Surface Properties and Hardness of Polypropylene Composites Filled With Sunflower Stalk Flour

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The effects of agricultural flour content on surface roughness, wettability, and surface hardness of injection molded polypropylene (PP) composites was investigated. Four content levels of the waste sunflower stalk flour (WSF) were mixed with the PP with and without maleic anhydride grafted PP (MAPP) as a coupling agent. Contact angle measurements were performed using a goniometer connected with a digital camera. Three roughness parameters, average roughness (R_a), mean peak-to-valley height (R_z), and maximum roughness (R_{max}), were used to evaluate surface roughness. The surface roughness increased with increasing WSF content while their wettability decreased. The unfilled (neat) PP composites had the lowest surface roughness, while the roughest surface was found for the PP composites filled with 60 wt% WSF. The surface smoothness of the composites was noticeably increased by addition of the compatibilizer MAPP while the wettability was decreased. The scratch hardness of the PP composites increased significantly with increasing WSF. The incorporation of the coupling agent increased the scratch hardness of the specimens. The Brinell hardness increased with increasing filler loading. At similar filler loading the composites with MAPP had lower Brinell hardness value than those without MAPP.

Key words: Polypropylene composites; Waste sun flower stalk; Surface roughness; Wettability; Hardness

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INTRODUCTION

Thermoplastic polymer composites filled with natural fillers can be successfully overlaid with decorative wood veneer sheets using a suitable adhesive (Jarusombuti and Ayrilmis 2011). When the thermoplastic composites are used as substrate for thin overlays and liquid surface coatings, their surface characteristics such as roughness and wettability play an important role in determining the quality of the final product. This is because any surface irregularities on the top surface may show through the overlay and influence the quality of the final product. (Ayrilmis 2011; Hiziroglu *et al.* 2004; Nemli *et al.* 2005). The wettability can be affected by various factors, such as surface roughness, polarity, heterogeneity, and porosity. Good wettability will lead to good bonding and smaller contact angles, indicating greater wettability (Aydin 2004). This analysis is very important for wood and wood-based composites. For this analysis, a comprehensive understanding of possible formulations on surface behavior of thermoplastic polymer composites is needed.

The contact angle technique to characterize wettability and stylus profilometer for surface roughness has recently been used to indicate compatibility between the wood and the polymer in wood-plastic composites (WPCs) (Jarusombuti and Ayrilmis 2011; Ayrilmis and Kaymakci 2013). The contact angle measurement method is probably the most definitive way to determine the hydrophobicity of material surfaces. The angle is very high for water if the substrate is hydrophobic. When the surface is hydrophilic, the droplet quickly spreads and the measured angle is low (Doyle 2000; Namen *et al.* 2008). Generally, if the water contact angle is smaller than 90°, the solid surface is considered to be hydrophilic.

Lignocellulosic fillers can constitute a major part of thermoplastic composites. Such fillers have high polarity, such that they may exhibit poor bonding with the non-polar synthetic polymers of the matrix. Therefore, compatibilizers are used to achieve stronger linkages between both polar and non-polar components of the composite. Maleic anhydride polypropylene (MAPP) is a kind of compatibilizer commonly used in natural fiber-filled thermoplastic composites. MAPP has low surface energy and is expected to give good compatibility between the lignocellulosic filler and the polymer by formation of stronger linkages in the interfaces. Adhesion performance between lignocellulosic filler and polymer is weaker when a compatibilizer has not been applied.

The paintability and overlaying of the natural filled thermoplastic composites has gained significant importance in outdoor furniture industry. Previous studies showed that the amount of wood filler significantly affects the surface properties of WPCs (Jarusombuti and Ayrilmis 2011; Ayrilmis and Kaymakci 2013). However, the surface properties and hardness of thermoplastic composites filled with agricultural waste has not been studied. The main objective of this study was to evaluate the effect of filler content and compatibilizer (MAPP) on the surface roughness, wettability, and hardness of polypropylene (PP) composites filled with waste sunflower stalk flour (WSF). A typical chemical composition of the WSF is presented in Table 1 (López *et al.* 2005).

