyethylene terephthalate (PET) was one of the few plastics that was actively sought for recycling. High density polyethylene (HDPE) generates a significantly greater amount of tonnage yearly than PET, therefore both are now being actively sought and recycled. HDPE is readily identified by consumers in the form of plastic milk jugs. In the State of Michigan, for example, over 12,000 tons of plastic milk jugs are discarded each year (Resource Integration Systems Ltd., 1987). HDPE is also used as packaging for household chemicals, bleach, detergent, and cosmetics. Barriers to the recovery of HDPE include contamination and health concerns. Recycled plastics are generally considered unsuitable for direct food content, due to fear of contaminants.

An advantage in recovering HDPE is that it is relatively easy to recycle compared with many other plastics. Products manufactured from recycled HDPE include: signs, toys, basecups for soft drink bottles, traffic barrier cones, pipe, and trash cans. Recycled HDPE resin has also been evaluated as a low cost matrix for structural polymer composites.

HDPE resin will be evaluated as a matrix material in the present investigation because of its low cost and abundance. HDPE by itself is limited in its use for structural applications, due to its low stiffness and high creep properties. However, if it is reinforced with a stiff and strong filler, these limitations may be overcome.

The type of filler used as a reinforcement is very important, since the ultimate properties of the composite are controlled by the properties and quantities of the component materials. The filler should provide maximum improvement of desired physical properties, and be inexpensive and readily available in controlled particle sizes, among other desired requirements.

The filler being investigated in this study is aspen hardwood fibers. Advantages of wood fiber include its low density, abundance, high strength-to-weight ratio, and low cost.

Previous studies which evaluated the mechanical properties of wood fiber/HDPE composites have shown very little improvement over filled but non-reinforced HDPE. This is not unexpected since wood fibers are polar and hydrophilic, while HDPE is non-polar and hydrophobic. The role of the matrix material or the continuous phase is to bind the fibers and protect them. Although limited bonding may occur as a result of physical entanglement across the interface between the continuous polymer phase and the discontinuous wood fiber phase, the extent of such bonding does not lead to an appreciable enhancement of the mechanical properties of the composites, as compared to non-reinforced HDPE. In the absence of a strong bond between the matrix and fibers, the two may separate. This type of failure is known as debonding.

With these factors in mind, many fibers and reinforcing agents are pre-treated before they are incorporated into a composite. A common pretreatment uses a coupling agent that acts as a bridge between the filler and matrix, thus creating a stronger bond between the two. Studies have shown that very small additions of a coupling agent are sufficient to promote good bonding and improve mechanical properties (Keal, 1990 and Childress, 1991).

To date, studies have focused on the inclusion of various modifiers with wood fiber/recycled plastic composites, and the effectiveness of the additives to improve the mechanical properties of the composites. The polymer matrices investigated include: (i) high density polyethylene; (ii) polypropylene; and (iii) recycled multi-layer polypropylene/adhesive/ ethylene-vinyl alcohol copolymer containers. The multi-component composite was found to have properties superior to those of a composite formed with polypropylene alone (Simpson, 1991). This was attributed to improved fiber adhesion, resulting from the polar functionality of the adhesive and ethylene-vinyl alcohol copolymer components. The inclusion of modifiers to high density polyethylene based composites was found to enhance the mechanical properties of the resultant composites by improving fiber/polymer matrix adhesion (Selke, et al., 1989; Childress, 1991). Two additives which showed promising results were maleic anhydride modified polypropylene, and ionomer modified polyethylene.

One of the major parameters governing the mechanical performance of composite materials is the interfacial adhesion between the reinforcing phase and the continuous matrix phase. It is generally accepted that adhesion between the reinforcing phase and the matrix phase in a composite material is dependent on the interfacial chemistry. However, while adhesion and the chemical bonding models of adhesion consider interfacial interactions, the exact nature of the chemistry and physics of adhesion, as they pertain to composite materials, is not fully understood.

Modification of the surface energy properties (dispersive and non-dispersive energies) of the HDPE matrix phase offers an opportunity to increase the strength of the adhesion between the reinforcing phase and matrix phase of the composite and enhance the mechanical performance of the composite material.

Sulfonation chemistry offers a new approach for chemically and structurally modifying the surface of polymers (Walles, 1989; Walles, 1973; Walles, 1971). Since the sulfonation process attaches the sulfonate groups along the polymer backbone, through a displacement reaction with hydrogen atoms, virtually any polymer except for fluorochloropolymers can be sulfonated. Further, the sulfonation process itself is not surface limited, i.e. the process can be extended under diffusion control below the surface to depths of a micron or more. Thus, modification of not only the surface but the surface region is possible. In principle, this process makes it possible to modify the surface of polymers, independent of their chemical composition, and can be applied to wood fiber/polymer composites resulting in enhanced interfacial adhesion between the fiber and polymer with a concomitant increase in mechanical properties.

In this study, surface sulfonation of HDPE will be carried out in order to determine its effect on the mechanical properties of wood fiber/surface sulfonated HDPE composites. Ammonium cation (NH_4^+) will be used to neutralize the sulfonated HDPE resins, which have been treated at different reaction times, to provide HDPE samples with various levels of sulfonation.

Success in this program will lead to the development of a method to modify the surface of polymers, independent of their chemical composition, and can be applied to co-mingled plastics resulting in enhanced compatibility.

The primary objectives of this study include: (i) To determine the density and distribution of sulfonate groups on HDPE following surface sulfonation.; (ii) To determine the effect of sulfonate group concentration and depth on the mechanical properties of wood fiber/surface sulfonated HDPE composites.

For this study, comparisons will be made between the sulfonated materials and controls formed using non-sulfonated resin with the same percentage of wood fiber.

LITERATURE REVIEW

1. Composite Materials

1.1 Introduction

Various types of materials such as metals, glass, and polymers, each processing specific physical, mechanical and barrier properties, have been utilized by modern industry. A supply of such materials is needed for the efficiency, comfort, and convenience of modern human life. Composite materials have also been researched and developed for over fifty years to meet the demands of society.

A composite material can be defined as any substance that is made by physically combining two or more existing materials to produce a multiphase system with different properties from the starting materials, but in which the constituents retain their identity (Richardson, 1977). For example, the basic constituents of a composite structure may consist of a polymeric matrix material and fibers. The matrix material is the continuous phase and the fibers are the reinforcing phase of the composite material. The fibers are embedded and surrounded by the matrix resulting in a higher, or enhanced mechanical strength to the composite. The tensile stress applied to the composite material can be transferred through the matrix to the fibers which accounts for the enhanced mechanical strength of the composite. Thus, the matrix phase typically exhibits lower tensile strength than the reinforcing fibers.

In addition to the two components, a continuous phase and the reinforcing fibers or discontinuous phase, the fiber-matrix interface is also a very important component of the resultant composite material. The mechanical properties of a composite are significantly affected by the structure and strength of the interface bonding in the composite material (Richardson, 1987). For example, the failure of the composite material may occur, due to the debonding of the fiber-matrix interface, with a corresponding weakening of the interfacial adhesion. In this case, the efficient transfer of stress between the matrix and fibers is not achieved. Surface-treatment of the respective components of the composite is often carried out to reduce the interfacial bonding problem and enhance interfacial adhesion.

There are three basic procedures to achieve enhanced interfacial adhesion between the fibers and matrix: (i) modification of the fibers, (ii) use of a coupling agent, and (iii) modification of the matrix (Krishnan and Narayan, 1992). With respect to the surface modification of the reinforcing fibers, the fibers can be coated with an additive which introduces suitable functional groups, resulting in the fiber surface being more compatible with that of the matrix (Krishnan and Narayan, 1992). A coupling agent, which acts as a bridge to promote adhesion between the fibers and matrix, may be added to the matrix when compounding the fibers. For hydrophobic, non-polar matrices such as polypropylene, or polyethylene, which exhibit incompatibility with hydrophilic polar fibers, modification of the continuous matrix phase can provide a means of introducing polar functionality to the characteristically hydrophobic polymer matrix. The polar nature of the polymers can provide a means of non-covalent interaction with the hydrophilic fiber surface.

1.2 Prediction of Properties

The mechanical properties of a composite material depend on the properties of its constituents, their distribution, and physical and chemical interactions (Agarwal and Broutman, 1980). The mechanical properties include the modulus, tensile strength, and impact strength. In order to determine these properties, theoretical estimation is considered an efficient measurement, while experimental measurement is not so due to its cost limitation, time requirements, and difficulty.

The modulus of a composite is the easiest property to estimate since it is a bulk property, which depends primarily on the geometry, modulus, particle size distribution, and concentration of the fibers (Bigg, 1987). The modulus of a continuous fiber composite material, E_c, can be predicted by the rule of mixtures as (Agarwal and Broutman, 1980):

$$E_c = E_f V_f + E_m V_m \qquad (1)$$

where: E_f, E_m = the modulus of the fibers and matrix phase, respectively

V_f, V_m = the volume fraction of the fibers and matrix phase, respectively

The average tensile stress applied to the continuous fiber composite materials, σ_c, can also be estimated (Agarwal and Broutman, 1980) by using the simple rule of mixture as:

$$\sigma_c = \sigma_f V_f + \sigma_m V_m \tag{2}$$

where: σ_f, σ_m = stress of the fibers and matrix phase, respectively

The rule of mixtures indicates that the load on the composite material will be distributed over the fibers and the matrix material according to the ratio of the volume of the fiber and matrix phase and their respective modulus or tensile strength. Moreover, the shared load between the constituents of the composite material will be expressed as:

$$\frac{P_f}{P_m} = \frac{E_f}{E_m} \frac{V_f}{V_m} \tag{3}$$

where: P_f, P_m = the loads carried by the fibers and matrix, respectively (Agarwal and Broutman, 1980)

Equation (3) indicates that a high ratio of the modulus between the constituents will result in the fibers carrying a higher proportion of the load, even with limited volume of fibers (Schliekelmann, 1982).

Usually, the loads on the composite materials are directly applied to the matrix and then transferred to the fibers through the fiber ends and also through the cylindrical surface of the fiber near the ends (Agarwal and Broutman, 1980).

The continuous fiber composite materials, as described above, are unidirectional composite materials that include similar fiber orientation, which result in the composite having greater tensile strength and a higher modulus in the direction of the fiber axis than in the transverse direction. In contrast, short-fiber (discontinuous fiber) composite materials have an advantageous property in that the stress on the composite is approximately equal in all directions. In addition, composites utilizing short fibers, which can be easily molded by injection or compression molding, are economical and can produce generally isotropic composite materials (Agarwal and Broutman, 1980).

The mechanical properties of the short-fiber composite materials were found to be a function of the fiber length. However, for continuous fiber composite materials, where the length of fiber is much greater, stress transfer is not related to the end effects. The length of the fiber which determines the ultimate strength of the fiber, is called the critical length (Holister and Thomas, 1966). The critical length, L_c, is obtained from the following equation (Agarwal and Broutman, 1980):

$$\frac{L_c}{d} = \frac{\sigma_{fu}}{2\tau_y} \qquad (4)$$

where: σ_{fu} = ultimate strength

d = fiber diameter

τ_y = shear stress

The shear stress can be either the shear strength of the matrix or the fiber-matrix interfacial shear strength, whichever is smaller (Schliekelmann, 1982).

In order to estimate the average ultimate strength of short-fiber composite materials, σ_{cu}, the following equations can be applied, which include two regions related to the fiber and critical length (Agarwal and Broutman, 1980):

$$\sigma_{cu} = \frac{\tau_y L}{d} V_f + \sigma_{mu} V_\pi \qquad (L < L_c) \qquad (5)$$

$$\sigma_{cu} = \sigma_{fu} \left(1 - \frac{L_c}{2L}\right) V_f + (\sigma_m)_{\varepsilon_f} V_m \qquad (L > L_c) \qquad (6)$$

where: L = length of fiber

σ_{mu} = ultimate stress of matrix

$(\sigma_m)_{\varepsilon_f}$ = stress of matrix at the fiber fracture strain ε_f

When the length of fibers is smaller than the critical length (L_c), the maximum fiber stress is less than the average strength of fibers, so that the fibers will not fracture (Agarwal and Broutman, 1980). In this case, therefore, failure of the composite material will occur as a result of failure of the matrix or fiber-matrix interface. When the length of the fiber is greater than the critical length, the fibers can be stressed to their average strength and fracture at the point when the maximum fiber stress reaches the ultimate strength of the fibers (Agarwal and Broutman, 1980).

The short-fiber composite materials involve randomly oriented fibers and promote basically an isotropic property. Thus, to predict the modulus of short-fiber composite materials, the longitudinal and transverse modulus in the materials should be considered. The following equation predicts the modulus of the short-fiber composite materials, E_{random} (Agarwal and Broutman, 1980):

$$E_{random} = \frac{3}{8}E_L + \frac{5}{8}E_T \qquad (7)$$

where: E_L, E_T = longitudinal and transverse modulus

There are no viable theoretical relationships between filler characteristics and concentration that can be used to predict the impact strength of reinforced polymers (Bigg, 1987). The impact strength of composite materials depends very strongly on the test procedure as well as other factors such as the rate of impact, shape of the impacting implement, existence of microdefects in the vicinity of the impact, fiber orientation, and interfacial adhesion (Bigg et al., 1988).

2. Sulfonation Process

2.1 Introduction

Sulfonation is useful technique to provide modification of a polymer surface. To accomplish the sulfonation of polymers such as polyethylene(PE), polypropylene(PP), and polystyrene(PS), the surface of polymers is treated with gaseous SO_3, fuming sulfuric acid, or SO_3 in chlorinated hydrocarbons (Ihata, 1988). Virtually any polymer except for fluorochloropolymers can be sulfonated because the sulfonation process attaches the sulfonate groups along the polymer backbone through a displacement reaction with hydrogen atoms. Olsen and Osteraas (1969) proposed from the infrared spectra of surface sulfonated PE films, that the process involved insertion of atomic sulfur into the carbon-hydrogen bonds of the PE surface. This results in the presence of sulfonic acid groups on the polymer surface.

Sulfonation of polymer surfaces has been applied to improve their barrier and physical properties. Sulfonated polymers contain a structurally modified surface layer due to the presence of sulfonate groups. The chemical nature of this thin layer results in an enhancement of surface properties of polymers. For example, Walles (1989) indicated an increase in gasoline vapor barrier properties of in-mold sulfonated high density polyethylene (HDPE) automotive gas tanks, with about 20% SO_3 in air followed by neutralization with NH_3 gas, as compared to untreated HDPE gas tanks. In addition, the exposure of PE to the sulfuric acid atmosphere substantially improved the hardness, surface tension, and conductivity of the thin sulfonated surface (Fonseca et al., 1985).

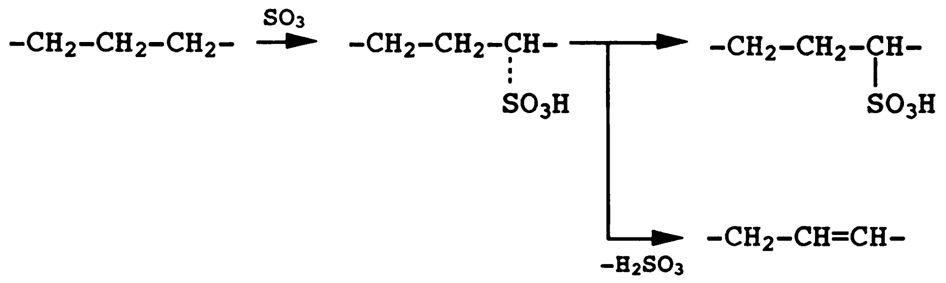
Surface sulfonation of polymers is usually carried out to enhance the hydrophilic (e.g. ion-exchange resin from styrene-divinylbenzene copolymers) or water soluble nature of the matrix (e.g. sulfonated PS) (Planche et al., 1988). The sulfonic acid groups attached to the polymer backbone account for the polar or hydrophilic nature of the surface sulfonated polymers.

Kinetic studies predicted that the sulfonation process is dependent on the concentration of the SO_3 species and time of exposure of the polymer to the SO_3 gas (Walles, 1989). The thickness of the thin surface layer that includes sulfonate groups can be extended under diffusion control below the matrix surface to depths of a micron or more. Thus the sulfonation process itself is not surface limited, and it is possible to modify not only the surface but the surface region of the polymers. The surface of polymer resins that are formed as pellets or powders may also be treated by the sulfonation process.

2.2 Reaction of polyethylene with SO_3

The behavior of SO_3 in chemical reactions shows that the sulfur atom is strongly electron-deficient while the oxygen agents are electron-rich (Gilbert, 1965). The nature of SO_3 leads to its activity as a sulfonating agent.

