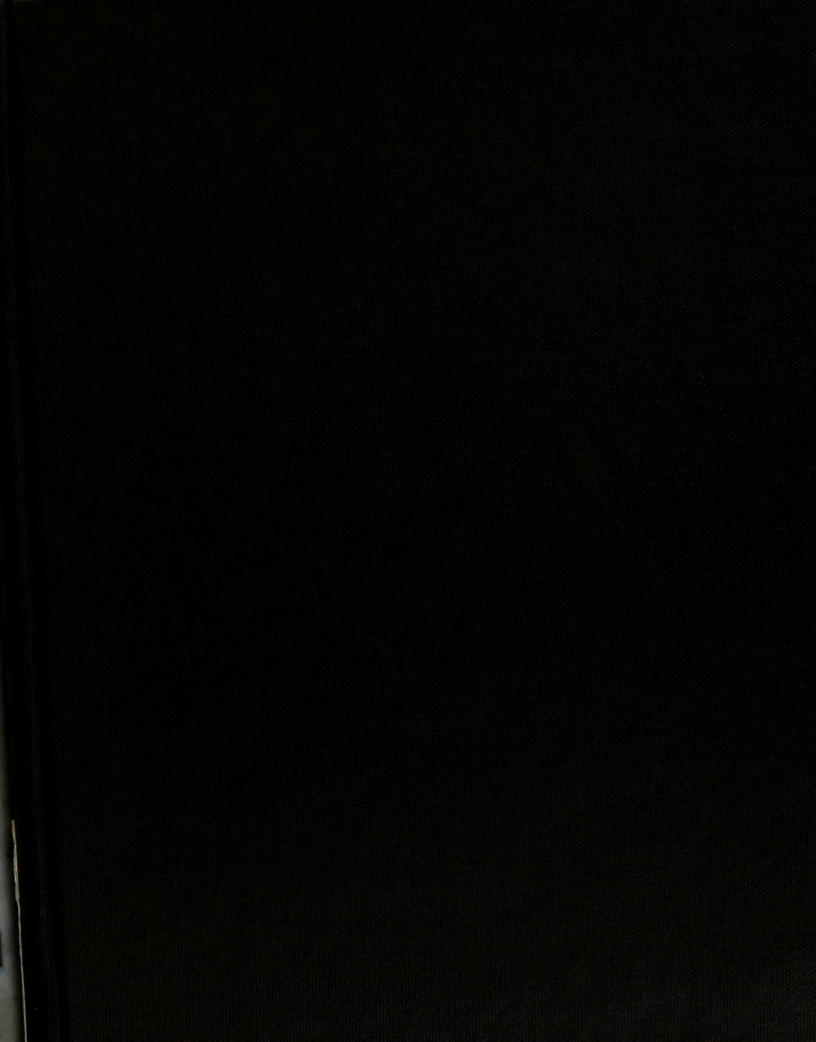




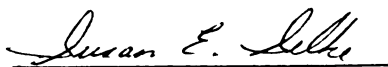
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COMPOSITE OF WOOD FIBER AND MIXED
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COMPOSITE OF WOOD FIBER AND MIXED RECYCLED THERMOPLASTICS

By

Sunetra Rojanarungtawee

A THESIS

**Submitted to
Michigan State University
in partial fulfillment of the requirements
for the degree of**

MASTER OF SCIENCE

School of Packaging

1998

ABSTRACT

COMPOSITE OF WOOD FIBER AND MIXED RECYCLED THERMOPLASTICS

By

Sunetra Rojanarungtawee

The effect of mixed resins in different proportions on the mechanical properties of a plastic/wood fiber composite was investigated. Polypropylene (PP) and high density polyethylene (HDPE) represented the mixed matrices. Aspen hardwood fiber served as the reinforcement. In the continuous phase, 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 processible. The comparison between two ratios revealed higher tensile strength with a 100% HDPE matrix. The modulus did not differ significantly at the two compositions investigated.

To my parents, Phong Rojanarungtawee and Pranee Euaritrakul.

ACKNOWLEDGEMENTS

I would like to express my gratitude to my major professor, Dr. Susan Selke, Ph.D. (School of Packaging, Michigan State University), and my committee members, Dr. Jack Giacin, Ph.D. (School of Packaging, Michigan State University) and Dr. Indrek Wichman (Department of Mechanical Engineering, Michigan State University) for their guidance and academic support.

I also wish to thank Dr. Mike Rich and Mr. Brian Rook from the Composite Materials and Structures Center for their instructions and frequent help in the use of the UTS and extruder and I would like to thank Adedejo B. Oladipo for taking the time to train me on extruder and compression molding operations. Special thanks to Ms. Ubonrat Siripatrawan and Ms. Kunlakarn Lekskul for their assistance in statistical analysis and statistics softwares. My appreciation also goes to Ms. Sudawan Supachokouychai.

I would especially like to thank my family for their incredible patience and support.

TABLE OF CONTENTS

LIST OF TABLES.....	vii
LIST OF FIGURES.....	xi
INTRODUCTION.....	1
CHAPTER 1	
LITERATURE REVIEW.....	6
Prediction Of Properties.....	16
Previous Research.....	23
CHAPTER 2	
MATERIALS.....	30
CHAPTER 3	
METHODS.....	34
CHAPTER 4	
RESULTS.....	38
180°C Compounding temperature.....	38
150°C Compounding temperature.....	39
Temperature effect.....	40
CHAPTER 5	
DISCUSSION.....	48
180°C Compounding temperature.....	48
150°C Compounding temperature.....	50
Temperature effect.....	51

CHAPTER 6	
CONCLUSIONS.....	52
RECOMMENDATIONS.....	53
APPENDICES.....	54
BIBLIOGRAPHY.....	71

LIST OF TABLES

Table 1- The effect of fiber orientation and stress direction on the reinforcement efficiency.....	12
Table 2- The relationship between fiber orientation and ϵ_0	20
Table 3- % Dry weight of organic components in woods.....	32
Table 4- Matrix composition.....	34
Table 5- Set temperature in the extruder under PP melting point.....	35
Table 6- Set temperature in the extruder under HDPE melting point.....	35
Table 7- Tensile strength in the lengthwise direction at 180°C.....	41
Table 8- Tensile strength in the crosswise direction at 180°C.....	41
Table 9- Tangent modulus in the lengthwise direction at 180°C.....	42
Table 10- Tangent modulus in the crosswise direction at 180°C.....	42
Table 11- Tensile strength in the lengthwise direction at 150°C.....	43
Table 12- Tensile strength in the crosswise direction at 150°C.....	43

Table 13- Tangent modulus in the lengthwise direction at 150°C.....	43
Table 14- Tangent modulus in the crosswise direction at 150°C.....	43
Table 15- Properties of PAXON® AD60-007.....	54
Table 16- Properties of PRO-FAX 7823.....	55
Table 17- Tensile strength (lb/in ²) data in the lengthwise direction under 180°C.....	58
Table 18- Tensile strength (lb/in ²) data in the crosswise direction under 180°C.....	59
Table 19- Tangent modulus (Mpsi) data in the lengthwise direction under 180°C.....	60
Table 20- Tangent modulus (Mpsi) data in the crosswise direction under 180°C.....	61
Table 21- Tensile strength (lb/in ²) data in the lengthwise direction under 150°C.....	62
Table 22- Tensile strength (lb/in ²) data in the crosswise direction under 150°C.....	62
Table 23- Tangent modulus (Mpsi) data in the lengthwise direction under 150°C.....	63
Table 24- Tangent modulus (Mpsi) data in the crosswise direction under 150°C.....	63
Table 25- One-way Analysis of Variance of tensile strength in the lengthwise direction over 7 treatments under 180°C.....	64
Table 26-- One-way Analysis of Variance of tensile strength in the crosswise direction over 7 treatments under 180°C.....	65
Table 27- One-way Analysis of Variance of tangent modulus in the lengthwise direction over 7 treatments under 180°C.....	65

Table 28- One-way Analysis of Variance of tangent modulus in the crosswise direction over 7 treatments under 180°C.....	66
Table 29- T-test of tensile strength in the lengthwise direction over two treatments under 150°C.....	67
Table 30- T-test of tensile strength in the crosswise direction over two treatments under 150°C.....	67
Table 31- T-test of tangent modulus in the lengthwise direction over two treatments under 150°C.....	67
Table 32- T-test of tangent modulus in the crosswise direction over two treatments under 150°C.....	68
Table 33- T-test of tensile strength of 0% PP/100% HDPE in the lengthwise direction over two different temperatures.....	68
Table 34- T-test of tensile strength of 0% PP/100% HDPE in the crosswise direction over two different temperatures.....	68
Table 35- T-test of tangent modulus of 0% PP/100% HDPE in the lengthwise direction over two different temperatures.....	69
Table 36- T-test of tangent modulus of 0% PP/100% HDPE in the crosswise direction over two different temperatures.....	69
Table 37- T-test of tensile strength of 30% PP/70% HDPE in the lengthwise direction over two different temperatures.....	69
Table 38- T-test of tensile strength of 30% PP/70% HDPE in the crosswise direction over two different temperatures.....	70
Table 39- T-test of tangent modulus of 30% PP/70% HDPE in the lengthwise direction over two different temperatures.....	70

Table 40- T-test of tangent modulus of 30% PP/70% HDPE in the crosswise direction over two different temperatures.....	70
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LIST OF FIGURES

Figure 1- Tensile strength in the lengthwise direction at 180°C.....	44
Figure 2- Tensile strength in the crosswise direction at 180°C.....	44
Figure 3- Tangent modulus in the lengthwise direction at 180°C.....	45
Figure 4- Tangent modulus in the crosswise direction at 180°C.....	45
Figure 5- Tensile strength in the lengthwise direction at 150°C.....	46
Figure 6- Tensile strength in the crosswise direction at 150°C.....	46
Figure 7- Tangent modulus in the lengthwise direction at 150°C.....	47
Figure 8- Tangent modulus in the crosswise direction at 150°C.....	47

INTRODUCTION

Disposal handling, landfills, has faced the problem of overwhelming garbage. The generation of municipal solid waste (MSW) has increased whereas the availability and the number of landfills have declined over the years. In 1996 there were only 3,091 landfills, a decrease from approximately 8,000 in 1988 (Goldstein, 1997). The decrease results from limited landfill capacity, strict Environmental Protection Agency (EPA) requirements and the difficulty of locating new sites due to the long approval process and public resistance (Fearncombe ,1995).

