Final Report Submitted to Nebraska Wheat Board

Project Title: Development of Wheat Based Automotive Composites Using Wheat Gluten as the Matrix Polymer and Wheat Straw as the Reinforcement Material

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Summary

We thank the Nebraska Wheat Board for their financial support to this project. We are glad to report that we have successfully completed the objectives of this project. We have for the first time, developed 100% biodegradable composites with excellent properties using wheat gluten as the matrix material. The wheat gluten composites were developed using water as a plasticizer and without any chemicals. Wheat gluten based composites have better properties than similar PP based composites. A manuscript on developing 100% biodegradable wheat gluten based composites is being prepared for submission to a peer reviewed journal.

Introduction

Extensive efforts are being made to develop biodegradable and environmentally friendly composites and to reduce or eliminate the need for synthetic polymers (Mohanty, 2000). Natural and synthetic polymers derived from plants have been used as matrix and reinforcing materials to develop biodegradable and green composites. Proteins and oils from soybeans, starch based polymers as matrix materials and synthetic polymers such as poly(lactic acid) that are derived from corn are some examples of plant based polymers that have been used to develop environmentally friendly composites. Although natural fibers such as jute and kenaf are being explored to replace synthetic materials such as fiber glass that have been traditionally used for composites, there has been limited success. Inferior properties of natural fibers compared to glass fibers, hydrophilicity of natural fibers and non-compatibility between the hydrophilic natural fibers and hydrophobic synthetic polymers used as matrix or adhesives for the composites are some of the major reasons for the poor properties of the natural fiber based composites. In addition, utilizing synthetic polymers as matrix and natural fibers as composites reduces the biodegradability of the natural fibers and the composites. Therefore, attempts have been made to develop 100% biodegradable composites by using both matrix and reinforcing materials from annually renewable and sustainable resources.

Bicomposites were developed using chemically modified soy oils as resin and kenaf, kayocell and other biofibers (Tran, 2006). However, this method requires extensive chemical modification of soy oils to obtain the resins and pretreatment of the biofibers to obtain composites with good properties. Wheat gluten was treated with urea and formaldehyde and used as a binder for particle board (El-Wakil, 2007). It was reported that wheat gluten based boards met the standard requirements. In another report, wheat gluten was plasticized using high concentration (30%) of glycerol and used as the matrix material with natural fibers as reinforcement (Kunanopparat, 2008). However, the composites suffered from excessive swelling and had poor mechanical properties.

So far, it has not been possible to obtain biocomposites with good properties using plant proteins as the matrix without using extensive and expensive chemical modifications. In this research, we have used wheat gluten as the matrix polymer and water as a plasticizer to develop composites with excellent properties.

Materials and Methods

Materials

Commercially available wheat gluten (Wheat Pro 80) was supplied by Archer Daniels Midlands company. Jute fibers were purchased from Bast Fibers LLC, (Cresskill, NJ). The jute fibers had a average denier of 32, tensile strength of 2.4 g/denier, breaking elongation of 1.4% and Young's modulus of 187 g/denier. Polypropylene fibers were supplied by Drake Extrusion (Martinsville, VA). The PP fibers were of 15 denier and 84 mm long and crimped, width is 45 mm, tensile strength is 4.0 g/denier, melting temperature is 162 °C, melt flow index is 20 g per 10 min measured at 230° C, crystallinity is 50%, and density is 0.90 g/cm^3.

Composite fabrication

Wheat gluten-Jute Composites

The jute fibers were opened by hand and carded twice to open and parallelize the fibers. Mats of jute fibers were removed from the carding machine and cut to 10 x 12 inch rectangular pieces. The jute fiber mats were weighed to obtain the required amount of fibers depending on the ratio of the jute fibers and wheat gluten used for each composite. The required amount of wheat gluten was weighed and evenly sprayed on the jute fiber mats. Water equivalent to twice the weight of the total jute fibers and wheat gluten used was sprayed onto the jute fibers and wheat gluten. The weight of the pre-preg was determined to ensure that the same weight was used for each composite. The pre-preg was placed between two aluminum foils and compressed in a composite press (Carver Inc) at a predetermined temperature and for a particular time at a pressure of 20,000 PSI. Spacers (3.2mm) thick were placed at the edges of the composite press to control the density of the composite. After compression, the press was cooled by running cold water and the composite formed was removed.

