Effect of Matrix Modification on Lignocellulosic Composite Properties

A.R. Sanadi, K. Walz, L. Wieloch, R.E. Jacobson, D.E Caulfield, and R.M. Rowell

Abstract

Several billion pounds of fillers and reinforcements are used annually in the plastics industry. Their use is likely to increase, to reduce the amount of plastics used in a product, with improved compounding technology and new coupling agents. Recent interest in reducing the environmental impact of materials is leading to the development of newer agricultural-based materials that can reduce the stress to the environment. The use of lignocellulosic fibers (e.g., kenaf, jute, etc.) as reinforcing fillers in plastics has generated significant interest in recent years. The use of lignocellulosic fibers permit the use of high volume fillings due to their lower densities and nonabrasive properties, and therefore reduces the use of plastics in a product. The matrix can be modified using maleated polypropylene to improve the interaction and adhesion between the fibers and polypropylene. The specific tensile and flexural moduli of a 50 percent by volume kenaf-polypropylene composite compares favorably with a 40 percent by weight glass fiber-polypropylene injection-molded composite and are superior to typical calcium carbonate or talc-based polypropylene composites. Results indicate that annual growth lignocellulosic wastes and fibers are viable reinforcing fillers as long as the right processing conditions and aids are used, and for applications where the higher water absorption of the agricultural fiber composite is not critical.

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Introduction

Newer materials or composites are being developed to reduce the stress to the environment. In light of petroleum shortages and pressures to decrease dependence on petroleum products, there is an increasing interest in maximizing the use of renewable materials. The use of agricultural materials as a source of raw materials to the industry not only provides a renewable source, but could also generate a nonfood source of economic development for farming and rural areas.

Several billion pounds of fillers and reinforcements are used annually in the plastics industry. The

use of these additives in plastics is likely to grow with the introduction of improved compounding technology and new coupling agents that permit the use of high filler/reinforcement content (6). As suggested by Katz and Milewski, fillings up to 75 pph could be common in the future: this could have a tremendous impact in lowering the usage of petroleum-based plastics. It would be particularly beneficial, both environmentally and socioeconomically, if a significant amount of the fillers were obtained from a renewable agricultural source. Ideally, of course, an agricultural/biobased renewable polymer reinforced with agricultural fibers would make the most environmental sense.

Advantages to using agricultural fibers in plastics

Advantages to using annual growth lignocellulosic fibers as fillers or reinforcements in plastics are their low density, nonabrasiveness, high filling levels possible resulting in high stiffness properties, high specific properties, easy recyclability, and tougher fibers resulting in reduced fiber attrition during processing. There are also some environmental and socioeconomic advantages that cannot be ignored. These include biodegradability, the wide variety of fibers available around the world, the generation of rural jobs, stimulation of nonfood agricultural/farm-based economy, low energy consumption, low cost, and low energy utilization.

Material cost savings due the incorporation of these relatively low-cost fibers and the higher filling levels possible, coupled with the advantage of being nonabrasive to the mixing and molding equipment, are benefits that are not likely to be ignored by the plastics industry for use in the automotive, building, appliance, and other applications.

Prior work on lignocellulosic fibers in thermoplastics has concentrated on wood-based flour or fibers and significant advances have been made by a number of researchers (1,7,8,10,13,18,23,25). A recent study on the use of annual growth lignocellulosic fibers indicate that these fibers have the potential of being used as reinforcing fillers in thermoplastics (1 9). The use of annual growth agricultural crop fibers such as kenaf has resulted in significant property advantages compared to typical wood-based fillers and fibers such as wood flour, wood fibers, and recycled newspaper. Properties of compatibilized polypropylene and kenaf has mechanical properties comparable with those of commercial polypropylene composites (1 9).

Limitations

The primary drawback of the use of these fibers is the lower processing temperature permissible due to the possibility of lignocellulosic degradation and/or the possibility of volatile emissions that could affect composite properties. The processing temperatures are limited to about 200°C, although it is possible to use higher temperatures for short periods. This limits the type of thermoplastics that can be used with agricultural fibers to commodity thermoplastics such as polyethylene, polypropylene, polyvinyl chloride, and polystyrene. However, it is important to note that these lower priced plastics constitute about 70 percent of the total thermoplastic consumed by the plastics industry, and subsequently the use of fillers and reinforcements presently used in these plastics far outweigh their use in other more expensive plastics.

