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HIGH DENSITY POLYETHYLENE/WOOD FIBER COMPOSITES : MEASURING THE EFFECT OF PROCESSING PARAMETERS ON THEIR PHYSICAL AND MECHANICAL PROPERTIES

presented by

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HIGH DENSITY POLYETHYLENE/WOOD FIBER COMPOSITES : MEASURING THE EFFECT OF PROCESSING PARAMETERS ON THEIR PHYSICAL AND MECHANICAL PROPERTIES

By

Kajiporn Uerkanarak

A THESIS

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ABSTRACT

HIGH DENSITY POLYETHYLENE/WOOD FIBER COMPOSITES : MEASURING THE EFFECT OF PROCESSING PARAMETERS ON THEIR PHYSICAL AND MECHANICAL PROPERTIES

By

Kajiporn Uerkanarak

High-density polyethylene (HDPE) is number one in the total amount used for plastic containers and packages. Recycling of HDPE is preferred but it cannot provide the same properties as pure HDPE does. Lately, the addition of natural fiber reinforcement to improve overall performance has been rising. This study investigated the effects of processing parameters (screw speed and position of fiber introduction) on the mechanical properties of HDPE/wood fiber composites. Tensile properties, impact strength, and water absorption were evaluated.

It was found that fiber-loading at port II provided higher tensile properties than at port I. Higher impact strength was obtained when wood fiber was loaded via port I. In the case of screw speed, the highest tensile properties were obtained when a moderate screw speed was used (100 RPM). The highest impact strength and water absorption were obtained when 80- RPM and 120-RPM were used, respectively.

То

My Dearest Parents Saovanee & Kajonsak Uerkanarak

The Biggest Sister and Brother Sutinee & Thanit Uerkanarak

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Chapter 1

INTRODUCTION

In 1998, approximately 220 million tons of municipal solid waste (MSW) was generated in the United State – an increase of 4 million tons from 1997. Paper and paperboard products made up the largest component of MSW generated (38%) and yard waste comprised the second largest material component (13%). Glass, metals, plastics, wood, and food wastes each constituted between 5% and 10% of the total MSW generated. Rubber, leather, and textiles combined made up about 7% of MSW, while other miscellaneous wastes made up about 2% of the MSW generated in 1998 [1].

The U.S. Environmental Protection Agency's (EPA) integrated waste management hierarchy includes the following three main techniques: <u>Source Reduction</u>, also known as "waste prevention", is the practice of designing, manufacturing, purchasing, or using materials (i.e. products and packaging) in ways that reduce the amount or toxicity of trash created. Reusing items is another way to stop waste at the source because it delays or avoids that item's entry in the waste collection and disposal system [2].

Source reduction, including reuse, can help reduce waste disposal and handling costs, because it avoids the costs of recycling, municipal composting, landfilling, and combustion. It also conserves resources and reduces pollution, including greenhouse gases that contribute to global warming.

Disposal includes waste combustion (incineration) and landfilling [3].

Combustion or incineration is used to reduce waste by a controlled burning process. In addition to minimizing volume, combustors can convert water into steam to fuel heating systems or generate electricity.

In 1997, about 55% of the MSW generated was disposed in landfills. The number of municipal solid waste landfills decreased substantially over the last ten years from about 8,000 in 1988 to 2,314 in 1998 while the average landfill size increased. <u>Recycling</u> is a series of activities that includes collecting recyclable materials that would be considered waste, sorting and processing recyclables into raw materials such as fibers, and manufacturing raw materials into new products [2].

There are three main recycling processes:

Step 1. Collecting and Processing

Collecting recyclables varies from community to community, but there are four primary methods: curbside, drop-off centers, buy-back centers, and deposit/refund programs. Then, recyclables are sent to a materials recovery facility to be sorted and prepared into marketable commodities for manufacturing.

Step 2. Manufacturing

Once cleaned and separated, the recyclables are ready to undergo the second part of the recycling loop, manufacturing. Recycled materials are used in common household items such as aluminum, plastic, and glass soft drink containers; and plastic laundry detergent bottles.

Step 3. Purchasing Recycled Products

Purchasing recycled products completes the recycling loop. By "buying recycled", governments, businesses and individual consumers each play an important role in making the recycling process a success.

During the past few decades, recycling has become popular and successful. Analysts project that Americans will be recycling at least 83 million tons, or 35% of all municipal waste, by 2005. In 1998, 27% of MSW was recovered and recycled [1].

While recycling has generally grown, recycling of specific materials has dramatically increased: 42% of all paper, 35.5% of all plastic soft drink bottles, 59.5% of all of aluminum beer and soft drink cans, 61% of all steel packaging, 92% of all automobiles and 64.3% of all major appliances were recycled in 1998 [1].

For the products in MSW, containers and packaging comprised the largest portion of products generated (33%). Nondurable goods were the second largest portion (27%). The third category is durable goods, which comprised 16% of total MSW generation [1].

Plastics are a rapidly growing segment of MSW. Plastics are found in durable and nondurable goods and in containers and packaging, with the latter being the largest category of plastics in MSW [3].

In durable goods, plastics are found in appliances, furniture, casings of lead-acid batteries, and other products. A wide range of resin types is found in durable goods.

Plastics are found in such nondurable products as disposable diapers, trash bags, cups, eating utensils, medical devices, etc. The plastic foodservice items are generally made of clear or foamed polystyrene, while trash bags are made of high-density polyethylene or low-density polyethylene.

Plastic resins are also used in a variety of container and packaging products such as polyethylene terephthalate (PET) soft drink bottles and high density polyethylene (HDPE) bottles for milk and water. Table 1 shows the six types of resins that are most often used in container and packaging products.

PET	14.44
HDPE	37.90
PVC	4.46
LDPE/LLDPE	30.15
РР	10.19
PS	2.23
Other resins	0.63

Table 1Plastic containers and packaging by resin type (%) [3]

High-density polyethylene (HDPE) is number one in the total amount used. HDPE is commonly used in packaging containers such as milk, juice and water containers [4]. These rigid containers are easy to separate and collect, thus making them become one of the top recycled materials. Recovery of high-density polyethylene milk and water bottles was estimated at about 31.3% in 1997 [3].

A number of recycling techniques have been developed to obtain well-sorted resins that can substitute for or blend with virgin resins in many applications [5]. There have been investigations of utilizing the recycled resins with reinforcements in the form of composite materials. The addition of up to 40% by weight of fiber reinforcement is common [6]. The step of compounding is also important to be considered. Compounding is a process of combining a number of different components into one. These different components combine to form a new material with properties of its own which are not necessarily those of its constituents. Therefore, the purpose of this study was to investigate the effect of processing parameters that are most suitable for the composite materials of resin and wood fiber, to evaluate the mechanical performance such as tensile strength and impact strength, as well as the effect of water absorption, of composite materials, and to compare the mechanical properties of composites when using different processing parameters.

Natural organic reinforcements such as wood fiber are slowly penetrating the fiber-reinforced thermoplastics market, presently dominated by glass fiber and other mineral reinforcements. This is the result of their low cost, low density, acceptable specific strength properties, renewable nature, comparative ease of processability, enhanced energy recovery, and biodegradability [7, 8, 9]. However, they have disadvantages in that they cannot withstand high temperature because of their low degradation temperature (200 °C), which restricts the range of plastic materials to be combined with, to those having low melting temperatures. The second is their water sorption, which weakens their adhesion to hydrophobic polymer matrices [9].

In this study, high density polyethylene functions as the matrix, and wood fiber from aspen hardwood is used as the reinforcement. HDPE was selected because of its high recycling rate. Wood fiber is a choice because it is plentiful, lightweight, non-toxic and has great strength [10]. It is also low cost, about 40% of the cost of glass fiber [11].

The processing parameters were varied, based on the different ports that were used for fiber loading and the different screw speeds of the extruder when loading the wood fiber through each port. The variations show how each processing parameter affects the mechanical performance of the composite materials and which parameter was the most suitable for the composites. The combination of resin and wood fiber was maintained at 60% resin and 40% wood. To control for variation in the quality of recycled resins, virgin resin was used [12]. Product performance was evaluated by the mechanical properties of tensile strength and impact strength and the physical property of water absorption.

Chapter 2

LITERATURE REVIEW

2.1 Background in Composite Materials

Demands of materials imposed by today's advanced technologies have become so diverse and severe that they often cannot be met by simple single-component materials acting alone. Therefore, it is necessary to combine several materials into a composite in which each constituent not only contributes its share, but also combines the action of the individual properties and provides new performance unattainable by the individual constituents [13].

Such composite material systems result in better overall performance and offer the great advantage of a flexible design; that is one can, in principle, tailor-make the material as per specifications of an optimum design [14].

When comparing conventional monolithic materials such as aluminum and steel with composite materials, it is found that composites provide the highest strength and stiffness as well as fatigue resistance, and they have the lowest thermal expansion. One important advantage of composites is their light weight compared with the heavy weight of steel.

The word composite has evolved over a long period. It may be considered as a system or process of combining two or more reinforcing materials in a matrix binder. On the other hand, it can be considered to be a new material having characteristics derived from its processing and from its microstructure [15].

Composites can be basically divided into two forms: (1) composite materials and (2) composite structures. Composite materials are composed of a reinforcing material, surrounded by a continuous matrix. They must be capable of arbitrary variation while composite structures are not capable of arbitrary variation.

Most composite materials have been produced to improve mechanical properties such as strength, stiffness, toughness, and high-temperature performance. Thus, it is usual to study the composites that have a common strengthening mechanism. The strengthening mechanism strongly depends on the geometry of the reinforcement. Therefore, it is quite convenient to classify composite materials on the basis of the geometry of a reinforcing material [16].

A common classification of composite materials consists of two types: (1) particle-reinforced composites and (2) fiber-reinforced composites. The distinguishing characteristic of a particle is that it is naturally nonfibrous. It may be spherical, cubic, tetragonal, a platelet, or of other regular or irregular shape, but it is approximately equiaxial. Particle-reinforced composites are sometimes referred to as *particulate composites*.

A fiber is characterized by its length being much greater than its cross-sectional dimensions. Fibers are small in diameter and will bend easily when pushed axially. Although they may have outstanding tensile strength, they must be supported to keep individual fibers from bending and buckling [15]. Fiber-reinforced composites are, understandably, called *fibrous composites* [16].

Three basic components in a fiber-reinforced composite are polymer matrices, reinforcements and interfaces [17].

2.1.1 Matrix

The matrix is the material that gives body, and grips or holds the reinforcements of the composite together. It usually has lower strength than the reinforcement. The matrix must be capable of being forced around the reinforcement during some stage in the composite process. The main functions of the matrix are (i) to protect the fiber from exposure to the environment as well as against fiber abrasion, (ii) to transfer and distribute stress loads onto the fiber, and (iii) to separate and keep fibers in the desired location and orientation [18, 19].