Ihata (1988) described the reaction of gaseous SO_3 with PE film, which was evaluated by spectrophotometric analyses including infrared (IR), ultraviolet (UV-VIS), and resonance Raman spectroscopy. The reaction which yields sulfonated PE is initiated by the abstraction of a hydrogen atom of the PE backbone by SO_3, as shown in Figure 1. This reaction then proceeds by further reactions which include: (i) SO_3 can react with a carbon atom of the PE backbone resulting in the sulfonic acid group being covalently bound to the PE surface; (ii) the sulfonated membrane can undergo elimination of two hydrogen atoms, resulting in an unsaturated bond.



$$-CH_2-CH_2-CH_2- \xrightarrow{SO_3} -CH_2-CH_2-\underset{\vdots\atop SO_3H}{CH}- \longrightarrow -CH_2-CH_2-\underset{|\atop SO_3H}{CH}- \quad (8)$$

$$\xrightarrow[-H_2SO_3]{} -CH_2-CH=CH- \quad (9)$$

Figure 1. Reaction of PE film with SO_3 (Ihata, 1988)

The sulfonic acid groups are then easily neutralized with NH_3. The neutralization results in the presence of $-C-SO_3^-NH_4^+$ on the PE backbone. Also, the barrier and physical properties of sulfonated polymers are strongly dependent upon the counterion utilized. Walles (1989) evaluated 27 common metal cations, in exchange for the NH_4^+ ion, for sulfonated PE gas tanks. The results showed that the best barrier properties were obtained with Li^+, Na^+, Cu^{++}, Mg^{++}, Sr^{++}, V^{++}, Mn^{++}, Co^{++}, and Ni^{++}. Meanwhile, PE film with 20 % $SO_3^-Li^+$ by weight, indicated good physical properties.

3. Prior Research

Raj et al. (1990) evaluated the influence of surface modification on the mechanical properties of three different cellulosic fiber/high density polyethylene (HDPE) composites. Chemithermomechanical pulp (CTMP) of aspen, wood flour, and cellulose fiber were respectively modified by polymethylenepolyphenyl isocyanate (PMPPIC) and vinyltri(2-methoxyethoxy)silane (Silane A-172). The composites prepared from pretreated fibers exhibited significantly improved mechanical properties as compared to those prepared with untreated fibers. The composite prepared with untreated fibers showed an increase in the tensile modulus, while the tensile strength, elongation, and impact strength were decreased when the respective properties were compared to these obtained for the unfilled HDPE. Raj et al. also concluded that as fillers, wood fibers were more cost-effective than glass fibers and mica, based on their respective cost and performance.

Krishnan and Narayan (1992) investigated the effects of modified polypropylene (PP)/low density hardwood residue blended with ground pecan shell (LDHW) composites. Two different modification processes were studied: (i) a two-step process in which maleated PP (MAPP) was compounded with LDHW, using catalyst of 0.05-0.1 % by weight, and (ii) a single step process where the PP, maleic anhydride, dicumylperoxide, LDHW, and catalyst were all combined. The levels of LDHW studied were 20 % and 30 % for both processes. Modification of PP by both processes resulted in improved mechanical properties, compared to unmodified PP/LDHW composites. Increase in content of the lignocellulosics significantly improved the tensile strength of the modified composites.

Raj et al. (1988) used a series of isocyanates as bonding agents to improve the tensile properties of wood fiber/linear low density polyethylene (LLPE) or high density polyethylene (HDPE) composites. The isocyanates investigated were: toluene-2-4-diisocyanate, 1-6 hexamethylene-diisocyanate, polymethylene(polyphenyl isocyanate), and ethyl isocyanate. The combination of isocyanates with the wood fiber/polyethylene composites significantly improved their tensile properties. HDPE based composites exhibited better mechanical properties than the LLDPE based composites.

Chtourou et al. (1992) evaluated the tensile properties of recycled polyolefin/chemicothermomechanical pulp fiber composites. The recycled polyolefins were comprised of 95% polyethylene (PE) and 5% PP. The surface of the pulp fibers was treated with acetic anhydride and phenol-formaldehyde. Tensile properties of the composites were also evaluated before and after storage in water. The addition of non-treated fibers enhanced the strength and toughness of the pure recycled polyolefins. Further, the composites fabricated with treated fibers showed lower water sorption as a function of storage time, and retained greater tensile properties, as compared to those of non-treated fibers.

Maldas and Kokta (1990) investigated the effects of coating treatments on mechanical properties of chemithermomechanical pulp (CTMP)/thermoplastic composites. The thermoplastics studied were high impact polystyrene (PS 525), high heat crystal polystyrene (PS 201), and polyvinyl chloride (PVC). The fibers were coated with polymer/silicate and isocyanate. The composites prepared from coated fibers showed an increase in the mechanical properties, as compared to both the individual polymers and composites fabricated with non-treated fibers. Composites of PS 525 exhibited a greater enhancement in mechanical properties than composites fabricated with PS 201.

Gogoi (1989) determined the mechanical properties of wood fiber/recycled HDPE composites, compounded by using a twin-screw extruder. The effects of fiber pretreatment, as well as screw configuration and temperature of the extruder were evaluated. The composites with acetylated and non-treated fibers indicated greater tensile and flexural yield strength than those with heat treated fibers. The screw configuration which had low conveying ability resulted in the best overall strength of the composites.

Simpson (1991) investigated the mechanical properties of aspen hardwood fiber/virgin polypropylene (PP) and aspen hardwood fiber/recycled PP based matrix. The reground multilayer squeezable ketchup bottle resin, which consists of PP, ethylene vinyl alcohol, and adhesive, was used as the recycled matrix phase. Aspen wood fiber of 30 %, 40 %, and 50 % by weight was incorporated into the matrix. The recycled resin/wood fiber composite exhibited greater mechanical properties than the virgin PP resin/wood fiber composite. An increase in the content of wood fibers improved the impact strength and flexural modulus of the composite, while the optimum tensile strength was found at the lowest content of wood fibers.

Childress (1991) studied the effects of the inclusion of additives in aspen hardwood fiber/recycled HDPE composites. Ionomer modified polyethylene (Surlyn), maleic anhydride modified polypropylene (MAPP), and two low molecular weight polypropylenes (Proflow 1000 and Proflow 3000) were respectively combined with the composites. The effects of Surlyn and MAPP were investigated at 1 %, 3 %, and 5 % by weight. Tensile properties increased with the addition of MAPP at all levels, with the highest increases in the tensile properties being observed at the 5 % by weight loading level.

There was no significant improvement in impact strength, using any of the above additives.

MATERIALS AND METHODS

1. Materials

1.1 Matrix

High density polyethylene (HDPE) was used as a matrix material for all composites. Both pelletized and powdered HDPE resins were evaluated in this study. The pelletized HDPE was supplied by Tredegar Film Products (Terre Haute, Indiana). The melting point and % crystallinity of each resin sample was determined by Differential Scanning Calorimetry (DSC) and the results are as follows: 136°C and 61.1 % for pelletized HDPE, and 136°C and 70.8 % for powdered HDPE, respectively. (See Tables 10 and 11, Appendix A)

1.2 Filler

As a reinforcing filler, aspen hardwood fibers were used for all composites. They were in the form of thermomechanical pulp (TMP) and supplied by Lionite Hardbord (Phillips, Wisconsin). Wood consists of three components: 40-50 % of cellulose, 20-35% of hemicellulose, and 15-35% of lignin (Wagenfuehr, 1984). The cellulose fraction has a high crystallinity, 50-83%, which contributes mainly to the strength of the wood. The average dimensions of aspen hardwood fibers are as follows: length of 1.04 mm and diameter of 0.01-0.027 mm. (Browning, 1968) The wood fibers are also hydrophilic and polar in nature. The structure of the cellulose fraction of the wood fiber is shown in Figure 2. The wood fibers were conditioned for at least 48 hours at 23°C and 50% RH before mixing with the polymer

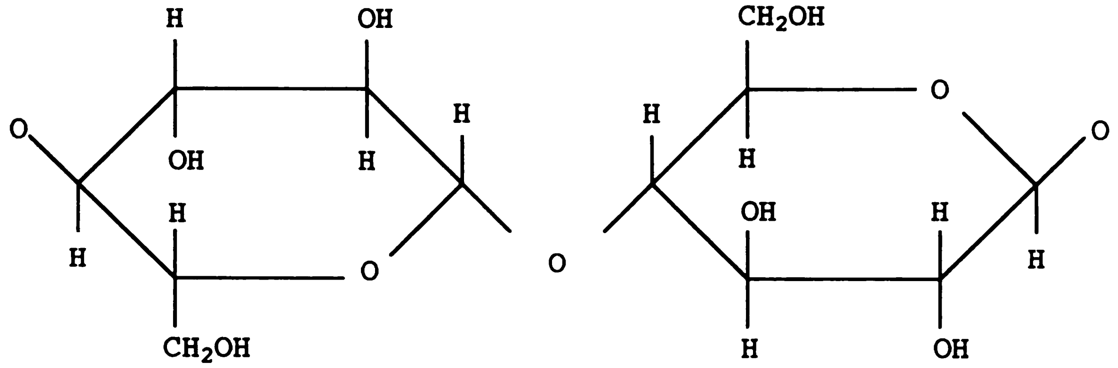


Figure 2. Molecular Structure of Cellulose

1.3 Coupling Agent

Maleic Anhydride Modified Polypropylene (MAPP) as a coupling agent was supplied by HIMONT Advanced Materials (Lansing, Michigan). MAPP was used to increase interfacial adhesion between the wood fibers and polymer. This is based on its ability to form a bonding network between bond both the wood fibers and HDPE. Figure 3 illustrates the structure of MAPP.

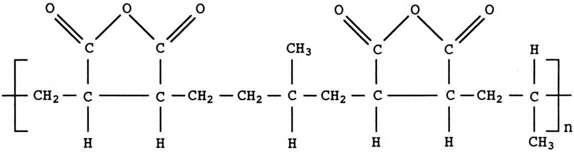


Figure 3. Molecular Structure of MAPP

2. Methods

2.1 Sulfonation

The surface sulfonation process for both the pelletized and powdered HDPE resins was carried out in the Solid Phase Sulfonator Reactor at the Composite Materials and Structures Center, Michigan State University. Figure 4 shows a schematic diagram of the sulfonation system. The procedure was based on the standard method for the sulfonation of polymer films, which has been described in detail by Ericson (1993).

Approximately 1500 grams of the respective HDPE resins were first dried for a period of 5 hours at a temperature of 60°C in a drying oven. The resin samples were then cooled to room temperature (23°C), and stored in a dessicator over calcium chloride. The dried resin (1500 gm) was placed into a rotating drum reactor that was connected to the SO_3 generator.

A nitrogen purge/vacuum/nitrogen purge cycle was used to reduce any active gas and water vapor in the reactor.

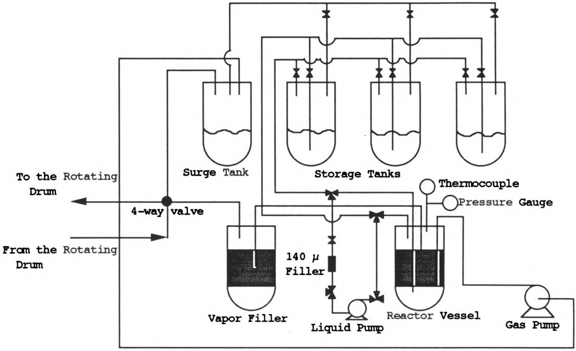


Figure 4. Schematic Diagram of Sulfonation System

The sulfonator lines and reactor were first flushed with nitrogen gas at 32 l/min for 10 minutes. The resins were then exposed to SO_3 gas (1% V/V), which was continuously circulated from the SO_3 generator to the reactor, for varying periods of time: 2 and 4 minutes for the pelletized HDPE resin; and 3 minutes for the powdered HDPE resin, respectively. After the exposure time, the system was flushed with nitrogen gas at 32 l/min for 10 minutes to remove the excessive SO_3 from the drum reactor. The sulfonated materials were then exposed to 99.99 % NH_3 gas (V/V) for approximately 30 seconds to neutralize the sulfonic acid groups. After the sulfonation and neutralization processes, the resin samples were washed with double-deionized water and dried for approximately 10 hours at a temperature of 50°C.

Elemental analysis was performed by Galbraith Laboratories, Inc. (Knoxville, Tennessee) to determine the sulfur content of the respective sulfonated HDPE resins.

2.2 Compounding

To achieve compounding of the polymer and wood fibers, a Baker-Perkins Model MPC/V-30 PE, 38 mm, 13:1 co-rotating twin-screw extruder (Baker-Perkins, Saginaw, Michigan) was used. A schematic of the extruder is presented in Figure 5. As shown, the extruder has three heating zones, and a die which is connected to the end of heating zone 3 to form the exiting material. The die was also heated. Their heating temperatures were maintained by circulating water. As shown in Figure 5, a hopper to feed the polymeric material was located at zone 1. The other feeding port, which was used to add wood fibers, was located at zone 2, as shown. The operating parameters of the extruder were as follows: heating temperature, 150°C; compounder speed, 100 rpm's; compounder % load, 105; feed rate, 3; discharge pressure, 900; discharge temperature, 150°C.

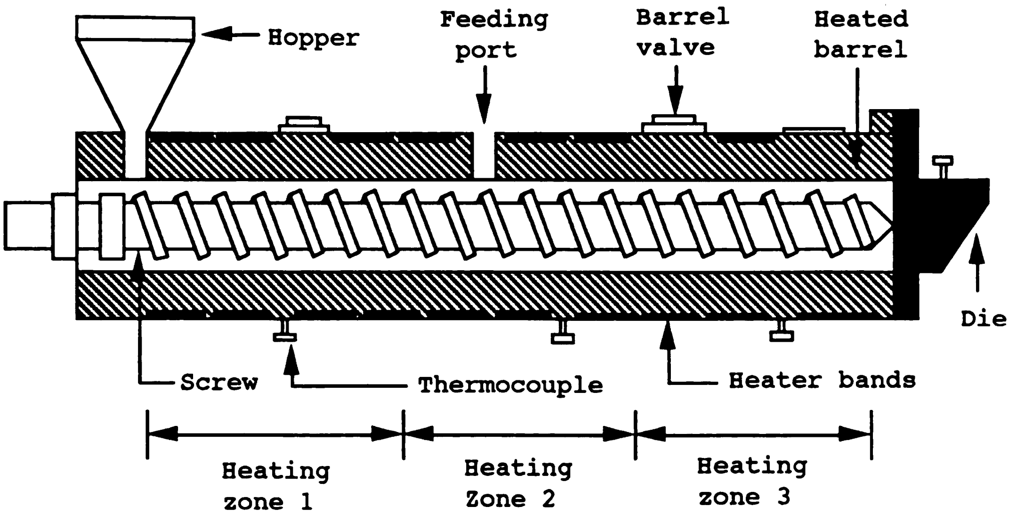


Figure 5. Schematic Cross Section of Extruder

Polymer resin was placed in the automatic feeder and fed into zone 1 through the hopper. For fabricating the composites which included the addition of MAPP, the HDPE was mixed with MAPP and then placed in the feeder. When the melted polymeric material reached zone 2 of the extruder barrel, wood fibers were carefully added by hand at a constant feeding rate. Before each extrusion process, to achieve an accurate mixing ratio of polymer to wood fibers by weight, the specific feed rate of the automatic feeder was first determined. Resin samples exiting through the automatic feeder during a two minutes period were collected. This procedure was repeated to give five replicate values which were then weighed to provide the specific feed rate, as expressed in gm/min. The range of the specific feed rate values of the automatic feeder was 4.1 to 6.7 gm/min. The feed rate for the fibers (gm/min) was then calculated, with regard to the % weight of wood fibers (40%) for all composites achieved. The range of the feed rate values for fibers was 2.7 to 4.5 gm/min. The compounded materials exited through the die were cut into bars of approximately 10 cm in length. The weight of each sample was approximately 24 grams.

A total of eight runs of the extrusion process was carried out to obtain a series of HDPE/wood fiber composites. Table 1 summarizes the composition by % weight of the respective composites fabricated.

