Blue-Agave Fiber-Reinforced Polypropylene Composites for Automotive Applications

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As consumer demand for more fuel-efficient vehicles increases, automakers are looking for innovative ways to reduce the weight of vehicles. Many automotive-grade plastics contain traditional reinforcing fillers, such as glass or talc, to improve the mechanical properties of the material. By replacing these high-density fillers with natural fibers, the material and corresponding weight can be reduced, which results in an improvement of the vehicle fuel economy. The objective of this study was to investigate the use of blue-agave bagasse fibers, which was sourced from tequila manufacturing waste, as a reinforcing agent in polypropylene composites. The effects of the fiber processing method, fiber loading level, and addition of a compatibilizer (polypropylene-grafted maleic anhydride) on the composite properties were determined. Samples were produced *via* twin-screw extrusion and injection molding. The resulting mechanical properties and morphology of the fracture surfaces were investigated. The fiber processing method (Agave C *vs.* Agave R) did not significantly affect the composite properties. Higher loading levels of fiber reduced both the elongation at break and impact strength, but increased the stiffness of the agave composites. The compatibilizer increased the fiber matrix adhesion, but reduced impact strength because the polymer matrix was softened.

Keywords: Composites; Natural fibers; Blue-agave; Morphology; Automotive; Melt compounding; Compatibility

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INTRODUCTION

Ford has an established leadership role in the research and implementation of plant-based materials, starting with company founder, Henry Ford. As resources become more constrained, the increasing environmental awareness from both consumers and governing bodies is driving manufacturers to focus on improving the fuel economy and reducing the carbon footprint of their vehicles. In the United States, the current Corporate Average Fuel Economy standards require that automakers increase the fuel economy of their cars and light duty trucks to an average of 54.5 mpg by 2025 (NHTSA 2012). In order to achieve this goal, automakers are in-part looking to develop lower-density materials. The use of polymers in vehicles has increased from 6% in 1970 to 16% in 2010, and is expected to continue to grow (Miller *et al.* 2014). Many of these polymers contain high-density fillers, such as glass fiber or minerals (talc, calcium carbonate, wollastonite, *etc.*), to boost the mechanical properties of the material without a remarkable increase in the cost. However, by removing or replacing these fillers with lightweight natural fibers, the density of each part can be reduced. Ahmad *et al.* (2015) reported that the use of natural fiber composites in the automotive industry has been growing at an annual rate of over 20%. For example, Ford Motor Company implemented a cellulose fiber-filled armrest and kenaf fiber-filled door substrate in the Lincoln MKX and Ford Escape, respectively. In addition to their low density, natural fiber composites have many advantageous characteristics, such as robust mechanical properties, low cost, being less abrasive than inorganic fibers to injection molding tooling, and lower energy consumption during

processing (Kiziltas *et al.* 2012; Ozen *et al.* 2012; Ahmad *et al.* 2015; Aydemir *et al.* 2015; González *et al.* 2015). Many natural fibers, such as cellulose, kenaf, flax, hemp, jute, and more, have been shown to be good lightweight replacements for traditional fillers (Ozen *et al*. 2013; Ahmad *et al.* 2015; Langhorst *et al.* 2015; Kiziltas *et al.* 2016).

In 2015, approximately 3.5 million vehicles were built in Mexico, making that country the seventh largest producer of vehicles in the world and the second largest vehicle supplier for the United States (Sada 2016; OICA 2017). The production in Mexico grew almost 6% between 2014 and 2015, with at least eleven major automakers outsourcing production to the country (Sada 2016; OICA 2017). Forbes estimates that production in Mexico will increase by at least 50% by 2020, and Mexico will become the sixth largest vehicle producer in the world (Sada 2016; Muller 2014).

The state of Jalisco in Mexico is the largest producer of tequila, an alcoholic beverage made from the blue-agave (*Agave tequilana* Weber var. Azul) plant, and 90% of the global production occurs in this state (Leduc *et al.* 2008). The plant takes approximately seven years to grow to full maturity. After maturing, the plant is harvested, and the nectar is extracted from the stem of the plant (commonly called the piña) and fermented to make tequila. The fibrous remains of the piña are commonly called bagasse, which is a term also used in the sugar cane industry to refer to the remains of sugarcane stalks after they are pressed to extract the sugar-bearing juice (Encyclopædia Britannica 2017). Over 940 thousand tons of blue-agave were used to produce tequila in Mexico in 2016 (Consejo Regulador del Tequila 2017). According to Leduc *et al.* (2008), the disposal of agave bagasse is an important issue within Jalisco. A lot of work has been performed to find novel value-added uses for agave bagasse (Iñiguez-Covarrubias *et al.* 2001a; Iñiguez-Covarrubias *et al.* 2001b; Consejo Regulador del Tequila 2017). One possible solution is the repurposing of the fibers for large-scale alternative uses, such as a reinforcing agent for polymer composites. According to the United States Environmental Protection Agency, more petroleum is used in the transportation of non-local goods than local goods because non-local goods must travel long distances to reach their customers. By using blue-agave fibers to reinforce polymeric parts that are molded and assembled for vehicles in Mexico, automakers could produce lightweight vehicle components, while appealing to consumer demand for locally produced products with small carbon footprints.

