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Polypropylene/cotton stalk biocomposites with enhanced characteristics

Abhishek Sachan, Veena Choudhary, K. K. Vimal, and G. S. Kapur

The influence of cotton stalk concentration, chemical treatment, and presence of a compatibilizer on the mechanical, rheological, and morphological properties of samples is investigated.

In recent years, it has become common practice to incorporate natural fibers—because of their low density and high specific properties—into polymer matrices as environmentally friendly reinforcing agents.[1–3] The use of natural fibers in this way also presents a number of other benefits over conventional synthetic-fiber-based composites. For example, the natural fibers pose fewer health risks during handling and manufacturing[4] and the natural composites require less energy for production (and thus cause less pollution to the environment).[5] To successfully use natural fibers as reinforcements in polymer matrices, it is vitally important to produce fibers that have reproducible physical properties. It can be extremely challenging, however, to obtain high-quality natural fibers (and to thus effectively replace traditional reinforcement materials) because the precise nature of the fibers varies from plant to plant, and depends on the cultivation conditions.[6–8]

It has previously been recognized that the substantial amount of biowaste produced every year in agriculture is very rich in lingocellulosic content[9,10] and thus has the potential to be used in polymeric composite materials.[11] For example, cotton stalk (CS) is a biowaste from the cultivation of cotton (an abundant crop in India), i.e., CS is the residue of cotton seed that remains after cotton fibers (used in textiles) have been obtained. Although there is nearly five times as much CS as cotton fibers, there has traditionally been no major use for this material and it has mostly been destroyed through burning, or as fuel for cooking in villages. CS, like other natural fibers, contains about 45% cellulose (along with small amounts of waxes, pectin, and water-soluble compounds),[12] and has a tendency to degrade at temperatures above 220°C.[13] CS can thus be mixed effectively with thermoplastics such as polypropylene (PP). In other words, CS can be used an economical natural-fiber reinforcement to further enhance the low-density and excellent mechanical properties of PP.[14–16] Indeed, the use of CS in this way has been the subject of several recent studies and these biocomposites are already being used, for instance, in automotive parts.[17] For example, it has been shown that the hydrophobic nature of CS can be reduced via chemical treatments (e.g., with alkalis, silane, permanganate, and stearic acid[18]) to improve the interaction between CS and the PP matrix, as well as with the incorporation of a compatibilizer. The effects of different CS chemical treatments on the mechanical, rheological, and dynamic mechanical properties of CS/biocomposites, however, have not yet been properly investigated.

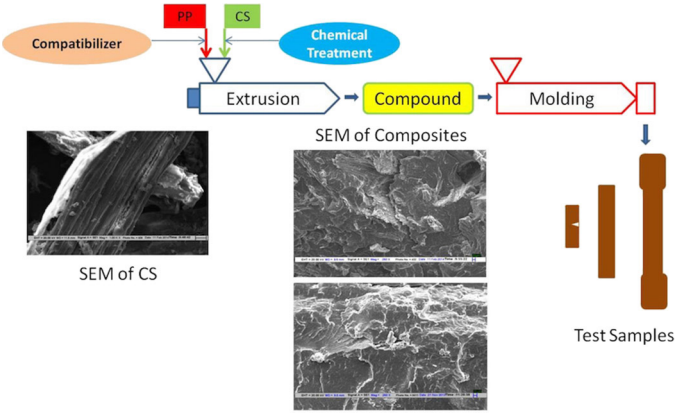

Figure 1. *Illustration of the full process chain for the preparation of polypropylene/cotton stalk (PP/CS) biocomposites used in this study. Scanning electron microscope (SEM) images of CS and the resultant composites are also shown.*

In this work,[19] we have thus conducted an experimental study to examine the effect of CS concentration, chemical treatment, and compatibilizer type on the mechanical, rheological, thermomechanical, and morphological properties of PP/CS biocomposites that we fabricated. We used the single-screw extrusion method with CS powder (ground from raw CS) and PP granules to prepare our biocomposites. To obtain the final specimens for the mechanical tests, we then injection molded the composites. The full process chain for our preparation method is illustrated in Figure 1.

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Table 1. *The thermal properties of neat PP and CS/PP biocomposites (without compatibilizer or chemical treatment). Data is given for the melting temperature (T_m), crystallization temperature (T_c), and crystallinity (X_c) of each sample.*

Sample CS concentration (wt%)	T_m (°C)	T_c (°C)	(X_c) (%)
0	164.2	114.4	49.6
10	164.7	117	47.1
20	151.2	122.1	49.5
30	165.2	120.1	48.3
40	164.8	120.3	50.5
50	163.9	118.9	49.3

For our initial tests, we included CS in the composites at concentrations of up to 50wt%—without any additional compatibilizer or chemical treatment—to try and improve the mechanical properties of PP. We found (see Figure 2) that this procedure did in fact improve the mechanical properties of the composites. For instance, we measured increases in the tensile modulus, flexural modulus, and impact strength of up to 83, 120, and 39%, respectively (for a CS concentration of 50wt%). In contrast, we observed a 23% decrease in the tensile strength for the 50wt% CS composite compared with neat PP (because of the poor interfacial interaction between the hydrophilic CS and the hydrophobic PP matrix). Furthermore, from our differential scanning calorimetry measurements, we found that the inclusion of CS (without any compatibilizer or chemical treatment) did not affect the thermal properties of the PP matrix (see Table 1).