Table 1. Composite Composition by % weight

Composite No.	**Composition (% weight)**
C1	60% pelletized HDPE/40% wood fiber
C2	60% sulf.(2min)pelletized HDPE/40% wood fiber
C3	60% sulf.(4min)pelletized HDPE/40% wood fiber
C4	60% powdered HDPE/40% wood fiber
C5	60% sulf.(3min)powdered HDPE/40% wood fiber
C6	55% pelletized HDPE/5% MAPP/40% wood fiber
C7	55% sulf.(2min)pelletized HDPE/5% MAPP/40% wood fiber
C8	55% sulf.(4min)pelletized HDPE/5% MAPP/40% wood fiber

2.3 Compression Molding

The compounded materials were compression molded by using a Carver Model M 25 ton laboratory press compression molding machine.

To form a sheet, three samples of the compounded materials were placed into a frame that was covered by metal plates. The specific frame size employed was dependent on the type of test to be performed on the fabricated composite sheet. The specific frame dimensions were as follows: 15 x 15 x 0.25 cm for the tensile test; and 12.7 x 12.7 x 0.3175 cm for the flexural and impact tests. A plastic film (Polyethylene terephthalate) was placed between the frame and metal plates to reduce any sticking to the surfaces of the frame and heated metal plates. The compounded material was heated at a temperature of 150°C for 10 minutes under a pressure of 30,000 psi, and then cooled down to a temperature of 60°C by circulating cold water, for approximately 7 minutes, through the press.

2.4 Tensile Test

Tensile strength at break, percent elongation at break, and modulus of elasticity were determined by an Instron Universal Tensile Tester (United Model SFM test system, United Calibration Corporation, Huntington Beach, California) at room conditions (23°C, 50% RH). The test procedure was based on ASTM Standard D638-87b, Standard Test Method for Tensile Properties of Plastics (ASTM, 1988). Dumbbell-shaped Type I specimens were produced by using a Tensilkut Model 10-13 specimen cutter (Tensilkut Engineering, Danbury, Connecticut). The dimensions of the specimens were: total length, 150 mm; width, 20 mm; thickness, 2.5 mm; width of narrow section, 10 mm. The test conditions of the Instron Universal Tester system were: full scale load, 1000 lbs; crosshead speed, 0.21 in./min. for the composites and 2 in/min for pure polymers, respectively; grip separation, 3.5 in.

The respective values of the tensile properties were automatically calculated by a computer system using the following equations:

Tensile strength at break, σ

$$\sigma = \frac{W}{A_0} \qquad (8)$$

where: W = maximum load

A_o = original cross-sectional area

Percent elongation at break, %El

$$\%El = \left[\frac{(L - L_0)}{L_0}\right] \times 100$$

$$= \varepsilon \times 100 \qquad (9)$$

where: L = extended grip separation at break

L_o = original grip separation

ε = strain

Modulus of elasticity E

$$E = \frac{\sigma_d}{\varepsilon_d} \tag{10}$$

where: σ_d = difference in stress corresponding to any segment of section on initial straight line portion of stress-strain curve

ε_d = difference in strain corresponding to any segment of section on initial straight line portion of stress-strain curve

2.5 Flexural Test

Flexural yield strength and flexural modulus were determined by an Instron Universal Tensile Tester (United Model SFM test system, United Calibration Corporation, California) at room conditions (23°C, 50% RH). The test procedure was based on ASTM Standard D790-86, Standard Test Method for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials (ASTM, 1986). Method I, which sets a loading nose and two supports to the specimen, was used. For the test specimens, the molded sheets were cut into samples with a length of 127 mm and a width of 12.7 mm. The following parameters were set on the Instron Universal Tester system: load cell, 20 or 1000 lbs; crosshead speed, 0.08 in/min; support span, 2.0 in.

The respective values of the properties were automatically calculated by the computer system, using the following equations:

Flexural yield strength, S

$$S = \frac{3\ P\ L}{2\ b\ d^2} \tag{11}$$

where: P = load at a point, which the load does not increase with an increase in deflection

L = support span

b = width of beam tested

d = depth of beam tested

Flexural modulus, E_B

$$E_B = \frac{L^3\ m}{4\ b\ d^3} \tag{12}$$

where: m = slope of the tangent to the initial straight line portion of the load-deflection curve

2.6 Impact Test

Izod impact strength was determined by a TMI 43-1 Izod Impact Tester (Testing Machines, Inc., Amityville, New York) at room conditions (23°C, 50% RH). The test procedure was based on ASTM standard D256-87, Standard Test Method for Impact Resistance of Plastics and Electrical Insulating Materials (ASTM, 1988). The molded sheets were cut into test specimens with a length of 63.5 mm and a width of 10 mm. The specimens were then notched using a TMI Notching Cutter, where the angle of the notch was 45 degrees with a radius of curvature at the apex of 0.25 mm. The pendulum load used was 5 ft-lbs. The impact strength was automatically calculated using the system of the tester, which was calibrated by the manufacture.

2.7 Density Measurement

Density of specimens was determined for all composites. The dimensions, which included length, width, and thickness, of un-notched specimens that were prepared for the impact test were measured using a micrometer to get the volume of the respective specimens. The test specimens were also accurately weighed, and the weight was divided by the volume, to obtain density values, as expressed in gm/cm^3.

2.8 Statistical Analysis

SPSS/PC+ statistical program (version 4.0, Michigan State University, 1990) was used to perform statistical analysis of test data for tensile strength, modulus of elasticity, percent elongation at break, flexural yield strength, flexural modulus, impact strength, and density measurement. The following statistical analysis procedures were performed: (i) a one way analysis of variance of mechanical properties and density measurement values for composites; (ii) a one way analysis of variance of mechanical properties values for matrix materials; and (iii) a one way analysis of variance of mechanical property values for composites with lengthwise fiber direction vs. crosswise fiber direction.

RESULTS AND DISCUSSION

1. Sulfonation of HDPE

Table 2 summarizes the results of elemental analysis performed on both sulfonated pelletized and powdered HDPE. As shown, very low levels of sulfonation were achieved for the respective HDPE samples, under the reaction conditions employed, which were ambient temperature and 1% SO_3 (V/V).

Table 2. Sulfur Concentration of Sulfonated HDPE

Type of HDPE	Reaction Time (min.)	Sulfur Concentration (%Sulfur(wt/wt))
Pelletized	2	0.0012
Pelletized	4	0.0034
Powdered	3	0.05

The presence of the low concentration levels of sulfur in the HDPE resins following reaction with SO_3, indicates a lack of reactivity of the surface region of the polymer resins to the sulfonation process. In the sulfonation process, SO_3 reacts with a carbon atom on the HDPE backbone and is neutralized with NH_3 gas. This results in the sulfonic acid groups being covalently bonded to the HDPE surface.

Comparing the two different reaction times with gaseous SO_3 in the sulfonation process, the 4 min. sulfonated pelletized HDPE exhibited approximately a three times higher sulfur concentration than the 2 min. sulfonated pelletized HDPE. These findings confirmed that a longer reaction time resulted in the sulfonated HDPE having a higher sulfur content.

In comparing sulfonation of the pelletized and powdered HDPE resins, the 3 min. powdered HDPE had approximately fifteen times higher sulfur content than the 4 min. sulfonated pelletized HDPE, despite the shorter reaction time. The greater surface area of the powdered HDPE resin, as compared to the pelletized resin, could account for the enhanced level of sulfonation achieved.

The effect of a longer reaction time and an increased surface area has been shown to result in increased levels of sulfonation. However, the extent of sulfonation achieved was quite low and, as described in the following sections, did not modify the dispersive and polar characteristics of the polymer to a level which would result in enhanced interfacial interaction between the HDPE and wood fibers, with a concomitant increase in mechanical properties.

2. Density Measurement of Composites

Ten replicate specimens of the respective composites were measured to determine their density values. Table 3 lists the mean density values for each composite, in units of gm/cm^3. There were no significant differences between the means of the respective composites, based on statistical analysis, as shown in Table 19, Appendix C. This result supports the achievement of uniformly compounded experimental materials for all composites.

Table 3. Results of Density Measurement of Composites

Composite No.	Composite (% weight)	Density (gm/cm^3) Mean	SD
C1.	60% Pel.HDPE/40% Fiber	1.06	0.01
C2.	60% Sulf.(2min)Pel.HDPE/40% Fiber	1.06	0.02
C3.	60% Sulf.(4min)Pel.HDPE/40% Fiber	1.05	0.02
C4.	60% Powd.HDPE/40% Fiber	1.08	0.01
C5.	60% Sulf.(3min)Powd.HDPE/40% Fiber	1.08	0.02
C6.	55% Pel.HDPE/5% MAPP/40% Fiber	1.06	0.03
C7.	55% Sulf.(2min)Pel.HDPE/5% MAPP/40% Fiber	1.07	0.01
C8.	55% Sulf.(4min)Pel.HDPE/5% MAPP/40% Fiber	1.06	0.04

3. Tensile Properties

The mean of 10 to 14 replicate samples of the respective composite materials was determined for tensile properties, which included both the lengthwise and crosswise fiber directions. The mean of 10 samples of the respective polymer matrices was also evaluated. The results of tensile property tests are summarized in Tables 4 to 6, where the respective tensile strength, modulus of elasticity, and percent elongation values are presented. The effect of sulfonation as well as the effect of the coupling agent (MAPP) on the mechanical properties of the respective composites is shown by the histograms presented in Figures 6 to 8. A one-way analysis of variance of tensile properties values was performed for the respective composites and matrix materials, to determine any significant difference between the means, at an alpha level of 0.05. The results of the statistical analysis are shown in Tables 20 to 25, 32 to 34, and 38, Appendix C.

3.1 Influence of fiber direction

For the composites tested, with the exception of the modulus of elasticity of the composites, C1, C6, C7, and C8, the means of the tensile properties in the lengthwise fiber direction showed higher values than those in the crosswise fiber direction. This may be due to orientation of fibers, where the loads on the composite were applied to the polymer and transferred to the fibers through the fiber ends more effectively in the direction of the fiber axis than in the transverse direction. However, theoretically, discontinuous fiber composites, as tested in this study, have the property that the stress on the composite is approximately equal in all directions (See literature review, 1.2). In the present study, the observed decrease in the tensile properties in the crosswise fiber direction may be due to the interfacial failure between the extruded bar during the process of compression molding, where three pieces of extruded bar were molded together to form a sheet for specimen sampling.

3.2 Influence of Sulfonated HDPE

In comparison of tensile properties of the non-sulfonated HDPE composite and sulfonated HDPE composite, the following pairs were evaluated: (i) C1 to C2 and C3; (ii) C4 to C5; and (iii) C6 to C7 and C8, respectively. For the respective pairs compared, the following significant differences between tensile properties were observed, which were based on statistical analysis: (i) an increase in tensile strength of C3 in crosswise fiber direction; (ii) a decrease in tensile strength of C3 in lengthwise fiber direction and of C5, C7, C8 in crosswise fiber direction; (iii) an increase in modulus of elasticity of C3 in lengthwise fiber direction and of C8 in both lengthwise and crosswise fiber direction; (iv) a decrease in modulus of elasticity of C2 in crosswise fiber direction; (v) an increase in percent elongation at break of C2 in crosswise direction; and (vi) a decrease in percent elongation at break of C3 in lengthwise fiber direction. The differences observed seemed to be of little or no practical value and not enough to find utility in the effect of sulfonation on the resultant composites. Thus, sulfonation at the achieved levels had little or no effect on enhancing the tensile properties of the respective composite. These results can be attributed to the low levels of sulfonation achieved and the resultant low level of enhanced interfacial

interaction between the HDPE and wood fibers.

3.3 Influence of a coupling agent

The composite fabricated with MAPP as a coupling agent, C6, showed a statistically significant increase in tensile strength and modulus of elasticity, as compared to the untreated composite, C1. This is shown graphically in Figures 6 and 7. A similar finding had been reported earlier by Childress (1991). A coupling agent acts as a bridge to promote interfacial adhesion between the fibers and matrix (Richardson, 1977). For the composite, C6, the stress applied on the composite was more effectively transmitted through the matrix to fibers, resulting in the higher tensile strength and modulus of elasticity. In contrast, the composite, C6, had a significant decrease in the percent elongation at break, as compared to the composite, C1 (See Figure 7). Miles and Rostami (1992) proposed that an increase in interfacial bonding resulted in a corresponding increase in the tensile strength, but a decrease in the elongation at break, due to the reduction of the toughness, for glass-filled polypropylene. This finding suggests that the inclusion of MAPP resulted in the composite having lower flexibility and toughness, as well as an increase in its brittleness. Conversely, the composite had a higher stiffness, as estimated by the tensile strength and modulus of elasticity.

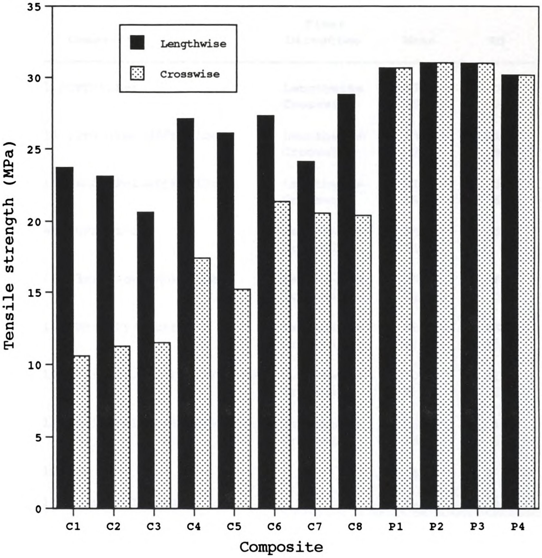
3.4 Influence of Powdered HDPE

The composite fabricated with powdered HDPE, C4, showed a statistically significant increase in the tensile strength and modulus of elasticity, as compared to that with pelletized HDPE, C1, as shown in Figures 6 and 7, respectively. These findings suggest that powdered HDPE may have a higher degree of compatibility with wood fibers, than pelletized HDPE resin. This can account for the observed enhanced tensile strength and modulus of elasticity values for the powdered HDPE based composites. The effective compatibility might be achieved in the compounding process of the wood fibers and powdered HDPE, where the fibers were uniformly dispersed throughout the matrix. The powdered HDPE seemed to have a more constant filling ability into the fibers than the pelletized HDPE resin, due to its smaller volume per piece. It should be noted that the increased tensile properties of the resultant composites may also be attributed in part to differences between pelletized and powdered HDPE in terms of the molecular weight and molecular weight distribution of the respective matrix materials. However, there were no statistically significant differences of tensile strength and modulus of elasticity values between the pelletized HDPE, P1, and powdered HDPE, P4. This finding suggests that the molecular weight and molecular weight distribution of the powdered and pelletized HDPE resin samples were similar.