PP-Jute Composites

The PP and jute fibers were carded separately. Later, the required ratio of PP/jute fibers was weighed and carded together three times to obtain homogenous mixing of the fibers. Mats of the fiber blends were removed from the carding machine and cut to 10 x 12 inch pieces. Several of the fiber mats were stacked to get the required weight/unit area and were later compression molded at 380 °F for 90 seconds. The temperature and time required to make the PP-jute composites was optimized in our earlier researches (Huda, 2008; 2009).

Optimizing conditions

The composite fabrication conditions such as the amount of reinforcing and matrix materials, time and temperature of compression molding were optimized. Initially, we optimized the amount of matrix and reinforcing material used in the composite. Later the temperature of processing the composite was optimized and finally, the optimum time of processing was determined.

Composite Characterization

The composites were conditioned in a standard testing atmosphere of 21 °C and 65% relative humidity for at least 24 hours before testing. Flexural tests were done according to ASTM standard D790-03 on a MTS (Model Q Test 10; MTS Corporation, Eden Prairie, MN) tensile tester equipped with a 500N load cell. The crosshead speed used was 10 mm/min. Tensile tests were performed on an Instron tensile tester (Model 4000, Instron, Norwood, MA) according to ASTM standard D638-03 using dog-bone shaped specimens. Crosshead speed was 5 mm/min.

Thermal Analysis

Wheat gluten without (unplasticized) and with water equivalent to twice the weight of wheat gluten (plasticized) was used to study the thermal behavior and effect of water on the plasticization of wheat gluten. A Mettler Toledo Differential Scanning Calorimeter (DSCS) was used for the thermal analysis. Sample was placed in closed pans and heated at 20 °C/min to obtain the DSC curves.

Results and Discussion

Effect of proportion of wheat gluten on flexural properties

Figure 1 shows the effect of increasing the proportion of wheat gluten on the flexural properties of the composites. Increasing the amount of wheat gluten affects the stiffness and flexural strength but the peak load and offset yield load are relatively unaffected. A wheat gluten concentration of 60% provides the highest flexural strength and stiffness. At low concentration of wheat gluten, there is insufficient binding material to hold the jute fibers together and therefore the composites have inferior properties. However, excessive wheat gluten makes the composite to be brittle and reduces the flexural strength.