In order to solve the disposal crisis, EPA recommended four alternatives: source reduction, recovery, incineration and landfilling. Source reduction or waste prevention is the top priority to diminish the quantity or toxicity of any material in the first place before it becomes solid waste (EPA, 1997). Recovery is basically to reprocess the material, which is either industrial scrap or post consumer waste. This method not only saves natural supplies and energy but also increases landfill space (Fearncombe, 1995). Incineration or waste combustion is simply to burn MSW in incinerators. This technique provides the benefits of substantial volume reduction and energy recovery. However, it requires well equipped facilities and expensive investment. In addition, air pollutants and toxic ash such as chlorinated dibenzodioxins and dibenzofurans released

after the burning process become health concerns (Denison & Ruston, 1990). Landfilling, the oldest and the most common method, is expected to take care of any material that is not qualified for the previous options (Fearncombe, 1995). The disadvantages of this solution are the limited capacities and public conflict about siting. In addition, the leakage of liquid from the waste into ground water leads to water contamination (Selke, 1994). On the whole, recycling, so far, seems to get the most public support, compared to other solutions for post consumer solid wastes. However, success depends on community participation, which is one part of the recovery procedure.

Recycling begins with collection, consisting of curbside collection, drop-off centers, buy-back centers, deposit systems and commercial collection. Each selection requires some simple separation from the participants, such as separation of paper from plastic. The more specific material sorting and recovery processes are done at Materials Recovery Facilities (MRFs) (EPA, 1997). Recycling can be classified into three groups: primary, secondary and tertiary. Primary recycling is to utilize recovered material to make the same product as the virgin material does. For instance, aluminum cans are reprocessed to make aluminum cans. Secondary recycling is to use recycled material to make lower quality products such as plastic lumber. Tertiary recycling is basically pyrolysis. The substrate's structure is broken into simple forms by the combination of heat and an air deficient condition. As a result, the small molecules in the form of liquid and gas become useful fuel (Selke, 1994).

Packaging and containers are the largest part of MSW in landfills. In 1995, they were

72.9 million tons or 35.0% of the 208 million tons of total wastes. It was estimated that they will continue to grow to 36% by 2000 and 38% by 2010. Among the variety of packaging and containers in landfills, plastic containers and packaging have shown a fast increase. In 1960, there was only 120,000 tons or, 0.1% of total generation and in 1995, it went up to 7.7 million tons or 3.7% of total waste generation. The constituents are polyethylene terephthalate (PET), high density polyethylene (HDPE), low density polyethylene (LDPE), linear low density polyethylene (LLDPE), polyvinyl chloride (PVC), polystyrene (PS), polypropylene (PP) and other resins. The sources mainly are from soft drink bottles, milk bottles, base cups, film goods such as bags and sacks, and other plastic packaging such as coatings and caps. In 1995, discarded plastics were 10.6% of total packaging and containers (EPA, 1997).

These post-consumer plastic wastes have worried the responsible organizations. Firstly, they last forever in the landfills because of their persistent property. Secondly, the light weight but large volume of plastics increases transportation costs of recovery processes. The loose plastic bottles occupy 100 cubic yards/ton whereas paper takes up only 4 cubic yards/ton (Steuteville, 1995). Obviously, shredding or crushing processes are needed to decrease the volume (Curlee & Das, 1991). Thirdly, the single use products for convenience increase the deposit of packaging materials into landfills. In order to relieve the problems, new technologies are developed to make the plastics susceptible to certain environments such as biodegradability and photodegradability. However, there are some arguments over whether or not it really functions (Wolf & Feldman, 1991).

The use of reclaimed resins is another effective effort. A number of reclamation techniques have been developed to obtain well-sorted resins that can substitute for or blend with the virgin resins in many applications (Bisio & Xanthos, 1994). However, chances are that the separated resin is contaminated by other resins. The adulteration usually causes adverse results in product performance, mainly due to the incompatibility of the resins.

There have been investigations of utilizing the reclaimed resins with a reinforcement in the form of composite materials. The addition up to 40% by weight of fiber reinforcement is common (Birley et al., 1992). It was found that the products are less sensitive to the impurities. As a result, the contamination does not significantly affect the product quality. On the other hand, it is interesting to see how much of the contaminant can appear in the dominant resin stream without lowering the product strength and there is also the possibility of synergistic effects at certain levels. Therefore, the purpose of this study was to investigate the performance of composite made of mixed plastics and wood fiber, and to compare the mechanical properties of mixed plastics-based composite with the single polymer-based composite.

For this project, polypropylene (PP) and high density polyethylene (HDPE), representing the impure resins, functioned as the matrix, and wood fiber from aspen hardwood was used as the reinforcement. HDPE was selected because of its high recycling rate. PP was chosen because it is often a contaminant in the recycled HDPE. Six to seven percent of PP in the HDPE stream does not make any significant change in HDPE properties (Rader

et al., 1995). The mixed ratios vary from 0 to 100% of each resin. The variation shows how one resin type affects the other as the contaminant and if the contaminant level affects the product performance. Wood fiber was a choice because it is plentiful, light weight, non-toxic and has great strength (Marcin, 1991). It is also low cost, about 40% the cost of glass fiber (Babyak, 1993). The combination of resins and wood fiber was maintained at 60% of mixed resins and 40% of wood. To control the variation of the quality of recycled resins, the virgin resins were used. Product performance was evaluated by the mechanical properties of tensile strength and modulus of elasticity.

Chapter 1

LITERATURE REVIEW

A composite can be defined in different ways depending on which principles and standpoints are used to identify it. It can be as broad as anything consisting of two or more dissimilar components on microscopic view (Hull, 1981). On the other hand, it can be narrowed down to the point that it is a composite only if, first, it illustrates a distinct property that is superior to the original materials; second, the volume fraction of one constituent is greater than 10%; third, the property of one part is about 5 times greater than the other (Agarwal & Broutman, 1990). It also can be described as a man-made multiphase material that consists of chemically distinct constituents having an obvious interface (Callister, 1994). Despite differences in definitions, the ultimate goal is to create a composite that unites the advantageous attributes from the constituents and illustrates superior properties to the individual substrates, regardless of any point of view (Hull, 1981).

The properties of composites greatly depend on the properties of the substrates, the

distribution of components and the interaction between them. Consequently, the geometry of the reinforcement, one of the components, needs to be specified in terms of shape, size, and size distribution. The shape of the discontinuous materials can be referred to as spheres, cylinders, and platelets. Size and size distribution determine the composite structure and, when volume fraction is included, they also determine the interfacial area between matrix and reinforcement (Agarwal & Broutman, 1990).

In addition, the properties also depend on the concentration, the concentration distribution and orientation of reinforcing materials. The concentration of the reinforcement is easily adjusted from the weight or volume fraction of the components, and it is considered the individual most powerful factor controlling the composite properties. The concentration distribution indicates the uniformity of the composite texture, which results in homogeneous physical and mechanical properties. A nonuniform characteristic lowers the material strength because the failure will take place at the weakest area. Therefore, the result does not represent the total strength of the material. The orientation of the reinforcement determines how the properties of the composite are in different directions in the system. If the properties of the material are the same regardless of the direction, such a material is an isotropic material. On the other hand, an anisotropic material illustrates different properties in different directions (Agarwal & Broutman, 1990).

The composite components can be classified into two types. The continuous phase is called the matrix and a discontinuous phase dispersed into the matrix, which can be one material or more, is named reinforcement or reinforcing material (Agarwal & Broutman,

1990). In this experiment, the polymers, HDPE and PP, are the matrices and wood fiber serves as reinforcement.

The matrix has several roles. First, it keeps the reinforcements together and protects them from surface imperfection as a result of environmental attack. A surface flaw leads to crack propagation and eventually to failure at a small extent of applied stress. Second, it takes an external load and evenly transfers most of the burden to the reinforcers. The geometry of reinforcement determines how much load the reinforcement can take. In the case of fiber reinforcement, the matrix not only shelters the fibers but also keeps fibers from damaging one another. The softness and plasticity of the matrix prevent crack spreading out from one fiber to another (Callister, 1994). Third, the continuous phase secures the reinforcer arrangement if there is any (Birley et al., 1992). They are commonly classified into polymer, metal and ceramic. The polymers are the focus of interest in this experiment. There are two types of polymers. One is thermosetting and the other is thermoplastic polymer. Thermosets develop cross linkages when they are heated in the so-called curing process. Once heated, they will not remelt but decompose. Examples of thermosets are epoxides, polyesters, phenolics, ureas and melamine. The polymer chains in thermoplastics are developed in linear or branched form. They remelt and can be reprocessed by heat and pressure. Examples of thermoplastics are PE, PP, PS, and Polyether-ether ketone (PEEK) (Agarwal & Broutman, 1990).

Thermosetting and thermoplastic matrices require different processing parameters. The operation for thermosets can be manually done at room temperature due to their low

viscous liquid state. Therefore, the composites made of thermosetting polymers are suitable for small production scales. The drawbacks of these matrices are part fragility, solvent sensitivity and need of chemical reaction leading to many problems. In addition, the reactive unprocessed thermosets have a relatively short shelf life, prolong the processing time and generate hard-to-recover material. Thermoplastic-based composites benefit economically from large production scales due to the need for a high temperature and pressure process which demands fully-automated manufacturing. The advantages of thermoplastic polymers are the promotion of composite toughness and long shelf life (Bigg et al., 1988).

The reinforcement, in particular its geometry, plays a significant role in composite strength. Particle and fiber reinforcements are two main categories which are also used as the composite classification. A particle reinforcement can be anything that has approximately equiaxial dimensions such as spheres, cubes, tetragons and platelets (Agarwal & Broutman, 1990). It can be divided into large and small particles which are referred to as large-particle composites and dispersion-strengthened composites, respectively. Large particles, or so-called fillers, are usually more rigid and solid than the matrix. These particles limit the movement of the matrix surrounding them. The amelioration of mechanical properties depends on the matrix-reinforcement adhesion. In this case, the applied load is taken by both matrix and reinforcement. Particles in dispersion-strengthened composite are quite small sizes. The diameter ranges from 0.01 to 0.1 μm or 10 to 100 nm. The strengthening mechanism microscopically takes place in the way that the matrix takes the major applied load and the reinforcement blocks the

dislocation movement in the matrix. As a result, plastic deformation is limited, which leads to the improvement of mechanical properties in terms of tensile strength, yield strength and hardness (Callister, 1994). On the whole, the particles work by promoting the stiffness of the particulate composites but they are not as effective in taking the load from the matrix as fiber reinforcements. Especially, when hard particles are included in a brittle matrix, they create stress concentration in the composite. Consequently, the system strength is reduced (Agarwal & Broutman, 1990).