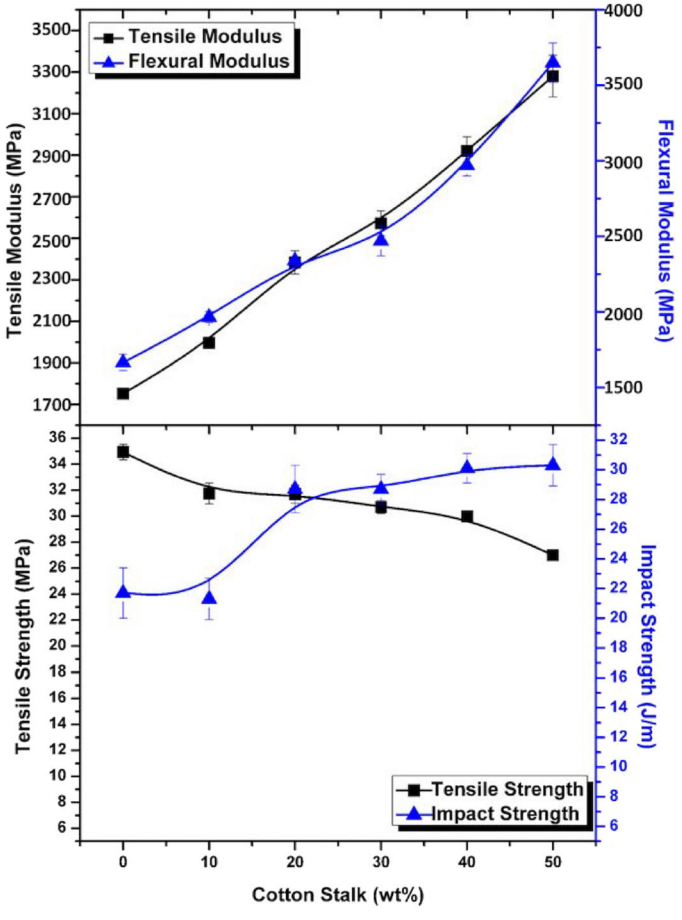

Figure 2. *Experimentally measured tensile and flexural modulus (top), and tensile and impact strength (bottom) of the PP/CS biocomposites (for CS concentrations up to 50wt%).*

In the next stage of our experiments, we added 6wt% of a compatibilizer—PP grafted with maleic anhydride (PP-g-MA)—to try and improve the tensile strength of our biocomposites. As a result, we measured tensile strength enhancements of 13 and 26% for the 30 and 40wt% CS composites, respectively (compared with the equivalent samples containing no compatibilizer). The tensile modulus and flexural modulus (rigidity) of the compatibilized samples was also improved, but we found that the impact strength of the composites decreased by 13–18% in the presence of PP-g-MA. We account for this decrease in impact strength by the improved adhesion at the CS–PP interface, i.e., which hindered energy absorption at the sudden loading that occurred at fiber pull-out from the matrix during the tests.

In our work we also attempted to optimize the tensile and impact strength of the biocomposites by examining the effect of different CS chemical treatments. To that end, we performed four simple chemical treatments on raw CS: an alkali treatment (with sodium hydroxide); an acid treatment (with stearic acid); a permanganate treatment (with potassium permanganate); and a silane treatment (with amino-propyl triethoxy silane). We thus used these differently treated CS fibers, with PP-g-MA, to prepare a set of composites. Overall, we found that the tensile properties of the chemically treated PP/CS biocomposites were better than those of the untreated and compatibilized samples. For example, the alkali-treated sample exhibited a 60% improvement in

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tensile strength (at 30wt% CS). The impact strength of this sample, however, showed no improvement from the untreated and compatibilized equivalents. The highest rigidity and tensile values we obtained were for the permanganate-treated samples, and the silane-treated composite exhibited the greatest improvement in tensile strength (and had a similar impact strength to the untreated and uncompatibilized samples). Our results therefore illustrate that the silane and permanganate treatments were the most effective (for the 30–40wt% CS composites).

In summary, we have systematically investigated how the use of different CS concentrations, chemical treatments, and a compatibilizer affect the properties of specifically synthesized PP/CS biocomposites. Our results demonstrate that the inclusion of CS (i.e., a biowaste material) improves the mechanical properties of PP. In addition, we find that by performing chemical modifications sequentially (i.e., the alkali treatment first, followed by the permanganate or silane treatment), we can optimize and enhance the mechanical characteristics of the samples. For our future work, we are planning to study the reactive extrusion of CS with PP, as well as the preparation of biocomposites that contain biodegradable polymers.

Author Information

Abhishek Sachan and Veena Choudhary
Indian Institute of Technology (IIT)
New Delhi, India

Abhishek Sachan received his MTech in polymer science and technology from IIT in 2014. He is now working as a PhD student in the Smart Plastics group at the European University of Brittany, France.

K. K. Vimal and G. S. Kapur
Research and Development Division
Indian Oil Corporation Limited
Faridabad, India

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