Polymers are generally used for matrices. They are found occurring in nature as amber, pitch, and resin; and a variety of synthetic polymers are available. Polymers are selected because they are easily processed and offer good mechanical and dielectric properties. They can wet the reinforcements as well, resulting in good adhesion. Although polymers have lower softening points than metals, they are low-density materials. It is because of the relatively low processing temperatures and production techniques that many organic reinforcements may be used [15].

All polymers are either thermoplastic or thermosetting. Probably one of the earliest distinctions between polymers was based on their reaction to heating and cooling. Thermoplastics are polymers that can be made to flow when heated and become solid when cooled. These materials may be softened repeatedly by heat and shaped into useful products. Most thermoplastic materials, including scrap or damaged pieces, may be

recycled. Continued heating above their melting points will cause them to degrade. Acrylics, cellulosics, polyamide, polystyrene, polyethylene, fluoroplastics, polyvinyls, polycarbonate, and polysulfone are examples of the thermoplastic group. Thermoplastic materials come in a variety of available forms and are generally fully polymerized.

Thermoplastic polymers in composites have high impact strength and higher resistance to failure, which provide a better withstanding of matrix microcracking in composite laminates. However, compared to thermoset matrices, thermoplastic polymers have developed slowly because of their high melt or solution viscosity, causing difficulty in incorporation of continuous fibers into the matrix [17].

Thermosetting materials cannot be reshaped or reformed once the material is set into a final structural framework. The heating and forming process causes them to undergo a curing reaction. Thermosets will char, burn, or degrade by continued heating, but they do not remelt. Thermosetting materials are particularly useful in producing many composite materials because they are available in a variety of forms. Thermosetting resins in a partially polymerized liquid state may facilitate penetration and wetting of the other constituents. Members of the thermosetting group include aminos, casein, epoxies, phenolics, polyesters, silicones, and polyurethanes.

2.1.2 Reinforcement

The reinforcement of thermoplastics and thermosets by ceramic, metallic or polymeric fibers is very important. It is easily achieved and the composite formation is often cheaper than the polymer matrix alone. These reinforced grades marry the strength and stiffness of such fibers with the good shock resistance of the thermoplastic matrix.

The fibers alone are usually very brittle and their strength and stiffness cannot be fully utilized. The matrix protects these fibers and transfers the load to them. This gives a material that combines the good properties of the fiber and the matrix, producing an improvement in the strength, stiffness and creep resistance over those properties for the matrix alone. These composite materials offer good competition to metals in many applications in the motor and aerospace industries and in domestic appliances.

The strengthening mechanism depends on the geometry of the reinforcing filler, which may be one of two types, particulate or fibrous. A particulate filler has no long dimension, platelets being a noted exception. As a long dimension discourages the growth of incipient cracks normal to the reinforcement in a brittle matrix, a particle does not improve the fracture toughness of such a matrix. The exception to this rule is when a rubberlike substance is dispersed in a brittle matrix. Under these conditions, considerable toughening occurs, and this method is standard for improving the impact behaviour of thermoplastics. Typical examples are high-impact polystyrene and ABS.

The particles will also share the load with the matrix, but to a lesser extent than a fiber. A particulate reinforcer will, therefore, improve stiffness but will not generally strengthen. Hard particles in a brittle matrix will cause localized stress concentrations in the matrix, which will reduce the overall impact strength.

Particulate fillers are employed to improve high-temperature performance, reduce friction, increase wear resistance, improve machinability and reduce shrinkage. In many cases, particulate fillers are used simply to reduce the cost. Under these conditions, the additive is a filler, whereas when a considerable improvement in properties of the composite occurs, the additive is a reinforcement.

Fiber reinforcement improves the three main weaknesses of a thermoplastic matrix: stiffness, strength and creep resistance. The measured strength of most materials is much less than that predicted by theory because flaws in the form of cracks perpendicular to the applied load are present in bulk materials. Fibers of non-polymeric materials have much higher longitudinal strengths in this form because the large flaws are not generally present in such small cross-sectional areas. However, these small crosssectional areas do not permit the use of fibers alone in engineering applications [20].

Composites reinforced with fibers are anisotropic, and properties depend on the direction of the stress [15]. There are three types of fibrous reinforced composites, namely particulate, continuous and discontinuous fibers [21].

Particulate composites are made of different sizes and shapes of particles randomly dispersed in the matrix. Due to the random distribution of particles, these composites can be considered as quasi-homogeneous on a scale larger than the particle size. Particulate composites may contain either nonmetallic or metallic particles in a nonmetallic or metallic matrix. Examples of this type are concrete and glass reinforced with mica flakes.

Continuous fiber composites are reinforced by long continuous fibers and are the most efficient for stiffness and strength. They also have greater strength and modulus in the fiber axis direction and generally lack physical strength in the transverse direction. The continuous fibers can be all parallel, can be oriented at right angles to each other, or can be oriented along several directions.

Discontinuous composites consist of short fibers or whiskers in the reinforcing phase. These short fibers can be either all oriented along one direction or randomly

oriented. In a discontinuous fiber composite, the stress along the fiber is not uniform. The length (l) to diameter (d) ratio of the fiber, commonly called the aspect ratio (l/d), determines the level of strength that the composite will reach. If the fiber is shorter than the critical length, the composite will fail at a low strength level [19, 21]. These short fiber reinforced thermoplastics are very important in replacing metals in applications requiring a combination of strength and lightness [20]. The principal fibers in commercial use are various types of glass and carbon [17].

2.1.3 Interface

The behavior of a composite material is a result of the combined behavior of the following [14]:

- (1) Fiber or the reinforcing element
- (2) Matrix

(3) The interface between the fiber and the matrix

To obtain desirable characteristics in the composite material, it is important that the fibers are not weakened by flaws (surface or internal) and the applied load is effectively transferred from the matrix to the fibers via the interface. Clearly, the interface generally has an important bearing in this situation. Specifically, in the case of a fiber reinforced composite material, the interface, commonly called the interfacial zone, consists of near surface layers of fiber and matrix and any layer(s) of material existing between these surfaces [14].

The reason why the interface is very important is that the internal surface area occupied by the interface is quite extensive. It can easily go as high as $3000 \text{ cm}^2/\text{cm}^3$ in a

composite containing a reasonable fiber volume fraction. Thus, it becomes extremely important to understand what exactly is going on in the interface region of any given composite system under a given set of conditions.

Wettability of the fiber by the matrix and the type of bonding between the two components constitute the primary considerations. Additionally, on should determine the characteristics of the interface and how they are affected by temperature, diffusion, residual stresses, and so on.

2.2 Natural Fiber Reinforced Thermoplastics

Originally, the purpose of fiber reinforcement was to improve the strength, stiffness and creep resistance of polymer matrices. It is interesting to analyze how well these intentions have been realized, to consider any additional, perhaps unexpected advantages accruing due to reinforcement, and to study the disadvantages that are involved in reinforcement.

Wood fibers are one of the potential reinforcements. They are naturally plentiful organic fiber [22]. In 1997, there were about 43.9 percent by weight of wood, paper and paperboard generated in MSW [3]. Wood fibers also offer strength that is close to that of the traditional reinforcing materials. The strength and modulus of wood pulp fibers are comparable with those of glass fibers at the same unit weight. The wood fiber composites show the same or higher stiffness per weight than the steel, aluminum, glass fiber composites and talc-contained polyolefins [23]. As a result, cost effectiveness is an outstanding advantage. The other benefits are low abrasion to machinery, no hazardous substance generation, low density and the possibility of surface modification [24].

However, there are some limitations. The low processing temperature, about 200°C, of wood fibers makes them unavailable for some polymers that require high melting temperatures. The water sorption of wood fibers causes a weak interface between the matrix and wood fiber [9], and leads to biodegradation after repeated exposure [11].

Wood fiber composites can be made by extrusion, compression, or injection molding to form a variety of products that can be used in packaging, paper products, building materials, automobile parts, etc. [25].

Composites of thermoplastic polymers and wood fiber yield poor mechanical properties due to the incompatibility between them. The thermoplastics, especially polyolefins, are hydrophobic whereas wood fibers are hydrophilic. The difference causes poor fiber distribution into the matrix and poor interfacial bonds between fiber and matrix. As a result, the fiber strength cannot contribute to the composite strength as much as it is supposed to. This phenomenon leads to easy failure of composites in mechanical testing [9].

According to Chtourou et al. [9], the interfacial bond between matrix and fiber plays an important role in the improvement of mechanical properties. It can be contributed to by five mechanisms: adsorption and wetting, interdiffusion, electrostatic attraction, chemical links, and mechanical adhesion. In the case of composites consisting of hydrocarbon polymer matrices and wood fibers, wetting and mechanical adhesion may be the main influences.

2.3 Compounding

Compounding is a process of combining a number of different components into one. These different components combine to form a new material with properties of its own which are not necessarily those of its constituents [26].

The extruder is an versatile machine that can be used to form composite materials. The extrusion process melts and mixes powdered or granular polymers into a continuous melt. The melt stream is then sent to the die, mold, or accumulator. Screw technology determines the output, milling rate, die pressure, and type of material that is to be plasticated. The screw rotates in the extruder barrel. The length (L) and barrel diameter (D) are expressed as an L/D ratio.

For many operations, multiple-screw extruders are used, for example, in the compounding and extrusion of many reinforced thermoplastic and thermosetting composite materials. With twin-screw machines, it is possible to introduce continuous strands of reinforcement at the hopper. The screw will break the fibers into short lengths. Adding fibers to a premelted polymer will reduce breakage and extruder wear, while increasing dispersion and fiber wet out.

Co-rotating, twin-screw extruders are very commonly used in the plastic industry for various compounds. They represent the standard for engineering thermoplastics and polymer alloy compounding. They can also be used in polymers ranging from high volume polyolefins to high performance liquid crystal polymers to specialty small-lot operations [26].

The primary advantages of these extruders are their modularity that makes them very flexible and adaptable to specific process requirements, coupled with their excellent

distributive and dispersive mixing. They also have short and narrow residence times, which can be as short as 5 to 10 seconds for high speed, high torque applications. Such features allow rapid purging and turnaround times.

The principal disadvantages are that they generate high peak shear rates and are inefficient as melt pumps. The higher shear rates can be a significant problem at higher screw speeds.

For the instrumentation control, the most important process parameters are melt pressure and temperature. They are the best indicators of how well or how poorly an extruder functions. Other important process parameters are: screw speed, motor load, barrel temperatures, die temperatures, power draw of the various heaters, cooling rate of the various cooling units, and vacuum level in vented extrusion [27].