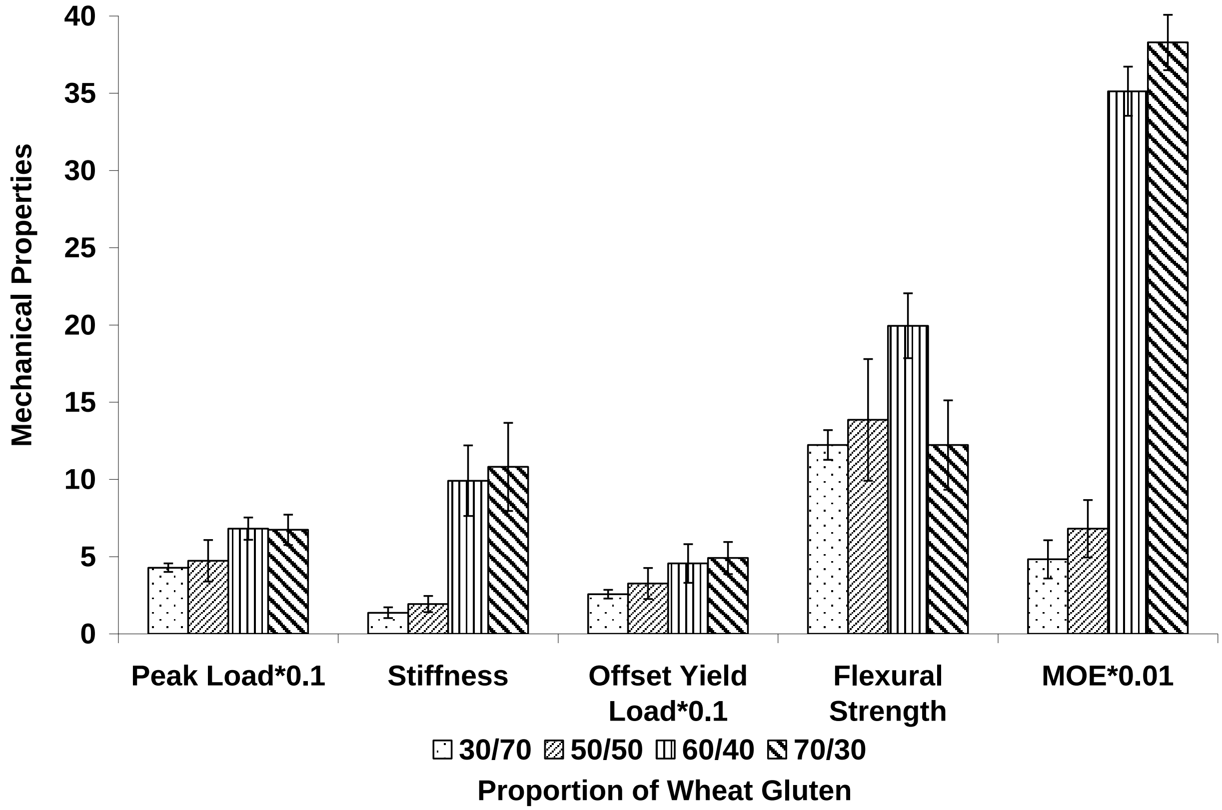

Figure 1 Effect of proportion of wheat gluten on the flexural properties of the composites. The composites were made at a temperature of 340 °F and compression time of 15 minutes.

Effect of compression time on flexural properties

The effect of increasing compression time on the flexural properties of the composites is shown in Figure 2. As seen from the figure, increasing compression increases the peak load, stiffness and flexural strength up to about 15 minutes and the properties decrease above a compression time of 15 minutes. The stiffness and flexural strengths show considerable increase when the compression time is increased from 5 to 10 minutes and from 10 to 15 minutes. However, there is a considerable decrease in the peak load, stiffness and flexural strength when the compression time is increased from 15 to 20 minutes. At short compression times, there is not enough time for the wheat gluten to plasticize and bind the jute fibers together. In addition, water used as plasticizers in the composites will not be completely removed at short compression times which may cause insufficient binding between the binding and reinforcing materials. At long compression times (20 minutes), wheat gluten becomes dehydrated and brittle leading to inferior composite properties.