Fiber, by nature, has two different dimensions. One, length, is relatively much longer than the other, cross sectional diameter (Agarwal & Broutman, 1990). The characteristic of a great length-to-diameter ratio makes the small fibers become stronger than the bulk material (Callister, 1994). Fiber reinforcers produce the fiber reinforced composites, or fibrous composites. Based on the standpoint of properties, there are two kinds of fibrous composites: single-layer and multi-layer. The idea of single layer is that the composite is composed of many separated layers whose orientation and properties are identical. Short fiber reinforced composites made from compression molding process are included in the single-layered category even though they do not show an obvious separation between each layer and have preferential fiber orientation. The multi-layer composites consist of single-layered composites having different orientations or even different components. To focus on one individual layer, the reinforcement can be continuous fibers, which are simply long fibers, or discontinuous fibers, as the name implied, short fibers. Long fibers generate continuous fiber reinforced composites and short fibers make up discontinuous fiber reinforced composites (Agarwal & Broutman, 1990).

Fiber length is the key factor to judge whether or not such fibers are either short or long fibers. Usually, the fiber length of continuous fiber is 15 times or more the critical length, and anything less falls into discontinuous fiber. The critical length can be calculated according to Equation 1.

$$l_c = \sigma_f d / \tau_c \quad (1)$$

l_c is critical length.

σ_f is ultimate or tensile strength of fiber.

d is fiber diameter.

τ_c is shear yield strength of the matrix or the fiber-matrix bond strength.

When the fiber length equals the critical fiber length, the middle of the fiber takes on the maximum applied stress. As the fiber is longer, the stress taking area is also broader. Consequently, the strengthening mechanism becomes more effective (Callister, 1994).

Fibers in continuous fiber reinforced composites primarily function as load carriers in the burden direction. The strength of the composites is determined by the fibers. The matrix is only the binder and protector. Continuous fibers are able to be oriented into either unidirection or bidirection. High strength:weight ratios and anisotropic adjustability are two beneficial attributes of oriented fiber-reinforced composites (Agarwal & Broutman, 1990). Strength:weight ratios are specific modulus and specific strength which are the ratio of modulus to density and the ratio of tensile strength to density, respectively (Nielsen & Landel, 1994). Fibrous composites show superior strength to metals on a

weight basis perspective. The anisotropic adjustability lets the composites take advantage of preferable properties in a certain direction. In addition, the flexibility of processing and manipulated structure of fibrous composites create new materials having certain properties that are impossible to achieve in conventional materials (Agarwal & Broutman, 1990). The relationship between fiber orientation, stress direction and reinforcement efficiency is shown in Table 1.

Table 1. The effect of fiber orientation and stress direction on the reinforcement efficiency.

Fiber orientation	Stress direction	Reinforcement efficiency
Unidirection	Parallel to fibers 90° to fibers	1 0
random orientation and even distribution on a certain plane	Any direction in the plane	3/8
random orientation and even distribution in three dimensions in space	Any direction	1/5

Source: H. Krenchel, Fibre Reinforcement, Copenhagen: Akademisk Forlag, 1964 (Callister, 1994).

Short or chopped fibers are similar to the particulate reinforcements. The properties of the short fiber composites are considerably sensitive to the fiber length and the matrix function is more important than just a binder. In general, random orientation is expected in short fiber reinforced composites (Agarwal & Broutman, 1990). The tensile strength is determined by the bond between matrix and fibers. In the case of wood flour and polymer composites, the fiber orientation is a more critical factor to the modulus than to tensile strength, whereas the polymer-fiber bonding plays a more important role in tensile strength than in modulus (Maldas & Kokta, 1994).

There are several advantages of short fiber reinforcement. At the same weight fraction, the short fiber possesses more specific surface area than do the long fibers. The fiber dispersion is quite even. It establishes the dimensional stability and fiber damage resistance during processing. Unlike long fibers, short fibers have no entanglement problem and the other typical properties of the fiber are still the same (Maldas & Kokta, 1994).

Wood fibers are one of the potential reinforcements. They are naturally plentiful organic fibers. In MSW, there was about 39 percent by volume of wood, paper and paperboard (Pieper, 1993). 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 in 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 (Woodhams et al., 1984). As a result, cost effectiveness is an outstanding advantage. The other benefits are low abrasions to machinery, non-hazardous substance generation, low density and the possibility of surface modification (Beshay et al., 1985). 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 temperature. The water sorption of wood fibers causes a weak interface between the matrix and wood fibers (Chtourou et al., 1992), and leads to biodegradation after repeated exposure (Babyak, 1993).

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 be. This phenomenon leads to easy failure of composites in mechanical testing (Chtourou et al., 1992).

According to Chtourou et al. (1992), the interfacial bond between matrix and fiber plays an important role in the improvement of mechanical properties. It can be contributed 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.

The equipment is another factor affecting the composite properties. The preferential orientation degree usually occurs from the flowing of molten streams in the injection molding machine. The outer layers face higher shear stress than do the inner layers. As a result, fibers on the surface of the molded piece are arranged along the flow direction whereas those inside are 90° to the flow. In a compression molding machine, a random orientation in three dimensional space should be observed (Chtourou et al., 1992).

It is apparently that mechanical properties are the important criteria for material evaluation and selection. Tensile testing, one type of mechanical testing, is the indicator

of the ability of a material to resist the tension force that elongates the object at a constant rate (Nielsen & Landel, 1994) until the breakage occurs (Shah, 1984). In tensile testing, a stress-strain diagram is established, and the following values can be obtained: stress (Y-axis), strain (X-axis), elongation, yield point, yield strength, proportional limit, elastic modulus, ultimate strength and secant modulus (Shah, 1984).

In this work, the point of interest is the tensile strength and modulus of elasticity of a composite of mixed PP/HDPE and wood fiber. The tensile strength or ultimate strength is the measurement of the maximum load taken by the specimen. In other words, it is the highest nominal stress (engineering stress), which is the ratio of maximum force to the cross-sectional area before the load is applied (Felbeck & Atkins, 1984). The modulus of elasticity or Young's modulus is simply the slope of the initial straight portion of the stress-strain curve. It determines the stiffness of the material (Shah, 1984). In the case of a non-linear curve, the tangent or secant modulus is applied. The secant modulus is the straight secant line drawn from the origin to a certain point of the non-linear curve. The tangent modulus is the linear line drawn barely to touch the curve at any segment to get the slope value (Callister, 1994). In this experiment, the tangent modulus equals the modulus of elasticity because it is taken at the very beginning of the stress-strain diagram.

The tensile results are influenced by molecular orientation, specimen making process, strain rate and temperature. A force application that is parallel to the molecular alignment is higher than a force application that is perpendicular to the orientation and vice versa for the elongation. The injection-molded samples result in higher tensile strength values than

the compression-molded samples. The tensile strength and modulus are proportional to the rate of strain: the higher the strain rate, the higher the tensile values. On the contrary, the increase in temperature reduces the tensile strength and modulus (Shah, 1984).

There are some drawbacks in the stress-strain tests, however. A difficulty in result interpretation can be the case if the stress keeps changing from place to place due to variation in the specimen. Also, chances are that the result may reflect interference by other possible reactions instead of predicted phenomena. For instance, the recorded stress may result from the spherulitic failure in crystalline polymer instead of amorphous portion rearrangement as usual (Nielsen & Landel, 1994).

Prediction of properties

There are several equations for property prediction of composite materials. The most popular equation is the rule of mixtures.

According to Callister (1994), in particulate composites, the upper and lower limits of elastic modulus can be estimated by applying the rule of mixtures as shown in Equation 2 and 3 respectively. The parameters affected the composite properties are the particle size, the particle distribution and the volume fraction of the components.

$$E_c = E_m V_m + E_p V_p \quad (2)$$

$$E_c = (E_m E_p) / (V_m E_p + V_p E_m) \quad (3)$$

E and V are elastic modulus and volume fraction respectively.

Subscripts c , m and p denotes composite, matrix and particle, respectively.

In fibrous composites, the properties are affected by the fiber orientation and fiber type. Continuous and oriented fibers result in an anisotropic system. The load applied in parallel to the fiber alignment is called longitudinal loading. The load applied perpendicular to the fiber alignment is called transverse loading. Again, the rule of mixtures is applied to predict the mechanical properties. In longitudinal loading, it is assumed that the continuous fibers are identical in properties and size, and oriented unidirectionally all over the matrix. The composite stress is predicted by Equation 4.

$$\sigma_c = \sigma_m V_m + \sigma_f V_f \quad (4)$$

V_f and V_m are volume fractions of fiber and matrix respectively.

$$V_f = A_f/A_c \quad (5)$$

$$V_m = A_m/A_c \quad (6)$$

Equation 4 is derived from Equation 7 by assuming that composite, matrix and fiber phases have the same length.

$$\sigma_c A_c = \sigma_f A_f + \sigma_m A_m \quad (7)$$

σ_c , σ_f and σ_m are stresses of composite, fiber and matrix respectively.

A_f , A_c and A_m are cross-sectional area of fiber, composite and matrix respectively.

If the interfacial bonding between matrix and fibers is perfect in such a way that both phases take an equal strain. The strain of composite is equivalent to that of matrix and fiber.

$$\epsilon_c = \epsilon_m = \epsilon_f \quad (8)$$

ϵ_c , ϵ_m , and ϵ_f are the strain of composite, matrix and fiber respectively.

If the system is characterized by elastic deformation, dividing the stress of each phase by the strain of each phase gives the modulus of elasticity of the composite.

$$E_c = E_m V_m + E_f V_f \quad (9)$$

E_c , E_m and E_f are elastic moduli of composite, fiber and matrix respectively.