2.4 **Prediction of Properties**

There are a number of variables that influence the properties of fibrous reinforced composite materials and structures: (1) interface bond between matrix and fiber, (2) properties of the fiber, (3) size and shape of the fiber, (4) loading of fiber, (5) processing technique, and (6) alignment or distribution of the fiber.

The prediction of mechanical properties of aligned, long fibers is difficult to achieve theoretically and further complications exist in dealing with short fiber reinforcement. This is due to the possibility of a spectrum of fiber lengths and orientations caused by processing into the final part.

The prediction of tensile modulus and tensile strength for long fiber reinforcement will be shown in the following equation [20]:

$$E_{c} = E_{m}\phi_{m} + E_{f}\phi_{f} \qquad (1)$$

$$\sigma_{\rm c} = \sigma_{\rm m}\phi_{\rm m} + \sigma_{\rm f}\phi_{\rm f} \tag{2}$$

Where;	E	=	tensile modulus
	σ	=	tensile strength
	φ	=	volume fractions
and subscrip	ots c	=	composite
	f	=	fiber
	m	=	matrix

Equation (1) represents a simple rule of mixtures, which gives values of E_c for long fiber composites close to values from more rigorous theoretical models and to actual experimental values. If the values of the Poisson's ratio, γ , of the fibers and the matrix are not identical, equation (1) is about 1% in error. For most thermoplastic matrices γ is approximately 0.4.

In the case of continuous fiber reinforcement all the fibers are working at maximum efficiency, with the average strain in the fiber being equal to that in the matrix. In aligned short fiber composites this is not so. This is because the fiber restricts the deformation of the surrounding matrix because it is stiffer than the matrix material. The load is transferred from the matrix to the fiber via the interfacial shear stresses. Thus, the calculation of the variation of the shear and tensile stresses along a short elastic fiber in an elastic matrix are included in the prediction of the tensile modulus of the composite.

The shear stress is greatest at the ends of the fiber and decays to zero somewhere along it. The tensile stress is zero at each end of the fiber and reaches a maximum at the center. If the fiber is just long enough, the maximum tensile stress reaches the tensile stress in the matrix. The ratio $(L/D)_c$ that occurs under these conditions is called the critical elastic aspect ratio. For values of L/D less than $(L/D)_c$ the tensile stress in the fiber is always less than that in the matrix, and clearly under these conditions the transfer of load from the matrix to the fiber is poor and the mechanical properties of the fiber are not fully utilized.

If $L/D > (L/D)_c$, the tensile stress at the interface remains at a maximum over a greater proportion of the fiber length. Here, the transfer of stress the matrix to the fiber is very efficient but the average tensile stress in the fiber is always less than that in the matrix because of the reduced tensile stress at the ends of the fiber. The efficiency of stress transfer is, therefore, never 100%.

In order to accommodate this change in reinforcement efficiency with fiber length, the term η_L , the length correction factor, is considered. Therefore, the tensile modulus of the composite can be predicted by the following equations [20]:

$$E_{c} = \eta_{L} E_{f} \phi_{f} + E_{m} \phi_{m}$$
(3)

where;

$$\eta_L = \frac{\left[1 - \tanh(\beta L/2)\right]}{(L/2)} \tag{4}$$

where L is the fiber length and

$$\beta = \frac{\sqrt{2\pi G_m}}{\left[E_f A_f \ln(R/r)\right]} \tag{5}$$

where;
$$G_m =$$
 shear modulus of the matrix
 $r =$ the radius of the fiber
 $R =$ the mean separation of the fibers normal to their
length
 $A_f =$ the cross-sectional area of all the fibers in the
composite

It is very important to remember that E_c depends on L/D rather than on L alone.

For the prediction of tensile strength for short fiber reinforcement, the following equation can be utilized with an additional factor of average tensile stress on the composite:

$$\sigma_{\rm c} = \sigma_{\rm m} \phi_{\rm m} + \sigma_{\rm f} \phi_{\rm f} \tag{6}$$

where σ_f is the average fiber stress, which is given by

$$\sigma_f = \frac{1}{L} \sigma f(x) dx \tag{7}$$

If the tensile stress builds up from the fiber ends in a non-linear way, then the tensile strength can be calculated by the following equation:

$$\sigma_f = \sigma_{fx} \left[1 - \left(1 - \beta \right) \frac{L_c}{L} \right] \qquad \text{for } L > L_c \qquad (8)$$

where; σ_{f} = the tensile stress in a continuous fiber in the same matrix under the same loading conditions. $\sigma_{f^{\infty}}$ = the average stress in the discontinuous fiber within a distance L_c/2 of either end L_c = critical fiber length When the fiber length is greater than the critical fiber length, it is assumed that the fiber failure occurs when $\sigma_f = \sigma_{f^{\infty}}$. Substituting the equation (8) in equation (6) gives the following equation:

$$\sigma_{c} = \sigma_{fx} \left[1 - \left(1 - \beta \right) \frac{L_{c}}{L} \right] \phi_{m} + \sigma_{m} \phi_{m}$$
(9)

A comparison between equation (2) and equation (9) shows that discontinuous fibers provide composites of less strength than continuous fibers. The value of L_c represents the shortest fiber length that may be broken in a given matrix. Fibers below this minimum length are not capable of receiving sufficient tensile stress to break them and composite failure occurs because of failure at the fiber-matrix interface.

The tensile strength of a short fiber reinforced thermoplastic decreases as the angle between the fiber axis and direction of loading increases. The tensile strength of the composite in a transverse direction is often less than that of the matrix material owing to the effect of the fibers.

Impact strength of a composite does not have a simple relationship with adhesion between filler and polymer, so it is very difficult to predict the impact strength of short fiber reinforced composites. When brittle fibers are added to brittle matrices, the impact strength of the composite is often reduced. This is because the matrix is bound by the fibers and cannot deform to absorb the impact load.

2.8 **Previous Research**

Several researchers have studied the properties of composites of polymer matrices with reinforcements from natural resources like cellulose fibers, wood fibers, and paper fibers. Some of those works are summarized as follows.

Yam et al. [28] investigated the mechanical properties of wood fiber/recycled HDPE composites in comparison to wood fiber/virgin HDPE. Aspen fiber, a hardwood, and spruce fiber, a softwood, were used. Recycled HDPE was made from chopped postconsumer milk bottles. The fiber and polymer were extruded through a co-rotating intermeshing twin screw extruder at 150°C. The extrudates were compression molded at 150°C and 4.22 MPa for 10 minutes and cooled under pressure for 15 minutes. The tensile strength and the elastic modulus of composites made from recycled HDPE and wood fiber were about the same as those of composites made from virgin HDPE. There was also no significant difference in mechanical properties between the hardwood composites and the softwood composites.

Kalyankar [29] investigated the mechanical properties of composites made from recycled HDPE obtained from milk bottles. Aspen fiber, a hardwood, was used. Recycled HDPE was made from chopped post-consumer milk bottles. Melt mixing of wood fibers with HDPE in a twin screw extruder was employed and gave a uniform blending. Mechanical property evaluation showed that the tensile modulus increased with increase in wood fiber content. The tensile strength, elongation at break and impact strength decreased. The composite also showed fair stability of dimensions and tensile strength at equilibrium water absorption. Addition of ethylene vinyl acetate (EVA) copolymer as a bonding agent improved the impact strength of the composite, but not the tensile strength.

Gogoi [25] investigated the effects of fiber pre-treatment, screw configuration of a twin-screw extruder, and compounding temperature on the mechanical properties of composites. Granulated HDPE milk bottles were used as the polymer matrix, while aspen wood fiber was used as the filler. The results showed that tensile strength decreased with an increase in fiber content. The effect of fiber pre-treatment in terms of tensile and flexural yield strength showed that acetylated and untreated aspen fibers were better than heat treated. The mechanical properties of the composite were sensitive to screw configuration and temperature.

Neiman [30] studied the mechanical properties of recycled HDPE and wood fiber composites. Five additives were used: low density polyethylene (LDPE), stearic acid, chlorinated polyethylene, maleic anhydride modified polypropylene (MAPP) and ionomer modified polyethylene. For tensile strength and modulus, only maleic anhydride modified polypropylene showed potential for improving adhesion between the polymer matrix and wood fibers. Ionomer modified polyethylene also displayed some positive results, while the others were determined ineffective for enhancing properties.

Keal [31] conducted research on the effect of dual additive systems on the mechanical properties of composites of wood fibers and recycled HDPE milk containers. The additive systems were two of stearic acid, maleic anhydride modified polypropylene and ionomer modified polyethylene; thus three combinations were used. The use of additives did improve tensile properties and creep but decreased impact strength. Only the stearic acid/ionomer additive system did not reduce impact strength. Compared to the

effect of single additives, none of the dual additive systems provided significantly better improvement.

Chtourou et al. [9] studied composites of recycled polyolefins and wood fibers. The polyolefins were 95% PE and 5% PP. A mixture of 45% spruce, 45% fir and 10% poplar produced by chemico-thermomechanical pulp (CTMP) was used as a reinforcement. The composites were made by injection molding and compression molding. The impact of fiber concentration, the effect of fiber surface modification by acetic anhydride and phenol formaldehyde, and the effect of moisture exposure on the composites were evaluated by tensile properties. The result was that the greater the nontreated fiber percentage, the higher the Young's modulus and the strength at yield. More than 30% fiber could be incorporated into the composites and at 30% fiber content by weight, Young's modulus increased 150%. Improvement in tensile strength was observed in the composites with 10% treated fiber. 10% treated fiber composites also displayed lower water sorption and higher mechanical properties than 10% non-treated fiber composites.

Shan Ren and David N.-S. Hon [32] evaluated the effects of components, processing and additives on the mechanical properties of composites made of newspaper fiber and polypropylene (PP). The test specimens were made by a mixing and molding process. The increase in the fiber from 0 to 10% proportionally reduced the strength. Then, the strength leveled off at 10 to 50% fiber content and started decreasing again when the fiber content was greater than 50% as a result of poor matrix-reinforcement adhesion. In contrast, the modulus of elasticity increased proportionally to the percent of fiber concentration. The optimum elastic modulus was achieved at the range of 40 to 50% fiber content. The different types of reinforcement, which were commingled newspaper fiber, TMP and chemical wood pulp, were also tested and showed an improvement of elastic modulus. The effect of processing temperature revealed that the higher the temperature, the higher the strength. The optimum temperature was between 190 and 205 °C. The tensile strength also increased proportionally to the addition of additives but there was no significant effect on modulus of elasticity. From the scanning electron micrographs, pulled-out fibers at the broken surface after tensile testing were evident. The presence of stretched fibers confirmed the poor interfacial bonding between matrix and fiber.