Many studies have investigated the use of agave fiber as a filler or reinforcement in polymeric matrices (Tronc *et al.* 2007; López-Bañuelos *et al.* 2012; Kaewkuk *et. al.* 2013, Moscoso-Sánchez *et al.* 2013, Pérez-Fonseca *et al.* Poly. Comp. 2014, Pérez-Fonseca *et al.* Mat. Des. 2014; Cisneros-López *et al.* 2016; Cisneros-López *et al.* 2017). Additionally, much work on the use of other fibers within the agave plant genus has been investigated recently as polymeric reinforcement (*i.e. Agave sisalana*, *A. americana*) (Zuccarello and Scaffaro 2017; Zuccarello and Zingales 2017). The chemical composition of *agave tequilana* Weber var. Azul in comparison to other agave species is shown in Table 1, and the single fiber properties of *A. tequilana* and *A. americana* are shown in Table 2.

Table 1. Chemical Composition of *Agave tequilana, A. sisalana*, and *A. americana* Fibers in Comparison to Pine (*Pinus oocarpa*) and Eucalyptus

	Cellulose (%)	Lignin (%)	Hemicelluloses (%)	Ash (%)	Source
Agave tequilana	64.9	16.8	5.5	2	Iñiguez-Covarrubias *et al*. 2001a
Agave sisalana	47-62	7-9	--	0.6-1	Han and Rowell 1997
Agave americana	63.19	2.72	32.09	--	Sghaier *et. al*. 2012
Pinus oocarpa	47.5	27.4	16.2	0.2	Zavala *et. al*. 1998
Eucalyptus	49.4	18.2	21.2	0.4	Fernandez *et. al*. 1999

Table 2. Single Fiber Tensile Properties of *Agave tequilana* and *Agave americana*

	Diameter (µm)	Tensile Strength (MPa)	Young's Modulus (GPa)	Elongation at Break (%)	Source
Agave tequilana	426.6 (63.3)	58.1 (21.0)	2.6 (0.8)	15 (7)	Flores-Sahagun *et al.* 2013
	328.7 (80.1)	41.5 (25.9)	2.7 (1.0)	11 (9)	
	345.8 (76.1)	49.9 (31.8)	2.9 (0.9)	12 (6)	
Agave americana	60-80	154 (47)	2.9 (1.2)	16.4 (8.2	Bessadok *et al*. 2008
	80-100	65 (42)	1.9 (0.6)	13.6 (11.9)	
	100-120	80 (12)	1.3 (0.2)	22.3 (6.0)	
	120-140	70 (21)	1.4 (0.3)	19.1 (10.7)	
	60-140	100 (32)	1.7 (1.1)	19.3 (8.2)	

Standard deviation is indicated in parentheses, when applicable.

However, to the knowledge of the authors, no studies investigating the mechanical behavior of injection-molded blue-agave fibers in polypropylene (PP) matrices have been published. Injection molding of natural fiber composites is especially relevant to the automotive industry because the majority of polymeric vehicle components are produced *via* injection molding techniques. This study evaluated the effect of blue-agave fiber-reinforcement on the mechanical behavior of injection-molded PP composites with the goal of producing a lightweight polymer system that could be applied to automotive parts. Additionally, several studies have shown that the use of a grafted maleic-anhydride polymer as a compatibilizer between hydrophilic cellulosic fibers and hydrophobic polymer matrices improves the fiber-matrix adhesion (Bataille *et al.* 1989; Qiao *et al.* 2004; Bullions *et al.* 2006; Niska and Sain 2008; Malkapuram *et al.* 2009; Langhorst *et al.* 2015). This study investigated the effectiveness of polypropylene-grafted-maleic anhydride (PPgMA) as a compatibilizer between agave fibers and a PP homopolymer matrix.

EXPERIMENTAL

Materials and Generation of the Composites

Agave fibers were supplied from the tequila manufacturer Jose Cuervo™ in Jalisco, Mexico. The fibers were dried and roughly chopped before shipment. Two types of fibers were supplied that varied in the way they were processed during the agave extraction process. Fibers labeled Agave C were washed with 85 °C water after agave nectar extraction, whereas fibers labeled Agave R were steam-treated before agave extraction. Prior to compounding, the fibers were chopped further using a Nelmor Grinder (AEC, New Berlin, USA) and filtered through a 4.76-mm (3/16 in) diameter mesh to control the fiber size. The PPgMA was supplied by Dupont (Fusabond P613, Wilmington, USA) with a grafting level of 0.5 wt.% maleic-anhydride. LyondellBasell Pro-fax 6523 virgin PP homopolymer (Rotterdam, Netherlands) was used in this study.

Fourteen samples were produced, and the formulations of each are given in Table 3. Both Agave C and Agave R fibers were blended with PP at loading levels of 10, 20, and 30 wt.% in the presence and absence of 5 wt.% compatibilizer (PPgMA). Control samples that consisted of 100% PP and PP with 5% PPgMA were also produced.

Samples were compounded using a twin-screw extruder (ThermoHaake Rheomex Model PTW25, Sigma-Aldrich, St. Louis, USA) with barrel temperatures that ranged from 160 to 180 °C. Prior to extrusion, the agave fibers and polymer pellets were dried for at least 12 h in a 60 °C conventional oven to reduce the moisture content. The fibers and pellets were separately starve fed

into the extruder using K-Tron gravimetric feeders (Coperion, Stuttgart, Germany), and the resulting samples were immediately quenched in a room temperature water bath. The compounded materials were granulated using a lab-scale grinder/chopper, and then dried in a conventional oven for 12 h at 60 °C to reduce the moisture content before being molded into test specimens with a Boy Machines Model 80M injection molding machine (Exton, USA) (barrel temperature range: 175 to 185 °C, mold temperature: 26 °C).