In the transverse direction, the external load is applied perpendicular to the fiber direction. The elastic modulus is estimated by Equation 12.

$$\sigma_c = \sigma_m = \sigma_f = \sigma \quad (10)$$

$$\epsilon_c = \epsilon_m V_m + \epsilon_f V_f \quad (11)$$

$$E_c = (E_m E_f) / [(1 - V_f) E_f + V_f E_m] \quad (12)$$

In the case of short and oriented fiber composites, the system is assumed to have an even fiber dispersion and the fiber is longer than the critical length. The longitudinal strength of the composite is predicted in Equation 13.

$$(TS)_c = (TS)_f V_f (1 - (l_c/2l)) + (TS)'_m (1 - V_f) \quad (13)$$

$(TS)_c$ is the longitudinal strength of the composite.

$(TS)_f$ is the fracture strength of the fiber.

$(TS)'_m$ is the stress in the continuous phase at failure.

l and l_c is fiber length and critical length respectively.

V_f is volume fraction of fiber phase.

If the fiber length is shorter than the critical length, the longitudinal strength is calculated by Equation 14.

$$(TS)_c = (l\tau_o/d) V_f + (TS)'_m (1 - V_f) \quad (14)$$

d is the fiber diameter.

For discontinuous and randomly aligned fiber composites, the elastic modulus is given in Equation 15.

$$E_c = KE_f V_f + E_m V_m \quad (15)$$

K is a fiber efficiency factor determined by V_f and E/E_m ratio. It is between 0.1 and 0.6.

According to Bigg et al. (1988), tensile strength of fiber reinforced thermoplastic-based composites can be predicted by applying the rule of mixtures shown in Expression 16.

$$\sigma_c = \sigma_p \phi_p + \sigma_f \phi_f \epsilon_1 \epsilon_0 \quad (16)$$

σ_c is composite tensile strength.

σ_p is polymer tensile strength.

σ_f is fiber tensile strength.

ϕ_p and ϕ_f are volume fraction of polymer and fiber in the composite respectively.

ϵ_1 is an efficiency factor related to the effectiveness of load transfer between the matrix and reinforcement.

ϵ_0 is an efficiency factor related to the orientation of the fibers in the composite.

Table 2. The relationship between fiber orientation and ϵ_0 .

Fiber orientation	ϵ_0
Unidirection	1.0
Random orientation in a plane	0.33

For continuous fibers, ϵ_1 is 1.0.

For discontinuous fibers, ϵ_1 is affected by the critical aspect ratio, which is the ratio of length to diameter of a fiber (Lee, 1989), of each matrix-fiber set.

$$(L/D)_c = \sigma_f / 2 \tau_l \quad (17)$$

$(L/D)_c$ is the critical aspect ratio.

σ_f is tensile strength of the fiber.

τ_l is fiber-matrix interfacial strength.

ε_1 value depends on each pair of matrix and fiber, and is strongly influenced by the efficacy of coupling agents which enhance matrix-fiber adhesion.

It was found that the random oriented fiber works most effectively when

$$L_f = 10(L/D)_c \quad (18)$$

L_f is average fiber length.

The critical aspect ratios of several sets of polymer-fiber composite are in a range of 20:1 to 50:1. In order to achieve the maximum load transfer capability, the aspect ratio of fibers should be at least 500:1.

For the elastic modulus, the rule of mixtures is also applied and exhibited in Equation 19.

$$E_c = E_p \phi_p + E_f \phi_f e \quad (19)$$

e is an efficiency factor.

E_c , E_p and E_f are the elastic moduli of the composite, polymer and fiber, respectively.

However, the factors that are taken into consideration are different between tensile strength and modulus. The fibers in woven form that do not reinforce the strength of the composite in the warp direction do enhance the modulus. The interbonding between fibers and matrix in the case of short fibers does not matter to the modulus as much as it does to the tensile strength. The fact that the addition of non-strengthening fillers results

in increase of modulus only and does nothing with polymer strength is the proof. Based on these different affected factors, there is no numerical relationship between the efficiency factors for modulus and tensile strength.

According to Chtourou (1992), there are two equations, based on rule of mixtures, used to estimate the elastic moduli and the strength at yield of short fiber reinforced composites.

$$E_c = k_{eff} * E_f * V_f + E_m * V_m \quad (20)$$

$$\sigma_c = K_{eff} * \sigma_f * V_f + \sigma_m V_m \quad (21)$$

E_c and σ_c are Young's moduli and the strength at yield of the composite.

E_f and σ_f are Young's moduli and the strength at yield of the fiber.

E_m and σ_m are Young's moduli and the strength at yield of the matrix.

V_f is the volume fraction.

K_{eff} and k_{eff} are the efficiency coefficients, depending upon the composite microstructures such as distribution, orientation and adhesion.

k_{eff} is 1/6 when the fiber orientation is random in three dimensional space.

k_{eff} is 1/3 when the fiber orientation is random in the plane.

k_{eff} is 1/2 when the fiber orientation is random in the angle of 90° in the plane.

The relationship between K_{eff} and fiber length shown in Equation 22.

$$K_{eff} = k'_{eff}(1-L_c/L) \quad (22)$$

L is the fiber length.

L_c is the critical length.

$$L_c = r * \sigma/Y \quad (23)$$

r is the fiber radius.

Y is the interfacial shear strength.

Previous research

Yam et al. (1988) 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 post-consumer milk bottles. The fiber and polymer were extruded through a corotating intermeshing twin 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.

Chtourou et al. (1992) studied the 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 non-treated fiber percentage, the higher the Young's modulus and the strength at yield. More than 30% of 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.

Maldas and Kokta (1994) studied the mechanical properties of the composites of recycled thermoplastics and wood flour. Three types of polymers were used: recycled LDPE, recycled PP and mixed plastics consisting of 50%LDPE, 15%HDPE, 15%PVC, 5%PS, 5% recycled PET and 10% maleated PP by weight. The reinforcement was maple wood flour precoated by maleated thermoplastics in different sizes. The degradation of the molten mixture during processing was reduced by the addition of flame retardant/heat resistant/antioxidant materials. There were four treatments of 0%, 20%, 30% and 40% per composite weight of treated and untreated wood flour mixed with each polymer. Mixing temperatures were 170°C to 175°C for PE, and 180°C to 185°C for PP and commingled plastics. Compression molding was done at 160°C for PE, 170°C for commingled plastics and 180°C for recycled PP, under 2.2 MPa pressure and 10 minute cooling time by cooling water under pressure. The treated fiber composite showed

substantial improvement in tensile strength, compared with the untreated fiber composites. The composites of PP/non treated fiber and PE/non treated fiber exhibited lower tensile strength than did the non-fiber contained polymers because of poor bonding between matrices and untreated fiber. On the other hand, the composites of commingled plastics and fibers, both with and without surface treatment, displayed improvement of tensile strength in comparison with the non-fiber contained polymer due to the maleated PP in the commingled plastics. Bifunctional acids and anhydrides in maleated PP served as the coupling agent between the hydrophilicity of fiber and hydrophobicity of polymer. Particularly in the pretreated fibers, the hydrophobic coating developed a soft film surrounding the hydrophilic fibers. The fiber dispersion in the matrix was also improved due to weakening of the intermolecular hydrogen bonds between fibers. The composites of polymers and fibers both with treatment and without treatment showed an improvement in modulus, compared to unfilled polymers. The elongation and tensile toughness (fracture energy/volume) of non-fiber contained polymers > treated wood flour/polymer composites > untreated wood flour/polymer composites was a consequence. The effect of thermoplastic types on the composite properties was that the increase of tensile strength in PE/treated fiber composites was observed up to 30% weight of fiber whereas the opposite result was found in PP based composites. In commingled based composites, the increase of tensile strength was associated with the increase of fiber content. The effect of the fiber concentration on the mechanical properties of composites was that the increase of % fiber content heightened the modulus but reduced elongation and tensile toughness. The fiber length was also an important factor to the composite properties, with the shorter the fibers, the better the properties. In other words,

the smaller size of wood flour particles increased the compatibility between polymer and fiber.

Youngquist et al. (1995) investigated the development of new techniques- air-laying and melt-blending- for producing composites from recycled plastics and wood fiber in comparison to the virgin materials. PET and HDPE, representing matrices, were compounded with different types of wood fiber by the air-laying method. The plastics and fibers were tested in both recycled and virgin forms. The results from PET-based composites made by this method revealed that there was no significant difference in mechanical properties, water resistance, and dimensional stability between virgin and recycled materials. The composites, consisting both of virgin and recycled wood fibers, had a higher modulus in the recycled PET matrix than in the virgin PET. The HDPE-based composites showed no difference in mechanical and physical properties between the products fabricated from virgin and recycled materials. There was no significant effect of wood fiber type and formulation on the impact strength.

The melt-blending procedure was to mix the fiber into the molten plastic stream by using an extruder or injection molding. The composites produced by this technique were able to include up to 50% wood fiber. Recycled matrices were PP from battery cases, PP from ketchup bottles and HDPE from milk bottles in comparison to virgin PP. Waste fibers were old newspaper and old magazines against pure cellulose fiber and wood flour. It was found that old newspaper composites had better properties than wood flour composites. Old magazines were also possible to use as a reinforcer but the clay and

impurities caused a difficulty in achieving uniform dispersion into the matrix. With the same reinforcer, the properties of recycled HDPE composites were inferior to virgin PP in terms of strength, stiffness, and unnotched impact energy. The composites of PP battery cases and HDPE milk bottles with old newspaper had a better impact performance than those of virgin PP with old newspaper. The effect of recyclability on the mechanical properties was tested by the comparison of the reground HDPE and non-refiberized HDPE panel from the air-laying technique. It turned out that the second cycle composites gave the same or even superior mechanical properties, water resistance and dimensional stability to the first cycle composites.