Chotipatoomwan [33] tested the processing and mechanical properties of paper fiber and high density polyethylene (HDPE) composites. Two kinds of paper fibers, mixed and deinked paper fibers, were used as fillers in composites. The mechanical and physical properties were studied by varying the fiber content and using different kinds of fibers at the same processing conditions. The addition of paper fibers to HDPE caused a decrease in tensile strength and impact strength, but an increase in tensile modulus. Water absorption increased due to the addition of paper fibers. Mixed paper fiber composites had higher strength than deinked paper fiber composites.

Rojanarungtawee [12] studied the effect of mixed resins in different proportions on the mechanical properties of a plastic/wood fiber composite. Polypropylene (PP) and high density polyethylene (HDPE) represented the mixed matrices. Aspen wood fiber served as the reinforcement. The composition of PP and HDPE was varied from 0% to 100% by weight. The mixed resins and wood fiber were compounded in a constant ratio
of 60% matrix and 40% reinforcement by a twin screw extruder operating at the PP and HDPE melting points, 180°C and 150°C, respectively. At 180°C, the maximum ultimate tensile strength was achieved at 30% PP/70% HDPE. 10% PP/90% HDPE had the highest modulus of elasticity. At 150°C, only two variations of 0% PP/100% HDPE and 30% PP/70% HDPE were processable. The comparison between two ratios revealed that the ratio of 0% PP/100% HDPE produced higher tensile strength than the ratio of 30% PP/70% HDPE did. The modulus did not differ significantly at the two compositions investigated.

Ricciardi [34] studied the impact of additives on the physical and mechanical properties of high density polyethylene and paper fiber composites. The additives used were maleic anhydride modified HDPE (MAHDPE), low molecular weight polypropylene (Proflow 1000), and low density polyethylene (LDPE) with a high melt flow index. The fiber loading level remained at approximately 35%. The effects of MAHDPE were studied at 3%, 6% and 10%. The effects of Proflow 1000 and LDPE were studied at 5% and 10%, while keeping the MAHDPE at 6%. The additives had no significant effect on modulus of elasticity, percent elongation, and Izod impact strength. MAHDPE was found to improve yield strength (at 10%) and tensile strength (at 3%), and appeared to decrease debonding in water (at 3%, 6% and 10%).

Recently, Thepwiwatjit [35] investigated the mechanical properties of composites of wood fiber and recycled HDPE bottles from household use. Aspen wood fiber was used as the reinforcement. The composite materials were made by combining the resins and wood fibers in 5 different ratios; 100% HDPE/0% fiber, 90% HDPE/10% fiber, 80%

HDPE/20% fiber, 70% HDPE/30% fiber, and 60% HDPE/40% fiber, and 60% virgin HDPE resin with 40% fiber. The compounding speed of the extruder was set at 120 RPM and the temperature of all 6 processing zones was set at 150°C. It was found that an increase of wood fiber content did not improve the tensile properties of the composites. With an increase in the fiber concentration, the tensile strength, yield strength and % elongation decreased, while the modulus and impact strength slightly increased. The fiber fraction in the composites had a very large effect on water absorption. The more the fiber content, the higher was the gain in weight.

Chapter 3

MATERIALS

High density polyethylene (HDPE) in pellet form was provided by Paxon Polymer Company under the trade name Paxon®AD 60-007. It was a virgin HDPE homopolymer having a medium molecular weight distribution. The virgin thermoplastic was able to be a substitute for the recycled HDPE because the properties of virgin HDPE composites were the same as those of recycled HDPE composites, according to Yam et al.[28].

HDPE is a milky-white nonpolar, linear thermoplastic. Its density ranges from 0.940 to 0.965 g/cm³. It is one of the most versatile polymers, and is the second most commonly used plastic in the packaging industry. The molecular chains of HDPE homopolymer are long and straight with little branching. This close packing produces HDPE with a crystallinity of 65-90% and contributes to HDPE's good moisture-barrier properties, chemical resistance and to its non-transparency. Glass transition and melting temperatures of HDPE are about -80±10 °C and 138 °C, respectively [12].

Aspen wood fiber was used as the reinforcement. Hardwoods are categorized in the subdivision angiospermae. Hardwood leaves are broad and change color in the fall season in temperate areas. Aspen is a generally recognized name applied to bigtooth aspen (*Populus grandidentata*) and to quaking aspen (*P. tremuloids*). Aspen wood is usually straight grained with a fine, uniform texture. It is easily worked. Well-seasoned aspen lumber does not impart odor or flavor foodstuffs. The wood of aspen is light

weight and soft. It is low in strength, moderately stiff, moderately low in resistance to shock, and has moderately high shrinkage [11].

Aspen hardwood fibers in this experiment were in the form of thermomechanical pulp (TMP). In this mechanical pulping process, wood chips are fed into a refiner at about 120°C, which grinds and defibrillates the chips into fibers. There is only a minimum amount of damage to the lignin or hemicellulose during this pulping process, so the wood fibers retain nearly all of their lignin and natural waxes, which can help in better dispersion of wood fiber into the non-polar hydrocarbon polymer matrix [23].

The fibers were conditioned for at least 40 hours at $23 \pm 2^{\circ}$ C and $50 \pm 5\%$ RH prior to the testing.

Chapter 4

METHODS

Extruding the Composite Material

Before extruding the composite material, the feed rate to the extruder was calibrated. It is important to know that the feed rates were impacted by the maximum amount of fiber that could be pushed down into the port. It was also affected by the screw speed of the extruder. In this experiment, there were 3 different screw speeds (80, 100 and 120 RPM), so the fiber feed rates were different for each screw speed. Using the following equation, the percent fiber-loading level was set:

$$\frac{X}{X+Y} = 0.4$$

Where:	Х	=	Feed rate of wood fiber (g/min)
	Y	=	Feed rate of resin (g/min)
	0.4	=	The chosen percent fiber loading of the composite

The feed rate of the resin was found experimentally by cutting and weighing the resin every minute. The feed rate of resin was then substituted into the above equation to calculate the fiber feed rate.

Screw Speed	Measured Resin Feed	Average (g/min)	Calculated Fiber
(RPM)	Rate (g/min)		Feed Rate (g/min)
	15.31		
80	16.40	15.91	10.61
	16.02		
	15.89		
100	16.05	15.93	10.62
	15.84		
	16.13		
120	15.97	16.29	10.86
	16.78		

<u>Table 2</u> Determination of the feed rate of fiber at each screw speed of the extruder

A Baker Perkins Model ZSK-30, 30 mm, 26:1 co-rotating twin-screw extruder (Werner & Pfleiderer Corporation, Ramsey, New Jersey) was employed to compound the polymers and wood fiber. It consists of three parts, the feed zone, compression zone and metering zone, functioning differently. The feed zone, attached below the feed hopper, works as the pathway for the resin pellets to get into the barrel. The compression zone is where some granules start melting. Then, all become liquid and ready to exit at the die at a constant rate in the metering zone [6]. The temperature was set at 150°C along the three zones of the extruder. The set temperature should be a little bit higher than the melting temperature of HDPE, 130-135°C. Too high a temperature is not desirable because charring of wood fibers increases. The wood fiber was loaded into the extruder through three different ports at three different screw speeds of the extruder (80, 100, and 120 RPM). At the die exit, a continuous stream of well-mixed composite flowed consistently out and was cut into six inches in length before it solidified at room temperature.

Nine treatments, as shown in Table 3, were performed.

Treatments	Screw Speed (RPM)	Port Position
1	80	I
2	100	I
3	120	Ι
4	80	II
5	100	II
6	120	II
7	80	III
8	100	III
9	120	III

Table 3Sample Treatments





Compression Molding

The extruded material was later compressed into plates using a Carver Laboratory Press, Model M (Fred S. Carver Inc., Menomonee Falls, Wisconsin). Three pieces of six inch extrudate were needed for each molding process. Two different sizes of frames were used for different mechanical tests. The first frame dimensions were $6 \times 6 \times 0.1$ inch, for tensile test samples. The second frame dimensions were $5 \times 5 \times 0.125$ inch, for both impact test and water absorption samples. For all samples, the machine direction of the pieces was noted. The compression molding steps in detail are described in Appendix A.

Specimen Preparation

Once the plates were made, they were cut into suitable pieces for tensile, impact and water absorption tests. When cutting, the machine direction was noted and kept consistent for all of the test pieces. The molded sheets were cut into $6 \times 0.75 \times 0.1$ inch for tensile tests and $2.5 \times 0.5 \times 0.125$ inch for both impact and water absorption tests using a New Hermes Safety Saw. Then, dumbbell-shape specimens (type I) were made for tensile test samples by using Tensilkut, Model 10-13 (Tensilkut Engineering Division Sieburg Industries, Inc., Danbury, Connecticut). The specimens for the impact test were then notched using a TMI notching cutter. The angle of the notch was $22.5^{\circ} \pm 0.5^{\circ}$ and the depth of the notch was 0.1 inch. The specimens were conditioned at 23 ± 2 °C and $50\pm$ 5 % RH for at least 40 hours before being tested for tensile strength and impact strength.

Tensile Strength Testing

ASTM 638-99 [36], Standard Test Method for Tensile Properties of Plastics, was followed. An Instron testing machine, Model SFM-20 (United Calibration Corp., Huntington Beach, California) was used to perform the tensile strength testing (Instron operation is in Appendix A). The parameters of the machine were set as follows: 1000 lb load cell, test speed 0.02 inch/minute, extension gage length 2 inches. All test samples were measured by a digital vernier caliper Digimatic (Mitutoyo Corporation, Japan).

At least five samples were tested. Tensile strength and modulus of elasticity were automatically calculated and the curves between load (lb) and % extension were plotted by a Graphtec XY plotter, type MP 3200. X axis or % extension was set at maximum 3% and Y axis or load was set at 250 lb maximum load. Statistical analysis of the tensile strength and elastic modulus was performed using the SPSS program for One Way ANOVA and Least Significant Difference (LSD). The comparisons between each composition were analyzed at the 95% confidence level.

Izod Impact Strength Testing

By following ASTM D 256-97 [37], Standard Test Method for Determining the Izod Pendulum Impact Resistance of Plastics, Izod impact strength was determined. At least ten samples were tested using a TMI 43-I IZOD impact tester with a 5-lb pendulum (impact test steps are in Appendix A). They were held as a vertical cantilever beam and broken with a single swing of the pendulum. When the sample failed, the type of failure

was classified following the ASTM standard. The statistical analysis and the comparison between compositions were analyzed by the method described above.