Table 3. Experimental Design for the Preparation of Agave Fiber-Filled Composites

Formulation	Final Composition		
	PP [wt.%]	**PPgMA** [wt.%]	**Agave Fiber** [wt.%]
PP	100	0	0
PP + PPgMA	100	5	0
PP + 10% Agave C	90	0	10
PP + 20% Agave C	80	0	20
PP + 30% Agave C	70	0	30
PP + 10% Agave C + PPgMA	85	5	10
PP + 20% Agave C + PPgMA	75	5	20
PP + 30% Agave C + PPgMA	65	5	30
PP + 10% Agave R	90	0	10
PP + 20% Agave R	80	0	20
PP + 30% Agave R	70	0	30
PP + 10% Agave R + PPgMA	85	5	10
PP + 20% Agave R + PPgMA	75	5	20
PP + 30% Agave R + PPgMA	65	5	30

Test Procedures

Mechanical testing

Tensile, flexural, and impact tests were performed according to ASTM D638-10 (2010), ASTM D790-10 (2010), and ASTM D256-10 (2010), respectively. The samples were tested in an environmentally conditioned room maintained at 23 °C and 50% relative humidity.

The tensile tests were performed on an Instron 3366 machine (Instron, Norwood, USA) using a 5-kN load cell, 50-mm extensometer, and extension rate of 5 mm/min. The three-point bend flexural tests were performed using a 50-mm wide support span and were run at a crosshead speed of 1.25 mm/min. The notched Izod impact tests were performed using a TMI machine (model 43-02-03, New Castle, USA) and a 2-lb pendulum. At least six tensile, five flexural, and ten impact specimens were tested for each sample.

The ultimate tensile strength, yield strength, elongation at break, and Young's modulus were determined from the tensile stress-strain curves. The flexural strength and flexural modulus were determined from the flexural stress-strain curves. The impact strength was determined from the notched Izod impact tests.

Microscopy

The fracture surfaces of the impact specimens were mounted vertically in epoxy resin to expose the composite cross-section perpendicular to the injection molding flow direction. After curing, the samples were ground and polished using successively finer grit sizes. The polished cross-sections were observed using an Olympus BX51 system microscope (Olympus Corporation, Shinjuku, Tokyo, Japan) and SPOT Insight Firewire 2 camera (SPOT Imaging, Sterling Heights, USA).

Water Absorption

Water immersion tests were performed according to ASTM D570. At least 8 specimens of each sample were immersed in distilled water maintained at 23°C. Samples were weighed to the nearest 0.00001 g before immersion, and weighed again after immersion 24 h, 7 days, and 21 days. Data points which fell more than 1.5 times the inner quartile range above the third quartile or below the first quartile were eliminated as outliers (1^{st} Quartile – (1.5*IQR); 3^{rd} Quartile + (1.5*IQR)).

Statistical analysis

To determine if the loading level, agave type, and compatibilizer had an effect on the mechanical properties, an analysis of variance (ANOVA) was performed using the Minitab statistical analysis program (Pennsylvania State University, State College, PA). A general linear model was created for each property. P-values were calculated for each model to determine if a factor or interaction between factors had a statistically significant effect on a property.

In statistical hypothesis testing, the p-value is the probability that a given statistical model would be the same as or more extreme than the actual observed results when the null hypothesis is true. In this case, the null hypothesis tested was that a factor (or a multiple factor interaction) did not effect a property. Specifically, this was performed by comparing the sample mean differences between groups of data. A small p-value (< 0.05) indicates that the null hypothesis can be rejected with 95% confidence, meaning that a factor has a statistically significant effect on the property. The p-values calculated for each property's general linear model were tabulated and reported in Table 5.

RESULTS AND DISCUSSION

Mechanical Properties

The tensile, flexural, and impact properties are presented in Table 4 for all of the samples. The mechanical property requirements of 20% talc-filled PP (PP20Talc) and 20% long glass fiber-filled PP (PP20LGF) were gathered from the Ford material specifications for interior applications (specification numbers M4D729-A3 and M4D865-B5, respectively). These requirements must be met by testing specimens at 23 °C after injection molding *via* a one-end gated mold, as was performed in this study.

In most cases, the addition of agave fibers to the PP matrices resulted in an increase in the stiffness and flexural strength, but reduced the elongation at break, tensile strength, and impact strength. However, a few samples exhibited impact strengths that were comparable with the controls. For example, the uncompatibilized 10% Agave C and Agave R samples had impact strengths of 4.1 (± 0.63) and 4.2 (± 0.31) kJ/m^2, respectively, whereas the unfilled PP had an impact strength of 4.9 (± 0.68) kJ/m^2 (Table 4).

The compatibilized 20% and 30% Agave C composites met the PP20Talc requirements for the yield strength, Young's modulus, and impact strength. However, all of the agave composites exhibited flexural moduli that were smaller than the PP20Talc requirement. The agave composites exhibited properties that were similar to those of a 25% wheat straw PP composite generated by Hornsby *et al*. (1997). However, the wheat straw composites had slightly higher tensile and flexural moduli values than the agave composites (Table 4). In comparison with the Ford material specification for PP20LFG, the agave composites exhibited values that were less than half that required for the tensile strength, Young's modulus, flexural modulus, and impact strength.

Overall, the compatibilized Agave C composites had a combination of properties that were similar to those of other natural fiber composites and could meet the requirements for PP20Talc. However, these composites may require some modification to improve the flexural modulus before replacing a traditional PP20Talc vehicle component. It is possible that an addition of a small amount of glass or talc to the 20% Agave C + PPgMA composite could cause an improvement in the flexural modulus and dimensional stability, while also helping to control the shrink of the polymer during molding.