Lovinger and Williams (1980) reinvestigated tensile properties and morphology of blends of high density polyethylene (HDPE) and polypropylene (PP). Two polymers in the pellet form were mixed by hand into the following ratios; 75/25, 50/50, 25/75, 20/80, and 10/90 by weight of PE/PP. A two-roll mill was employed to mix the polymers at 200°C for 15 minutes. The mixture was compression molded to 1.25 mm thickness. The tensile samples were specimened according to Type IV of ASTM D 638 and tested by an Instron instrument. Polarized-light and scanning electron microscopes were used to study morphology. The polymer blends in the form of thin film were also studied by a transmission electron microscope. It was found that the ultimate elongation of all blends was inferior to those of an individual polymer due to a molecular incompatibility between PP and HDPE. The gradual increase in tensile strength at yield, along with the increase in PP percentage, implied that the elongation at yield of each ratio did not vary greatly. The composition between 75 to 90% PE expressed the highest ultimate tensile strength. The

maximum modulus at 1% elongation was achieved at 80%PP. The morphological study of the tensile samples revealed the different sizes of PP and PE spherulites. Spherulites of PE were much smaller than those of PP due to fast growth rates and high nucleation. From the micrograph, the average size of spherulites of all PE/PP blends decreased due to the PE effect. The study of the broken area at liquid nitrogen temperature showed that the failure took place at the PE/PP interphase. Transmission electron microscopy on PE/PP thin films demonstrated that at a 75/25 PE/PP ratio, the distinct interphase between PE and PP disappeared whereas a network was created throughout the space. At 50%PP, the PE part dispersed in PP was more obvious and at higher %PP, the PE characteristic was clearly seen. Based on the microstructural studies, it was concluded that at lower than 50%PP, the permeating network of PP and PE was formed and at 50%PP or more, PE was distributed in a continuous PP matrix. The mechanical properties can be explained by these morphological discoveries. The immiscible phases and incompatibility were responsible for poor mechanical properties in terms of early yielding point and failure occurrence at the interphase. The size reduction of spherulites, the increase in crystallinity, and the forming of a permeating network were synergistic effects. It was harder for the failure, taking place at the interboundary, to occur at the small spherulite size than it was at the big spherulite size. As a result, an increase in the stress at yield and ultimate strength was observed. The increase in total crystallinity by adding PE to PP yielded the improvement of modulus. The interlinking network throughout the structure created a better load transfer at interphase and consequently improved the strength. On the whole, the synergistic effect functioned at the low % elongation proportional to PP concentration, and the incompatibility played a major role at the high % elongation.

Shan Ren and David N.-S. Hon (1993) evaluated the effects of components, processing and additives on the mechanical properties of the composites made of newspaper fiber and 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 % 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 test were evident. The presence of stretched fibers confirmed the poor interfacial bonding between matrix and fiber.

Chapter 2

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. (1988). HDPE is the product of the polymerization of ethylene monomers under moderate temperature and pressure. It is a highly crystallized and non-polar thermoplastic (Toensmeier, 1994). The linear structure with a few side branches allows the molecular chains to get close and pack to one another (Hanlon, 1992) in the high structural regularity manner (Brydson, 1989). As a result, the % crystallinity can be up to 95% with stiffness and impermeability (Hanlon, 1992). Glass transition and melting temperatures of HDPE are about -90°C (-130°F) and 137°C (279°F) respectively (Callister, 1994). The properties of Paxon® AD 60-007 are shown in Appendix A.

Polypropylene (PP) in the pellet form was supplied by Himont U.S.A, Inc. under the trade

name Pro-Fax 7823. This PP type was recommended for high extrudate strength. According to the product data, it gives good melt strength and very good toughness, which is a characteristic of low flow propylene copolymers. In general, PP is a linear addition polymer of propylene monomers if it is a homopolymer. The problem of homopolymer PP is the occurrence of the brittleness as the temperature reaching about 0°C. The solution is to block copolymerise propylene with 4 to 15 % of ethylene to decrease the brittle temperature and increase strength, and it is called propylene copolymers. PP can be classified into three forms according to the methyl group arrangement on the main chain. The isotactic form has all methyl groups on the same side of carbon backbone. The syndiotactic structure has methyl groups alternately on the hydrocarbon chain. The atactic form is the random arrangement of methyl groups on the carbon chain. The first and second structures are possible to crystallize whereas the last form is completely amorphous. Isotactic PP has high stiffness, melting point and crystallinity. Atactic PP has a rubbery texture and low value. 90 to 95% PP in commercial resins is in the isotactic form. PP glass transition temperature and melting point are -20°C (-4°F) and 175°C (347°F) respectively (Callister, 1994). In comparison with HDPE, isotactic PP has a higher softening point, no problem with environmental stress cracking (ESC), higher brittle temperature, more sensitivity to oxidation and lower density, which is about 0.9 g/cc (Brydson, 1989). From a morphological standpoint, the complex structure of crystalline PP is consisted of the carbon backbone chain folding back and forth into crystallite structures surrounded by the amorphous structure. Each polymer crystallite is connected into a ribbon-like form by amorphous-like chains. The conglomeration of a number of wrenched ribbons around a nucleating origin forms a

spherulite (Nielsen & Landel, 1994). The properties of Pro-Fax 7823 are shown in Appendix A.

Aspen hardwood fiber was used as the reinforcement. Hardwoods are categorized in the subdivision angiospermae attributing to ovary wrapped seeds. Hardwood leaves are broad and color changed in the fall season in temperate areas. Aspen is in the genus *Populus* and divided into Bigtooth aspen, *Populus grandidentata*, and Quaking aspen, *Populus tremuloides*. The main difference between hardwoods and softwoods is that hardwoods have a vessel element whereas softwoods do not. The organic components of hardwoods are cellulose, hemicellulose and lignin. The approximate dry weight percentage of each in hardwood and softwood are shown in Table 3.

Table 3. % Dry weight of organic components in woods.

Type	Cellulose	Hemicellulose (% dry weight)	Lignin
Hardwood	40-44	15-35	18-25
Softwood	40-44	20-32	25-35

(Haygreen and Bowyer, 1982)

Cellulose ($C_6H_{10}O_5$)_n, is made of 5,000 to 10,000 units of glucose anhydride connected by β type linkage and characterizes the straight polymer chain. The linkage results from glucose hydrolysis. The longest cellulose chain is approximately 5 microns or 1/2000 cm in length and 8 Angstroms or 1/10,000,000 cm in diameter. Hemicelluloses are the low molecular weight and mostly branched-chain polymers composed of 150 or less sugar

anhydride units. Lignin is made of phenylpropane units into the complex and high molecular weight polymer. It has a phenolic structure even though it consists of carbon, hydrogen and oxygen like those first components. Lignin can be found between and within cell walls. It is a part of the strength inside cell walls and also glues cells together. Lignin possesses thermoplastic characteristics. It is hard at cool temperatures, and becomes soft and flexible as the temperature increases (Haygreen and Bowyer, 1982). The reaction between the matrix and the fiber in the composite materials takes place at lignin containing methoxyl groups. The level of methoxyl groups in hardwoods is higher than in softwoods, although the lignin content in hardwoods is a little lower than in softwoods. The higher level of methoxyl groups make hardwoods be the better choice than softwoods (Babyak, 1993). Aspen hardwood fiber in this experiment was produced by a thermomechanical pulping (TMP) method. Wood chips were fed into the approximately 120°C refiner which ground and defibrillated those chips into the fibers. The TMP fibers still hold a lot of lignin and natural wax which help to disperse the fibers into the hydrophobic polymers (Woodhams et al., 1984).

Chapter 3

METHODS

In order to form the continuous phase, PP and HDPE pellets were manually mixed following the ratios illustrated in Table 4. Each treatment was compounded and incorporated with wood fiber at two temperatures representing PP and HDPE melting points. The proportion of the continuous phase and wood fiber in each treatment was maintained constantly at 60% polymers and 40% wood fiber by weight. The calculation was described in Appendix B.

Table 4. Matrix composition.

%HDPE	%PP
0	100
10	90
30	70
50	50
70	30
90	10
100	0

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 is consisted of three parts, 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 in a constant rate at the metering zone (Birley et al., 1992). The temperatures were set differently in six ports along three zones of the extruder.

Table 5. Set temperature in the extruder under PP melting point.

Port	1	2	3	4	5	6
Temperature (°C)	180	180	155	155	155	155

Table 6. Set temperature in the extruder under HDPE melting point.

Port	1	2	3	4	5	6
Temperature (°C)	150	150	155	155	155	155

The temperature at port three to six was adjusted to be lower than port one and two for PP to reduce the burning of wood fiber. The resin pellets were fed at port one and wood fiber was incorporated at port three. This technique decreases fiber breakage, results in fiber distribution and lessens wear of equipment (Agarwal & Broutman, 1990). The screw speed was set at 100 rpm. At the die exit, the continuous stream of well-mixed composite flowed consistently out and was cut into six inches in length before it

solidified at room temperature. The extrudates were compression molded by 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. They were put into a 6*6*0.125 inch square mold which was topped and bottomed with Mylar sheets and steel platens. This mirror-like structure was heated up to 160°C within approximately 12 minutes and held under a pressure of 25,000 psi for 15 minutes. Then, the system was cooled down by cooling water to room temperature. The diagram was shown in Appendix C. The molded sheet was taken out, and kept at 23±2°C and 50±5%RH for about 40 hours to obtain the uniform distribution of internal stress (Ren & Hon, 1993) before test specimens were cut.

Tensile property testing conformed to ASTM D638-94b (Standard Test Method for Tensile Properties of Plastics). The test specimens were made in the shape of dumbbells. In order to perform the test, the molded sheets were cut into 6*0.75*0.125 inch strips using a New Hermes Safety Saw, in both crosswise and lengthwise directions, relative to the orientation of the extrudates in the sheet. Then, dumbbell-shape specimens were made by using Tensilkut, Model 10-13 (Tensilkut Engineering Division Sieburg Industries, Inc., Danbury, Connecticut). The specimens were conditioned at 23±2°C and 50±5%RH for at least 40 hours before being tested for tensile strength and modulus of elasticity. The tensile properties were performed on the United Testing Systems (U.T.S) SFM-20 Mechanical Test System, using laser extensometer model no. EXT 62LOE, and laser power source model no. EXT-62-LHMO (United Calibration Corp. 5802 Engineer Dr. Huntington Beach, CA). The specimen was elongated by moving the upper gripper at

0.02 inch/minute by gripper mover model no. SFM-20 (United Calibration Corp. 5802 Engineer Dr. Huntington Beach, CA). The parameters of the U.T.S 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, made in Japan). Tensile strength and modulus of elasticity were automatically calculated and the curves between load (lb) and % extension were plotted by the plotter Graphtec XY plotter, type MP 3200 (Made in Japan). 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 by using One Way ANOVA from STATG program and T-test from SPSS software. The comparison between each composition was made and analyzed at the 95 % confidence level.