Table 4. Results of Tensile Strength (MPa)

Comp. No.	Composite	Fiber Direction	Mean	SD
C1.	Pel.HDPE/Fiber	Lengthwise	23.7	2.1
		Crosswise	10.6	0.2
C2.	Sulf.(2min)Pel.HDPE/Fiber	Lengthwise	23.1	1.2
		Crosswise	11.2	0.2
C3.	Sulf.(4min)Pel.HDPE/Fiber	Lengthwise	20.6	0.2
		Crosswise	11.5	0.4
C4.	Powd.HDPE/Fiber	Lengthwise	27.1	0.1
		Crosswise	17.4	0.2
C5.	Sulf.(3min)Powd.HDPE/Fiber	Lengthwise	26.1	1.8
		Crosswise	15.2	0.1
C6.	Pel.HDPE/MAPP/Fiber	Lengthwise	27.3	1.4
		Crosswise	21.4	0.2
C7.	Sulf.(2min)Pel.HDPE/MAPP/Fiber	Lengthwise	24.1	1.1
		Crosswise	20.6	1.3
C8.	Sulf.(4min)Pel.HDPE/MAPP/Fiber	Lengthwise	28.8	0.6
		Crosswise	20.4	0.8
P1.	Pel.HDPE		30.7	1.6
P2.	Sulf.(2min)Pel.HDPE		31.1	0.2
P3.	Sulf.(4min)Pel.HDPE		31.0	0.7
P4.	Sulf.(3min)Powd.HDPE		30.2	0.9

Figure 6. Tensile Strength



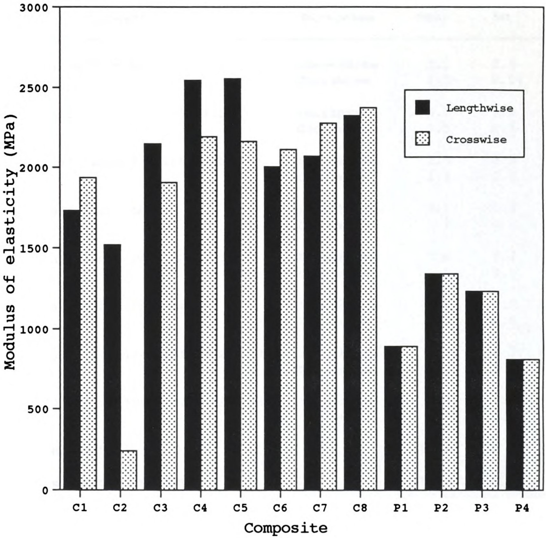
C1. Pel.HDPE/Fiber
C2. Sulf.(2min)Pel.HDPE/Fiber
C3. Sulf.(4min)Pel.HDPE/Fiber
C4. Powd. HDPE/Fiber
C5. Sulf.(3min)Powd.HDPE/Fiber
C6. Pel.HDPE/MAPP/Fiber
C7. Sulf.(2min)Pel.HDPE/MAPP/Fiber
C8. Sulf.(4min)Pel.HDPE/MAPP/Fiber

P1. Pel.HDPE
P2. Sulf.(2min)Pel.HDPE
P3. Sulf.(4min)Pel.HDPE
P4. Sulf.(3min)Powd.HDPE

Table 5. Results of Modulus of Elasticity (MPa)

Comp. No.	Composite	Fiber Direction	Mean	SD
C1.	Pel.HDPE/Fiber	Lengthwise	1730	91
		Crosswise	1934	38
C2.	Sulf.(2min)Pel.HDPE/Fiber	Lengthwise	1518	210
		Crosswise	243	91
C3.	Sulf.(4min)Pel.HDPE/Fiber	Lengthwise	2150	53
		Crosswise	1905	67
C4.	Powd.HDPE/Fiber	Lengthwise	2544	36
		Crosswise	2194	5
C5.	Sulf.(3min)Powd.HDPE/Fiber	Lengthwise	2555	69
		Crosswise	2165	58
C6.	Pel.HDPE/MAPP/Fiber	Lengthwise	2004	436
		Crosswise	2112	453
C7.	Sulf.(2min)Pel.HDPE/MAPP/Fiber	Lengthwise	2072	212
		Crosswise	2280	87
C8.	Sulf.(4min)Pel.HDPE/MAPP/Fiber	Lengthwise	2328	14
		Crosswise	2378	33
P1.	Pel.HDPE		890	148
P2.	Sulf.(2min)Pel.HDPE		1342	36
P3.	Sulf.(4min)Pel.HDPE		1235	66
P4.	Sulf.(3min)Powd.HDPE		807	265

Figure 7. Modulus of Elasticity

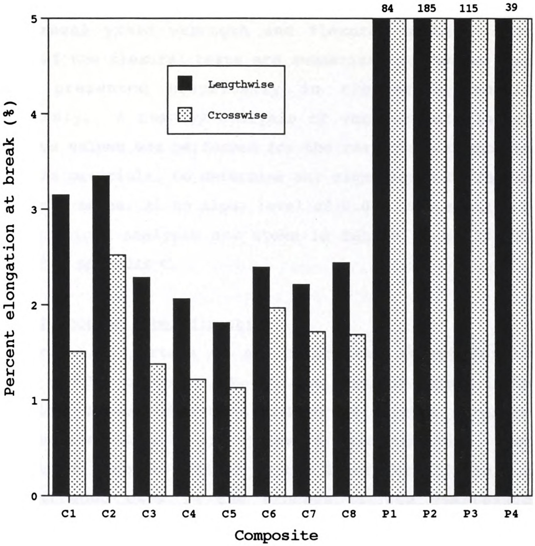


C1.	Pel.HDPE/Fiber	P1.	Pel.HDPE
C2.	Sulf.(2min)Pel.HDPE/Fiber	P2.	Sulf.(2min)Pel.HDPE
C3.	Sulf.(4min)Pel.HDPE/Fiber	P3.	Sulf.(4min)Pel.HDPE
C4.	Powd. HDPE/Fiber	P4.	Sulf.(3min)Powd.HDPE
C5.	Sulf.(3min)Powd.HDPE/Fiber		
C6.	Pel.HDPE/MAPP/Fiber		
C7.	Sulf.(2min)Pel.HDPE/MAPP/Fiber		
C8.	Sulf.(4min)Pel.HDPE/MAPP/Fiber		

Table 6. Results of Percent Elongation at Break (%)

Comp. No.	Composite	Fiber Direction	Mean	SD
C1.	Pel.HDPE/Fiber	Lengthwise	3.2	0.4
		Crosswise	1.5	0.04
C2.	Sulf.(2min)Pel.HDPE/Fiber	Lengthwise	3.4	0.3
		Crosswise	2.5	0.1
C3.	Sulf.(4min)Pel.HDPE/Fiber	Lengthwise	2.3	0.3
		Crosswise	1.4	0.3
C4.	Powd.HDPE/Fiber	Lengthwise	2.1	0.3
		Crosswise	1.2	0.1
C5.	Sulf.(3min)Powd.HDPE/Fiber	Lengthwise	1.8	0.3
		Crosswise	1.1	0.1
C6.	Pel.HDPE/MAPP/Fiber	Lengthwise	2.4	0.5
		Crosswise	2.0	0.5
C7.	Sulf.(2min)Pel.HDPE/MAPP/Fiber	Lengthwise	2.2	0.3
		Crosswise	1.7	0.2
C8.	Sulf.(4min)Pel.HDPE/MAPP/Fiber	Lengthwise	2.4	0.01
		Crosswise	1.7	0.2
P1.	Pel.HDPE		84.4	75.5
P2.	Sulf.(2min)Pel.HDPE		185.3	92.9
P3.	Sulf.(4min)Pel.HDPE		115.1	77.8
P4.	Sulf.(3min)Powd.HDPE		39.2	11.6

Figure 8. Percent Elongation at Break



C1. Pel.HDPE/Fiber
C2. Sulf.(2min)Pel.HDPE/Fiber
C3. Sulf.(4min)Pel.HDPE/Fiber
C4. Powd. HDPE/Fiber
C5. Sulf.(3min)Powd.HDPE/Fiber
C6. Pel.HDPE/MAPP/Fiber
C7. Sulf.(2min)Pel.HDPE/MAPP/Fiber
C8. Sulf.(4min)Pel.HDPE/MAPP/Fiber

P1. Pel.HDPE
P2. Sulf.(2min)Pel.HDPE
P3. Sulf.(4min)Pel.HDPE
P4. Sulf.(3min)Powd.HDPE

4. Flexural Properties

The mean of 5 replicate samples of the respective composites, which included the lengthwise and crosswise fiber directions, as well as the mean of the homopolymer matrix was determined for flexural yield strength and flexural modulus. The results of the flexural tests are summarized in Tables 7 and 8, and presented graphically in Figures 9 and 10, respectively. A one-way analysis of variance of flexural properties values was performed for the respective composites and matrix materials, to determine any significant difference between the means, at an alpha level of 0.05. The results of the statistical analysis are shown in Tables 26 to 29, 35, 36, and 38, Appendix C.

4.1 Influence of fiber direction

For flexural properties of all composites except for the flexural modulus of composite, C7, the means of values in the lengthwise fiber direction exhibited higher flexural properties than those in the crosswise fiber direction. This result might also be related to the interfacial failure of mixed extruded bars in the process of the compression molding, as described in the previous section (3.1).

4.2 Influence of Sulfonated HDPE

In comparison of flexural yield strength and flexural modulus, between the non-sulfonated HDPE composites and the sulfonated HDPE composite structures, the following pairs were used: (i) C1 to C2 and C3; (ii) C4 to C5; and (iii) C6 to C7 and C8. For the respective pairs compared, the following significant differences of flexural yield strength were observed, which were based on statistical analysis: an increase in flexural yield strength of C7 in cross fiber direction; and a decrease in flexural strength of C2, C3, and C5 in crosswise fiber direction. In addition, no statistically significant differences of flexural modulus were exhibited, for the respective sample pairs compared. Thus, sulfonation of the HDPE resins had little or no effect on the flexural properties of the resultant composites. As previous discussed, these findings may be attributed to the low levels of sulfonation achieved, which resulted in little change in the dispersive and polar characteristics of the polymer and therefore little increase in interfacial interaction between the polymer and wood fibers.

4.3 Influence of Coupling Agent

Both flexural yield strength and flexural modulus were determined to evaluate the stiffness of the composites tested, which is a measure of the ability of the composite to resist bending forces without failure. The stiffness is very dependent on the interfacial bonding between fiber and polymer, as a higher bonding network will increase the stiffness of the sample. As shown in Figure 9, the addition of MAPP as a coupling agent significantly improved the flexural yield strength of the composite, C6, as compared to the untreated HDPE composite, C1, for both the lengthwise and crosswise fiber direction. The differences were confirmed by statistical analysis. These findings show the effect of the coupling agent on increasing the interfacial bonding between

wood fiber and HDPE. For flexural modulus, there were no statistically significant differences between the respective composites.

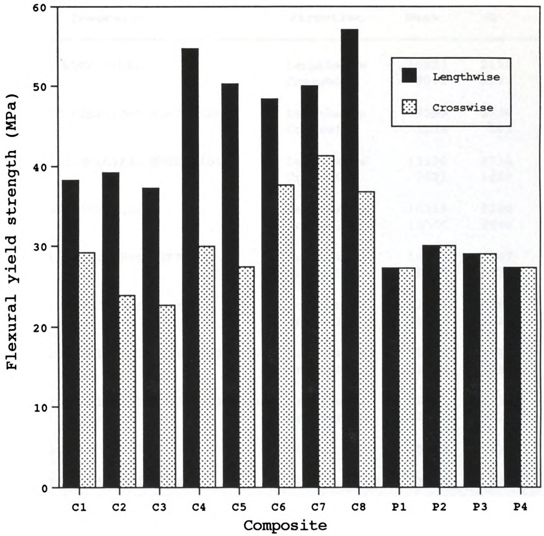
4.4 Influence of Powdered HDPE

As shown in Figure 9, the composite prepared with powdered HDPE, C4, exhibited a significant increase in the flexural yield strength, as compared to that fabricated with pelletized HDPE, C1, for the lengthwise direction. As comparison between the flexural strength of the respective matrix materials used to fabricate the composites, pelletized HDPE, P1, and 3 min. sulfonated powdered HDPE, P4, were evaluated. This result illustrates the effect of powdering of the polymer resin on the flexural yield strength of the resultant composites, and may be attributed to the effective compatibility between powdered HDPE and wood fibers. It should be noted that the increased flexural yield strength of the resultant composites may also be attributed in part to differences between pelletized and powdered HDPE, in terms of the molecular weight and molecular weight distribution of the respective matrix materials. However, the observed flexural strength values of pelletized HDPE, P1, and powdered HDPE, P4, were not found to be statistically different. This finding suggests that there was no difference of molecular weight and molecular weight distribution between the pelletized and powdered HDPE resins. Based on statistical analysis, a significant increase in the flexural modulus of composite, C4, in the crosswise direction, was exhibited, as compared to composite, C1.

Table 7. Results of Flexural Yield Strength (MPa)

Comp. No.	Composite	Fiber Direction	Mean	SD
C1.	Pel.HDPE/Fiber	Lengthwise	38.3	3.9
		Crosswise	29.2	1.5
C2.	Sulf.(2min)Pel.HDPE/Fiber	Lengthwise	39.2	5.3
		Crosswise	23.9	1.1
C3.	Sulf.(4min)Pel.HDPE/Fiber	Lengthwise	37.3	7.3
		Crosswise	22.7	0.9
C4.	Powd.HDPE/Fiber	Lengthwise	54.6	5.1
		Crosswise	30.0	1.5
C5.	Sulf.(3min)Powd.HDPE/Fiber	Lengthwise	50.2	2.4
		Crosswise	27.4	0.6
C6.	Pel.HDPE/MAPP/Fiber	Lengthwise	48.4	4.4
		Crosswise	37.7	1.6
C7.	Sulf.(2min)Pel.HDPE/MAPP/Fiber	Lengthwise	50.0	1.0
		Crosswise	41.3	2.9
C8.	Sulf.(4min)Pel.HDPE/MAPP/Fiber	Lengthwise	57.0	5.8
		Crosswise	36.8	1.6
P1.	Pel.HDPE		27.2	0.7
P2.	Sulf.(2min)Pel.HDPE		30.0	0.3
P3.	Sulf.(4min)Pel.HDPE		29.0	1.2
P4.	Sulf.(3min)Powd.HDPE		27.3	1.0

Figure 9. Flexural Yield Strength

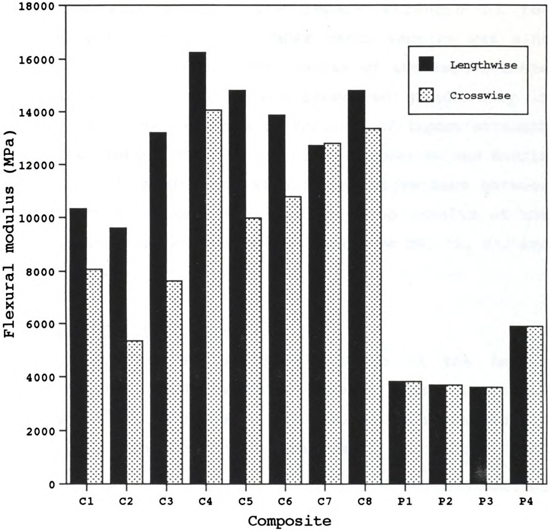


C1. Pel.HDPE/Fiber
C2. Sulf.(2min)Pel.HDPE/Fiber
C3. Sulf.(4min)Pel.HDPE/Fiber
C4. Powd. HDPE/Fiber
C5. Sulf.(3min)Powd.HDPE/Fiber
C6. Pel.HDPE/MAPP/Fiber
C7. Sulf.(2min)Pel.HDPE/MAPP/Fiber
C8. Sulf.(4min)Pel.HDPE/MAPP/Fiber
P1. Pel.HDPE
P2. Sulf.(2min)Pel.HDPE
P3. Sulf.(4min)Pel.HDPE
P4. Sulf.(3min)Powd.HDPE

Table 8. Results of Flexural Modulus (MPa)

Comp. No.	Composite	Fiber Direction	Mean	SD
C1.	Pel.HDPE/Fiber	Lengthwise	10323	2134
		Crosswise	8076	1977
C2.	Sulf.(2min)Pel.HDPE/Fiber	Lengthwise	9599	2630
		Crosswise	5339	505
C3.	Sulf.(4min)Pel.HDPE/Fiber	Lengthwise	13190	2724
		Crosswise	7621	1438
C4.	Powd.HDPE/Fiber	Lengthwise	16235	2308
		Crosswise	14060	2890
C5.	Sulf.(3min)Powd.HDPE/Fiber	Lengthwise	14801	4507
		Crosswise	9975	1289
C6.	Pel.HDPE/MAPP/Fiber	Lengthwise	13871	2760
		Crosswise	10786	3120
C7.	Sulf.(2min)Pel.HDPE/MAPP/Fiber	Lengthwise	12713	2424
		Crosswise	12793	2725
C8.	Sulf.(4min)Pel.HDPE/MAPP/Fiber	Lengthwise	14801	6113
		Crosswise	13361	3021
P1.	Pel.HDPE		3814	475
P2.	Sulf.(2min)Pel.HDPE		3693	425
P3.	Sulf.(4min)Pel.HDPE		3609	460
P4.	Sulf.(3min)Powd.HDPE		5889	1706

Figure 10. Flexural Modulus



C1.	Pel.HDPE/Fiber
C2.	Sulf.(2min)Pel.HDPE/Fiber
C3.	Sulf.(4min)Pel.HDPE/Fiber
C4.	Powd. HDPE/Fiber
C5.	Sulf.(3min)Powd.HDPE/Fiber
C6.	Pel.HDPE/MAPP/Fiber
C7.	Sulf.(2min)Pel.HDPE/MAPP/Fiber
C8.	Sulf.(4min)Pel.HDPE/MAPP/Fiber
P1.	Pel.HDPE
P2.	Sulf.(2min)Pel.HDPE
P3.	Sulf.(4min)Pel.HDPE
P4.	Sulf.(3min)Powd.HDPE

5. Impact Resistance

The means of 9 to 10 replicate samples of the respective composites, which included the lengthwise and crosswise fiber directions, were obtained to compare the impact strength of the various structures. The impact strength of the sulfonated and non-sulfonated HDPE resin samples was also determined for comparison. The results of the impact tests are summarized in Table 9, and presented graphically in Figure 11. A one-way analysis of variance of impact strength values was performed for the respective composites and matrix materials, to determine any significant difference between the means, at an alpha level of 0.05. The results of the statistical analysis are summarized in Tables 30, 31, 37, and 38, Appendix C.