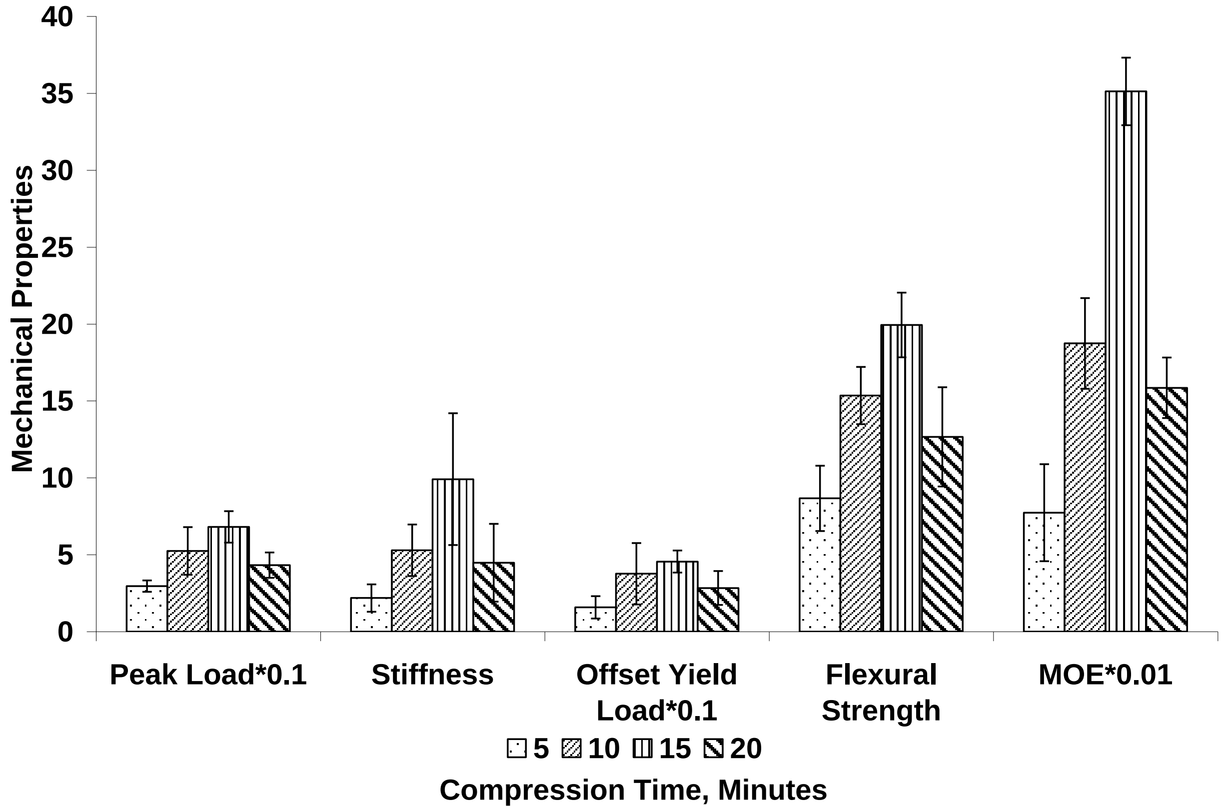

Figure 2 Effect of compression time on the flexural properties of the composites. The composites were made using 60% wheat gluten and 40% jute fibers at a temperature of 340 °F.

Effect of compression temperature on the flexural properties

Figure 3 shows the effect of compression temperature on the flexural properties of the composites. Compression temperature considerably affects the stiffness and flexural strength but the peak load and offset yield load remain relatively unaffected. As with the compression time, lower temperatures do not provide enough plasticization and removal of water from the composites resulting in poor flexural strength. However increasing temperature makes the wheat gluten brittle and hence reduces the strength of the composites.