Table 1. Chemical Composition of Waste Sunflower Stalk Flour (WSF)

Chemical Composition	Value
Hot water solubles (%)	22.1
1 % NaOH solubles (%)	50.4
Holocellulose (%)	66.9
α-cellulose (%)	37.6
Lignin (%)	10.8

EXPERIMENTAL

Materials

Waste sunflower stalk

The waste sunflower stalks were supplied from a local farm located in Nilufer, Bursa, Western Turkey. The waste sunflower stalks were first dried in a laboratory oven at 60 °C for 10 h to a moisture content of 20 to 30% based on the oven-dry solid weight. Following the drying, the raw material was processed in a rotary grinder. Finally, it was passed through a U.S. 35-mesh screen and was retained by a U.S. 80-mesh screen. The waste sunflower stalk flour (WSF) was then dried in a laboratory oven at 100 °C for 24 h to a moisture content of 1 to 2%.

Polymer matrix and coupling agent

The polypropylene (MFI/230 °C/2.16 kg = 5.5 g/10 min, melting point: 161 °C) produced by Likom PP Corporation in Ukraine, was used as the polymeric material. The coupling agent, maleic anhydride-grafted polypropylene (MAPP-Optim-425, MFI/ 190 °C, 2.16 kg = 120 g/10 min), was supplied by Pluss Polymers Pvt. Ltd. in India.

Preparation of Injection-Molded PP Composites

The WSF, polypropylene, and MAPP granulates were processed in a 30 mm co-rotating twin screw extruder with a length-to-diameter (L/D) ratio of 30:1. The barrel temperatures of the extruder were controlled at 170, 180, 185, and 190 °C for zones 1, 2, 3, and 4, respectively. The temperature of the extruder die was held at 200 °C. The extruded strand was passed through a water bath and was subsequently pelletized. The pellets were stored in a sealed container and then dried to the moisture content of 1 to 2% in a laboratory oven before the injection molding. The temperature used for injection molded specimens was 180 to 200 °C from feed zone to die zone. The thermoplastic composite specimens were injected at an injection pressure between 5 to 6 MPa with a cooling time of about 30 s. Finally, the specimens were conditioned at a temperature of 23 °C and relative humidity (RH) of 50% according to ASTM D 618. The injection-molded specimens were in the form of a disk 50.8 mm in diameter and 3.2 mm in thickness. Density values of the specimens varied from 0.87 to 1.05 g/cm^3. The raw material formulations used for the PP composites are presented in Table 2.

Table 2. Compositions of the Unfilled and Filled PP Composites

Composite Type	Composite Composition		
	Waste Sunflower Stalk Flour (WSF) (wt %)	Polypropylene (wt %)	Coupling Agent (MAPP) (wt %)
A	30	70	-
B	40	60	-
C	50	50	-
D	60	40	-
E	30	67	3
F	40	57	3
G	50	47	3
H	60	37	3
I	-	100	-

Property Testing

Determination of surface roughness

Ten specimens were used from each type of PP composites filled with the WSF for surface roughness measurements. A total of 40 roughness measurements, four from each of ten specimens, were performed for each type of formulation. A Mitutoyo SJ-301 surface roughness tester, stylus type profilometer, was used for the surface roughness tests. Three roughness parameters characterized by ISO 4287: 1997, respectively, average roughness (R_a), mean peak-to-valley height (R_z), and maximum peak-to-valley height (R_y) were considered to evaluate the surface characteristics of the thermoplastic composites. The roughness values were measured with a sensitivity of 0.5 μm. Measuring speed, pin diameter, and pin top angle of the tool were 10 mm/min, 4 μm, and 90°, respectively. The length of tracing line (L_t) was 12.5 mm and the cut-off was λ = 2.5 mm. Measuring force of the scanning arm on the specimens was 4 mN (0.4 gf). Measurements were done at room temperature and the pin was calibrated before the tests.

Determination of Wettability

The contact angle was defined as the angle through the liquid phase formed between the surface of a solid and the line tangent to the droplet radius from the point of contact with the solid. The contact angles were obtained using a KSV Cam-101 Scientific Instrument (Helsinki, Finland). A sessile drop method was used to measure the contact angle (θ) of a 5-µL distilled water drop that was applied to the surface by means of a pipette. Image analysis software was used to measure contact angle and the shape and size of water droplets for the tested surfaces of thermoplastic composite specimens. The contact angle measurements were obtained by using a goniometer system connected with a digital camera and computer system. The liquid used for the measurements was distilled water at 20 °C with a surface tension of 72.80 mN/m. After the 5-µL droplet of distilled water was placed on the sample surface, the contact angles from the images were measured at 3 sec time intervals up to 120 sec total. Ten specimens were taken from each treatment type for contact angle measurements.