5.1 Influence of fiber direction

For all composites evaluated, the means of the impact strength in the lengthwise and crosswise fiber directions showed similar values and were not found to be statistically different with respect to fiber direction.

5.2 Influence of Sulfonated HDPE

In comparison of the impact strength of the untreated HDPE composite and sulfonated HDPE composite structures, the following pairs were evaluated: (i) C1 to C2 and C3; (ii) C4 to C5; and (iii) C6 to C7 and C8, respectively. Based on statistical analysis, no significant differences within treatments were observed. These findings are attributed to the low levels of sulfonation achieved (See section 1).

5.3 Influence of Coupling Agent

A statistically significant decrease in the impact strength for the composite fabricated with MAPP as a coupling agent, C6, was exhibited, when compared to the untreated HDPE composite, C1, as shown in Figure 11. Further, the composite, C6, was found to have lower impact strength than the powdered HDPE composite, C4. Good interfacial adhesion between the filler and polymer improves the tensile strength of the resultant composites. However, it also increases the tendency for brittle failure and makes the composite more notch sensitive (Richardson, 1977). Therefore, these results confirmed that the inclusion of MAPP promoted a higher degree of interfacial bonding between fibers and HDPE. In other words, inclusion of a coupling agent resulted in the composite having less flexibility and toughness, due to the brittleness.

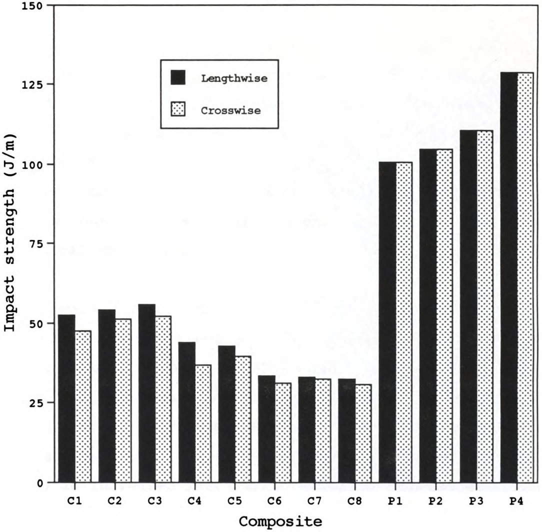
5.4 Influence of Powdered HDPE

As shown in Figure 11, the powdered HDPE, P4, exhibited higher impact strength than the pelletized HDPE, P1. Further, the powdered HDPE composite, C4, exhibited a significant decrease in impact strength, as compared to the pelletized HDPE composite, C1. This was confirmed by statistical analysis. These findings indicate that the composite, C4, experienced a reduction in toughness. This may be due to an increased compatibility of the fibers within the polymer matrix, resulting in a more rigid and brittle composite structure.

Table 9. Results of Impact Strength (J/m)

Comp. No.	Composite	Fiber Direction	Mean	SD
C1.	Pel.HDPE/Fiber	Lengthwise	52.6	6.7
		Crosswise	47.7	5.5
C2.	Sulf.(2min)Pel.HDPE/Fiber	Lengthwise	54.2	7.9
		Crosswise	51.4	7.0
C3.	Sulf.(4min)Pel.HDPE/Fiber	Lengthwise	55.9	5.0
		Crosswise	52.3	7.9
C4.	Powd.HDPE/Fiber	Lengthwise	44.0	4.6
		Crosswise	36.9	2.7
C5.	Sulf.(3min)Powd.HDPE/Fiber	Lengthwise	42.9	3.7
		Crosswise	39.7	5.6
C6.	Pel.HDPE/MAPP/Fiber	Lengthwise	33.4	4.4
		Crosswise	31.1	6.0
C7.	Sulf.(2min)Pel.HDPE/MAPP/Fiber	Lengthwise	33.0	4.0
		Crosswise	32.4	6.1
C8.	Sulf.(4min)Pel.HDPE/MAPP/fiber	Lengthwise	32.4	2.3
		Crosswise	30.7	1.3
P1.	Pel.HDPE		100.6	4.8
P2.	Sulf.(2min)Pel.HDPE		104.8	10.9
P3.	Sulf.(4min)Pel.HDPE		110.6	15.2
P4.	Sulf.(3min)Powd.HDPE		128.6	11.0

Figure 11. Impact Strength



C1. Pel.HDPE/Fiber
C2. Sulf.(2min)Pel.HDPE/Fiber
C3. Sulf.(4min)Pel.HDPE/Fiber
C4. Powd. HDPE/Fiber
C5. Sulf.(3min)Powd.HDPE/Fiber
C6. Pel.HDPE/MAPP/Fiber
C7. Sulf.(2min)Pel.HDPE/MAPP/Fiber
C8. Sulf.(4min)Pel.HDPE/MAPP/Fiber

P1. Pel.HDPE
P2. Sulf.(2min)Pel.HDPE
P3. Sulf.(4min)Pel.HDPE
P4. Sulf.(3min)Powd.HDPE

SUMMARY AND CONCLUSION

In sulfonation of HDPE resin, the effect of a longer reaction time and an increased surface area was shown to result in increased levels of sulfonation. However, the extent of sulfonation achieved was quite low and did not modify the dispersive and polar characteristics of the polymer to a level which would result in enhanced interfacial interaction between the HDPE and wood fibers with a concomitant increase in mechanical properties.

The HDPE/wood fiber composite fabricated with MAPP as a coupling agent showed a statistically significant increase in tensile strength, modulus of elasticity, and flexural yield strength, but decreased in percent elongation at break and impact strength, as compared to the untreated composite. These findings could be attributed to the inclusion of MAPP resulting in increased interfacial adhesion between HDPE and wood fibers, with an associated increase in stiffness and brittleness but a decrease in toughness and flexibility of the resultant composite structures.

The sulfonated HDPE based composite fabricated with MAPP exhibited little or no practical differences in mechanical properties, as compared to the non-sulfonated HDPE based composite fabricated with MAPP. Thus, sulfonation at the achieved levels had little or no effect on enhancing the mechanical properties of the HDPE composite fabricated with MAPP.

The composite fabricated with powdered HDPE exhibited statistically significant increases in tensile strength, modulus of elasticity, and flexural yield strength, but showed a decrease in percent elongation at break and impact strength, as compared to the pelletized HDPE based composite. This result may be attributed to the increased compatibility between HDPE and wood fibers.

The composite fabricated with sulfonated powdered HDPE showed no significant differences of mechanical properties, when compared to that with non-sulfonated powdered HDPE. Thus, sulfonation at the achieved levels had little or no effect on enhancing the mechanical properties of the powdered HDPE composite.

RECOMMENDATION FOR FURTHER RESEARCH

In order to obtain conclusive results for enhanced interfacial interaction between HDPE and wood fibers with a concomitant increase in mechanical properties of surface sulfonated HDPE/wood fiber composites, the following investigations are proposed for further study: (i) a much longer reaction time or higher temperature in sulfonation, to achieve increased levels of sulfonation, which can effectively modify dispersive and non-dispersive energies on the HDPE surface; (ii) utilize powdered HDPE resin samples to obtain the increased levels of sulfonation and also the increased compatibility with wood fibers in compounding process.

Additionally, the effect of surface sulfonation of polypropylene (PP) on mechanical properties of PP/wood fiber composites should be considered. Sulfonation of PP has been found to easily modify the surface energy properties on PP surface, since the presence of tertiary carbons in the molecule obtains active sites for SO_3 insertion (Wangwiwatsilp, 1993). The effective modification of the surface energy properties of the PP may offer an opportunity to increase the interfacial interaction between PP and wood fibers.

APPENDIX A

APPENDIX A

Table 10. Results of DSC for Pelletized HDPE Resin

Var. No.	Type of HDPE	Tm (°C)	ΔH_f (J/g)	%crystallinity
1	Pelletized	135.16	176.1	61.51
2	Pelletized	135.96	173.9	60.74
	Mean	135.56	175.0	61.12
	SD	0.57	1.6	0.54

Table 11. Results of DSC for Powdered HDPE Resin

Var. No.	Type of HDPE	Tm (°C)	ΔH_f (J/g)	%crystallinity
1	Powdered	136.07	204.6	71.46
2	Powdered	136.17	200.9	70.17
	Mean	136.12	202.8	70.81
	SD	0.07	2.6	0.91

Figure 12. DSC Data for Pelletized HDPE Resin

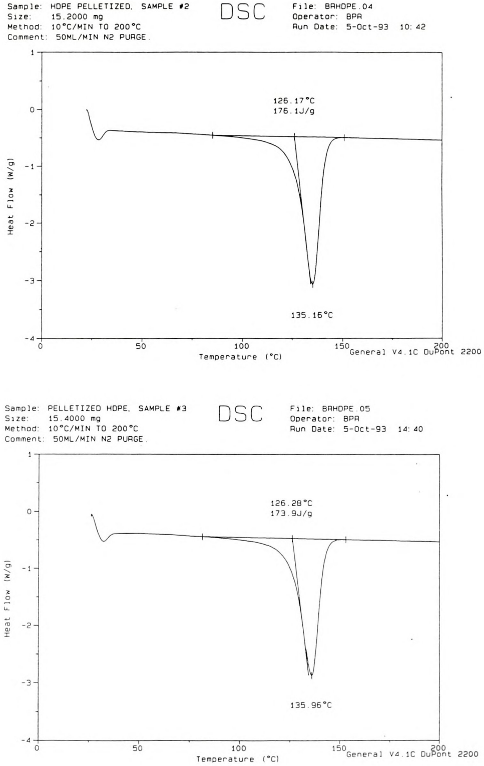
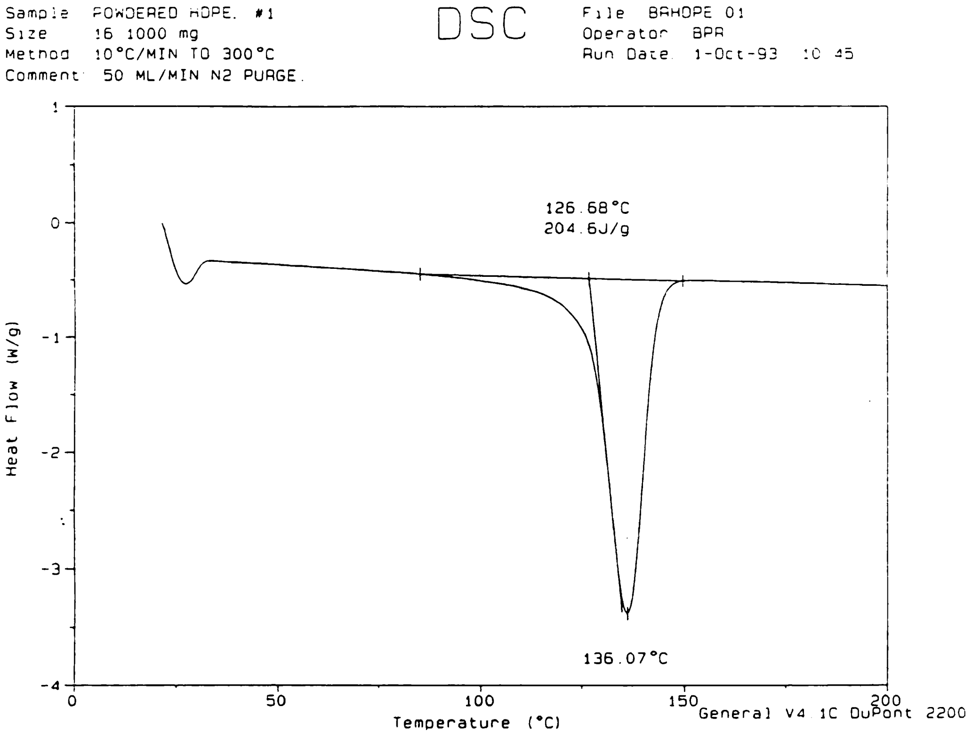
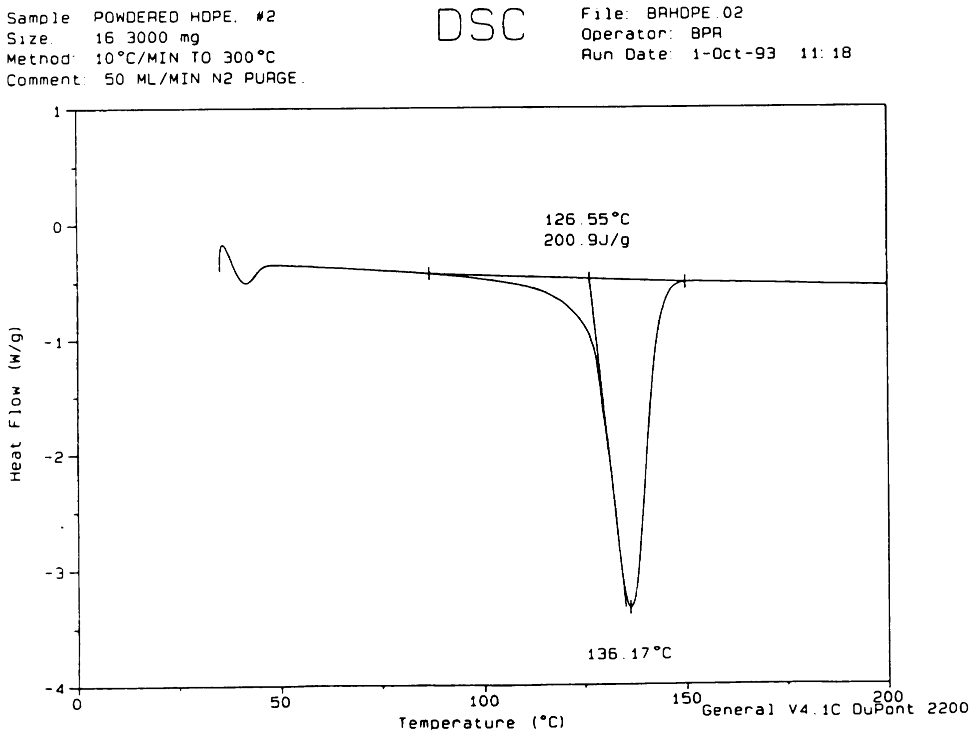


Figure 13. DSC Data for Powdered HDPE Resin





APPENDIX B

APPENDIX B

Table 12. Density Measurement Data (gm/cm^3)

Comp. No.	Replications									
	1	2	3	4	5	6	7	8	9	10
C1	1.05	1.06	1.06	1.06	1.03	1.06	1.08	1.04	1.08	1.06
C2	1.10	1.08	1.08	1.05	1.07	1.06	1.08	1.05	1.02	1.06
C3	1.06	1.02	1.07	1.04	1.06	1.02	1.06	1.04	1.04	1.06
C4	1.07	1.09	1.04	1.09	1.07	1.03	1.08	1.04	1.03	1.09
C5	1.06	1.09	1.08	1.08	1.05	1.08	1.09	1.07	1.09	1.08
C6	1.09	1.06	1.12	1.05	1.04	1.04	1.09	0.99	1.02	1.11
C7	1.09	1.05	1.06	1.07	1.07	1.08	1.10	1.09	1.08	1.08
C8	1.06	1.07	1.07	1.09	1.10	1.06	1.06	1.08	1.09	1.09

Table 13. Tensile Strength Data (MPa)

Comp. No.	Fiber Dir.	Replications						
		1	2	3	4	5	6	7
C1	Length	25.75	25.57	28.36	24.00	29.11	22.34	21.18
C1	Length	23.58	19.55	25.03	22.30	19.64	22.06	23.30
C1	Cross	9.46	10.11	10.71	11.10	11.31	11.63	10.92
C1	Cross	10.73	11.36	10.99	11.18	9.80	9.50	9.46
C2	Length	26.65	20.69	25.66	20.28	23.40	21.21	18.13
C2	Length	26.80	21.17	27.64	24.95	23.41	21.77	21.85
C2	Cross	10.69	10.90	11.06	11.56	11.24	11.16	11.21
C2	Cross	12.38	11.85	11.36	12.31	10.29	11.41	9.98
C3	Length	18.03	16.42	19.93	20.26	26.50	24.29	19.90
C3	Length	18.84	23.23	15.82	18.08	25.74	20.58	20.85
C3	Cross	10.56	11.78	11.40	10.65	11.81	11.09	
C3	Cross	11.94	12.40	11.05	11.42	12.16		
C4	Length	21.34	25.97	26.32	25.32	42.36		
C4	Length	24.46	26.08	25.71	27.66	27.68		
C4	Cross	19.35	19.75	20.93	23.18	23.05		
C4	Cross	19.57	20.15	21.75	22.61	23.46		
C5	Length	24.82	21.55	24.50	25.44	20.48		
C5	Length	26.37	22.70	23.16	24.23	28.05		
C5	Cross	19.72	19.16	21.64	17.68	19.99		
C5	Cross	22.38	23.39	19.70	20.15	22.30	21.05	
C6	Length	30.78	29.08	22.03	30.45	29.63		
C6	Length	28.70	33.56	28.96	29.55	25.33		
C6	Cross	20.61	20.39	20.32	21.81	21.95		
C6	Cross	23.37	16.84	19.65	20.09	19.17		
C7	Length	29.10	25.64	26.64	26.85	26.76	27.65	
C7	Length	28.27	29.15	27.68	26.96	22.99		
C7	Cross	17.62	17.61	17.71	16.91	17.80		
C7	Cross	16.91	16.51	17.59	18.93	16.48		