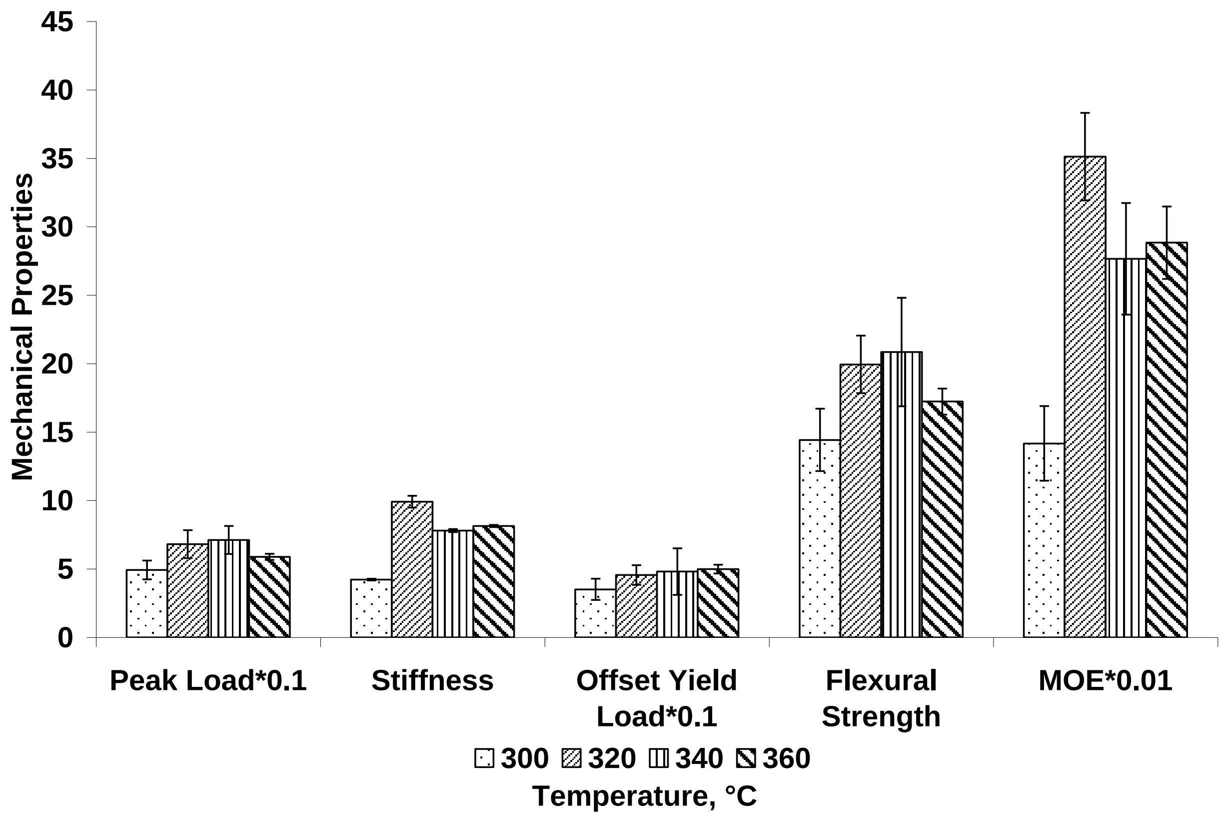

Figure 3 Effect of compression temperature on the flexural properties of the composites. The composites were made using 60% wheat gluten and 40% jute and a compression time of 15 minutes.

Effect of composite fabrication conditions on tensile properties

Table 1 shows the tensile properties of the wheat gluten/jute fiber composites at various composite forming conditions. As seen from the table, increasing the proportion of wheat gluten to 70% decreases the strength and modulus compared to composites containing 60% wheat gluten. However, the tensile strength and modulus of the composites remains similar at 320, 340 and 360 °F whereas increasing temperature from 5 to 10 and 10 to 15 minutes considerably increases the tensile strength and tensile modulus of the composites. Longer compression time (20 minutes) considerably decreases the tensile strength and modulus of the composites. As with the flexural properties, lower concentrations of wheat gluten results in insufficient plasticizer and high concentration (70%) of wheat gluten results in insufficient reinforcing material to provide good properties to the composites. Similarly, lower temperature or time do not provide enough plasticization and higher temperatures and longer time degrade the wheat gluten resulting in composites with inferior properties. Overall a wheat gluten concentration of 60%, compression temperature of 340 °F and compression time of 15 minutes provides composites with the best tensile strength and tensile modulus.

Table 1 Effect of proportion of wheat gluten, compression time and temperature on the tensile strength and tensile modulus of the composites

Composite property	Wheat gluten/Jute (%w/w)				Temperature, °F				Time, Minutes			
	30/70	50/50	60/40	70/30	300	320	340	360	5	10	15	20
Peak load, MPa	22.3 ±4.8	14.8 ±4.8	20.4 ±3.0	15.0 ±3.3	15.8 ± 1.7	20.4 ± 3.0	20.2 ± 1.8	20.4 ± 3.0	10.7 ± 4.4	17.6 ± 5.7	20.4 ± 3.0	11.6 ± 3.1
Modulus, MPa	1367 ±305	1133 ± 288	1548 ±138	1334 ± 109	1003 ± 314	1548 ± 139	1646 ± 82	1548 ± 139	935 ± 235	1365 ± 245	1548 ± 139	1127 ± 118

Thermal behavior

The thermal behavior of wheat gluten without (unplasticized) and with water (plasticized) equivalent to two times its weight is shown in Figure 4. As seen from the figure, the unplasticized wheat gluten has a relatively small and broad peak between 140 and 160 °C with a melting enthalpy of 110 J/g that is probably due to the removal of moisture in the wheat gluten. The plasticized wheat gluten has a sharp and narrow peak between 50 and 60°C and a large broad peak between 100 to 160°C that has a melting enthalpy of 2270 J/g nearly twenty times higher than the melting enthalpy of the unplasticized wheat gluten. The DSC curves show that water is able to plasticize wheat gluten and make it thermoplastic. The broad melting range suggests that the smaller molecular weight proteins could melt earlier and those with high molecular weight melt later. Thermal analysis showed that the unplasticized wheat gluten had a s