Chapter 4

RESULTS

180°C compounding temperature

Tensile strength

The results of tensile strength in both lengthwise and crosswise directions were shown in Tables 7 and 8, as well as in Figures 1 and 2, respectively. It turned out that a ratio of 30% PP and 70% HDPE gave the highest tensile strength in both directions. Statistical analysis showed a significant difference between the 30% PP/70% HDPE and other proportions. In the lengthwise direction, 30% PP/70% HDPE was significantly different from 50% PP/50% HDPE, 70% PP/30% HDPE, 90% PP/10% HDPE and 100% PP/0% HDPE. On the other hand, it was not significantly different from 0% PP/100% HDPE and 10% PP/90% HDPE. In the crosswise direction, 30% PP/70% HDPE was significantly different from all other proportions.

Modulus of elasticity

The values obtained for the modulus of elasticity in both lengthwise and crosswise directions were tabulated in Tables 9 and 10, as well as presented graphically in Figures 3

and 4, respectively. The ratio of 10% PP/90% HDPE showed the best performance in both directions. In the lengthwise direction, a significant difference was found between 10% PP/90% HDPE and every ratio, excluding the ratio of 50% PP/50% HDPE. In the crosswise direction, the 10% PP/90% HDPE was significantly different from every proportion but 30% PP/70% HDPE.

150°C compounding temperature

Tensile strength

The results of tensile strength in both the lengthwise and crosswise directions were summarized in Table 11 and 12 respectively. They were also presented graphically in Figure 5 for the lengthwise direction and in Figure 6 for the crosswise direction. Both directions revealed a good agreement that 0% PP/100% HDPE had a higher tensile strength than 30% PP/70% HDPE. The statistical analysis also showed a significant difference between these two ratios in both directions.

Modulus of elasticity

The values determined for the modulus of elasticity were summarized in Tables 13 and 14, as well as in Figures 7 and 8 for the lengthwise and the crosswise directions, respectively. In the lengthwise direction, the ratio of 0% PP/100% HDPE exhibited a higher elastic modulus than 30%PP/70%HDPE and vice versa in the crosswise direction. However, statistical analysis showed no significant differences in both directions.

Temperature effect

The effect of temperature was determined by a comparison of the mechanical properties between 180°C and 150°C of 0% PP/100% HDPE and 30% PP/70% HDPE. For 0% PP/100% HDPE, there were no significant differences between two temperatures in both tensile strength and modulus of elasticity. In the contrast, there were significant differences between two temperatures in both tensile strength and modulus of elasticity for 30% PP/70% HDPE.

The raw data for all tensile values were shown in Appendix D and the statistical analyses were reported in Appendix E.

Table 7. Tensile strength in the lengthwise direction at 180°C.

%PP	%HDPE	Tensile strength (psi)	
		average	std. dev.
0	100	2723	303
10	90	2621	538
30	70	2880	336
50	50	2460	314
70	30	2327	360
90	10	2304	426
100	0	2119	443

Table 8. Tensile strength in the crosswise direction at 180°C.

%PP	%HDPE	Tensile strength (psi)	
		average	std. dev.
0	100	1745	96
10	90	2134	149
30	70	2288	207
50	50	1963	144
70	30	1804	27
90	10	1253	67
100	0	1804	95

Table 9. Tangent modulus in the lengthwise direction at 180°C.

%PP	%HDPE	Tangent Modulus (Mpsi)	
		average	std. dev.
0	100	0.3740029	0.0683980
10	90	0.4965418	0.0533967
30	70	0.4337601	0.0716733
50	50	0.4513923	0.0616853
70	30	0.4036430	0.0332676
90	10	0.4190089	0.0587326
100	0	0.4227439	0.0600575

Table 10. Tangent modulus in the crosswise direction at 180°C.

%PP	%HDPE	Tangent Modulus (Mpsi)	
		average	std. dev.
0	100	0.3783813	0.0834753
10	90	0.4805773	0.0876116
30	70	0.4303778	0.0754111
50	50	0.4004511	0.0673725
70	30	0.3702770	0.0629460
90	10	0.3263411	0.0609941
100	0	0.4000848	0.0269124

Table 11. Tensile strength in the lengthwise direction at 150°C.

%PP	%HDPE	Tensile strength (psi)	
		average	std. dev.
0	100	2441	379
30	70	2022	245

Table 12. Tensile strength in the crosswise direction at 150°C.

%PP	%HDPE	Tensile strength (psi)	
		average	std. dev.
0	100	1809	86
30	70	1702	38

Table 13. Tangent modulus in the lengthwise direction at 150°C.

%PP	%HDPE	Tangent Modulus (Mpsi)	
		average	std. dev.
0	100	0.360235	0.056787
30	70	0.328016	0.033399

Table 14. Tangent modulus in the crosswise direction at 150°C.

%PP	%HDPE	Tan Modulus (Mpsi)	
		average	std. dev.
0	100	0.310325	0.043495
30	70	0.355487	0.054241

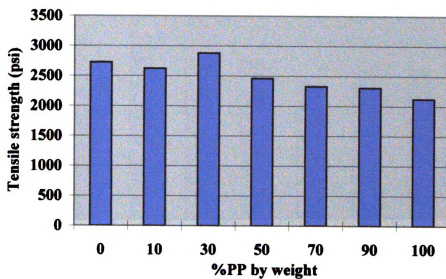


Figure 1. Tensile strength in the lengthwise direction at 180°C.

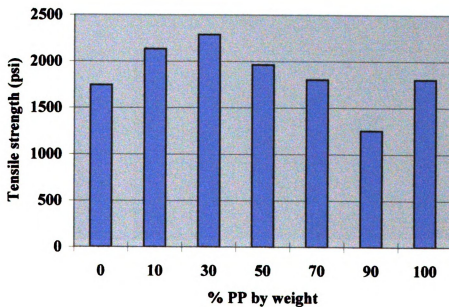


Figure 2. Tensile strength in the crosswise direction at 180°C.

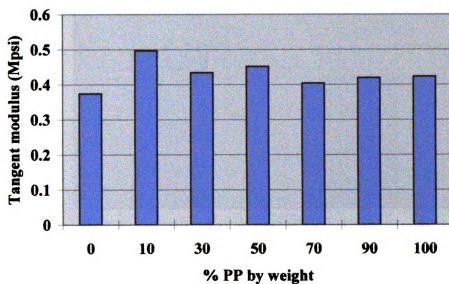


Figure 3. Tangent modulus in the lengthwise direction at 180°C.

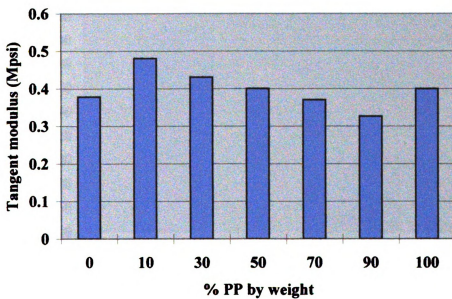


Figure 4. Tangent modulus in the crosswise direction at 180°C.

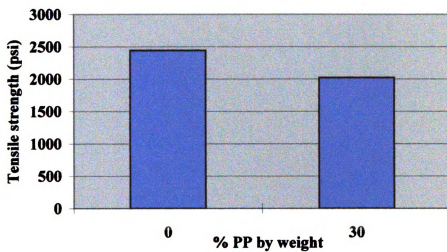


Figure 5. Tensile strength in the lengthwise direction at 150°C.

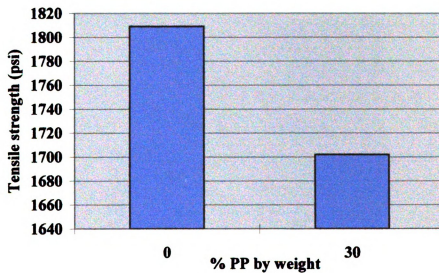


Figure 6. Tensile strength in the crosswise direction at 150°C.

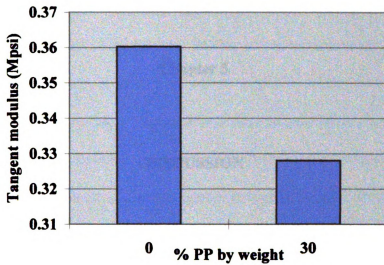


Figure 7. Tangent modulus in the lengthwise direction at 150°C.

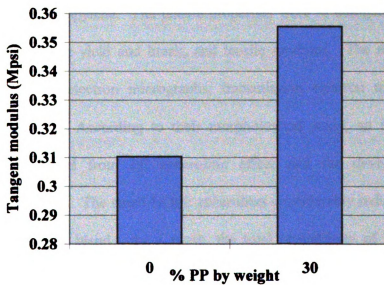


Figure 8. Tangent modulus in the crosswise direction at 150°C.

Chapter 5

DISCUSSION

180°C compounding temperature

Tensile strength values reported in the present study referred to an average of the ultimate strength. The results had a good agreement with the previous work of Lovinger and Williams (1980). They investigated tensile properties and morphology of blends of polyethylene and polypropylene. The tensile properties were % elongation at yield and break, tensile strength at yield and break, and tensile modulus. The morphology was consisted of scanning electron micrographs, transmission electron micrographs and polarizing microscope. According to their morphological study, an improvement of tensile strength resulted from PE spherulitic effect and the development of an interpenetrating network. The small HDPE spherulites considerably reduced the average spherulite size in every blend. As a result, the total crystallinity of the system was increased and intercrystalline linkage was developed. Based on this previous study, the development of the network without the distinct boundaries between PP and HDPE were found at 25%PP/75%HDPE. The strong linkage between PP and HDPE created at PP proportions lower than 50% contributed to the strength of the material through the

increase in the interphase adhesion yielding load transfer improvement between crystalline regions, where the failure usually occurred. In this experiment, the maximum tensile strength was found at 30% PP/70% HDPE, which was very close to that of 25% PP/75% HDPE. Therefore, a synergistic effect might be taking place in the mixed plastics-based composite in the same manner with the polymer blend, but the composite had lower tensile strength than did the blends. The lower strength was mainly caused by the poor adhesion, as a result of the incompatibility between the hydrophobic polymers and the hydrophilic wood fiber. According to statistical analysis in the lengthwise direction, the PP impurity not only could be allowed into the HDPE main stream up to 30% by weight but the contamination also brought about the synergistic effect to the system. Therefore, the HDPE adulterated with up to 30% PP could be utilized in the composite making process instead of using 100% HDPE. Consequently, the cost of raw material was reduced due to less separation process. On the other hand, PE was easier to handle than PP, because it had less stiffness than PP. The result in the crosswise direction might be interfered with the incomplete melting of PP in the extrudates, due to the low compression temperature (160°C). The pieces of extrudate did not bond as well as they were supposed to. The failure often occurred at the interpiece area.