Effect of the loading level on the properties

An increase in the agave loading level (from 10% to 30% agave) resulted in a more than 20% increase in the Young's modulus and 25% increase in the flexural modulus, while the elongation at break decreased by approximately 50% and the impact strength decreased by 30%-40% (Tables 4 and 5). The loading level had very little effect on the tensile or flexural strengths (Table 4).

These results were consistent with those seen in studies that examined the effect of the natural fiber loading level on polymer composites (Bigg 1987; Landel and Nielsen 1993; Faud *et al.* 1995; Zaini *et al*. 1996). Bledzki *et al.* (2002) observed a reduction in the impact strength and elongation at break with increased loading levels for both hardwood and softwood composites. Additionally, Stark and Berger (1997) noted that an increase in the loading level of wood flour in wood flour-PP composites resulted in a reduction in the tensile strength, but an increase in the flexural strength and Young's modulus. The fiber-matrix interfacial region is generally weak, allowing cracks to propagate more easily than through the polymer bulk (Zaini *et. al*. 1996). Also, the addition of filler to a polymer matrix limits the mobility of polymer chains, resulting in lower impact properties (Karmarkar *et al.* 2007). Impact strength of the composites can be improved with surface functionalization of agave fibers (silane, acetylation, NaOH treatment), the use of different coupling agents (styrene maleic-anhydride (SMA)), and the addition of impact modifiers (Karmarkar 2007).

Effect of the agave type on the properties

There were small statistically significant differences between the mechanical properties of the Agave C and Agave R composites (Tables 4 and 5). However, these differences can be considered negligible in large-scale manufacturing.

For example, the 10% Agave C + PPgMA composite exhibited an average ultimate tensile strength of 30.5 MPa, which was 2.7 MPa higher than that of the 10% Agave R + PPgMA composite (Table 4).

While this increase was statistically significant, the Agave C composites had 10% higher tensile strengths than the Agave R composites, at best (Table 5). In general, Agave C and Agave R resulted in very similar composites, which suggested that the waste streams could be combined if needed without a significant change in the properties.

Effect of the compatibilizer on the properties

The compatibilizer interacted with the agave type and loading level and affected the mechanical properties of the composites. The 30% Agave C composites responded most favorably to the addition of the compatibilizer, while the properties of the Agave R composites degraded or did not significantly improve (Tables 4 and 5).

Table 4. Summary of the Mechanical Properties of the Agave Composites and Control Samples

Sample	Ultimate Tensile Strength [MPa]		Yield Strength [MPa]		Elongation at Break [%]		Young's Modulus [MPa]	
PP + 20% Talc	-	-	26.5	-	-	-	2000	-
PP + 20% Long Glass Fiber	64.0*	-	-	-	-	-	4100	-
PP + 25% Wheat Straw[1]	-	-	29.7	(0.17)	3.60	(0.1)	2630	(103)
PP	29.1	(0.10)	29.1	(0.10)	11.1	(0.16)	1526	(104)
PP + PPgMA	29.0	(0.10)	29.0	(0.10)	11.1	(0.23)	1632	(32.2)
PP + 10% Agave C	27.1	(0.17)	27.1	(0.17)	8.28	(0.25)	1776	(80.7)
PP + 20% Agave C	25.1	(0.40)	25.1	(0.40)	5.56	(0.21)	2046	(55.3)
PP + 30% Agave C	23.2	(0.43)	23.2	(0.43)	4.17	(0.29)	2221	(60.4)
PP + 10% Agave C + PPgMA	30.5	(0.28)	30.5	(0.28)	8.79	(0.17)	1876	(31.5)
PP + 20% Agave C + PPgMA	31.2	(0.08)	31.2	(0.08)	6.72	(0.12)	2181	(49.3)
PP + 30% Agave C + PPgMA	31.7	(0.15)	31.7	(0.15)	4.81	(0.11)	2484	(15.9)
PP + 10% Agave R	27.4	(0.04)	27.4	(0.04)	9.13	(0.09)	1786	(59.2)
PP + 20% Agave R	25.8	(0.09)	25.8	(0.09)	6.75	(0.39)	1984	(54.7)
PP + 30% Agave R	22.9	(0.18)	22.9	(0.18)	4.86	(0.09)	2184	(171)
PP + 10% Agave R + PPgMA	27.8	(0.39)	27.8	(0.39)	8.30	(0.21)	1768	(40.9)
PP + 20% Agave R + PPgMA	28.1	(0.36)	28.0	(0.36)	5.02	(0.18)	2235	(110)
PP + 30% Agave R + PPgMA	25.0	(0.47)	25.0	(0.47)	3.56	(0.22)	2319	(155)