Table 13. (cont'd)

Comp. No.	Fiber Dir.	Replications 1	2	3	4	5	6
C8	Length	22.44	31.78	29.21	20.91	19.84	
C8	Length	36.84	20.57	28.27	27.69	23.25	
C8	Cross	14.49	15.00	15.93	15.20	15.03	
C8	Cross	15.18	15.40	15.51	15.95	14.51	
P1		29.78	32.82	33.13	31.97	31.36	
P1		30.83	29.74	29.78	29.06	28.41	
P2		31.12	32.00	30.90	31.23	31.58	30.42
P2		30.81	31.41	30.81	31.07	30.03	31.18
P3		31.34	31.83	32.12	32.38	32.27	29.47
P3		30.48	30.13	31.32	29.87	30.79	
P4		31.96	30.29	30.77	30.79	30.12	31.13
P4		30.83	29.74	29.78	29.06	28.41	

Table 14. Modulus of Elasticity Data (MPa)

Comp. No.	Fiber Dir.	Replications 1	2	3	4	5	6	7
C1	Length	2091.8	1838.8	1487.2	1390.6	1220.3	2481.4	2051.8
C1	Length	2243.5	1769.8	1175.5	1634.7	1428.6	1672.6	1734.0
C1	Cross	1717.4	2084.9	2050.5	1657.5	1893.3	2233.9	2091.8
C1	Cross	1370.0	1929.1	2080.1	2012.5	2107.0	1920.8	1931.2
C2	Length	2342.8	1294.1	763.9	1187.9	1088.0	1305.9	1608.5
C2	Length	1623.0	853.6	2322.8	1800.9	1725.7	1781.6	1559.6
C2	Cross	490.9	151.7	153.8	252.3	316.6	397.8	387.5
C2	Cross	133.1	255.8	183.4	226.1	96.5	126.9	226.1
C3	Length	1991.9	2099.4	1922.9	2206.3	2337.3	2019.4	2209.7
C3	Length	2005.0	1913.3	2531.7	2181.5	2173.2	2225.6	2280.0
C3	Cross	1803.6	1967.0	1856.7	1868.4	1945.0	1704.4	
C3	Cross	2167.0	2048.4	1826.4	1962.9	1754.0		

Table 14. (cont'd)

Comp.	Fiber	Replications					
No.	Dir.	1	2	3	4	5	6
C4	Length	1652.0	1496.1	1618.9	1697.5	2012.5	
C4	Length	1836.7	2429.0	2259.4	2613.1	2420.0	
C4	Cross	1505.8	2315.2	1680.2	1658.8	1800.9	
C4	Cross	2373.1	2473.1	2290.4	2510.3	2514.5	
C5	Length	1917.4	1938.8	1885.7	2104.2	1765.0	
C5	Length	2098.0	2175.3	1991.2	2104.9	2737.9	
C5	Cross	2229.7	2294.5	1895.3	2246.3	2429.0	
C5	Cross	2378.0	2493.1	2304.9	2206.3	2308.3	2360.7
C6	Length	2423.5	2135.3	2433.1	2416.6	2178.7	
C6	Length	2160.1	2333.1	2049.1	2810.2	2336.6	
C6	Cross	2551.0	2120.1	2435.2	2401.4	2264.9	
C6	Cross	2574.4	2021.5	2521.4	2222.1	2668.2	
C7	Length	2632.4	2531.7	2768.2	3360.4	1887.7	2235.2
C7	Length	2941.9	2611.0	2273.8	2411.7	2354.5	
C7	Cross	2133.2	1771.9	2444.8	2148.4	2489.0	
C7	Cross	2152.5	2124.2	2506.2	2080.8	2089.8	
C8	Length	2221.4	3212.9	2909.5	2438.6	2235.2	
C8	Length	2571.7	2253.2	2643.4	2673.0	2389.0	
C8	Cross	2370.4	1971.2	1917.4	2265.6	2096.0	
C8	Cross	2199.4	2282.1	2275.2	2335.9	1937.4	
P1		697.7	729.5	727.4	653.6	1118.3	
P1		1025.9	875.6	964.6	1045.9	1059.0	
P2		1254.8	1345.8	1471.3	1395.5	1194.1	1238.3
P2		1361.0	1387.9	1271.4	1405.8	1236.9	1545.1
P3		1289.3	1237.6	1290.7	1261.7	1386.5	1221.0
P3		1065.2	1085.2	1128.7	1221.0	1442.4	
P4		631.6	610.9	552.3	649.5	657.1	613.6
P4		1025.9	875.6	964.6	1045.9	1059.0	

Table 15. Percent Elongation at Break Data (%)

Comp. No.	Fiber Dir.	Replications 1	2	3	4	5	6	7
C1	Length	3.94	3.40	3.76	3.01	3.44	2.70	3.55
C1	Length	2.79	2.72	3.32	2.73	2.46	3.02	3.21
C1	Cross	1.16	1.13	1.38	1.61	1.46	1.49	2.16
C1	Cross	2.11	2.14	1.53	1.62	1.16	1.21	1.03
C2	Length	4.60	3.16	4.51	2.82	3.67	3.49	2.83
C2	Length	3.59	2.72	3.56	3.13	2.83	2.87	3.14
C2	Cross	1.89	2.41	2.19	2.79	2.27	2.62	3.12
C2	Cross	2.95	2.52	2.17	2.73	2.79	2.97	1.83
C3	Length	2.71	2.34	2.22	2.38	2.63	2.71	2.28
C3	Length	1.90	2.59	1.50	1.68	2.21	2.27	2.49
C3	Cross	1.14	1.22	1.05	1.04	1.25	1.10	
C3	Cross	2.17	1.54	1.66	1.11	1.61		
C4	Length	2.18	2.93	2.59	2.36	3.77		
C4	Length	2.34	1.85	1.91	1.80	2.17		
C4	Cross	2.28	1.67	2.88	2.36	2.44		
C4	Cross	1.24	1.43	1.98	1.80	1.58		
C5	Length	3.03	2.42	2.47	2.31	1.70		
C5	Length	2.41	1.66	2.01	2.20	1.87		
C5	Cross	1.57	1.53	1.92	1.39	1.34		
C5	Cross	2.13	1.48	1.72	2.27	1.69	2.04	
C6	Length	2.95	2.21	1.53	2.63	2.94		
C6	Length	2.76	3.31	2.55	1.75	1.81		
C6	Cross	1.73	1.72	2.12	1.73	1.93		
C6	Cross	1.62	1.57	1.55	1.54	1.38		
C7	Length	2.26	1.87	1.69	1.48	2.13	1.81	
C7	Length	2.08	2.19	2.63	2.20	2.15		
C7	Cross	1.48	1.41	1.26	1.27	1.08		
C7	Cross	1.07	1.19	1.07	1.41	0.99		

Table 15. (cont'd)

Comp. No.	Fiber Dir.	Replications 1	2	3	4	5	6
C8	Length	1.66	1.59	1.94	1.29	1.52	
C8	Length	2.37	1.64	2.17	2.08	1.88	
C8	Cross	1.25	1.08	1.21	1.14	1.26	
C8	Cross	1.12	0.95	1.01	1.18	1.07	
P1		178.0	75.1	252.0	133.0	50.5	
P1		37.5	23.9	43.1	24.1	26.5	
P2		95.0	89.0	116.0	95.9	115.0	207.0
P2		219.0	273.0	141.2	382.0	294.0	196.7
P3		120.0	78.8	581.0	104.0	95.4	41.4
P3		18.0	36.6	116.0	24.6	105.0	
P4		28.5	62.4	45.3	40.2	54.2	53.9
P4		37.5	23.9	43.1	24.1	26.5	

Table 16. Flexural Yield Strength Data (MPa)

Comp. No.	Fiber Dir.	Replications 1	2	3	4	5
C1	Length	34.58	42.59	33.74	40.73	39.71
C1	Cross	29.31	28.83	31.64	28.26	27.80
C2	Length	35.42	42.84	36.64	34.47	46.64
C2	Cross	23.71	24.39	24.60	24.65	22.01
C3	Length	42.11	27.82	39.26	31.75	45.31
C3	Cross	23.66	22.08	22.01	23.63	22.01
C4	Length	49.76	41.16	47.94	50.32	52.63
C4	Cross	35.93	38.25	36.25	37.95	39.98
C5	Length	49.13	51.78	49.35	49.82	49.93
C5	Cross	41.42	38.49	39.64	46.01	41.15
C6	Length	64.02	62.62	51.46	54.07	53.03
C6	Cross	34.08	37.91	38.09	37.02	36.91
C7	Length	59.65	49.55	52.41	60.62	50.90
C7	Cross	29.61	28.59	31.69	31.36	28.50
C8	Length	49.34	50.70	49.66	47.45	53.93
C8	Cross	27.08	27.59	28.43	27.02	26.88
P1		27.96	27.39	26.13	26.86	27.76
P2		30.02	30.36	29.76	29.83	30.20
P3		30.07	27.57	27.96	29.83	29.61
P4		26.17	26.39	27.93	27.96	28.14

Table 17. Flexural Modulus Data (MPa)

Comp. No.	Fiber Dir.	Replications 1	2	3	4	5
C1	Length	11811.4	13226.8	8003.3	9154.9	9419.8
C1	Cross	6656.8	11279.2	7679.4	6324.7	8437.8
C2	Length	8322.2	7551.5	13428.2	7079.2	11016.1
C2	Cross	5523.6	5368.7	4803.3	6069.4	4931.4
C3	Length	13190.4	10402.8	14353.2	10177.2	14905.9
C3	Cross	9980.0	6784.6	7240.2	7826.7	6271.2
C4	Length	17084.2	12378.9	15860.3	10135.2	13895.1
C4	Cross	12942.4	15176.8	8737.2	9129.6	7943.7
C5	Length	12961.1	14735.1	11468.9	9260.0	15137.7
C5	Cross	16810.2	13254.0	12572.3	12120.9	9208.6
C6	Length	22008.1	20487.8	10302.4	8397.6	12808.2
C6	Cross	17149.2	11740.2	11673.2	10244.4	16000.1
C7	Length	15563.3	13888.4	19018.6	18299.0	14406.1
C7	Cross	19145.6	13137.9	12066.3	12548.1	13403.3
C8	Length	12462.2	8878.6	17852.5	14418.0	20391.1
C8	Cross	10626.8	9401.2	11899.8	9281.8	8667.5
P1		4120.1	3409.6	3529.1	4501.7	3509.6
P2		4246.7	3835.5	3853.2	3307.8	3221.2
P3		3200.1	3836.8	3144.6	4249.7	3612.7
P4		6308.6	4008.6	8273.8	6380.3	4472.7

Table 18. Impact Strength Data (J/m)

Comp. No.	Fiber Dir.	Replications 1	2	3	4	5
C1	Length	41.56	52.65	45.18	60.82	60.87
C1	Length	47.10	53.13	51.11	53.56	60.12
C1	Cross	39.48	45.56	53.51	49.19	41.50
C1	Cross	54.73	51.53	50.68	42.84	
C2	Length	49.93	60.76	58.31	55.32	60.92
C2	Length	60.49	57.56	38.25	43.32	56.97
C2	Cross	52.01	54.89	52.87	42.62	45.45
C2	Cross	63.00	59.21	41.82	54.89	47.26
C3	Length	49.99	50.36	60.55	64.07	54.73
C3	Length	57.61	60.01	49.19	55.59	57.03
C3	Cross	66.47	54.57	58.47	46.84	57.03
C3	Cross	36.33	49.56	51.43	50.36	51.59
C4	Length	35.05	31.79	30.67	36.65	31.10
C4	Length	28.70	42.46	29.18	35.32	
C4	Cross	25.07	24.27	42.25	26.78	33.98
C4	Cross	35.05	37.50	30.89	29.29	26.19
C5	Length	31.63	40.28	31.10	27.85	36.44
C5	Length	29.39	36.49	31.10	32.97	
C5	Cross	41.61	39.69	32.81	30.62	23.21
C5	Cross	32.54	25.98	35.32	36.44	25.98
C6	Length	32.54	34.73	32.17	33.50	30.14
C6	Length	29.66	29.66	36.17	33.34	
C6	Cross	30.62	30.14	32.33	32.54	32.33
C6	Cross	29.39	29.66	29.66	29.93	
C7	Length	48.81	40.92	44.28	43.48	38.94
C7	Length	39.58	52.87	38.94	45.56	46.84
C7	Cross	34.73	37.61	32.65	36.17	35.85
C7	Cross	35.32	42.20	39.58	37.45	37.29

Table 18. (cont'd)

Comp. No.	Fiber Dir.	Replications 1	2	3	4	5
C8	Length	37.50	45.88	38.20	46.84	39.26
C8	Length	43.16	45.61	40.92	46.84	44.86
C8	Cross	29.29	42.46	45.08	39.58	42.89
C8	Cross	30.62	45.02	38.20	40.92	43.00
P1		96.24	109.68	108.35	101.41	95.92
P1		98.37	98.85	100.98	97.57	98.74
P2		100.98	101.47	112.45	103.23	94.32
P2		121.42	99.70	102.37	88.93	122.70
P3		87.97	106.21	137.37	126.86	116.62
P3		116.08	108.35	109.57	88.66	108.03
P4		150.01	126.86	130.33	143.66	116.35
P4		126.91	120.35	117.90	132.57	121.31

APPENDIX C

APPENDIX C

Table 19. One-Way Analysis of Variance of Density Measurement Values for Composites

Analysis of Variance Table

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	7	0.008	0.0011	2.2857	0.0368
Within groups	72	0.036	0.0005		
Total	79	0.044			

Group	Count	Mean	Standard Deviation	Standard Error	95% Confidence Interval for Mean
C1	10	1.06	0.01	0.0032	1.0528 To 1.0672
C2	10	1.06	0.02	0.0063	1.0457 To 1.0743
C3	10	1.05	0.02	0.0063	1.0357 To 1.0643
C4	10	1.08	0.01	0.0032	1.0728 To 1.0872
C5	10	1.08	0.02	0.0063	1.0657 To 1.0943
C6	10	1.06	0.03	0.0095	1.0385 To 1.0815
C7	10	1.07	0.01	0.0032	1.0628 To 1.0772
C8	10	1.06	0.04	0.0126	1.0314 To 1.0886
Total	80	1.065	0.0236	0.0026	1.0597 To 1.0703

Multiple Comparison Test

Tukey-HSD procedure
Range for the 0.05 level
Table range: 4.41
The value actually compared with Mean (J) - Mean (I) is
0.0158 x Range x Sqrt (1/N(I) + 1/N(J))

No two groups are significantly different at the 0.05 level

Table 20. One-Way Analysis of Variance of Tensile Strength Values for Composites (Lengthwise Fiber Direction)

Analysis of Variance Table

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	7	602.5361	86.0766	54.1604	0.0000
Within groups	85	135.0897	1.5893		
Total	92	737.6258			

Group	Count	Mean	Standard Deviation	Standard Error	95% Confidence Interval for Mean		
C1	14	23.70	2.11	0.5639	22.4817	To	29.9183
C2	14	23.11	1.17	0.3127	22.4345	To	23.7855
C3	14	20.61	0.22	0.0588	20.4830	To	20.7370
C4	11	27.06	0.07	0.0211	27.0130	To	27.1070
C5	10	26.08	1.76	0.5566	24.8210	To	27.3390
C6	10	27.29	1.38	0.4364	26.3028	To	28.2772
C7	10	24.13	1.09	0.3447	23.3503	To	24.9097
C8	10	28.81	0.58	0.1834	28.3951	To	29.2249
Total	93	24.78	2.83	0.2936	24.1979	To	25.3642

Multiple Comparison Test

Tukey-HSD procedure
Range for the 0.05 level
Table range: 4.39
The value actually compared with Mean (J) - Mean (I) is
0.8914 x Range x Sqrt (1/N(I) + 1/N(J))