Water Absorption Testing

ASTM D570-98 [38], Standard Test Method for Water Absorption of Plastics, was followed. The specimens were conditioned by drying in an oven for 24 hours at 50±3 °C, cooled in a desiccator, and immediately weighed to the nearest 0.001 g. Twenty-four hour Immersion and Long-Term Immersion procedures were employed (both procedures are in Appendix A). The change in weight was monitored and used to calculate the % water absorption.

Percent water absorbed was calculated after 24 hours and when equilibrium was reached, by the formula:

% water absorption = <u>wet weight - conditioned weight × 100</u> conditioned weight

Chapter 5

RESULTS AND DISCUSSION

The mechanical properties of the composite samples were evaluated by the test methods described in the previous chapter. At least five specimens of each treatment were tested for tensile properties and water absorption. Twelve specimens of each composition were tested for Izod impact strength.

As mentioned before, nine treatments were performed. However, the last three treatments (treatment 7, 8 and 9) did not work as well. This is because the HDPE resins were still in granular form; not melted, when the wood fiber was loaded into the port. That resulted in a non-uniform mixture coming out of the die. Thus, the extrudate could not be molded into uniform plates for testing.

The results of the first six treatments are shown for the following mechanical properties: tensile strength, yield strength, modulus of elasticity, percent elongation, Izod impact strength and water absorption.

4.1 Tensile Strength

Table 4 presents the tensile strength results for the different six treatments. It was found that the tensile strength of the composite was higher when fiber was loaded into the extruder via port II than when via port I (Figure 1). It was also found that the 100 RPM screw speed gave the highest tensile strength of the composite when compared with 80 and 120 RPM, for each fiber-loading port. The one-way analysis of variance method (ANOVA – see Appendix B for statistical analysis results) confirmed that there was a significant difference in the tensile strength between the treatments. However, the result by the least significant difference method (LSD) at the 95% confidence level showed no significant difference between treatments 1, 2 and 3, no significant difference between treatments 3 and 6, and no significant difference between treatments 4 and 5. This might be affected by the wide range of the tensile strength values of treatments 2, 3 and 6 (see data in Table 10 in Appendix A).

<u>Table 4</u>	Tensile S	Strength	(psi)
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Treatment	Fiber Loading	Screw Speed	Average	SD
1		80 RPM	2504.33ª	332.92
2	Port I	100 RPM	2774.21 ^{ab}	286.75
3		120 RPM	2563.85 ^{ac}	278.55
4		80 RPM	3387.78 ^d	257.98
5	Port II	100 RPM	3434.36 ^d	208.31
6		120 RPM	2888.76°	229.79

Note: Values with the same superscript letter are not significantly different.





Treatment 1	=	Fiber was loaded via port I and at 80 rpm screw speed
Treatment 2	=	Fiber was loaded via port I and at 100 rpm screw speed
Treatment 3	-	Fiber was loaded via port I and at 120 rpm screw speed
Treatment 4	=	Fiber was loaded via port II and at 80 rpm screw speed
Treatment 5	=	Fiber was loaded via port II and at 100 rpm screw speed
Treatment 6	=	Fiber was loaded via port II and at 120 rpm screw speed

4.2 Yield Strength

The yield strength results of the test samples are shown in Table 5 and Figure 2. Similar to the results for tensile strength, the yield strength of the composite was higher when fiber was loaded into the extruder via port II than when loaded via port I, and a screw speed of 100 RPM gave the highest tensile strength of the composite when compared with 80 and 120 RPM for each fiber-loading port.

The ANOVA result, again, showed there was a significant difference in the yield strength between treatments. The LSD results indicated no significant difference between treatments 1, 2, and 3, between treatments 2 and 6, and between treatments 4 and 5. However, the group of treatments 1, 2 and 3 had a significant difference in yield strength from the group of treatments 4 and 5, and treatments 1 and 3 from treatment 6.

The data for yield strength testing and the statistical analysis results can be seen in Appendices A and B respectively.

Treatment	Fiber Loading	Screw Speed	Average	SD
1		80 RPM	2441.15ª	351.51
2	Port I	100 RPM	2545.16 ^{ac}	369.08
3		120 RPM	2449.49 ^a	307.31
4		80 RPM	3282.55 ^b	298.75
5	Port II	100 RPM	3417.73 [⊾]	223.25
6		120 RPM	2871.33°	222.68

Table	5	Yield Strength	(nsi)	
Table		i iela buengui	(psi)	

Note: Values with the same superscript letter are not significantly different.

Figure 3 Yield Strength



Treatment 1	=	Fiber was loaded via port I and at 80 rpm screw speed
Treatment 2	-	Fiber was loaded via port I and at 100 rpm screw speed
Treatment 3	=	Fiber was loaded via port I and at 120 rpm screw speed
Treatment 4	=	Fiber was loaded via port II and at 80 rpm screw speed
Treatment 5	=	Fiber was loaded via port II and at 100 rpm screw speed
Treatment 6	=	Fiber was loaded via port II and at 120 rpm screw speed

4.3 Modulus of Elasticity

Table 6 and Figure 3 demonstrate the results for modulus of elasticity for the wood fiber reinforced HDPE composite system.

Unlike tensile strength and yield strength, the ANOVA results showed there was no significant difference between treatments. Therefore, statistical analysis at the 95% confidence level by the LSD method was not performed.

Treatment	Fiber Loading	Screw Speed	Average	SD
1		80 RPM	250,070	19092.13
2	Port I	100 RPM	246,633	13543.50
3		120 RPM	228,685	31164.17
4		80 RPM	268,094	26139.53
5	Port II	100 RPM	287,587	72818.70
6		120 RPM	225,600	35694.02

Table 6 Modulus of Elasticity (psi)

Figure 4 Modulus of Elasticity



Treatment 1	=	Fiber was loaded via port I and at 80 rpm screw speed
Treatment 2	=	Fiber was loaded via port I and at 100 rpm screw speed
Treatment 3	=	Fiber was loaded via port I and at 120 rpm screw speed
Treatment 4	=	Fiber was loaded via port II and at 80 rpm screw speed
Treatment 5	=	Fiber was loaded via port II and at 100 rpm screw speed
Treatment 6	=	Fiber was loaded via port II and at 120 rpm screw speed

4.4 Percent Elongation

The test results for percent elongation of wood fiber reinforced recycled HDPE composite systems are presented in Table 7 and Figure 4.

The ANOVA results showed there was a significant difference in the percent elongation between treatments. The highest percent elongation of the composite was produced in the process at a screw speed of 80 RPM and fiber loading at port II.

Treatment	Fiber Loading	Screw Speed	Average	SD
1		80 RPM	1.48 ^{ab}	1.15
2	Port I	100 RPM	2.96 ^{bc}	0.54
3		120 RPM	1.97 ^{abc}	0.47
4		80 RPM	5.91 ^ª	0.60
5	Port II	100 RPM	3.46°	1.40
6		120 RPM	2.97 ^{bc}	1.73

Table 7Percent Elongation (%)

Note: Values with the same superscript letter are not significantly different.

Statistical analysis at the 95% confidence level by the LSD method confirmed that the percent elongation of the composites was affected by both the fiber loading location and the screw speed.

Figure 5 Percent Elongation



Treatment 1	=	Fiber was loaded via port I and at 80 rpm screw speed
Treatment 2	=	Fiber was loaded via port I and at 100 rpm screw speed
Treatment 3	=	Fiber was loaded via port I and at 120 rpm screw speed
Treatment 4	=	Fiber was loaded via port II and at 80 rpm screw speed
Treatment 5	=	Fiber was loaded via port II and at 100 rpm screw speed
Treatment 6	=	Fiber was loaded via port II and at 120 rpm screw speed

4.5 Izod Impact Strength

The results of Izod impact strength testing tabulated in Table 8 and Figure 5, show that the location of fiber loading had an effect on the impact strength of the test specimens. The ANOVA results indicated that there was a significant difference between the treatments. Statistical analysis at the 95% confidence level by the LSD method confirmed that there was a significant difference between the group of treatments 1, 2, and 3 and the group of treatments 4, 5, and 6.

However, the different screw speeds at the same location of fiber loading had no effect on the Izod impact strength of the composite (no significant difference was found at the 95% confidence level by the LSD method).

Fiber Loading	Screw Speed	Type of Failure	Izod Impact Strength (ft-lb/in)				
			Average	SD			
	80 RPM	Partial and Hinge	1.367ª	0.380			
Port I	100 RPM	Partial, Hinge and Complete	1.083 ^b	0.328			
	120 RPM	Partial, Hinge and Complete	1.034 ^b	0.129			
	80 RPM	Partial, Hinge and Complete	0.683°	0.069			
Port II	100 RPM	Partial, Hinge and Complete	0.699°	0.100			
	120 RPM	Partial, Hinge and Complete	0.577°	0.081			

I dole o izoa impacti su enga	Table	8	Izod	impact	strengtl
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Note: Values with the same superscript letter are not significantly different





Treatment 1	=	Fiber was loaded via port I and at 80 rpm screw speed
Treatment 2	=	Fiber was loaded via port I and at 100 rpm screw speed
Treatment 3	=	Fiber was loaded via port I and at 120 rpm screw speed
Treatment 4	=	Fiber was loaded via port II and at 80 rpm screw speed
Treatment 5	=	Fiber was loaded via port II and at 100 rpm screw speed
Treatment 6	=	Fiber was loaded via port II and at 120 rpm screw speed

4.6 Water Absorption

Table 9 shows the percent water absorption in terms of % increase in weight for each treatment. The effect of fiber loading location and screw speed is illustrated in Figure 6.

As the screw speed increased, the gain in weight due to water absorption increased. Results from ANOVA (see Appendix B) showed that there was a significant difference in these six treatments, which means both the fiber loading location and the screw speed had an effect on water absorption.

Treatment	Fiber Loading	Screw Speed	SD		
1		80 RPM	0.0234 ^{ab}	0.0065	
2	Port I	100 RPM	0.0562°	0.0263	
3		120 RPM	0.0823 ^d	0.0132	
4		80 RPM	0.0081 ^{be}	0.0017	
5	Port II	100 RPM	0.0141 ^{abe}	0.0020	
6		120 RPM	0.0138 ^{abe}	0.0054	

<u>Table 9</u> Percent increase in weight due to water absorption

Note: Values with the same superscript letter are not significantly different.

The LSD method at the 95% confidence level confirmed that there was a significant difference between treatments 1, 2, and 3, but no difference between treatments 4, 5, and 6. (Details of the data for water absorption testing and the statistical analysis are shown in Appendices A and B). That means the fiber loading locations have more effect on water absorption test than does the screw speed.