Sample	Flexural Strength [MPa]		Flexural Modulus [MPa]		Impact Strength [kJ/m^2]	
PP + 20% Talc	-	-	2300	-	1.7	-
PP + 20% Long Glass Fiber	-	-	4300	-	14.0	-
PP + 25% Wheat Straw[1]	-	-	2350	(40.0)	2.22**	(0.17)
PP	34.5	(0.29)	1140	(11.0)	4.9	(0.68)
PP + PPgMA	35.2	(0.50)	1166	(15.0)	4.2	(1.52)
PP + 10% Agave C	37.2	(0.71)	1312	(22.4)	4.1	(0.63)
PP + 20% Agave C	39.7	(0.17)	1552	(27.5)	3.3	(0.39)
PP + 30% Agave C	39.2	(0.53)	1776	(36.3)	2.8	(0.28)
PP + 10% Agave C + PPgMA	39.7	(0.35)	1353	(21.0)	2.8	(1.23)
PP + 20% Agave C + PPgMA	43.6	(0.49)	1547	(13.4)	2.8	(0.80)
PP + 30% Agave C + PPgMA	48.8	(0.33)	1801	(13.4)	2.8	(0.86)
PP + 10% Agave R	36.8	(0.32)	1273	(21.5)	4.2	(0.31)
PP + 20% Agave R	38.9	(0.29)	1469	(36.9)	3.7	(0.36)
PP + 30% Agave R	37.5	(0.59)	1641	(36.1)	2.7	(0.76)
PP + 10% Agave R + PPgMA	38.6	(0.24)	1311	(13.9)	3.2	(0.64)
PP + 20% Agave R + PPgMA	44.6	(0.57)	1654	(11.3)	3.3	(0.23)
PP + 30% Agave R + PPgMA	42.4	(0.92)	1795	(30.7)	1.9	(0.32)

[1] Hornsby *et al*. (1997)

* Ford requirement for tensile strength at break

** Charpy impact strength (Hornsby *et al*. 1997)

Parentheses indicate the standard deviation

Table 5. P-values from the General Linear Models

Property	Ultimate Tensile Strength	Elongation at Break	Young's Modulus	Flexural Strength	Flexural Modulus	Impact Strength
Factors						
Agave Loading Level	0.000	0.000	0.000	0.000	0.000	0.000
Agave Type	0.000	0.179	0.030	0.297	0.008	0.580
Compatibilizer	0.000	0.002	0.000	0.000	0.351	0.000
Interactions						
Loading Level & Compatibilizer	0.000	0.418	0.006	0.002	0.777	0.020
Loading Level & Agave Type	0.000	0.029	0.360	0.117	0.296	0.008
Compatibilizer & Agave Type	0.000	0.000	0.354	0.000	0.351	0.642
Loading Level & Compatibilizer & Agave Type	0.000					

The Agave C composites experienced significant increases in the tensile and flexural strengths with the addition of the compatibilizer, especially at high loading levels. For example, the 30% Agave C + PPgMA composite exhibited an average tensile strength that was nearly 30% higher than that of the uncompatibilized 30% Agave C composite (Table 4, Fig. 1). The addition of the compatibilizer to the Agave R composites resulted in small increases to the tensile and flexural strengths (< 15%) (Table 4). This interaction effect between the agave type and compatibilizer could have been the result of differences in the surface chemistry. It is possible that because the Agave C fibers were washed with hot water prior to agave extraction, the surface contaminates were removed, which enabled better compatibilization. An evaluation of the surface chemistry *via* X-ray photoelectron spectroscopy or Fourier transform infrared spectroscopy will be performed in the future.

These results were consistent with previous studies, which observed improved dispersion and fiber matrix adhesion upon the use of maleic anhydride grafted polymers in natural fiber composites (Bataille *et al.* 1989; Qiao *et al.* 2004; Bullions *et al.* 2006; Niska and Sain 2008; Malkapuram *et al.* 2009; Langhorst *et al.* 2015). Niska and Sain (2008) and Qiu *et al.* (2003) experienced similar improvements to the tensile strength after the addition of 2 wt.% PPgMA to 50% wood flour–PP composites, and the addition of PPgMA to high-crystalline fibrous cellulose–PP composites, respectively. It has been shown that PPgMA forms covalent linkages between maleic anhydride groups and hydroxyl groups on the cellulosic portions of natural fibers (Hedenberg and Gatenholm 1995).

Efficient stress transfer will occur when there is significant bonding of PPgMA onto the hydroxyl groups of the cellulose portions of a natural fiber, and when the PPgMA contains a sufficiently long chain length such that it entangles with the PP matrix.

The compatibilizer slightly decreased the impact strength of the composites. A similar result was also reported by Karmaker *et al.* (2007) for wood fiber filled PP composites with a novel compatibilizer containing isocyanate groups. It is also believed that low impact properties can be the result of thermal degradation of the fiber during processing, or the over compatibilization of the composite.

Similarly, the compatibilizer reduced the elongation at break of the Agave R composites. For example, the elongation at break of the 30% Agave R + PPgMA composite was nearly 40% lower than that of the uncompatibilized 30% Agave R composite (Table 4).