Determination of Hardness

Scratch hardness

Ten specimens were used from each type of PP composites filled with the WSF for hardness measurements. A total of 40 hardness measurements, four from each of ten specimens, were performed for each type of composite formulation according to ISO 4586-2 (2004). An Elcometer 3092 Sclerometer hardness tester was used for the hardness tests. For determination hardness; a spring (grey spring (0-3 N), red spring (1-10 N), blue spring (0-20 N) and green spring (0-30 N) force set by the collar; compressing the spring increases the force with which the tip was pushed to the surface of the test piece. By making short and straight movements while gradually increasing the load, the force was observed at which the tip left a mark or destroys the surface.

Brinell hardness

In order to measure the Brinell hardness of each type of PP, composites filled with the WSF were used for a total ten specimens for each type of composite. Brinell hardness of the composites was measured according to EN 1534 (2000) using a Llyod-Ametek material testing machine. The measurements were done using a steel ball of 10 mm diameter and load of 3 kN. It took 15 seconds to reach the maximum load of 3 kN; the load was maintained for 25 seconds, then within 15 seconds the load gradually was decreased to zero. The diameter of the remaining indentation opened through the sphere was then measured with a Brinell microscope.

Statistical Analysis

An analysis of variance, ANOVA, was conducted ($p< 0.01$) to evaluate the effect of the WSF content and coupling agent (MAPP) on surface roughness, wettability, and hardness properties of the PP composites filled with WSF flour. Significant differences among the average values of the composite types were determined using Duncan's multiple range tests.

RESULTS AND DISCUSSION

Surface Roughness

The R_a, R_y, and R_z values of the PP composites filled with WSF are presented in Table 3. The surface roughness values of the composites decreased with increasing polypropylene content. As the polypropylene is melted by press platens in the hot press, it fills capillaries (micropores) in the filler. This results in the smoother surface. Similar findings were also reported by Gupta *et al.* (2007). Statistical analysis revealed some significant differences ($p<0.01$) among the filled PP composite means for R_a, R_y, and R_z values. The results of Duncan's multiple range tests are indicated by letters in Table 3. Among the composites containing WSF, the composite type E had the smoothest surface with an R_a value of 1.66 µm, while the roughest surface was found for the composite type D having an R_a value of 2.34 µm. The surface roughness of the PP composites with and without MAPP significantly increased with increasing content of the WSF. Similar results were also observed for the R_y and R_z values of the filled composites with and without MAPP. Differences in the average surface roughness of the PP composites were most likely due to the amount of the WSF (Ozdemir *et al.* 2009; Akbulut *et al.* 2000; Ayrilmis *et al.* 2006). This was mainly attributable to the anatomical structure of the filler such as cavities inside (vessels and cell lumens). The lower surface roughness of the composites having higher polymer content can be explained as polymer melt at injection temperature. The polypropylene can crystallize on the filler and thereby wrap WSF better and leave less exposed particles on the composite surface. This results in lower surface roughness on the composite surface.

The filled PP composites without MAPP were found to have higher surface roughness than those with MAPP. The coupling agents, also known as compatibilizers, have the primary function in composites of improving the blend homogeneity of dissimilar or incompatible materials. Lack of homogeneity can prevent the development of satisfactory structural properties in the end product; hence the use of these materials improves physical and mechanical properties of the composites. The WSF-filled PP composites without MAPP were found to have higher surface roughness than those with MAPP and neat polypropylene (Fig. 1).