Figure 7 Water Absorption



Treatment 1	=	Fiber was loaded via port I and at 80 rpm screw speed
Treatment 2	=	Fiber was loaded via port I and at 100 rpm screw speed
Treatment 3	=	Fiber was loaded via port I and at 120 rpm screw speed
Treatment 4	=	Fiber was loaded via port II and at 80 rpm screw speed
Treatment 5	=	Fiber was loaded via port II and at 100 rpm screw speed
Treatment 6	=	Fiber was loaded via port II and at 120 rpm screw speed

4.7 Discussion

There are many factors that influence the properties of composites, such as fiber type, length of fiber, aspect ratio (length to diameter ratio), fiber alignment, interface, matrix resin morphology, processing procedure and environmental effects.

4.7.1 Tensile properties

Tensile testing results were used to determine the mechanical strength of the composites. In this experiment, each test specimen's tensile strength, yield strength, modulus of elasticity, and percent elongation were determined. These data are useful for qualitative characterization and development.

Basically, fiber reinforcement is used to improve the strength of polymer matrix [39]. However, weak adhesion between the polymer and reinforcement can cause difficulty in development of a composite property.

As the tensile strength and yield strength of the composite are influenced by the fiber loading location, it indicates that the tensile strength of the composite was lower when fiber was loaded into the extruder via port I than when loaded via port II because the time for the polymer matrix mixing with the wood fiber was too short, resulting in poor adhesion at the interface between HDPE and wood fiber or it may be due to poor adhesion of the fibers in the matrix.

The wide range of tensile properties in the composites, which can be seen in the high standard deviations, may result from poor fiber dispersion. The high viscosity of the matrix during the composite fabrication and the polarity of the fiber, which tend to hold the fibers together, may cause poor distribution of fibers. Instead of spreading out evenly

in the polymer, the fibers were more likely to crowd randomly in the matrix, leading to lack of uniformity of the composite systems. In respect to mixing time, the shorter the mixing time, the higher was the variation, resulting in lower mechanical strength of the composite.

The modulus of elasticity or Young's modulus is simply the slope of the initial straight portion of the stress-strain curve. According to Bigg [40], the reinforcement concentration can affect the modulus of the composite. The same mixing ratio of polymer matrix (60%) and wood fiber (40%) was used in every treatment. It was found that there was no significant difference in the modulus between treatments.

When the results were compared in the case of screw speed, it was found that the highest mechanical strength (tensile strength and yield strength) was obtained from the composite produced at 100 RPM screw speed. This might be because the 80-RPM screw speed allows too long a mixing time between polymer and wood fiber, resulting in fiber damage, and the 120-RPM screw speed allows too short a mixing time, resulting in difficulty mixing the HDPE and wood fiber.

4.7.2 Impact strength

The impact strength of such composites depends on the nature of the composite and the type of impact test [41]. According to Chotipatumwan [33], this test method is used to determine the resistance to breakage by flexural shock of composites. The notch on the samples was made to concentrate the stress, minimize the deformation, and direct the fracture to the part of the specimen behind the notch.

For non-brittle polymer matrices, the addition of fiber is generally found to reduce the impact strength of composite materials because it reduces the surface energy and then decreases the volume fraction of matrix in the plastic zone.

However, the highest impact strength can be obtained in the case of the poorest adhesion between the matrix and the wood fiber. This is because the maximum energy can be dissipated by mechanical friction during the pull-out process and by debonding of the fibers [41].

In this investigation, the highest impact strength was obtained when a fiberloading location of port I and 80-RPM screw speed were used. That was not expected. The 120-RPM screw speed and the same fiber-loading port location should provide the highest impact strength because the time for mixing was shorter than that occurring at 80 RPM and so provided the poorest adhesion. But the shear rate would be higher and that could compensate by producing greater mixing force, even though less time.

Fibers also can reduce the impact strength by reducing the elongation to break and thus may reduce the area under the stress-strain curve. Therefore, the higher percent elongation of such composites contributes to the higher impact strength.

This does not explain the higher impact strength of the composites produced when the fiber-loading location of port I was used, compared to the composites produced when the fiber-loading location of port II was used. The higher percent elongation of composites produced when the fiber-loading location of port II was used should provide higher impact strength than the lower percent elongation of composites produced when the fiber-loading location of port I was used.

4.7.3 Water absorption

This test was used to determine the relative rate of water absorption, and had two main functions. First, it is a guide to the proportion of water absorbed by a material and consequently, in those cases where the relationships between moisture and electrical or mechanical properties, dimensions, or appearance have been determined, as a guide to the effects of exposure to water or humid conditions on such properties. Secondly, it is used as a control test on the uniformity of a product.

Results showed that a higher amount of water was absorbed when fiber loading port I was used than when fiber loading port II was used. As the structure of the wood fiber is hydrophilic, it does not adhere very well to the hydrophobic structure of the polymer matrix, especially when the mixing time is too short. After processing of the composites, the thermal shrinkage of the matrix results in a gap surrounding the fiber. Then the composite materials can take up a large amount of water because the hydroxyl groups in the fiber structure interact with the surrounding water. This causes swelling of the fibers which can fill the gap between the fibers and the polymer matrix. Then, mechanical properties might be decreased.

Composite materials, when the fiber was loaded via port I, had more gaps between the polymer matrix and wood fiber than when loaded via port II. This resulted in higher water absorption in the composites when fiber was loaded via port I than when loaded via port II.

When comparing the effect of screw speeds on water absorption, it seems that there was not much difference between treatments.

Chapter 6

CONCLUSIONS AND RECOMMENDATIONS

6.1 Conclusions

In the investigation of the fiber loading location, it was found that tensile strength, yield strength, modulus of elasticity and percent of elongation were higher when fiber was loaded into the extruder via port II than via port I. This is because of the longer mixing time of the HDPE and wood fiber composites. But higher impact strength and water absorption were obtained when fiber was loaded into the extruder via port I than via port II because of the poorer adhesion between the matrix and the wood fiber.

In the case of the screw speed, the 100-rpm screw speed provided the highest tensile properties except for percent elongation. The highest impact strength was obtained when the 80-rpm screw speed was used. The highest percent water absorption was found when the 120-rpm screw speed was used.

Overall, most properties of composites were improved when the fiber loading location was port II and the 100-rpm screw speed was used.

6.2 Recommendations for future research

As the incompatibility of the fibers and the matrix, including poor dispersion of the fibers in the matrix, causes adhesion between HDPE and wood fiber to be relatively poor, so it produces poor result for the mechanical and physical properties.

Therefore, the recommendation for further research is to improve the interface adhesion by adding coupling agents or surface modification such as maleic anhydride modified HDPE (MAHDPE), Proflow 1000, etc.

Moreover, the fiber might be treated to obtain a better distribution of fiber in the matrix. This method may be a way of improving the mechanical properties of the composites.

Appendix A

Sample	le Port I Port II						
	80 RPM 100 RPM		120 RPM	80 RPM	100 RPM	120 RPM	
1	2819.84	3092.98	2208.75	3449.22	3347.29	2572.29	
2	2175.80	2392.15	2925.79	3319.24	3626.63	2935.93	
3	2543.31	2565.81	2375.07	3228.10	3150.40	3198.42	
4	2833.41	2932.23	2631.65	3801.59	3396.45	2946.46	
5	2149.29	2887.90	2677.98	3140.77	3651.05	2790.71	
Average	2504.33	2774.21	2563.85	3387.78	3434.36	2888.76	
SD	332.92	286.75	278.55	257.98	208.31	229.79	
Maximum	2833.41	3092.98	2925.79	3801.59	3651.05	3198.42	
Minimum	2149.29	2392.15	2208.75	3140.77	3150.40	2572.29	

Table 10	Tensile Strength	Data (psi)
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Table 11Yield Strength Data (psi)

Sample		Port I	Port II					
	80 RPM	100 RPM	120 RPM	80 RPM	100 RPM	120 RPM		
1	2816.49	3067.36	2084.67	3039.51	3342.37	2571.04		
2	2175.80	2310.88	2792.36	3309.29	3608.03	2876.93		
3	2234.32	2116.93	2167.77	3161.25	3095.90	3183.38		
4	2832.70 2725.00		2630.02	3787.15	3393.71	2935.52		
5	2146.43 2505.64		2572.61	3115.55	3648.65	2789.80		
Average	2441.15 2545.1		2449.49	3282.55	3417.73	2871.33		
SD	351.51	369.08	307.31	298.75	223.25	222.68		
Maximum	2832.70	3067.36	2792.36	3787.15	3648.65	3183.38		
Minimum	2146.43	2116.93	2084.67	3039.51	3095.90	2571.04		

Sample		Port I		Port II					
	80 RPM	100 RPM	120 RPM	80 RPM	100 RPM	120 RPM			
1	271,026	243,315	216,709	262,920	264,343	202,785			
2	270,506	229,353	207,456	262,920	416,369	178,581			
3	239,331	240,246	195,872	310,612	236,657	265,288			
4	231,897 263,224		265,873	265,558	260,707	227,725			
5	237,593 257,027		257,513 239,088		259,861	253,623			
Average	250,070 246,633		228,685	268,094	287,587	225,600			
SD	19092.13 13543.50		31164.17	26139.53	72818.70	35694.02			
Maximum	271,026	263,224	265,873	310,612	416,369	265,288			
Minimum	231,897	229,353	195,872	239,088	236,657	178,581			

Table 12Modulus of Elasticity Data (psi)

Table 13Elongation Data (%)

Sample		Port I				
	80 RPM	100 RPM	120 RPM	80 RPM	100 RPM	120 RPM
1	1.82	3.52	1.43	5.75	5.75 2.34	
2	0.66	3.29	2.73	6.56	2.61	2.39
3	1.14	1.14 2.54		5.02	2.59	5.98
4	3.31 2.24		1.95	6.35	4.19	2.77
5	0.48 3.20		1.88	5.86	5.59	1.83
Average	1.48	2.96	1.97	5.91	3.46	2.97
SD	1.15 0.54		0.47	0.60	1.40	1.73
Maximum	3.31 3.52		2.73	6.56	5.59	5.98
Minimum	0.48	2.24	1.43	5.02	2.34	1.83

Table 14 Izod Impact Strength Data (ft-lb/in)