The modulus of elasticity or Young's modulus was simply the slope of the initial straight portion of the stress-strain curve. In this study, the tangent modulus was equivalent to the Young's modulus, because the slope was taken at the very beginning of the stress-strain diagram. According to Bigg (1987), the parameters that affected the modulus of filled polymers were filler geometry, particle size dispersion, and filler concentration. In

addition, the modulus also depended on fiber orientation, crystallinity and spherulite size of the polymer components. According to Maldas & Kokta (1994), fiber orientation was a more critical factor to the modulus than to the tensile strength, whereas the interbonding between polymer and fiber contributed more to the tensile strength than to the modulus. The addition of wood flour to the polymer improved the modulus in comparison to the pure polymer. The increase in the crystallinity also improved the modulus (Nielsen & Landel, 1994). According to the previous research of Lovinger and Williams (1980), the investigation of the effect of reducing the size of the PP spherulite on its tensile modulus revealed that the modulus increased as the PP spherulite size was decreased. Besides these molecular effects, the strain, which measured how far the specimens could be pulled apart until broken, might also affect the modulus values since the modulus was the ratio of stress over strain. Therefore, the reduction of PP spherulite size, due to PE acting as nucleating agent and the low elongation resulting in low strain, might be responsible for the highest modulus achieved at 10%PP/90%HDPE. Based on the statistical point of view, there was no significant difference between 10% PP/90% HDPE and 50%PP/50%HDPE in the lengthwise direction. Thus, either 10%PP or 50%PP impurity in HDPE stream gave the same performance in term of elastic modulus. Again, the result in the lengthwise direction should be more precise than in the crosswise direction.

150°C compounding temperature

The lower tensile strength values in 30% PP/70% HDPE than in 100% HDPE might be a consequence of the partial melting of PP and incompatibility between PP and HDPE. Since PP melting temperature was about 175 to 185°C (Ren & Hon, 1993), the

compounding temperature at 150°C was apparently too low for PP to melt during processing and resulted in incomplete melt PP which led to inconsistent fiber distribution (Ren & Hon, 1993). In addition, the low temperature also caused difficulty in taking a piece of extrudate out at the die exit. The higher the percentage of PP, the harder it was to process at this condition due to the stiffness of PP. As a result, only up to 30% PP was processible in this work.

In contrast, there was no significant difference between the two ratios in the case of modulus of elasticity. At this point, up to 30% PP impurity in HDPE system did not affect the modulus of elasticity of the composite under this condition.

Temperature effect

HDPE obviously worked well in both temperatures. The higher temperature at 180°C was above its melting point but was not high enough to cause significant thermal degradation of the polymer or the fiber, at the residence times used. In contrast, an introduction of 30% PP into 70% HDPE at 150°C significantly lowered the strength at 150°C compared to 180°C, probably as a result of the partial melt of PP leading to non-uniform fiber distribution and encapsulation of poorly bonded PP particles in the HDPE matrix.

Chapter 6

CONCLUSIONS

Inclusion of PP in a HDPE/wood fiber composite resulted in an improvement in the mechanical properties at certain proportions. Under the 180°C compounding temperature, the maximum ultimate tensile strength and elastic modulus in both directions were achieved at 30% PP/70% HDPE and 10% PP/90% HDPE respectively. An increase in total crystallinity due to an addition of one polymer to the other and an intercrystalline network were believed to play major roles in a synergistic effect on the tensile strength. Under the 150°C compounding temperature, the data were available only at 0% PP and 30% PP due to the difficulty in operation. 0% PP/100% HDPE displayed a higher tensile strength than 30% PP/70% HDPE. The inferior performance in 30% PP/70% HDPE might result from an incompatibility between PP and HDPE, and the low processing temperature leading to non-uniform fiber distribution and partial melt of the PP. 0% PP/100% HDPE statistically showed the same performance at both 180°C and 150°C. However, the inclusion of 30% PP required compounding at 180°C to result in acceptable properties.

RECOMMENDATIONS

Since synergistic performance occurred in the mixed plastics-based composites under the 180°C processing temperature, the recommendation for further research is to improve the interphase adhesion through the addition of coupling agents or surface modification of either the continuous phase or the reinforcement. Also the specimen preparation should be switched from compression molding to injection molding in order to improve the precision of experimental data.

APPENDICES

APPENDIX A

Table 15. Properties of PAXON® AD60-007

Properties	AD60-007 (U.S. system)	AD60-007 (Metric system)	Test Method
Melt index	0.7 g/10 min	0.7 g/10 min	ASTM D 1238-89
Density	60.0 lbs/ft ³	0.960 g/cm ³	ASTM D 1505-85
Tensile strength at yield	4,400 psi	30 MPa	ASTM D 638-89
Elongation at break	>600 %	>600%	ASTM D 638-89
Tensile modulus of elasticity	155,000 psi	1,070 MPa	ASTM D 638-89
Flexural stiffness, Cantilever Beam	145,000 psi	1,000 MPa	ASTM D 747-86
Tensile impact	90 ft-lbs/in ²	19 J/cm ²	ASTM D 1822-89
Impact brittleness temperature	<-105°F	<-76°C	ASTM D 746-79
Environmental stress crack resistance	10 hrs	10 hrs	ASTM D 1693-70
Hardness, Shore D	70	70	ASTM D 2240-86
Vicat softening temperature	260°F	127°C	ASTM D 1525-87
Heat deflection temperature, 66 psi load	175°F	79°C	ASTM D 648-82
Coefficient of linear thermal expansion	6*10 ⁻⁵ in/in/°F	1.1*10 ⁻⁴ cm/cm/°C	ASTM D 696-79
Bulk density	37 lbs/ft ³	590 kg/m ³	ASTM D 1895-89

(HIMONT U.S.A., Inc, 1988)

Table 16. Properties of PRO-FAX 7823

Properties	PRO-FAX 7823 (U.S. system)	PRO-FAX 7823 (Metric system)	Test Method
Melt flow rate	0.5 dg/min	0.5 dg/min	ASTM D 1238
Density	55.9 lbs/ft ³	.897 g/cm ³	ASTM D 792A-2
Tensile strength at yield	4,100 psi	28.5 MPa	ASTM D 638
Elongation at yield	15%	15%	ASTM D 638
Flexural modulus	185,000 psi	1,280 MPa	ASTM D 790B
Notched Izod impact strength	10 ft-lbs/in at 73°F	530 J/m at 23°C	ASTM D 256A
Rockwell hardness, R scale	80	-	ASTM D 785A
Deflection temperature at 66 psi (455 kPa)	185°F	85°C	ASTM D 648
Drop-weight impact at -20°F (-29°C)	17 ft-lbs	23 J/m	HIMONT method

(PAXON POLYMER COMPANY, 1990)

APPENDIX B

In order to obtain the proportion of 60% matrix and 40% wood fiber in the compounding process, the feed rate of wood fiber and polymer were calculated as follows:

1. Fiber feed rate

- Adjust the fiber feeder speed to 2000rpm and fill with wood fiber. Then, turn the feeder on until some of the fibers dropped from the delivered pipe to make sure that the pipe was full with fibers.
- Weigh the empty tray (T grams) and put under the pipe.
- Turn the feeder on and start timing simultaneously. Wait about 10 minutes or more (t seconds) to cover all the fluctuated feeding cycle.
- Weigh the wood fiber-filled tray (F grams).
- Calculate the amount of wood fibers from

$$\text{Fiber weight (grams)} = F - T \text{ grams/t seconds}$$

$$\text{Fiber feed rate (lb/hr)} = [(F - T)/t] * [3600/454]$$

2. Polymer feed rate

- Calculate the polymer feed rate from

$$\text{Polymer feed rate (lb/hr)} = \text{Fiber feed rate} * (60/40)$$

- Input the polymer feed rate in the polymer feeder.

APPENDIX C

Diagram of compression molding process

Three pieces of the extrudate in the mirror-like structure of Mylar sheets and the metal platens



Wait for 12 to 15 minutes to heat the system up to 160°C



Remain at 160°C for 5 minutes while pushing up the lower platens



Put 25,000 psi load on and hold for 15 minutes



Adjust the temperature to 60°C and turn on the cooling water



Wait for 5 to 7 minutes in order to let the system cool down to 60°C



Hold at 60°C for 10 minutes



Adjust the temperature to 30°C and hold for 20 minutes

APPENDIX D

Table 17. Tensile strength (lb/in²) data in the lengthwise direction under 180°C.

Replications	Variation PP/HDPE						
	0/100	10/90	30/70	50/50	70/30	90/10	100/0
1	2363	2406	2570	2200	2612	3141	2122
2	3104	2654	2716	2620	2400	2310	2631
3	3061	2169	3295	2472	2481	3229	2795
4	2265	2500	3207	2274	2384	2176	2479
5	2760	2434	2908	2518	2884	2318	2690
6	2764	2871	2759	2560	2365	2244	1609
7	3023	3938	2429	2391	2403	2154	1516
8	2620	2425	2667	1727	2423	2438	1975
9	2547	2194	3366	2656	1742	1944	1925
10				2841	2614	2014	2146
11				2804	1908	2059	1951
12					1709	2140	1594
13						1789	

Table 18. Tensile strength (lb/in²) data in the crosswise direction under 180°C.

Replications	Variation PP/HDPE						
	0/100	10/90	30/70	50/50	70/30	90/10	100/0
1	1721	1971	2218	2061	1782	1172	1901
2	1683	2268	2118	2036	1797	1204	1680
3	1646	2413	2222	2067	1834	1171	1719
4	1840	2204	2114	2035		1304	1855
5	1691	1955	2087	1732		1232	1901
6	1887	2051	2072	1751		1347	1767
7		2039	2055	1908		1288	
8		2179	2498	1890		1305	
9		2122	2556	1921			
10			2448	1816			
11			2558	2225			
12			2509	1978			
13				2100			

Table 19. Tangent modulus (Mpsi) data in the lengthwise direction under 180°C.