The second drawback is the high moisture absorption of the natural fibers. Moisture absorption can result in swelling of the fibers; concerns about the dimensional stability of the agricultural fiber composites cannot be ignored. The absorption of moisture by the fibers is minimized in the composite due to encapsulation by the polymer. It is difficult to entirely eliminate the absorption of moisture without using expensive surface barriers on the composite surface. If necessary, the moisture absorption of the fibers can be dramatically reduced through the acetylation of some of the hydroxyl groups present in the fiber (15), but with some increase in the cost of the fiber. Good fiber-matrix bonding can also decrease the rate and amount of water absorbed by the composite. Research on this area is presently underway at the University of Wisconsin and the Forest Products Laboratory.

It is important to keep these limitations in perspective when developing end use applications. We believe that by understanding the limitations and benefits of these composites, these renewable fibers are not likely to be ignored by the plastics/composites industry for use in the automotive, building, appliance, and other applications.

Sources of agricultural fibers

A wide variety of agricultural fibers and wastes are available. Our work has concentrated on fibers that are easily available, those that are already an important source for clothing, rope, sacking, and rugs in Asia and Africa. Fibers such as kenaf are now being grown in the United States and the quantity and the

types of fibers grown is likely to increase with new uses being developed for natural fibers.

Processing considerations

Separation of the fibers from the original plant source is an important step to ensure the high quality of fibers. The limiting processing temperatures when using lignocellulosic materials with thermoplastics is important in determining processing techniques. High processing temperatures (> 200°C) that reduces melt viscosity and facilitates good mixing cannot be used (except for short periods) and other routes are needed to facilitate mixing of the fibers and matrix in agricultural fiber thermoplastics.

The ultimate fiber lengths present in the composite depends on the type of compounding and molding equipment used. The properties of the agricultural fiber-based thermoplastic composites therefore depend on the manufacturing process. Yam et al. (25) at Michigan State University studied the effect of twin-screw blending of wood fibers and high-density polyethylene (HDPE) and concluded that the level of fiber attrition depended on the screw configuration and the processing temperature. Use of stearic acid in HDPE-wood fiber composites improved fiber dispersion and improved wetting between the fiber and the matrix (23) and resulted in significant improvement in mechanical properties. Work by Raj and Kokta (14) indicate the importance of using surface modifiers to improve fiber dispersion in cellulose fibers/polypropylene composites. Use of a small amount of stearic acid during the blending of cellulose fibers in polypropylene decreased both the size and number of fiber aggregates formed during blending in an internal mixer (Brabender roll mill).

Another technique that is gaining acceptance is the high intensity compounding using a turbine mixer (thermokinetic mixer). Woodhams et al. (24) and Myers et al. (1 3) found the technique effective in dispersing lignocellulosic fibers in thermoplastics. Addition of dispersion aids/coupling agents further improved the efficiency of mixing. The high shearing action developed in the mixer decreased the lengths of fibers in the final composite. However, the improved fiber dispersion resulted in improved composite properties. Recent work using a thermokinetic mixer to blend kenaf in polypropylene (19) has confirmed the usefulness of the compounding technique in effectively dispersing natural fibers in the thermoplastic matrix. An added advantage is that no predrying of the fibers is needed prior to the blending stage in the mixer.

Properties of composites

Cellulosic fillers and fibers can be classified under three categories depending on their performance when incorporated in a plastic matrix. Wood flour and other low-cost, agricultural-based flour can be considered as particulate fillers that enhance the tensile and flexural moduli of the composite with little effect on the composite strength. Wood fibers and recycled newspaper fibers have higher aspect ratios and contribute to an increase in the moduli of composite, and can also improve the strength of the composite when suitable additives are used to improve stress transfer between the matrix and the fibers. The improvement in modulus is not significantly different than the cellulosic particulate fillers. The most efficient cellulosic additives are some natural fibers such as kenaf, jute, flax, etc. The specific Young's modulus and specific flexural modulus, the ratio of the composite modulus to the composite specific gravity of composites with natural fibers such as kenaf are significantly higher than those possible with wood fibers. The specific moduli (the ratio of the composite modulus to the composite specific gravity) of high fiber volume fraction bast fiber-polypropylene composites are high and in the same range as glass fiber-polypropylene composites. The most efficient natural fibers are those that have a high cellulose content coupled with a low microfibril angle resulting in high filament mechanical properties.