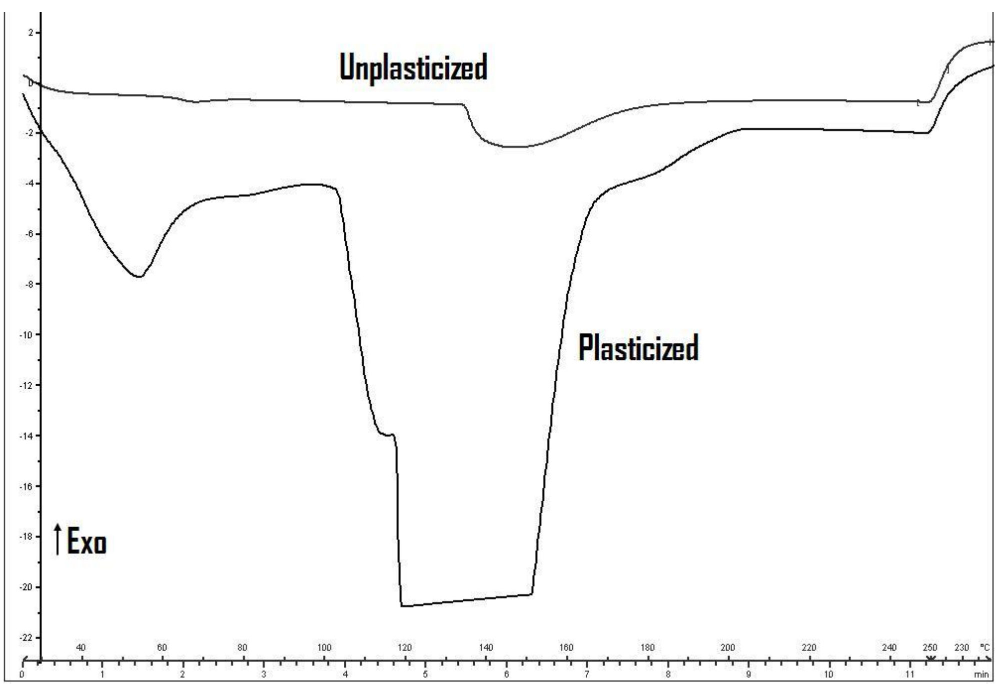

Figure 4 DSC curves of unplasticized and plasticized wheat gluten. Water equivalent to two times the weight of the wheat gluten was used as the plasticizer.

Comparison of wheat gluten/jute and polypropylene/jute composites

Table 2 provides a comparison of the flexural and tensile properties of composites developed using wheat gluten and PP as the matrix. Composites developed using wheat gluten as the matrix have much higher flexural strength and tensile properties compared to the PP composites. However, the wheat gluten composites are much stiffer than PP composites mainly due to the brittleness of wheat gluten whereas PP will be flexible in the composite.

Table 2 Comparison of properties of wheat gluten and polypropylene composites. The composites contain 60% matrix (wheat gluten/PP) and 40% reinforcing (jute) materials

Matrix Material	Flexural strength, MPa	Stiffness, N/mm	Modulus of Elasticity, MPa	Tensile Strength, MPa	Tensile Modulus, MPa
Gluten	20.0 ± 2.1	11.2 ±3.7	3962 ± 130	20.4 ± 3.0	1548 ± 139
Polypropylene	15.1 ± 1.0	1.8 ± 0.2	620 ± 72	13.5 ± 1.3	979 ± 98

Conclusions

We have successfully developed 100% biodegradable composites with wheat gluten as the matrix and jute fibers as reinforcement and without using any chemicals. The composites developed have excellent flexural and tensile properties, better than similar PP composites. The wheat gluten composites could be useful for various applications. Further research is in progress to improve the properties of the composites developed and identify potential applications for the wheat gluten based composites.

Acknowledgements

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