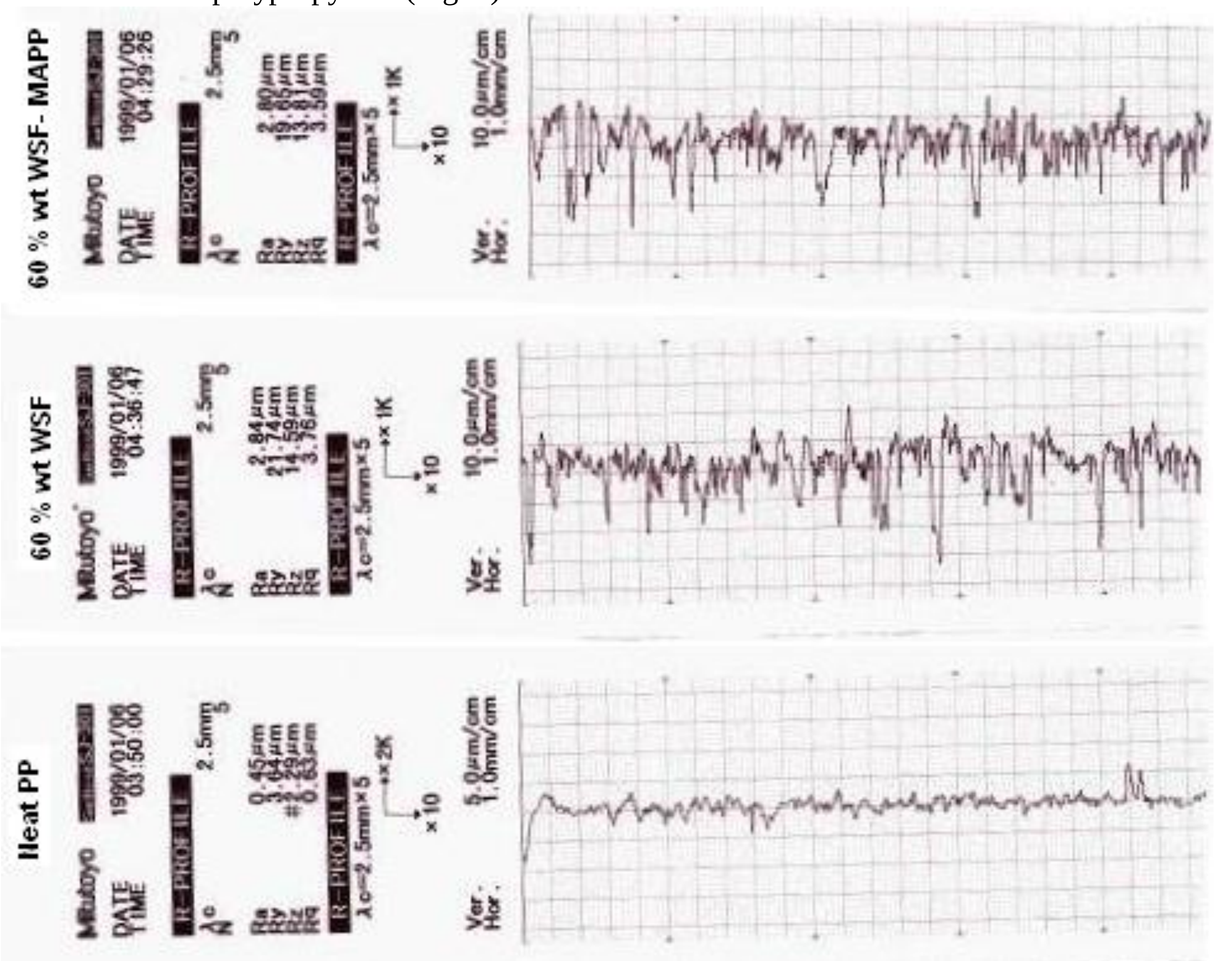

Fig 1. Typical surface roughness profiles of composite types H and D, and neat PP

The generally favorable results shown in Fig. 1 were consistent with previous studies (Jarusombuti and Ayrilmis 2011; Ozdemir and Mengeloglu 2008). For example, Ozdemir and Mengeloglu (2008) found that injection-molded WPC specimens made without MAPP had higher surface roughness (R_a: 14.91 µm) than those with MAPP (4% wt.) (R_a: 8.28 µm). Their study indicated that modification of the composites with MAPE coupling agent increased the surface smoothness. Similar results were also found by several researchers (Jarusombuti and Ayrilmis, 2011; Ayrilmis *et al*, 2006; Ayrilmis, 2011; Gupta *et al.* 2007). Gupta *et al.* (2007) reported that in the absence of a coupling agent between wood and polymer, the lower interfacial adhesion could result in removal of larger material chunks upon sanding and therefore higher surface roughness. It appears that the coupling agent decreased surface roughness of the composites due to having well-developed contact between WSF and polymer on the surface layers. This was also due to good dispersion of the WSF in the polymer matrix as the MAPP was incorporated in the composite.

Wettability

The wettability of PP composites increased with increasing content of the WSF. The neat PP composite had the lowest contact angle value, (58.1° for 5 s), while the highest contact angle value was found for composite type E (113.1° for 5 s). As shown in Table 3, the WSF filled composites having higher surface roughness showed better wettability than ones having lower surface roughness. It should be noted that lower wettability of rough surfaces may be due to the higher amount of peaks and valley points on the surface where liquid can be captured by capillary force.