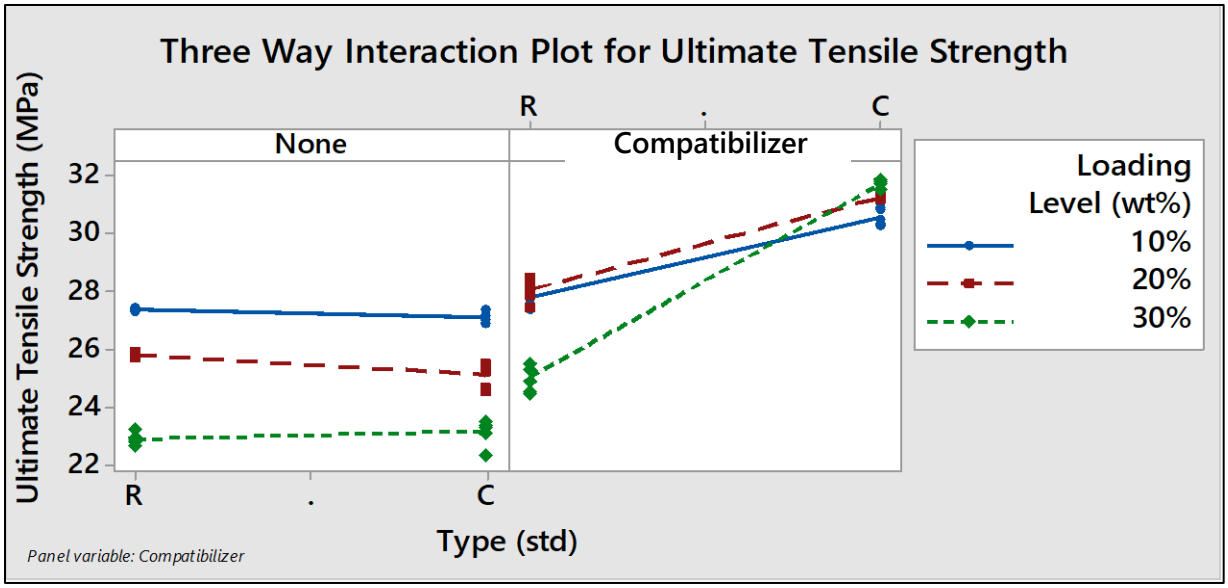

Fig. 1. Three-way interaction plot for the ultimate tensile strength

Microscopy

Optical micrographs were collected to investigate the dispersion and fiber-matrix adhesion of the agave fibers, with and without compatibilizer. Figure 2 shows the impact fracture surfaces of the 10% agave fibers within a PP matrix (50x magnification). For all samples, the fibers were uniformly dispersed throughout the matrix. However, Figure 2c (fracture surface of the 10% Agave C + PPgMA composite) showed better distributive and dispersive mixing of the agave fibers.

It is possible that, in compatibilized agave C composites, the fibers were more readily dispersed during processing. The presence of maleic-anhydride based compatibilizer has been shown to prevent fiber agglomeration, resulting in enhanced mechanical mixing, and better fiber dispersion (Felix and Gatenholm 1991, Hendenberg, *et. al* 1995). The compatibilizer bonds with the hydroxyl groups on the fiber surfaces, preventing hydrogen bonding between the fibers. By preventing agglomeration, the fibers are thoroughly dispersed as the melt undergoes sheer during processing (Felix and Gatenholm 1991; Hendenberg *et al.* 1995). It is believed that due to surface chemistry differences between agave C and Agave R fibers, the compatibilizer is better able to bond more effectively with agave C than Agave R fibers, resulting in improved dispersion.

Figure 3 shows an individual Agave C fiber within the PP matrix after failure. Without the compatibilizer, there was a visible gap between the agave fiber and PP matrix (Fig. 3b). However, when the compatibilizer was added, this gap was eliminated (Fig. 3a), which indicated that the presence of the compatibilizer improved the interfacial adhesion between the Agave C fibers and PP matrix.

Overall, compatibilized Agave C composites showed better tensile and flexural performance than compatibilized Agave R composites (Table 4). Several studies have shown that the formation of composites with good mechanical properties depends on good fiber dispersion throughout the matrix and strong fiber-matrix interfacial adhesion (Felix and Gatenholm 1991). Figures 2 and 3 show that compatibilized Agave C composites contain superiorly dispersed and adhered fibers than Agave R composites, causing the improved tensile and flexural performance exhibited by the composites.