Replications	Variation PP/HDPE						
	0/100	10/90	30/70	50/50	70/30	90/10	100/0
1	0.3486166	0.4644395	0.4344537	0.3767412	0.4531586	0.4655113	0.4312822
2	0.4560768	0.5602943	0.3200044	0.4646209	0.3804117	0.3093931	0.4074772
3	0.4573495	0.4987969	0.3969684	0.3247268	0.4223617	0.3489718	0.4483089
4	0.3538164	0.4588165	0.5128456	0.4657305	0.4219338	0.3167362	0.3449261
5	0.2394241	0.4559348	0.4554398	0.4900326	0.4578639	0.4337922	0.4173105
6	0.4296247	0.4199073	0.4227754	0.4565800	0.3635188	0.5015044	0.3673371
7	0.3501414	0.5877076	0.4410043	0.4988192	0.3806679	0.4542786	0.4817018
8	0.3458416	0.5096734	0.3639745	0.3873344	0.3806616	0.4631735	0.4960938
9	0.3851348	0.5133056	0.5563747	0.4866557	0.3541729	0.4485103	0.4741886
10				0.5263295	0.4168977	0.4413261	0.3177987
11				0.4877445	0.3946897	0.4325031	0.4989465
12					0.4173779	0.4102132	0.3875557
13						0.4212017	

Table 20. Tangent modulus (Mpsi) data in the crosswise direction under 180°C.

Replications	Variation PP/HDPE						
	0/100	10/90	30/70	50/50	70/30	90/10	100/0
1	0.4749848	0.5807528	0.4711415	0.3392065	0.4422742	0.2239746	0.3718728
2	0.3720138	0.4940500	0.4173815	0.3848750	0.3256472	0.3379837	0.4392896
3	0.4615679	0.5009187	0.3651493	0.4534989	0.3429095	0.2461646	0.3970512
4	0.3956999	0.3837360	0.3346263	0.3747366		0.3832713	0.4257525
5	0.2776831	0.3645246	0.3431248	0.4830866		0.3346625	0.3885158
6	0.2883382	0.6105639	0.4160498	0.3440493		0.3947271	0.3780266
7		0.5194026	0.4117317	0.3052333		0.3575835	
8		0.4879864	0.5428627	0.4018511		0.3323618	
9		0.3832604	0.5903392	0.5189132			
10			0.4068566	0.3265231			
11			0.4455095	0.3731373			
12			0.4197614	0.4870637			
13				0.4136896			

Table 21. Tensile strength (lb/in²) data in the lengthwise direction under 150°C.

Replications	Variation PP/HDPE	
	0/100	30/70
1	2397	1958
2	1714	1736
3	2610	1843
4	1822	1820
5	2589	2246
6	2502	2378
7	2786	2128
8	2996	2387
9	2363	2130
10	2638	1722
11	2436	1893

Table 22. Tensile strength (lb/in²) data in the crosswise direction under 150°C.

Replications	Variation PP/HDPE	
	0/100	30/70
1	1823	1628
2	1682	1707
3	1812	1722
4	1767	1713
5	1802	1735
6	1971	1704

Table 23. Tangent modulus (Mpsi) data in the lengthwise direction under 150°C.

Replications	Variation PP/HDPE	
	0/100	30/70
1	0.2994451	0.3284089
2	0.3141345	0.3288897
3	0.3815890	0.3001226
4	0.3153619	0.3814775
5	0.4000295	0.3494355
6	0.2652121	0.3294654
7	0.3597577	0.2921225
8	0.3813177	0.2803313
9	0.3649814	0.3488516
10	0.4443442	0.2959661
11	0.4364102	0.3731069

Table 24. Tangent modulus (Mpsi) data in the crosswise direction under 150°C.

Replications	Variation PP/HDPE	
	0/100	30/70
1	0.3915240	0.3409836
2	0.2781950	0.3360364
3	0.2913819	0.3027846
4	0.3190162	0.3998013
5	0.3265355	0.4417873
6	0.2552978	0.3115257

APPENDIX E

Table 25. One-way Analysis of Variance of tensile strength in the lengthwise direction over 7 treatments under 180°C.

Analysis of variance

Source of Variation	Sum of Squares	d.f.	Mean Square	F-ratio	Sig-level
Between groups	4360468	6	726744.69	4.616	0.0005
Within groups	10706592	68	157449.89		
Total (corrected)	15067060	74			

Multiple range analysis

Method: 95 percent LSD

Level (%PP)	Count	Average	Homogeneous groups
100	12	2119.4167	x
90	13	2304.3077	xx
70	12	2327.0833	xx
50	11	2460.2727	xx
10	9	2621.2222	xxx
0	9	2723.0000	xx
30	9	2879.6667	x

Table 26. One-way Analysis of Variance of tensile strength in the crosswise direction over 7 treatments under 180°C.

Analysis of variance

Source of Variation	Sum of Squares	d.f.	Mean Square	F-ratio	Sig-level
Between groups	5919889.9	6	986648.32	48.192	0.0000
within groups	1023673.8	50	20473.48		
Total (corrected)	6943563.7	56			

Multiple range analysis

Method: 95 percent LSD

Level (%PP)	Count	Average	Homogeneous groups
90	8	1252.8750	x
0	6	1744.6667	x
100	6	1803.8333	x
70	3	1804.3333	xx
50	13	1963.0769	x
10	9	2133.5556	x
30	12	2287.9167	x

Table 27. One-way Analysis of Variance of tangent modulus in the lengthwise direction over 7 treatments under 180°C.

Analysis of variance

Source of Variation	Sum of Squares	d.f.	Mean Square	F-ratio	Sig-level
Between groups	.0833286	6	.0138881	4.060	0.0015
Within groups	.2326275	68	.0034210		
Total (corrected)	.3159560	74			

Table 27 (cont'd)
Multiple range analysis
Method: 95 percent LSD

Level (%PP)	Count	Average	Homogeneous groups
0	9	.3740029	x
70	12	.4036430	xx
90	13	.4190089	xx
100	12	.4227439	xx
30	9	.4337601	x
50	11	.4513923	xx
10	9	.4965418	x

Table 28. One-way Analysis of Variance of tangent modulus in the crosswise direction over 7 treatments under 180°C.

Analysis of variance

Source of Variation	Sum of Squares	d.f.	Mean Square	F-ratio	Sig-level
Between groups	.1169157	6	.0194859	3.884	0.0029
Within groups	.2508585	50	.0050172		
Total (corrected)	.3677742	56			

Multiple range analysis
Method: 95 percent LSD

Level (%PP)	Count	Average	Homogeneous groups
90	8	.3263411	x
70	3	.3702770	xx
0	6	.3783813	xx
100	6	.4000848	xx
50	13	.4004511	x
30	12	.4303779	xx
10	9	.4805773	x

Table 29. T-test of tensile strength in the lengthwise direction over two treatments under 150°C.

%PP	N	Mean	Standard deviation	t
0	11	2441.1818	379.4443	3.080*
30	11	2021.9091	244.6252	

***denotes a statistically significant difference.**

Table 30. T-test of tensile strength in the crosswise direction over two treatments under 150°C.

%PP	N	Mean	Standard deviation	t
0	6	1809.5	94.2226	2.607*
30	6	1701.5	37.7187	

***denotes a statistically significant difference.**

Table 31. T-test of tangent modulus in the lengthwise direction over two treatments under 150°C.

%PP	N	Mean	Standard deviation	t
0	11	0.36023484	0.05678685	1.622
30	11	0.32801618	0.03339860	

***denotes a statistically significant difference.**

Table 32. T-test of tangent modulus in the crosswise direction over two treatments under 150°C.

%PP	N	Mean	Standard deviation	t
0	6	0.31032506	0.04764612	-1.532
30	6	0.35548648	0.05424078	

*denotes a statistically significant difference.

Table 33. T-test of tensile strength of 0% PP/100% HDPE in the lengthwise direction over two different temperatures.

Temperature (°C)	N	Mean	Standard deviation	t
150	11	2441.1818	379.4443	-1.805
180	9	2723	302.7796	

* denotes a statistically significant difference.

Table 34. T-test of tensile strength of 0% PP/100% HDPE in the crosswise direction over two different temperatures.

Temperature (°C)	N	Mean	Standard deviation	t
150	6	1809.5	94.2226	1.179
180	6	1744.7	96.2552	

* denotes a statistically significant difference.

Table 35. T-test of tangent modulus of 0% PP/100% HDPE in the lengthwise direction over two different temperatures.

Temperature (°C)	N	Mean	Standard deviation	t
150	11	0.36023484	0.05678685	-.492
180	9	0.37400287	0.06839803	

* denotes a statistically significant difference.

Table 36. T-test of tangent modulus of 0% PP/100% HDPE in the crosswise direction over two different temperatures.

Temperature (°C)	N	Mean	Standard deviation	t
150	6	0.31032506	0.04764612	-1.734
180	6	0.37838128	0.08347528	

* denotes a statistically significant difference.

Table 37. T-test of tensile strength of 30% PP/70% HDPE in the lengthwise direction over two different temperatures.

Temperature (°C)	N	Mean	Standard deviation	t
150	11	2021.9091	244.6252	-6.609*
180	9	2879.6667	335.8832	

* denotes a statistically significant difference.

Table 38. T-test of tensile strength of 30% PP/70% HDPE in the crosswise direction over two different temperatures.

Temperature (°C)	N	Mean	Standard deviation	t
150	6	1701.5	37.7187	-9.487*
180	12	2287.9167	207.3738	

* denotes a statistically significant difference.

Table 39. T-test of tangent modulus of 30% PP/70% HDPE in the lengthwise direction over two different temperatures.

Temperature (°C)	N	Mean	Standard deviation	t
150	11	0.32801618	0.03339860	-4.367*
180	9	0.43376008	0.07167331	

* denotes a statistically significant difference.

Table 40. T-test of tangent modulus of 30% PP/70% HDPE in the crosswise direction over two different temperatures.

Temperature (°C)	N	Mean	Standard deviation	t
150	6	0.35548648	0.05424078	-2.155*
180	12	0.43037785	0.07541109	

* denotes a statistically significant difference.

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BIBLIOGRAPHY

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