	RPM	Izod	Strength	(ft-lb/in)	0.689	0.574	0.535	0.612	0.612	0.535	0.535	0.612	0.651	0.535	0.651	0.382	0.577	0.081
	120	Type	of	failure	Н	С	С	С	С	С	c	С	Р	С	C	C		
ort II	RPM	Izod	Strength	(ft-lb/in)	0.689	0.612	0.805	0.574	0.651	0.805	0.651	0.651	0.689	0.921	0.612	0.728	0.699	0.100
Pc	100	Type	of	failure	С	С	С	Н	Р	Н	c	Н	Н	Н	Н	Ρ		
	RPM	Izod	Strength	(ft-lb/in)	0.651	0.612	0.651	0.728	0.805	0.651	0.728	0.689	0.574	0.612	0.728	0.766	0.683	0.069
	80	Type	of	failure	Ρ	Р	Н	Р	Ρ	Ρ	Ρ	Р	Ρ	Ρ	Ρ	ပ		
	RPM	Izod	Strength	(ft-lb/in)	0.882	4.240*	1.038	1.121	1.076	0.805	1.056	0.923	1.115	0.961	1.238	1.155	1.034	0.129
	120	Type	of	failure	С	Ρ	Ρ	Н	Ρ	Н	Ρ	Р	ပ	Н	c	Ч		
ort I	RPM	Izod	Strength	(ft-lb/in)	1.310	0.728	0.921	0.921	1.976	0.844	1.154	1.271	0.960	0.999	0.999	0.921	1.084	0.328
P	100	Type	of	failure	Ρ	Н	Н	Н	Ρ	С	Р	С	Р	Ρ	Р	Ь		
	RPM	Izod	Strength	(ft-lb/in)	1.223	1.193	1.766	1.787	0.855	1.115	1.193	1.038	1.961	1.877	1.388	1.009	1.367	0.380
	80	Type	of	failure	Р	Р	Р	Р	Р	Р	Р	Ч	Р	Н	Р	Р		
Sample					1	2	3	4	5	9	7	∞	6	10	11	12	Average	SD

Sample	Port I			Port II		
	80 RPM	100 RPM	120 RPM	80 RPM	100 RPM	120 RPM
1	0.0308	0.1137	0.0740	0.0057	0.0129	0.0191
2	0.0213	0.0297	0.0987	0.0078	0.0164	0.0067
3	0.0280	0.0392	0.0662	0.0097	0.0155	0.0115
4	0.0246	0.0369	0.0919	0.0089	0.0147	0.0166
5	0.0306	0.0647	0.0628	0.0105	0.0157	0.0053
6	0.0224	0.0588	0.0853	0.0078	0.0140	0.0167
7	0.0127	0.0593	0.0859	0.0060	0.0140	0.0161
8	0.0169	0.0476	0.0938	0.0085	0.0099	0.0185
Average	0.0234	0.0562	0.0823	0.0081	0.0141	0.0138
SD	0.0065	0.0263	0.0132	0.0017	0.0020	0.0054
Maximum	0.0308	0.1137	0.0987	0.0105	0.0164	0.0185
Minimum	0.0127	0.0297	0.0628	0.0057	0.0099	0.0053

Table 15Water Absorption Test

Compression Molding Process

- 1. Set both platens (top and bottom) to the desired temperature (150 °C)
- 2. Assemble the frame and chrome platens in the mirror-like structure of Mylar sheets and the metal platens
- 3. Wait for 15 minutes to heat the system up to 150°C
- 4. Place the assembly on the bottom platen

Close the hydraulic chamber by turning the knob at the base clockwise

Use pump handle to apply pressure up to 30,000 psi

Hold for 5 minutes (if pressure drops, apply more pressure using handle)

5. Turn off the heat by setting temperature to 50°C

Turn on cooling water slowly

- 6. When the temperature reaches 50°C (around 5 minutes), turn off cooling water, release the pressure by turning the knob counterclockwise.
- 7. Disassemble the assembly
- 8. Clean up: set lower platen temperature at 200°C
 - place the frame on the lower platen for about 5 minutes
 - use scraper to scrape out resin
Izod Impact Strength Test

- 1. Press left soft key and hold while turning power switch on. Release soft key.
- 2. Press the key labeled "ID", 1

Enter "Test ID", "Data", and "Sample ID" by pressing a soft key under corresponding choices. After each selection press "enter" key to store the information in computer memory.

- 3. Bring the 5 lb. pendulum to its latched position.
- 4. Press pendulum key. Enter pendulum weight. Press "Enter".
- 5. Release pendulum without specimen. Press "enter".
- 6. Select type of test (Izod) using soft key below test type as viewed on LCD display.
- 7. Enter the average sample width in "mil". This is "width" at the notch—what we would normally call the thickness. Press "enter".
- 8. Enter temperature of testing. Press "enter".
- Press "enter" again. You will be asked to enter the specimen width. Measure thickness at notched position with vernier caliper. Enter in mils, then press "enter".
- 10. Place specimen in the clamp; use the jig for positioning the notch to align in the center, with the notch facing the pendulum. Remove the jig.
- 11. Release the pendulum. Catch it after the beep, before it hits the sample again.
- 12. Identify the type of breakage as a complete break, hinge break, partial break or nonbreak.
- 13. Repeat steps 9-12 until all specimens are finished.

14. To print out your data, turn on the printer. Press the key "print". Press the right soft key under "report". The display will change. Press the right soft key again under the display "print". The printer will start to generate the report.

Water Absorption Test

1. Twenty-Four Hour Immersion

The conditioned specimens shall be placed in a container of distilled water maintained at a temperature of 23 ± 1 °C (73.4 ± 1.8 °F), and shall rest on edge and be entirely immersed. At the end of 24, +1/2, -0 h, the specimens shall be removed from the water one at a time, all surface water wiped off with a dry cloth, and weighed to the nearest 0.001 g immediately.

2. Long-Term Immersion

To determine the total water absorbed when substantially saturated, the conditioned specimens shall be tested as described in Twenty-Four Hour Immersion except that at the end of 24 h they shall be removed from the water, wiped free of surface moisture with a dry cloth, weigh to the nearest 0.001 g immediately, and then replaced in the water. The weighings shall be repeated at the end of the first week and every two weeks thereafter until the increase in weight per two-week period, as shown by three consecutive weighings, averages less than 1% of the total increase in weight or 5 mg, whichever is greater; the specimen shall then be considered substantially saturated.

APPENDIX B

TENSILE STRENGTH

Source	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	4025312.622	5	805062.524	11.145	.000
Within Groups	1733620.006	24	72234.167		
Total	5758932.628	29			

Multiple Comparisons

Dependent Variable: TENSILE STRENGTH LSD

Treatment	Treatment	Mean	Std. Error	Sig.	95% Confid	ence Interval
(I)	(J)	Difference				
		(I-J)				
					Lower Bound	Upper Bound
1	2	-269.8840	169.981	.125	-620.7083	80.9403
	3	-59.5180	169.981	.729	-410.3423	291.3063
	4	-883.4540*	169.981	.000	-1234.2783	-532.6297
	5	-930.0340*	169.981	.000	-1280.8583	-579.2097
	6	-384.4320*	169.981	.033	-735.2563	-33.6077
2	1	269.8840	169.981	.125	-80.9403	620.7083
	3	210.3660	169.981	.228	-140.4583	561.1903
	4	-613.5700*	169.981	.001	-964.3943	-262.7457
	5	-660.1500 *	169.981	.001	-1010.9743	-309.3257
	6	-114.5480*	169.981	.507	-465.3723	236.2763
3	1	59.5180	169.981	.729	-291.3063	410.3423
	2	-210.3660	169.981	.228	-561.1903	140.4583
	4	-823.9360*	169.981	.000	-1174.7603	-473.1117
	5	-870.5160*	169.981	.000	-1221.3403	-519.6917
	6	-324.9140	169.981	.068	-675.7383	25.9103
4	1	883.4540°	169.981	.000	532.6297	1234.2783
	2	613.5700 [*]	169.981	.001	262.7457	964.3943
	3	823.9360 [•]	169.981	.000	473.1117	1174.7603
	5	-46.5800	169.981	.786	-397.4043	304.2443
	6	499.0220 [•]	169.981	.007	148.1977	849.8463
5	1	930.0340 [*]	169.981	.000	579.2097	1280.8583
	2	660.1500 [*]	169.981	.001	309.3257	1010.9743
	3	870.5160	169.981	.000	519.6917	1221.3403
	4	46.5800	169.981	.786	-304.2443	397.4043
	6	545.6020 [•]	169.981	.004	194.7777	896.4263
6	1	384.4320 [*]	169.981	.033	33.6077	735.2563
	2	114.5480	169.981	.507	-236.2763	465.3723
	3	324.9140	169.981	.068	-25.9103	675.7383
	4	-499.0220*	169.981	.007	-849.8463	-148.1977
	5	-545.6020*	169.981	.004	-896.4263	-194.7777

YIELD STRENGTH

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	4644715.690	5	928943.138	10.266	.000
Within Groups	2171599.296	24	90483.304		
Total	6816314.986	29			

Multiple Comparisons

Dependent Variable: YIELD STRENGTH LSD

Treatment	Treatment	Mean	Std. Error	Sig.	95% Confide	ence Interval
(I)	(J)	Difference		-		
		(I-J)				
					Lower Bound	Upper Bound
1	2	-104.0140	190.245	.590	-496.6613	288.6333
	3	-8.3380	190.245	.965	-400.9853	384.3093
	4	-841.4020 [•]	190.245	.000	-1234.0493	-448.7547
	5	-976.5840 [•]	190.245	.000	-1369.2313	-583.9367
	6	-430.1860*	190.245	.033	-822.8333	-37.5387
2	1	104.0140	190.245	.590	-288.6333	496.6613
	3	95.6760	190.245	.620	-296.9713	488.3233
	4	-737.3880*	190.245	.001	-1130.0353	-344.7407
	5	-872.5700 [•]	190.245	.000	-1265.2173	-479.9227
	6	-326.1720	190.245	.099	-718.8193	66.4753
3	1	8.3380	190.245	.965	-384.3093	400.9853
	2	-95.6760	190.245	.620	-488.3233	296.9713
	4	-833.0640 [•]	190.245	.000	-1225.7113	-440.4167
	5	-968.2460 [•]	190.245	.000	-1360.8933	-575.5987
	6	-421.8480*	190.245	.036	-814.4953	-29.2007
4	1	841.4020 [•]	190.245	.000	448.7547	1234.0493
	2	737.3880 [•]	190.245	.001	344.7407	1130.0353
	3	833.0640 [•]	190.245	.000	440.4167	1225.7113
	5	-135.1820	190.245	.484	-527.8293	257.4653
	6	411.2160 [•]	190.245	.041	18.5687	803.8633
5	1	976.5840 [•]	190.245	.000	583.9367	1369.2313
	2	872.5700 [•]	190.245	.000	479.9227	1265.2173
	3	968.2460 [•]	190.245	.000	575.5987	1360.8933
	4	135.1820	190.245	.484	-257.4653	527.8293
	6	546.3980 [•]	190.245	.008	153.7507	939.0453
6	1	430.1860*	190.245	.033	37.5387	822.8333
	2	326.1720	190.245	.099	-66.4753	718.8193
	3	421.8480 [•]	190.245	.036	29.2007	814.4953
	4	-411.2160*	190.245	.041	-803.8633	-18.5687
	5	-546.3980*	190.245	.008	-939.0453	-153.7507