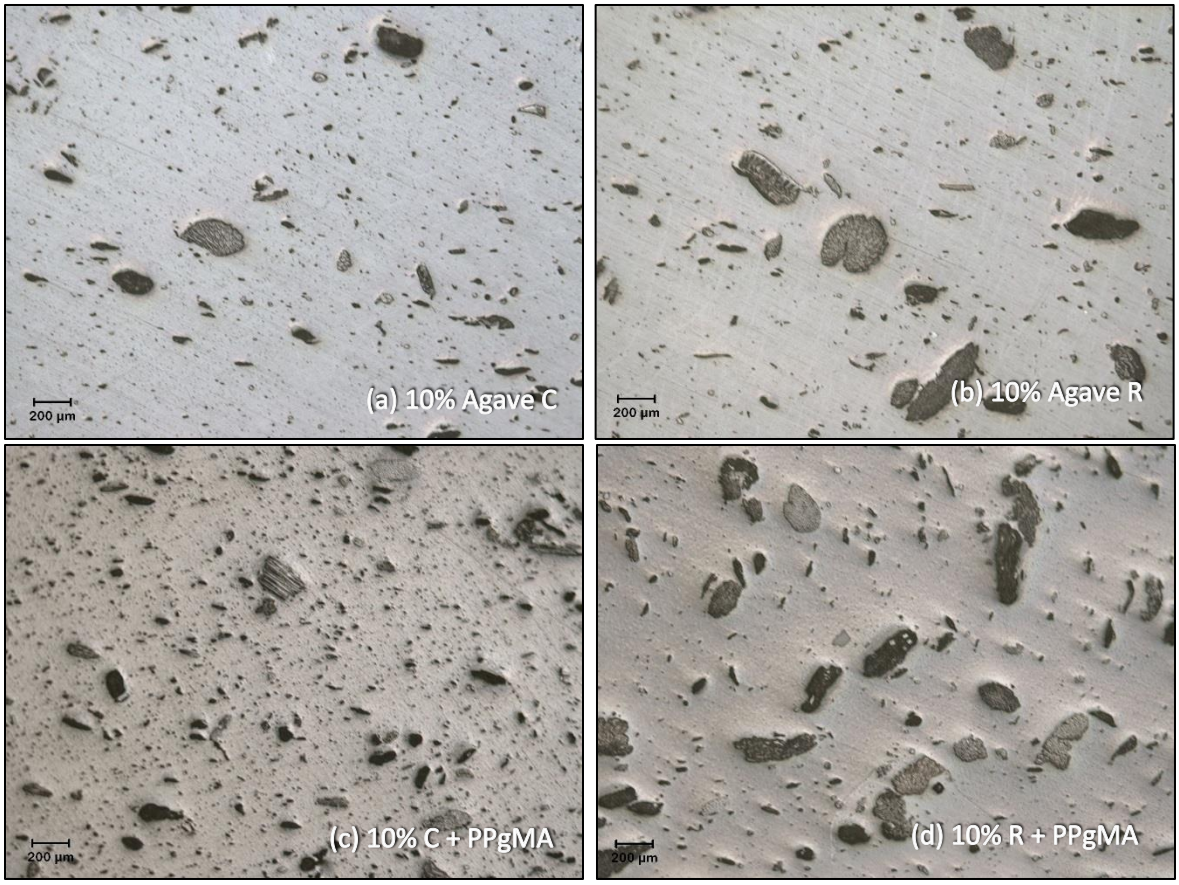

Fig 2. Optical micrographs (50x magnification) of the fracture surface of the 10% Agave + PP impact specimens: (a) 10% Agave C; (b) 10% Agave R; (c) 10% Agave C + PPgMA; and (d) 10% Agave R + PPgMA

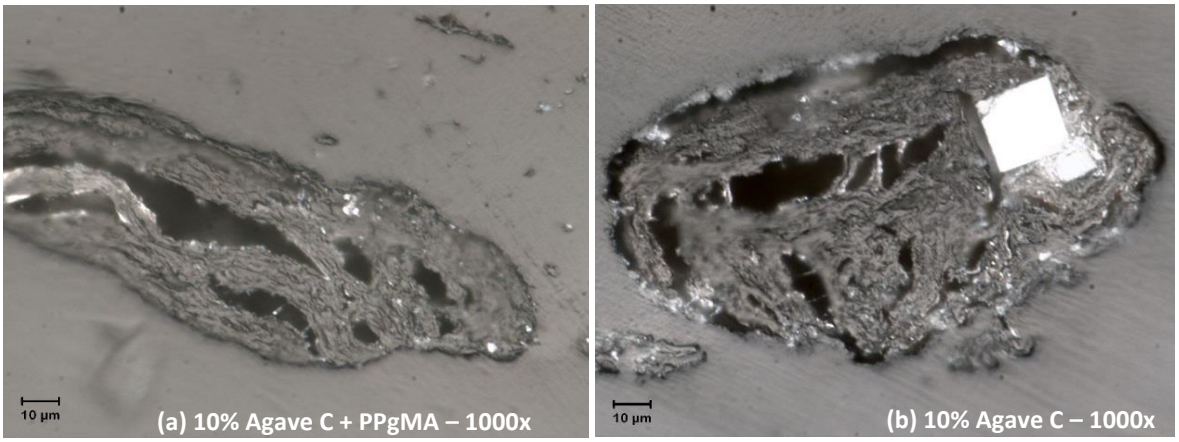

Fig. 3. Optical micrographs (1000x magnification) of single agave fibers in the 10% Agave C + PP impact specimens: (a) with the compatibilizer and (b) without the compatibilizer

Water Absorption

Water absorption of the agave composites is shown in Table 6. The water uptake by weight was measured after 24 h, 7 days, and 21 days of immersion, and the percentage weight change was calculated.

Absorption increased as immersion time increased, and as fiber content increased. For example, after 21 days of immersion, the weight of compatibilized samples containing 10%, 20%, and 30% Agave R increased by approximately 0.2%, 0.5%, and 1% respectively (Table 6, Fig. 4).

Additionally, Agave C composites absorbed more water over 21 days of immersion than Agave R composites. This suggests that the processing of the fibers during tequila production significantly affects the fiber surface chemistry. The Agave R fibers undergo a steam treatment during agave nectar extraction.

It has been shown that steam treatment or steam explosion of natural fibers results in the decomposition of the main components of the fiber: cellulose, hemicellulose, and lignin, with hemicellulose depolymerizing first (Jakobsons *et al.* 1995). Additionally, hemicellulose is the most hydrophilic component of the fiber (Saheb and Jog 1999). It is possible that during steam treatment of the Agave R fibers, some of the hemicellulose was removed, resulting in a more hydrophobic fiber.

The addition of compatibilizer had little to no effect on the absorption of the composites. The carboxylic groups of PPgMA react with surface hydroxyl groups of the cellulosic fiber, forming ester linkages. However, due to the large number of hydroxyl groups within a cellulosic fiber, even with maleic anhydride capping some surface hydroxyl groups, water can still interact with the bulk of the fiber. This result is consistent with previous work examining the water absorption of PPgMA treated cellulose fibers (Gauthier *et al.* 1998).

Overall, this result clearly shows that water absorption within agave composites is controlled by the fibers. The large number of hydroxyl groups within cellulosic fibers, such as agave, cause the hydrophilic nature of the fiber. Chemical changes to the fiber during processing may have an effect on the extent of absorption, as apparent between Agave C and Agave R composites. Figure 3 shows a noticeable gap between agave fiber and the polypropylene matrix. However, because there was no noticeable difference in water absorption between composites containing and lacking compatibilizer, water absorption was likely not occurring at the fiber, matrix interface, but within the fiber itself.