Cellulosic fillers and fibers have been incorporated in a wide variety of thermoplastics such as polypropylene, polyethylene, polystyrene, polyvinyl chloride, and polyamides (7,8). In general, dispersing agents and/or coupling agents are necessary for property enhancement when fibers are incorporated in thermoplastics. Grafting chemical species onto the fiber surface has also been reported to improve the interaction between the fibers and matrix. Although grafting can improve the properties of the composite to a significant extent, this process increases the material cost of system. The use of dispersing agents and/or coupling agents is a cheaper route to improve properties and makes more practical sense for high-volume, low-cost composite systems.

In general, cellulosic fillers or fibers have a higher Young's modulus as compared to commodity thermoplastics thereby contributing to the higher stiffness of the composites. The increase in the Young's modulus with the addition of cellulosics depends on many factors such as the amount of fibers used, the orientation of the fibers, the interaction and adhe-

sion between the matrix, the ratio of the fiber to matrix Young's modulus, etc. The Young's modulus of the composite can be crudely estimated through the simple rule of mixtures and other simple models if the Young's modulus of the filler/fiber is known (5). The use of dispersing or coupling agents can change the molecular morphology of the polymer chains both at the fiber-polymer interphase and also in the bulk matrix phase. Crystallites have much higher moduli compared to the amorphous regions and can increase the modulus contribution of the polymer matrix to the composite modulus.

In order to use any models to estimate composites properties, it is necessary to know the property of the fibers. In general, natural fibers such as kenaf and jute are in the form of filaments that consist of discrete individual fibers, generally 2 to 6 mm long, which are themselves composites of predominantly cellulose, lignin, and hemicelluloses. Filament and individual fiber properties can vary widely depending on the source, age, separating techniques, moisture content, speed of testing, history of the fiber, etc. The properties of the individual fibers are very difficult to measure. Earlier work on a natural bast filament, sun hemp *(Crotalaria juncea)* suggested that the filament properties ranged widely.

Although several plastics have been used with cellulosic fibers, the major part of the work at the University of Wisconsin-Madison and the Forest Products Laboratory has been on polypropylene. The work reported here will concentrate on this versatile plastic in combination with kenaf.

Experimental methods

Kenaf filaments harvested from mature plants were obtained from AgFibers Inc., Bakersfield, Calif., and cut into lengths of about 1 cm. The fibers were not dried, and the moisture content of the fibers varied from 6 to 9 weight percent (wt%). In all our experiments the wt% and volume percent (vol%) reported is the amount of dry fiber present in the blend. The homopolymer was Fortilene- 1602 (21) with a melt flow index of 12 g/10 mm as measured by ASTM D 1238. A maleic anhydride grafted polypropylene (MAPP) was used as a coupling agent to improve the compatibility and adhesion between the fibers and matrix.

The filaments were not pulped prior to compounding as the former procedure can consume a significant amount of energy. The short fibers, MAPP, and polypropylene (the latter two in pellet form) were compounded in a high-intensity kinetic mixer (Synergistics Industries Ltd., Canada) where the only source of heat is generated through the kinetic energy of rotating blades. The blending was accomplished at 4600 rpm and then automatically discharged at 190°C. A total weight of fibers, polypropylene, and MAPP of 150 g were used for each batch and about 1.5 kg of blended material was prepared for each set of experiments. Fiber weight varied from 20 to 60 percent and coupling agent weight varied from 0 to 3 percent. The total residence time of the blending operation depended on the proportions of fiber and polypropylene present and averaged about 2 minutes.

The mixed blends were then granulated and dried at 105°C for 4 hours. Test specimens were injection-molded at 190°C using a Cincinnati Milacron Molder and injection pressures varied from 2.75 MPa to 8.3 MPa depending on the constituents of the blend. Specimen dimensions were according to the respective ASTM standards. The specimens were stored under controlled conditions (20% relative humidity and 32°C) for 3 days before testing. The crosshead speed during the tension and flexural testing was 5 mm/min. Although all the experiments were designed around the wt% of kenaf in the composites, fiber volume fractions can be estimated from composite density measurements and the weights of dry kenaf fibers and matrix in the composite. The density of the kenaf present in the composite was estimated to be 1.4 g/cc.