MODULUS OF ELASTICITY

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	13990490985.200	5	2798098197.040	1.914	.129
Within Groups	35092021182.000	24	1462167549.250		
Total	49082512167.200	29			

Multiple Comparisons

Dependent Variable: MODULUS OF ELASTICITY LSD

Treatment	Treatment	Mean	Std. Error	Sig.	95% Confide	ence Interval
(I)	(J)	Difference (I-J)				
					Lower Bound	Upper Bound
1	2	3437.6000	24184.024	.888	-46475.7725	53350.9725
	3	21386.0000	24184.024	.385	-28527.3725	71299.3725
	4	-18149.0000	24184.024	.460	-68062.3725	31764.3725
	5	-37516.8000	24184.024	.134	-87430.1725	12396.5725
	6	24470.2000	24184.024	.322	-25443.1725	74383.5725
2	1	-3437.6000	24184.024	.888	-53350.9725	46475.7725
	3	17948.4000	24184.024	.465	-31964.9725	67861.7725
	4	-21586.6000	24184.024	.381	-71499.9725	28326.7725
	5	-40954.4000	24184.024	.103	-90867.7725	8958.9725
	6	21032.6000	24184.024	.393	-28880.7725	70945.9725
3	1	-21386.0000	24184.024	.385	-71299.3725	28527.3725
	2	-17948.4000	24184.024	.465	-67861.7725	31964.9725
	4	-39535.0000	24184.024	.115	-89448.3725	10378.3725
	5	-58902.8000 [•]	24184.024	.023	-108816.1725	-8989.4275
	6	3084.2000	24184.024	.900	-46829.1725	52997.5725
4	1	18149.0000	24184.024	.460	-31764.3725	68062.3725
	2	21586.6000	24184.024	.381	-28326.7725	71499.9725
	3	39535.0000	24184.024	.115	-10378.3725	89448.3725
	5	-19367.8000	24184.024	.431	-69281.1725	30545.5725
	6	42619.2000	24184.024	.091	-7294.1725	92532.5725
5	1	37516.8000	24184.024	.134	-12396.5725	87430.1725
	2	40954.4000	24184.024	.103	-8958.9725	90867.7725
	3	58902.8000°	24184.024	.023	8989.4275	108816.1725
	4	19367.8000	24184.024	.431	-30545.5725	69281.1725
	6	61987.0000 [•]	24184.024	.017	12073.6275	111900.3725
6	1	-24470.2000	24184.024	.322	-74383.5725	25443.1725
	2	-21032.6000	24184.024	.393	-70945.9725	28880.7725
	3	-3084.2000	24184.024	.900	-52997.5725	46829.1725
	4	-42619.2000	24184.024	.091	-92532.5725	7294.1725
	5	-61987.0000 [•]	24184.024	.017	-111900.3725	-12073.6275

PERCENT ELONGATION

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	59.780	5	11.956	10.058	.000
Within Groups	28.530	24	1.189		
Total	88.309	29			

Multiple Comparisons

Dependent Variable: PERCENT ELONGATION LSD

Treatment	Treatment	Mean	Std. Error	Sig.	95% Confidence Interval	
(I)	(J)	Difference				
		(I-J)				
					Lower Bound	Upper Bound
1	2	-1.4760 [•]	.690	.043	-2.8992	-5.2813E-02
	3	4840	.690	.489	-1.9072	.9392
	4	-4.4260 *	.690	.000	-5.8492	-3.0028
	5	-1.9 8 20*	.690	.008	-3.4052	5588
	6	-1.4840 *	.690	.042	-2.9072	-6.0813E-02
2	1	1.4760 [•]	.690	.043	5.281E-02	2.8992
	3	0.9920	.690	.163	4312	2.4152
	4	-2.9500 [•]	.690	.000	-4.3732	-1.5268
	5	-0.5060	.690	.470	-1.9292	.9172
	6	-8.0000E-03	.690	.991	-1.4312	1.4152
3	1	0.4840	.690	.489	9392	1.9072
	2	-0.9920	.690	.163	-2.4152	.4312
	4	-3.9420 °	.690	.000	-5.3652	-2.5188
	5	-1.4980 [•]	.690	.040	-2.9212	-7.4813E-02
	6	-1.0000	.690	.160	-2.4232	.4232
4	1	4.4260 [•]	.690	.000	3.0028	5.8492
	2	2.9500 [•]	.690	.000	1.5268	4.3732
	3	3.9420*	.690	.000	2.5188	5.3652
	5	2.4440	.690	.002	1.0208	3.8672
	6	2.9420 [•]	.690	.000	1.5188	4.3652
5	1	1.9820*	.690	.008	.5588	3.4052
	2	0.5060	.690	.470	9172	1.9292
	3	1.4980*	.690	.040	7.481E-02	2.9212
	4	-2.4440*	.690	.002	-3.8672	-1.0208
	6	0.4980	.690	.477	9252	1.9212
6	1	1.4840	.690	.042	6.081E-02	2.9072
	2	8.000E-03	.690	.991	-1.4152	1.4312
	3	1.0000	.690	.160	4232	2.4232
	4	-2.9420*	.690	.000	-4.3652	-1.5188
	5	-0.4980	.690	.477	-1.9212	.9252

IMPACT STRENGTH

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	5.521	5	1.104	22.611	.000
Within Groups	3.174	65	4.883E-02		
Total	8.695	70			

Multiple Comparisons

Dependent Variable: IMPACT STRENGTH LSD

Treatment	Treatment	Mean	Std. Error	Sig.	95% Confide	ence Interval
(I)	(J)	Difference				
		(I-J)				
					Lower Bound	Upper Bound
1	2	.2834	.090	.003	.1032	.4636
	3	.3334	.092	.001	.1492	.5177
	4	.6842*	.090	.000	.5040	.8643
	5	.6681	.090	.000	.4879	.8483
	6	.7903*	.090	.000	.6101	.9704
2	1	2834 [•]	.090	.003	4636	1032
	3	5.003E-02	.092	.589	1342	.2343
	4	.4008*	.090	.000	.2206	.5809
	5	.3847*	.090	.000	.2045	.5648
	6	.5068*	.090	.000	.3267	.6870
3	1	3334*	.092	.001	5177	1492
	2	-5.0030E-02	.092	.589	2343	.1342
	4	.3507*	.092	.000	.1665	.5349
	5	.3346*	.092	.001	.1504	.5189
	6	.4568*	.092	.000	.2726	.6410
4	1	6842 [*]	.090	.000	8643	5040
	2	4008 [•]	.090	.000	5809	2206
	3	3507*	.092	.000	5349	1665
	5	-1.6083E-02	.090	.859	1963	.1641
	6	.1061	.090	.244	-7.4089E-02	.2863
5	1	6681°	.090	.000	8483	4879
	2	3847*	.090	.000	5648	2045
	3	3346*	.092	.001	5189	1504
	4	1.608E-02	.090	.859	1641	.1963
	6	.1222	.090	.180	-5.8006E-02	.3023
6	1	7903*	.090	.000	9704	6101
	2	5068 [•]	.090	.000	6870	3267
	3	4568 *	.092	.000	6410	2726
	4	1061	.090	.244	2863	7.409E-02
	5	1222	.090	.180	3023	5.801E-02

WATER ABSORPTION

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	3.527E-02	5	7.053E-03	44.911	.000
Within Groups	6.596E-03	42	1.570E-04		
Total	4.186E-02	47			

Multiple Comparisons

Dependent Variable: WATER ABSORPTION LSD

Treatment	Treatment	Mean	Std. Error	Sig.	95% Confide	ence Interval
(I)	(J)	Difference				
		(I-J)				
					Lower Bound	Upper Bound
1	2	-3.2825E-02*	.006	.000	-4.5470E-02	-2.0180E-02
	3	-5.8912E-02*	.006	.000	-7.1558E-02	-4.6267E-02
	4	1.530E-02*	.006	.019	2.655E-03	2.795E-02
	5	9.275E-03	.006	.146	-3.3701E-03	2.192E-02
	6	9.600E-03	.006	.133	-3.0451E-03	2.225E-02
2	1	3.282E-02*	.006	.000	2.018E-02	4.547E-02
	3	-2.6088E-02*	.006	.000	-3.8733E-02	-1.3442E-02
	4	4.812E-02*	.006	.000	3.548E-02	6.077E-02
	5	4.210E-02*	.006	.000	2.945E-02	5.475E-02
	6	4.243E-02*	.006	.000	2.978E-02	5.507E-02
3	1	5.891E-02*	.006	.000	4.627E-02	7.156E-02
	2	2.609E-02*	.006	.000	1.344E-02	3.873E-02
	4	7.421E-02*	.006	.000	6.157E-02	8.686E-02
	5	6.819E-02*	.006	.000	5.554E-02	8.083E-02
	6	6.851E-02*	.006	.000	5.587E-02	8.116E-02
4	1	-1.5300E-02*	.006	.019	-2.7945E-02	-2.6549E-03
	2	-4.8125E-02*	.006	.000	-6.0770E-02	-3.5480E-02
	3	-7.4213E-02*	.006	.000	-8.6858E-02	-6.1567E-02
	5	-6.0250E-03	.006	.342	-1.8670E-02	6.620E-03
	6	-5.7000E-03	.006	.368	-1.8345E-02	6.945E-03
5	1	-9.2750E-03	.006	.146	-2.1920E-02	3.370E-03
	2	-4.2100E-02*	.006	.000	-5.4745E-02	-2.9455E-02
	3	-6.8188E-02*	.006	.000	-8.0833E-02	-5.5542E-02
	4	6.025E-03	.006	.342	-6.6201E-03	1.867E-02
	6	3.250E-04	.006	.959	-1.2320E-02	1.297E-02
6	1	-9.6000E-03	.006	.133	-2.2245E-02	3.045E-03
	2	-4.2425E-02*	.006	.000	-5.5070E-02	-2.9780E-02
	3	-6.8512E-02*	.006	.000	-8.1158E-02	-5.5867E-02
	4	5.700E-03	.006	.368	-6.9451E-03	1.835E-02
	5	-3.2500E-04	.006	.959	-1.2970E-02	1.232E-02

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