Table 6. Percentage Weight Change of Agave Composites upon Extended Water Immersion: Sample Weight was Measured Prior to Immersion and after Immersion for 24 Hours, 7 Days, and 21 Days

	Percent Weight Change					
	24 hours (%)		7 days (%)		21 days (%)	
PP	0.05	(0.03)	0.06	(0.11)	0.10	(0.15)
PP + PPgMA	0.03	(0.00)	0.02	(0.03)	0.05	(0.11)
PP + 10% Agave R + PPgMA	0.00	(0.05)	0.16	(0.04)	0.19	(0.04)
PP + 20% Agave R + PPgMA	0.33	(0.02)	0.19	(0.08)	0.45	(0.11)
PP + 30% Agave R + PPgMA	0.26	(0.02)	0.53	(0.00)	0.94	(0.02)
PP + 10% Agave C + PPgMA	0.11	(0.11)	0.17	(0.03)	0.34	(0.15)
PP + 20% Agave C + PPgMA	0.09	(0.02)	0.32	(0.01)	0.55	(0.09)
PP + 20% Agave R	0.11	(0.06)	0.20	(0.03)	0.38	(0.07)
PP + 20% Agave C	0.19	(0.08)	0.36	(0.04)	0.54	(0.07)

The weight change was recorded and the percentage difference was calculated. Parenthesis indicate standard deviation.

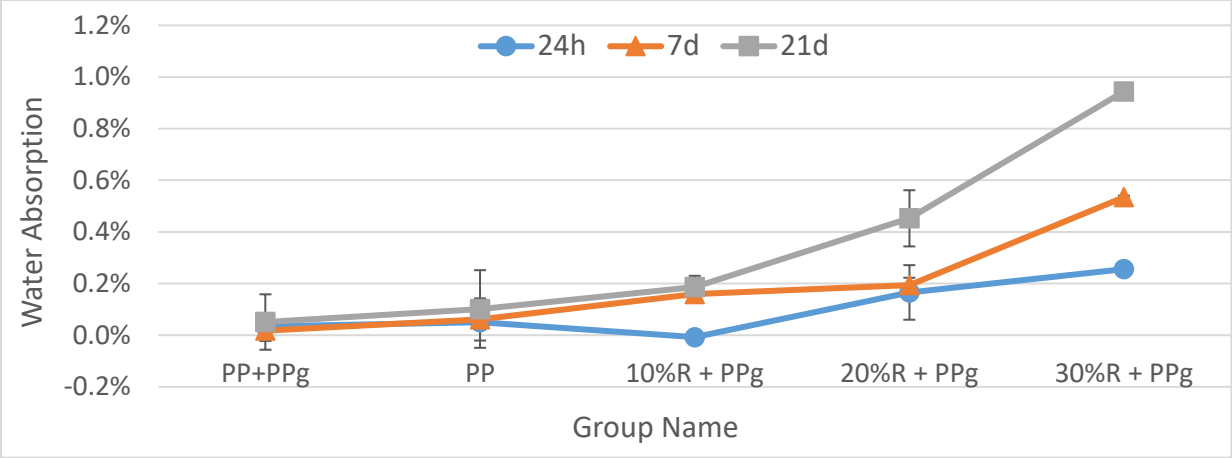

Fig. 4. Water absorption of compatibilized agave R composites with increasing fiber content. Water absorption increased with fiber content and immersion time.

CONCLUSIONS

1. The mechanical properties and morphologies of agave fiber-filled PP composites were investigated. Higher loading levels of agave fiber caused a reduction in the impact strength and elongation at break, but increased the stiffness.
2. The agave type did not have a significant effect on the mechanical properties. However, after the addition of the compatibilizer, the Agave C composites experienced enhanced tensile and flexural strengths, while the tensile and flexural strengths of the Agave R composites improved only slightly. The difference in the performances between the agave types in the presence of the compatibilizer could have been the result of differences in the surface chemistry caused by the fiber processing method. The Agave C fibers were washed with 85 °C water during processing. It is possible that this washing eliminated surface contaminates, which enabled better compatibilization.
3. Differences in performance between the agave types in the presence of compatibilizer is visually apparent: micrographs of Agave C samples show improved fiber-matrix adhesion and improved fiber dispersion. Compatibilization prevents fiber agglomeration, enabling better dispersion while improving fiber-matrix adhesion.
4. Water absorption within blue-agave composites increased with fiber content and immersion time, but compatibilizer had no effect on absorption. Agave C composites absorbed more water than Agave R, suggesting that the processing of the fibers during tequila production significantly affects the fiber surface chemistry.
5. Future studies will analyze the surface chemistry and roughness of the agave fibers to understand the effect of fiber processing. A fiber-debonding test will also be performed to better quantify fiber-matrix adhesion. Additionally, a hybrid composite with agave fiber and talc or glass will be created using a thermoplastic olefin matrix with the goal of improving the flexural modulus and impact strength of the system. Improvement of these properties will allow the system to meet the requirements for application in automotive interior components.

6. In summary, blue-agave PP composites have the potential to replace talc-filled PP components, resulting in a lower part density and lower overall vehicle carbon footprint. By optimizing the effects and interactions of the compatibilizer, agave type, and loading level, the composite could meet the required mechanical properties of an automotive composite system.

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