Results and discussion

Use of additives to modify fiber-matrix interactions

The matrix can be modified to improve the interaction and adhesion between the fiber and matrix and significant property enhancement can thus be achieved. A small amount of the MAPP (0.5 wt%) improved the flexural and tensile strength, tensile energy absorption, failure strain, and unnotched Izod impact strength. The anhydride groups present in the MAPP can covalently bond to the hydroxyl groups of the fiber surface. Any maleic anhydride that has been converted to the acid form can interact with the fiber surface through acid-base interactions. The improved interaction and adhesion between the fibers and the matrix leads to better matrix-to-fiber stress transfer. There was little difference in the properties obtained between the 2 and 3 wt% MAPP systems. The drop in tensile modulus with the addition of the MAPP is probably due to polymer morphology. Transcrystallization and changes in the ap-

parent modulus of the bulk matrix can result in changes in the contribution of the matrix to the composite modulus and will be discussed later. There is little change in the notched impact strength with the addition of the MAPP, while the improvement in unnotched impact strength is significant.

Use of the MAPP increases the failure strain and the tensile energy absorption. Thermodynamic segregation of the MAPP toward the interphase can result in covalent bonding to the -OH groups on the fiber surface. Entanglement between the polypropylene and MAPP molecules results in improved interphase properties and the strain-to-failure of the composite. There is a plateau after which further addition of coupling agent results in no further increase in ultimate failure strain. Any further increase in the amount of MAPP does not increase the failure strain past the critical amount. However, a minimum amount of entanglements are necessary through the addition of about 1.5 wt% for the critical strain to be reached.

Strength and modulus

There is little difference in the tensile strength of uncoupled composites compared with the unfilled polypropylene, irrespective of the amount of fiber present. This suggests that there is little stress transfer from the matrix to the fibers due to incompatibilities between the different surface properties of the polar fibers and nonpolar polypropylene. The tensile strengths of the coupled systems increases with the amount of fiber present. Strengths up to 74 MPa were achieved with the higher fiber loading of 60 wt%, which is about 49 vol%. As with tensile strength, the flexural strength of the uncoupled composites were approximately equal for all fiber loading levels, with a small improvement compared to the unfilled polypropylene.

The specific tensile and flexural moduli of the 50 wt% kenaf coupled composites were equivalent to or higher than typical reported values of 40 wt% coupled glass-polypropylene injection-molded composites. Typical commercial polypropylene composites were compared with kenaf-polypropylene composites (Table 1). Data on mineral-filled systems came from various sources (11,12). The specific flexural moduli of the kenaf composites with fiber contents greater than 40 percent were extremely high and even stiffer than a 40 percent mica-polypropylene composite.

Failure strain and tensile energy of absorption

The failure strain decreases with the addition of the fibers. Addition of a rigid filler/fiber restricts the mobility of the polymer molecules to flow freely past one another and thus causes premature failure. Addition of MAPP followed a similar trend to that of the uncoupled system, although the drop in failure strain with increasing fiber amounts was not as severe. The decrease in the failure strain with increasing amounts of kenaf for the coupled systems is apparent. The distribution of the fiber lengths present in the composite can influence the shape of the stress-strain curve since the load taken up by the fibers decreases as the strain increases (5). The tensile energy absorption, the integrated area under the stress-strain curve up to failure, behaves in roughly the same manner as the tensile failure strain The difference between the coupled and uncoupled composites increases with the amount of fibers present, although the drop in energy absorbed for the coupled composites levels off after the addition of about 35 vol% of fiber.

Impact properties

The impact strength of the composite depends on the amount of fiber and the type of testing (i.e., whether the samples were notched or unnotched).

TABLE 1.-*Results of previous work on biodegradable fiber in polypropylene (comparison with data on commercially available polypropylenes).*

Filler/reinforcement in polypropylene	ASTM standard	No filler	Kenaf	Talc[a]	Glass[a]	Mica[a]
Percent filler by weight (%)		0	50	40	40	40
Percent filler by volume (estimated) (%)		0	39	18	19	18
Tensile modulus (GPa)	D 638	1.7	8.46	4	9	7.6
Specific tensile modulus (GPa)		1.9	7.9	3.1	7.3	6.0
Tensile strength (MPa)	D 638	33	69	35	110	39
Elongation at break (%)	D 638	>>10	2.2	X	2.5	2.3
Notched Izod impact (J/m)	D 256A	24	37	32	107	27
Specific gravity		0.9	1.07	1.27	1.23	1.26

[a] Data from *Modern Plastics Encyclopedia and Materials Selector 1994*.