(*) Denotes pairs of group significantly different at the 0.05 level

Group	C1	C2	C3	C4	C5	C6	C7	C8
C1			*	*	*	*		*
C2			*	*	*	*		*
C3	*	*		*	*	*	*	*
C4	*	*	*				*	*
C5	*	*	*				*	*
C6	*	*	*				*	
C7			*	*	*	*		*
C8	*	*	*	*	*		*	

Table 21. One-Way Analysis of Variance of Tensile Strength Values for Composites (Crosswise Fiber Direction)

Analysis of Variance Table

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	7	1674.7496	239.2499	726.4737	0.0000
Within groups	82	27.0051	0.3293		
Total	89	1701.7547			

Group	Count	Mean	Standard Deviation	Standard Error	95% Confidence Interval for Mean
C1	14	10.59	0.23	0.0615	10.4572 To 10.7228
C2	14	11.24	0.18	0.0481	11.1361 To 11.3439
C3	11	11.50	0.41	0.1236	11.2246 To 11.7754
C4	10	17.41	0.17	0.0538	17.2884 To 17.5316
C5	10	15.22	0.13	0.0411	15.1270 To 15.3130
C6	10	21.38	0.18	0.0569	21.2512 To 21.5088
C7	11	20.57	1.31	0.3950	19.6899 To 21.4501
C8	10	20.42	0.84	0.2656	19.8191 To 21.0209
Total	90	15.59	4.37	0.4609	14.6696 To 16.5013

Multiple Comparison Test

Tukey-HSD procedure
Range for the 0.05 level
Table range: 4.40
The value actually compared with Mean (J) - Mean (I) is
0.4058 x Range x Sqrt (1/N(I) + 1/N(J))

(*) Denotes pairs of group significantly different at the 0.05 level

Group	C1	C2	C3	C4	C5	C6	C7	C8
C1			*	*	*	*	*	*
C2				*	*	*	*	*
C3	*			*	*	*	*	*
C4	*	*	*		*	*	*	*
C5	*	*	*	*		*	*	*
C6	*	*	*	*	*		*	*
C7	*	*	*	*	*	*		
C8	*	*	*	*	*	*		

Table 22. One-Way Analysis of Variance of Modulus of Elastisity Values for composites (Lengthwise Fiber Direction)

Analysis of Variance Table

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	7	11490850.15	1641550.021	48.3563	0.0000
Within groups	85	2885492.949	33946.9759		
Total	92	14376343.1			

Group	Count	Mean	Standard Deviation	Standard Error	95% Confidence Interval for Mean		
C1	14	1730.05	91.24	24.3894	1677.3696	To	1782.7304
C2	14	1518.44	209.70	56.0447	1397.3628	To	1639.5172
C3	14	2149.79	52.86	14.1274	2119.2695	To	2180.3105
C4	11	2543.94	35.83	10.8032	2519.8691	To	2568.0109
C5	10	2554.79	68.94	21.8007	2505.4733	To	2604.1067
C6	10	2003.50	435.75	137.7962	1691.7834	To	2315.2166
C7	10	2071.83	211.58	66.9075	1920.4749	To	2223.1851
C8	10	2327.62	14.43	4.5632	2317.2974	To	2337.9426
Total	93	2076.74	395.30	40.9910	1995.3266	To	2158.1500

Multiple Comparison Test

Tukey-HSD procedure
Range for the 0.05 level
Table range: 4.39
The value actually compared with Mean (J) - Mean (I) is
130.2823 x Range x Sqrt (1/N(I) + 1/N(J))

(*) Denotes pairs of group significantly different at the 0.05 level

Group	C1	C2	C3	C4	C5	C6	C7	C8
C1			*	*	*	*	*	*
C2			*	*	*	*	*	*
C3	*	*		*	*			
C4	*	*	*			*	*	
C5	*	*	*			*	*	
C6	*	*		*	*			*
C7	*	*		*	*			
C8	*	*				*		

Table 23. One-Way Analysis of Variance of Modulus of Elastisity Values for composites (Crosswise Fiber Direction)

Analysis of Variance Table

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	7	43962861.77	6280408.024	241.6535	0.0000
Within groups	82	2131123.587	25989.3120		
Total	89	46093985.36			

Group	Count	Mean	Standard Deviation	Standard Error	95% Confidence Interval for Mean		
C1	14	1934.28	38.24	10.2201	1912.2009	To	1956.3591
C2	14	242.75	91.18	24.3689	190.1042	To	295.3958
C3	11	1904.62	66.61	20.0837	1859.8708	To	1949.3692
C4	10	2194.07	4.78	1.5116	2190.6506	To	2197.4894
C5	10	2165.04	57.92	18.3159	2123.6065	To	2206.4735
C6	10	2112.23	452.62	143.1310	1788.4453	To	2436.0147
C7	11	2280.41	86.91	26.2044	2222.0231	To	2338.7969
C8	10	2378.02	33.25	10.5146	2354.2344	To	2401.8056
Total	90	1833.42	719.66	75.8588	1682.6852	To	1984.1448

Multiple Comparison Test

Tukey-HSD procedure
Range for the 0.05 level
Table range: 4.40
The value actually compared with Mean (J) - Mean (I) is
113.9941 x Range x Sqrt (1/N(I) + 1/N(J))

(*) Denotes pairs of group significantly different at the 0.05 level

Group	C1	C2	C3	C4	C5	C6	C7	C8
C1		*		*	*		*	*
C2	*		*	*	*	*	*	*
C3		*		*	*		*	*
C4	*	*	*					
C5	*	*	*					
C6		*						*
C7	*	*	*					
C8	*	*	*			*		

Table 24. One-Way Analysis of Variance of Percent Elongation at Break Values for composites (Lengthwise Fiber Direction)

Analysis of Variance Table

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	7	24.5694	3.5099	34.3764	0.0000
Within groups	85	8.6787	0.1021		
Total	92	33.2481			

Group	Count	Mean	Standard Deviation	Standard Error	95% Confidence Interval for Mean		
C1	14	3.15	0.36	0.0962	2.9421	To	3.3579
C2	14	3.35	0.33	0.0882	3.1595	To	3.5405
C3	14	2.28	0.27	0.0722	2.1241	To	2.4359
C4	11	2.06	0.27	0.0814	1.8786	To	2.2414
C5	10	1.81	0.30	0.0949	1.5954	To	2.0246
C6	10	2.39	0.53	0.1676	2.0109	To	2.7691
C7	10	2.21	0.25	0.0791	2.0312	To	2.3888
C8	10	2.44	0.01	0.0032	2.3932	To	2.4472
Total	93	2.52	0.60	0.0623	2.3932	To	2.6408

Multiple Comparison Test

Tukey-HSD procedure
Range for the 0.05 level
Table range: 4.39
The value actually compared with Mean (J) - Mean (I) is
0.2259 x Range x Sqrt (1/N(I) + 1/N(J))

(*) Denotes pairs of group significantly different at the 0.05 level

Group	C1	C2	C3	C4	C5	C6	C7	C8
C1			*	*	*	*	*	*
C2			*	*	*	*	*	*
C3	*	*			*			
C4	*	*						
C5	*	*	*			*		*
C6	*	*			*			
C7	*	*						
C8	*	*			*			

Table 25. One-Way Analysis of Variance of Percent Elongation at Break Values for composites (Crosswise Fiber Direction)

Analysis of Variance Table

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	7	17.2699	2.4671	42.3691	0.0000
Within groups	82	4.7748	0.0582		
Total	89	22.0447			

Group	Count	Mean	Standard Deviation	Standard Error	95% Confidence Interval for Mean
C1	14	1.51	0.04	0.0107	1.4869 To 1.5331
C2	14	2.52	0.07	0.0187	2.4796 To 2.5604
C3	11	1.38	0.34	0.1025	1.1516 To 1.6084
C4	10	1.22	0.11	0.0348	1.1413 To 1.2987
C5	10	1.13	0.09	0.0285	1.0656 To 1.1944
C6	10	1.97	0.51	0.1613	1.6052 To 2.3348
C7	11	1.72	0.24	0.0724	1.5588 To 1.8812
C8	10	1.69	0.22	0.0696	1.5326 To 1.8474
Total	90	1.67	0.50	0.0525	1.5693 To 1.7778

Multiple Comparison Test

Tukey-HSD procedure
Range for the 0.05 level
Table range: 4.40
The value actually compared with Mean (J) - Mean (I) is
0.1706 x Range x Sqrt (1/N(I) + 1/N(J))

(*) Denotes pairs of group significantly different at the 0.05 level

Group	C1	C2	C3	C4	C5	C6	C7	C8
C1		*			*	*		
C2	*		*	*	*	*	*	*
C3		*				*	*	
C4		*				*	*	*
C5	*	*				*	*	*
C6	*	*	*	*	*			
C7		*	*	*	*			
C8		*		*	*			

Table 26. One-Way Analysis of Variance of Flexural Yield Strength Values for composites (Lengthwise Fiber Direction)

Analysis of Variance Table

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	7	2061.0964	294.4423	12.9177	0.0000
Within groups	32	729.3996	22.7937		
Total	39	2790.4960			

Group	Count	Mean	Standard Deviation	Standard Error	95% Confidence Interval for Mean
C1	5	38.27	3.91	1.7486	33.4152 To 43.1248
C2	5	39.20	5.28	2.3613	32.6441 To 45.7559
C3	5	37.25	7.28	3.2557	28.2108 To 46.2892
C4	5	54.63	5.14	2.2987	48.2480 To 61.0120
C5	5	50.22	2.38	1.0644	47.2649 To 53.1751
C6	5	48.36	4.36	1.9499	42.9464 To 53.7736
C7	5	50.00	1.05	0.4696	48.6963 To 51.3037
C8	5	57.04	5.83	2.6073	49.8012 To 64.2788
Total	40	46.87	8.46	1.3375	44.1660 To 49.5765

Multiple Comparison Test

Tukey-HSD procedure
Range for the 0.05 level
Table range: 4.58
The value actually compared with Mean (J) - Mean (I) is
3.3759 x Range x Sqrt (1/N(I) + 1/N(J))

(*) Denotes pairs of group significantly different at the 0.05 level

Group	C1	C2	C3	C4	C5	C6	C7	C8
C1				*	*	*	*	*
C2				*	*		*	*
C3				*	*	*	*	*
C4	*	*	*					
C5	*	*	*					
C6	*		*					
C7	*	*	*					
C8	*	*	*					

Table 27. One-Way Analysis of Variance of Flexural Yield Strength Values for composites (Crosswise Fiber Direction)

Analysis of Variance Table

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	7	1610.0478	230.0068	89.6612	0.0000
Within groups	32	82.0892	2.5653		
Total	39	1692.1370			

Group	Count	Mean	Standard Deviation	Standard Error	95% Confidence Interval for Mean		
C1	5	29.17	1.50	0.6708	27.3075	To	31.0325
C2	5	23.87	1.11	0.4964	22.4918	To	25.2482
C3	5	22.68	0.88	0.3935	21.5874	To	23.7726
C4	5	29.95	1.51	0.6753	28.0751	To	31.8249
C5	5	27.40	0.64	0.2862	26.6053	To	28.1947
C6	5	37.67	1.64	0.7334	35.6337	To	39.7063
C7	5	41.32	2.88	1.2880	37.7441	To	44.8959
C8	5	36.80	1.61	0.7200	34.8010	To	38.7990
Total	40	31.11	6.59	1.0415	29.0009	To	33.2141

Multiple Comparison Test

Tukey-HSD procedure
Range for the 0.05 level
Table range: 4.40
The value actually compared with Mean (J) - Mean (I) is
1.1254 x Range x Sqrt (1/N(I) + 1/N(J))

(*) Denotes pairs of group significantly different at the 0.05 level

Group	C1	C2	C3	C4	C5	C6	C7	C8
C1		*	*			*	*	*
C2	*			*	*	*	*	*
C3	*			*	*	*	*	*
C4		*	*		*	*	*	*
C5		*	*	*		*	*	*
C6	*	*	*	*	*		*	
C7	*	*	*	*	*	*		*
C8	*	*	*	*	*		*	

Table 28. One-Way Analysis of Variance of Flexural Modulus Values for composites (Lengthwise Fiber Direction)

Analysis of Variance Table

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	7	181313884.9	25901983.55	2.1721	0.0638
Within groups	32	381588770.0	11924649.06		
Total	39	562902654.8			

Group	Count	Mean	Standard Deviation	Standard Error	95% Confidence Interval for Mean		
C1	5	10323.23	2133.58	954.1660	7674.0835	To	12972.3765
C2	5	9599.44	2630.12	1176.2254	6333.7676	To	12865.1124
C3	5	13190.40	2724.26	1218.3261	9807.8393	To	16572.9607
C4	5	16235.08	2308.18	1032.2495	13369.1425	To	19101.0175
C5	5	14800.49	4506.99	2015.5872	9204.4135	To	20396.5665
C6	5	13870.73	2759.76	1234.2022	10444.0909	To	17297.3691
C7	5	12712.56	2423.88	1083.9921	9702.9643	To	15722.1557
C8	5	14800.83	6113.41	2734.0001	7210.1520	To	22391.5080
Total	40	13191.60	3799.13	600.6955	11976.5737	To	14406.6163

Multiple Comparison Test

Tukey-HSD procedure
Range for the 0.05 level
Table range: 4.58
The value actually compared with Mean (J) - Mean (I) is
2441.7872 x Range x Sqrt (1/N(I) + 1/N(J))

No two groups are significantly different at the 0.05 level

Table 29. One-Way Analysis of Variance of Flexural Modulus Values for composites (Crosswise Fiber Direction)

Analysis of Variance Table

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	7	333931816.2	47704545.17	8.9728	0.0000
Within groups	32	170131139.2	5316598.101		
Total	39	504062955.4			

Group	Count	Mean	Standard Deviation	Standard Error	95% Confidence Interval for Mean		
C1	5	8075.58	1976.75	884.0295	5621.1605	To	10529.9995
C2	5	5339.27	505.34	225.9949	4711.8177	To	5966.7223
C3	5	7620.57	1438.19	643.1781	5834.8502	To	9406.2898
C4	5	14060.24	2889.86	1292.3847	10472.0631	To	17648.4169
C5	5	9975.42	1289.27	576.5791	8374.6058	To	11576.2342
C6	5	10785.94	3120.44	1395.5032	6911.4648	To	14660.4152
C7	5	12793.20	2724.84	1218.5855	9409.9191	To	16176.4809
C8	5	13361.38	3020.94	1351.0054	9610.4484	To	17112.3116
Total	40	10251.45	3595.09	568.4341	9101.6836	To	11401.2164

Multiple Comparison Test

Tukey-HSD procedure
Range for the 0.05 level
Table ranges: 4.58
The value actually compared with Mean (J) - Mean (I) is
1630.4291 x Range x Sqrt (1/N(I) + 1/N(J))

(*) Denotes pairs of group significantly different at the 0.05 level

Group	C1	C2	C3	C4	C5	C6	C7	C8
C1				*				*
C2				*		*	*	*
C3				*			*	*
C4	*	*	*					
C5								
C6		*						
C7		*	*					
C8	*	*	*					

Table 30. One-Way Analysis of Variance of Impact Strength Values for composites (Lengthwise Fiber Direction)

Analysis of Variance Table

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	7	6499.4524	928.4932	35.3048	0.0000
Within groups	69	1814.6527	26.2993		
Total	76	8314.1051			

Group	Count	Mean	Standard Deviation	Standard Error	95% Confidence Interval for Mean
C1	10	52.61	6.69	2.1156	47.8243 To 57.3957
C2	10	54.18	7.86	2.4856	48.5573 To 59.8027
C3	10	55.91	4.96	1.5685	52.3618 To 59.4582
C4	10	44.02	4.63	1.4641	40.7079 To 47.3321
C5	10	42.91	3.65	1.1542	40.2989 To 45.5211
C6	9	33.44	4.39	1.4633	30.0656 To 36.8144
C7	9	33.03	3.96	1.3200	29.9861 To 36.0739
C8	9	32.43	2.29	0.7633	30.6698 To 34.1902
Total	77	43.98	10.46	1.1919	41.6053 To 46.3532

Multiple Comparison Test

Tukey-HSD procedure
Range for the 0.05 level
Table range: 4.42
The value actually compared with Mean (J) - Mean (I) is
3.6262 x Range x Sqrt (1/N(I) + 1/N(J))