In case of notched samples, the impact strength increases with the amount of fibers added until a plateau is reached at about 45 percent of the fiber weight, irrespective of whether MAPP was used or not. The fibers bridge cracks and increase the resistance of the propagation of the crack. Contribution from fiber pullout is limited since the aspect ratio of the fibers in the system are well below the estimated critical aspect ratio of about 0.4 mm (1 7). In case of the unnotched impact values of the uncoupled composites, the presence of the fibers decrease the energy absorbed by the specimens. Addition of the fibers creates regions of stress concentrations that require less energy to initiate a crack. Improving the fiber-matrix adhesion through the use of MAPP increases the resistance to crack initiation at the fiber-matrix interface and the fall in impact strength with the addition of fibers is not as dramatic. The impact strength can be increased by providing flexible interphase regions in the composite or by using impact modifiers. Some work has been done in this area by the University of Wisconsin and the Forest Products Laboratory.

Physical properties

Water absorption and specific gravity of lignocellulosic fiber composites are important characteristics that determine end use applications of these materials. Water absorption could lead to a decrease in some of the properties and needs to be considered when selecting applications. It is difficult to entirely eliminate the absorption of moisture in the composites without using expensive surface barriers on the composite surface. Water absorption in lignocellulosic-based composites can lead to a build-up of moisture in the fiber cell wall and also in the fiber-matrix interphase region. Moisture build-up in the cell wall could result in fiber swelling and concerns about the dimension stability cannot be ignored. A typical 50 wt% of kenaf-homopolymer polypropylene blend absorbed about 1.05 wt% of water in a 24-hour water soak test. This is considerably higher than any mineral-filled systems. It is therefore very important to select applications where this high water absorption is not a critical factor, such as in electrical housing components.

The specific gravity of lignocellulosic-based composites is much lower than the mineral-filled thermoplastic systems. The apparent density of the lignocellulosic fibers in polypropylene is about 1.4 g/cc compared to mineral filler/fibers (about 2.5 g/cc). The specific gravity of a 50 wt% kenaf-polypropylene composite is about 1.07, while that of a 40 wt% glass-polypropylene composite is 1.23. The specific mechanical properties of kenaf-polypropylene composites compare favorably to other filled commodity plastics. Since raw materials are bought in units of weight, and finished articles are in general sold by the piece, more articles can be made with lignocellulosic fibers compared to the same weight of mineral fibers. This could result in significant material cost savings in the high-volume and low-cost commodity plastic market.

Conclusion

The cost of natural fibers are in general less than that of plastic. High fiber loading can result in significant material cost savings. The cost of compounding is unlikely to be much more than for conventional mineral/inorganic-based fillers presently used by the plastics industry. Due to the lower specific gravities of the cellulosic-based additives (approx. 1.4 compared to about 2.5 for mineral-based systems), the composite properties may have an advantage for automotive and other transportation applications. Furthermore, using the same weight of plastic/natural fiber as, for example, plastic/glass fiber, about 20 percent more pieces are possible. Cellulosic fibers are soft and nonabrasive and high filling levels are possible; 60 wt% of fiber has been incorporated in polypropylene at the Forest Products Laboratory and the University of Wisconsin. Reduced equipment abrasion and the subsequent reduction of retooling costs through the use of agricultural-based fibers is a factor that will be considered by the plastics industry when evaluating these natural fibers. Agricultural-based fillers and fibers won't completely replace conventional fillers and fibers, but these natural materials will develop their own niche in the plastics filler/fiber market in the future.

The volume of thermoplastics used in the housing, automotive, packaging, and other low-cost, high-volume applications is enormous. Recent interest in reducing the environmental impact of materials is leading to the development of newer materials or composites that can reduce the stress to the environment. In light of petroleum shortages and pressures to decrease dependence on petroleum products, there is increasing interest in maximizing the use of renewable materials. The use of agricultural materials as a source of raw materials to the industry not only provides a renewable source, but could also generate a nonfood source of economic development for farming and rural areas. Appropriate research and devel-

opment in the area of agricultural-based fillers and fibers in fiber-plastic composites could lead to new value-added, nonfood uses of agricultural materials.

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