(*) Denotes pairs of group significantly different at the 0.05 level

Group	C1	C2	C3	C4	C5	C6	C7	C8
C1				*	*	*	*	*
C2				*	*	*	*	*
C3				*	*	*	*	*
C4	*	*	*			*	*	*
C5	*	*	*			*	*	*
C6	*	*	*	*	*			
C7	*	*	*	*	*			
C8	*	*	*	*	*			

Table 31. One-Way Analysis of Variance of Impact Strength Values for composites (Crosswise Fiber Direction)

Analysis of Variance Table

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	7	5557.9097	793.9871	24.6166	0.0000
Within groups	70	2257.7875	32.2541		
Total	77	7815.6972			

Group	Count	Mean	Standard Deviation	Standard Error	95% Confidence Interval for Mean		
C1	9	47.67	5.51	1.8367	43.4346	To	51.9054
C2	10	51.40	7.02	2.2199	46.3782	To	56.4218
C3	10	52.26	7.91	2.5014	46.6015	To	57.9185
C4	10	36.88	2.65	0.8380	34.9843	To	38.7757
C5	10	39.71	5.58	1.7646	35.7183	To	43.7017
C6	10	31.13	5.95	1.8816	26.8736	To	35.3864
C7	10	32.42	6.08	1.9227	28.0706	To	36.7694
C8	9	30.73	1.30	0.4333	29.7307	To	31.7293
Total	78	40.30	10.07	1.1408	38.0310	To	42.5741

Multiple Comparison Test

Tukey-HSD procedure
Range for the 0.05 level
Table range: 4.42
The value actually compared with Mean (J) - Mean (I) is
4.0159 x Range x Sqrt (1/N(I) + 1/N(J))

(*) Denotes pairs of group significantly different at the 0.05 level

Group	C1	C2	C3	C4	C5	C6	C7	C8
C1				*		*	*	*
C2				*	*	*	*	*
C3				*	*	*	*	*
C4	*	*	*					
C5		*	*			*		*
C6	*	*	*		*			
C7	*	*	*					
C8	*	*	*		*			

Table 32. One-Way Analysis of Variance of Tensile Strength Values for Matrix Materials

Analysis of Variance Table

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	3	5.3682	1.7894	1.9391	0.1388
Within groups	40	36.9108	0.9228		
Total	43	42.2790			

Group	Count	Mean	Standard Deviation	Standard Error	95% Confidence Interval for Mean
P1	10	30.69	1.59	0.5028	29.5526 To 31.8274
P2	12	31.05	0.23	0.0664	30.9039 To 31.1961
P3	11	31.04	0.74	0.2231	30.5429 To 31.5371
P4	11	30.20	0.90	0.2714	29.5954 To 30.8046
Total	44	30.75	0.99	0.1495	30.4517 To 31.0546

Multiple Comparison Test

Tukey-HSD procedure
Range for the 0.05 level
Table range: 3.79
The value actually compared with Mean (J) - Mean (I) is
0.6793 x Range x Sqrt (1/N(I) + 1/N(J))

No two groups are significantly different at the 0.05 level

Table 33. One-Way Analysis of Variance of Modulus of Elasticity Values for Matrix Materials

Analysis of Variance Table

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	3	2273044.229	757681.4097	31.6631	0.0000
Within groups	40	957180.3940	23929.5098		
Total	43	3230224.623			

Group	Count	Mean	Standard Deviation	Standard Error	95% Confidence Interval for Mean		
P1	10	889.75	147.72	46.7132	784.0775	To	995.4225
P2	12	1342.32	36.32	10.4847	1319.2434	To	1365.3966
P3	11	1234.81	65.51	19.7520	1190.7998	To	1278.8202
P4	11	806.67	265.21	79.9638	628.4995	To	984.8405
Total	44	1078.67	274.08	41.3196	995.3443	To	1162.0020

Multiple Comparison Test

Tukey-HSD procedure
Range for the 0.05 level
Table range: 3.79
The value actually compared with Mean (J) - Mean (I) is
109.3835 x Range x Sqrt (1/N(I) + 1/N(J))

(*) Denotes pairs of group significantly different at the 0.05 level

Group	P1	P2	P3	P4
P1		*	*	
P2	*			*
P3	*			*
P4		*	*	

Table 34. One-Way Analysis of Variance of Percent Elongation at Break Values for Matrix Materials

Analysis of Variance Table

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	3	129908.0811	43302.6937	8.3270	0.0002
Within groups	40	208010.3334	5200.2583		
Total	43	337918.4145			

Group	Count	Mean	Standard Deviation	Standard Error	95% Confidence Interval for Mean		
P1	10	84.37	75.45	23.8594	30.3963	To	138.3437
P2	12	185.32	92.87	26.8093	126.3132	To	244.3268
P3	11	115.07	77.82	23.4636	62.7898	To	167.3502
P4	11	39.22	11.59	3.4945	31.4337	To	47.0063
Total	44	108.29	88.65	13.3643	81.3377	To	135.2409

Multiple Comparison Test

Tukey-HSD procedure
Range for the 0.05 level
Table range: 3.79
The value actually compared with Mean (J) - Mean (I) is
109.3835 x Range x Sqrt (1/N(I) + 1/N(J))

(*) Denotes pairs of group significantly different at the 0.05 level

Group	P1	P2	P3	P4
P1		*		
P2	*			*
P3				
P4		*		

Table 35. One-Way Analysis of Variance of Flexural Yield Strength Values for Matrix Materials

Analysis of Variance Table

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	3	27.9385	9.3128	13.1393	0.0001
Within groups	16	11.3404	0.7088		
Total	19	39.2789			

Group	Count	Mean	Standard Deviation	Standard Error	95% Confidence Interval for Mean		
P1	5	27.22	0.74	0.3309	26.3012	To	28.1388
P2	5	30.03	0.25	0.1118	29.7196	To	30.3404
P3	5	29.01	1.15	0.5143	27.5821	To	30.4379
P4	5	27.32	0.95	0.4249	26.1404	To	28.4996
Total	20	28.40	1.44	0.3215	27.7221	To	29.0679

Multiple Comparison Test

Tukey-HSD procedure
Range for the 0.05 level
Table range: 4.04
The value actually compared with Mean (J) - Mean (I) is
0.5953 x Range x Sqrt (1/N(I) + 1/N(J))

(*) Denotes pairs of group significantly different at the 0.05 level

Group	P1	P2	P3	P4
P1		*	*	
P2	*			*
P3	*			*
P4		*	*	

Table 36. One-Way Analysis of Variance of Flexural Modulus Values for Matrix Materials

Analysis of Variance Table

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	3	17986500.18	5995500.059	6.7941	0.0036
Within groups	16	14119389.08	882461.8175		
Total	19	32105889.26			

Group	Count	Mean	Standard Deviation	Standard Error	95% Confidence Interval for Mean		
P1	5	3814.03	475.39	212.6009	3223.7649	To	4404.2951
P2	5	3692.87	425.28	190.1910	3164.8237	To	4220.9163
P3	5	3608.77	459.70	205.5841	3037.9863	To	4179.5537
P4	5	5888.80	1706.36	763.1074	3770.1086	To	8007.4914
Total	20	4251.12	1299.92	290.6702	3642.7377	To	4859.4973

Multiple Comparison Test

Tukey-HSD procedure
Range for the 0.05 level
Table range: 4.04
The value actually compared with Mean (J) - Mean (I) is
664.2521 x Range x Sqrt (1/N(I) + 1/N(J))

(*) Denotes pairs of group significantly different at the 0.05 level

Group	P1	P2	P3	P4
P1				*
P2				*
P3				*
P4	*	*	*	

Table 37. One-Way Analysis of Variance of Impact Strength Values for Matrix Materials

Analysis of Variance Table

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	3	4574.6060	1524.8687	12.3415	0.0000
Within groups	36	4448.0367	123.5566		
Total	39	9022.6427			

Group	Count	Mean	Standard Deviation	Standard Error	95% Confidence Interval for Mean		
P1	10	100.61	4.77	1.5084	97.1977	To	104.0223
P2	10	104.76	10.94	3.4595	96.9340	To	112.5860
P3	10	110.57	15.17	4.7972	99.7180	To	121.4220
P4	10	128.62	11.03	3.4880	120.7296	To	136.5104
Total	40	111.14	15.21	2.4049	106.2755	To	116.0045

Multiple Comparison Test

Tukey-HSD procedure
Range for the 0.05 level
Table range: 3.81
The value actually compared with Mean (J) - Mean (I) is
7.8599 x Range x Sqrt (1/N(I) + 1/N(J))

(*) Denotes pairs of group significantly different at the 0.05 level

Group	P1	P2	P3	P4
P1				*
P2				*
P3				*
P4	*	*	*	

Table 38. One Way Analysis of Variance of Mechanical Property Values for Composites with Lengthwise Fiber Direction vs. Crosswise Fiber Direction

A. Composite #1 (C1)

1. Tensile Strength

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	1203.1047	1203.1047	534.1197	0.0000
Within groups	26	58.5650	2.2525		
Total	27	1261.6697			

2. Modulus of Elasticity

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	291969.2503	291969.2503	59.6645	0.0000
Within groups	26	127231.4576			
Total	27	419200.7079			

3. Percent Elongation at Break

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	18.8272	18.8272	287.0000	0.0000
Within groups	26	0.0656	0.0656		
Total	27	0.044			

4. Flexural Yield Strength

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	207.0250	207.0250	23.6086	0.0013
Within groups	8	70.1524	8.7691		
Total	9	277.1774			

5. Flexural Modulus

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	12629826.31	12629826.31	2.9859	0.1223
Within groups	8	33838816.72	4229852.089		
Total	9	46468643.02			

6. Impact Strength

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	115.5960	115.5960	3.0435	0.0991
Within groups	17	645.6857	37.9815		
Total	18	761.2817			

B. Composite #2 (C2)

1. Tensile Strength

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	984.6172	984.6172	1405.2911	0.0000
Within groups	26	18.2169	0.7006		
Total	27	1002.8341			

2. Modulus of Elasticity

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	11391694.83	11391694.83	435.7298	0.0000
Within groups	26	679742.4712	26143.9412		
Total	27	12071437.30			

3. Percent Elongation at Break

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	4.8223	4.8223	84.7504	0.0000
Within groups	26	1.4794	0.0569		
Total	27	6.3017			

4. Flexural Yield Strength

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	587.5222	587.5222	40.3650	0.0002
Within groups	8	116.4420	14.5553		
Total	9	703.9642			

5. Flexural Modulus

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	45372621.07	45372621.07	12.6511	0.0074
Within groups	8	28691598.92	3586449.865		
Total	9	74064219.99			

6. Impact Strength

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	38.6420	38.6420	0.6959	0.4151
Within groups	17	999.5400	55.5300		
Total	18	1038.1820			

C. Composite #3 (C3)

1. Tensile Strength

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	511.2313	511.2313	5089.7415	0.0000
Within groups	23	2.3102	0.1004		
Total	24	513.5415			

2. Modulus of Elasticity

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	370267.3060	370267.3060	105.5373	0.0000
Within groups	23	80693.2558	3508.4024		
Total	24	450960.5618			

3. Percent Elongation at Break

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	4.9896	4.9896	54.5519	0.0000
Within groups	23	2.1037	0.0915		
Total	24	7.0933			

4. Flexural Yield Strength

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	530.7123	530.7123	19.7391	0.0022
Within groups	8	215.0912	26.8864		
Total	9	745.8035			

5. Flexural Modulus

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	77557515.57	77557515.57	16.3451	0.0037
Within groups	8	37959932.09	4744991.512		
Total	9	115517447.7			

6. Impact Strength

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	66.6125	66.6125	1.5283	0.2323
Within groups	18	784.5273	43.5848		
Total	19	851.1398			

D. Composite #4 (C4)

1. Tensile Strength

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	48.7845	487.7845	29983.5197	0.0000
Within groups	19	0.3091	0.0163		
Total	20	488.0936			

2. Modulus of Elasticity

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	641190.0885	641190.0885	933.9969	0.0000
Within groups	19	13043.5246	686.5013		
Total	20	654233.6131			

3. Percent Elongation at Break

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	3.6960	3.6960	83.8095	0.0000
Within groups	19	0.8379	0.0441		
Total	20	4.5339			

4. Flexural Yield Strength

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	1522.7560	1301.8810	428.6734	0.0000
Within groups	8	114.7988	3.0370		
Total	9	1637.5548			

5. Flexural Modulus

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	11824822.56	11824822.56	1.7289	0.2250
Within groups	8	54715942.93	6839492.866		
Total	9	66540765.49			

6. Impact Strength

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	254.8980	254.8980	17.9131	0.0005
Within groups	18	256.1346	14.2297		
Total	19	511.0326			

E. Composite #5 (C5)

1. Tensile Strength

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	589.6980	589.6980	378.6791	0.0000
Within groups	18	28.0305	1.5573		
Total	19	617.7285			

2. Modulus of Elasticity

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	759525.3125	759525.3125	187.3648	0.0000
Within groups	18	72967.0500	4053.7250		
Total	19	832492.3625			

3. Percent Elongation at Break

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	2.3120	2.3120	47.1356	0.0000
Within groups	18	0.8829	0.0490		
Total	19	3.1949			

4. Flexural Yield Strength

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	1301.8810	1301.8810	428.6734	0.0000
Within groups	8	24.2960	3.0370		
Total	9	1326.1770			

5. Flexural Modulus

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	58203251.26	58203251.26	5.2972	0.0503
Within groups	8	87900703.97	10987588.00		
Total	9	146103955.2			

6. Impact Strength

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	51.2000	51.2000	2.3033	0.1465
Within groups	18	400.1301	22.2295		
Total	19	451.3301			

F. Composite #6 (C6)

1. Tensile Strength

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	174.6405	174.6405	180.3392	0.0000
Within groups	18	17.4312	0.9684		
Total	19	192.0717			

2. Modulus of Elasticity

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	59111.0645	59111.0645	0.2995	0.5909
Within groups	18	3552686.342	197371.4635		
Total	19	3611797.407			

3. Percent Elongation at Break

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	0.8820	0.8820	3.2606	0.0877
Within groups	18	4.8690	0.2705		
Total	19	5.7510			

4. Flexural Yield Strength

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	285.6903	285.6903	26.3319	0.0009
Within groups	8	86.7968	10.8496		
Total	9	372.4871			

5. Flexural Modulus

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	23789823.36	23789823.36	2.7418	0.1363
Within groups	8	69413684.20	8676710.526		
Total	9	93203507.57			

6. Impact Strength

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	25.2763	25.2763	0.9088	0.3538
Within groups	18	472.7993	27.8117		
Total	19	498.0756			

G. Composite #7 (C7)

1. Tensile Strength

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	66.3855	66.3855	45.2836	0.0000
Within groups	19	27.8539	1.4660		
Total	20	94.2394			

2. Modulus of Elasticity

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	227886.5621	227886.5621	9.0501	0.0072
Within groups	19	478428.3486	25180.4394		
Total	20	706314.9107			

3. Percent Elongation at Break

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	1.2577	1.2577	20.9887	0.0002
Within groups	19	1.1385	0.0599		
Total	20	2.3962			

4. Flexural Yield Strength

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	188.3560	188.3560	40.0890	0.0002
Within groups	8	37.5876	4.6984		
Total	9	225.9436			

5. Flexural Modulus

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	16257.024	16257.0240	0.0024	0.9618
Within groups	8	53199789.12	6649973.64		
Total	9	53216046.14			

6. Impact Strength

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	1.7626	1.7626	0.0654	0.8012
Within groups	17	458.1504	26.9500		
Total	18	459.9130			

H. Composite #8 (C8)

1. Tensile Strength

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	351.1220	351.1220	673.9386	0.0000
Within groups	18	9.3780	0.5210		
Total	19	360.5000			

2. Modulus of Elasticity

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	12700.8000	12700.8000	19.3346	0.0003
Within groups	18	11824.0866	656.8937		
Total	19	24524.8866			

3. Percent Elongation at Break

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	2.8125	2.8125	115.9794	0.0000
Within groups	18	0.4365	0.0243		
Total	19	3.2490			

4. Flexural Yield Strength

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	1024.1440	1024.144	55.9932	0.0001
Within groups	8	146.3240	18.2905		
Total	9	1170.4680			

5. Flexural Modulus

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	5180040.756	5180040.756	0.2228	0.6495
Within groups	8	185999441.2	23249930.16		
Total	9	191179482.1			

6. Impact Strength

Source	Degree of Freedom	Sum of Squares	Mean Square	F Ratio	F Probability
Between groups	1	4087.8882	4087.8882	1179.0681	0.0000
Within groups	16	55.4728	3.4671		
Total	17	4143.3610			

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