Table 3. Variations in the Values of Average Surface Roughness and Contact Angle of Unfilled and Filled PP composites

Composite Type[1]	Density g/cm³	Surface Roughness Parameters			Contact Angle Measuring Intervals					
		R_a (µm)	R_y (µm)	R_z (µm)	5 s degree (°)	10 s degree (°)	30 s degree (°)	60 s degree (°)	90 s degree (°)	120 s degree (°)
A	0.98a (0.02)	1.72a[2] (0.2)	13.6a (2.06)	8.2a (0.94)	107.0ab (1.44)	105.4a (0.88)	104.5ab (1.66)	103.8a (1.45)	102.5a (3.44)	101.8a (3.50)
B	0.99a (0.01)	1.95bc (0.3)	17.6bc (2.92)	9.3ab (1.33)	105.3b (1.82)	104.3a (0.73)	104.0ab (0.84)	103.7a (0.79)	103.3a (0.75)	102.6a (0.97)
C	1.00a (0.01)	2.18de (0.2)	19.2cd (2.2)	10.5b (2.48)	95.9c (2.20)	93.2b (5.64)	92.4c (7.18)	89.4b (5.59)	88.6b (5.56)	87.3b (5.05)
D	1.04a (0.02)	2.34e (0.3)	24.3e (2.40)	14.4c (2.82)	94.6c (2.15)	93.1b (5.71)	91.9c (6.74)	90.4b (8.76)	89.3b (9.26)	87.7b (6.19)
E	0.99a (0.05)	1.66a (0.1)	12.1a (2.73)	8.0a (1.46)	113.2a (5.68)	112.0a (6.52)	111.7a (6.26)	109.6a (0.84)	108.3a (1.69)	107.0a (1.71)
F	1.00a (0.01)	1.83ab (0.5)	16.5b (2.79)	9.2ab (2.62)	108.0ab (1.65)	106.8a (1.81)	105.3ab (0.56)	104.1a (0.40)	103.5a (1.56)	102.9a (2.87)
G	1.01a (0.02)	2.08cd (0.3)	18.7cd (2.43)	10.1b (2.34)	98.1c (1.51)	95.9b (1.25)	94.8bc (5.59)	93.4b (4.72)	90.4b (4.27)	89.3b (6.10)
H	1.05a (0.04)	2.20de (0.4)	20.2d (1.61)	12.7d (1.35)	97.4c (3.77)	94.9b (3.60)	93.5bc (1.37)	91.3b (1.32)	89.4b (3.18)	88.8b (1.20)
I	0.87b (0.01)	0.45f (0.1)	3.6f (0.2)	2.3e (0.2)	58.1d (1.78)	57.6c (1.91)	57.4d (4.46)	56.9c (1.25)	56.6c (1.47)	54.6c (1.83)

[1]See Table 2 for composite formulation.
[2]Groups with same letters in column indicate that there is no statistical difference ($p<0.01$) between the specimens according to Duncan's multiple range test. The values in the parentheses are standard deviations.

Surface roughness has been proposed to enhance intrinsic adhesion by providing greater interfacial area and some mechanical interlocking mechanism for natural fiber filled PP composites (Jarusombuti and Ayrilmis 2011). A low contact angle is very important to capillary flow in the complex porous structure of wood to achieve a strong bond between adhesive and material surface. The incorporation of the coupling agent in the composites decreased the wettability of the specimens. Among the filled PP composites, the lowest contact angle with a value of 94.6° (5 s) was obtained from the PP composites containing 60 wt% WSF.

The contact angle values of the specimens were maximum at the beginning of the test but they decreased with increasing time (Fig. 2). The highest contact angle value with a value of 113.2° (5 s) was found for samples containing 40% WSF and 3 wt% coupling agent (MAPP). The contact angle of the specimens was significantly affected by increasing the WSF portion. This is expected because the WSF has a hydrophilic nature due to the presence of cellulose and hemicelluloses.

The chemical reaction of the hydrophilic hydroxyl groups of the lignocellulosics and acid anhydride groups of the MAPP form ester linkages and reduce the number of free hydrophilic groups (Mohanty *et al.* 2005). This indicates that chemical bonding of hydroxyl groups of the WSF with functional groups of the MAPP at the interface reduces surface water absorption of the specimens. The MAPP improves the interfacial adhesion between the WSF and polymer matrix, leading to less micro-voids and filler-polypropylene debondings in the interphase region. Better wettability of the composites without MAPP was mainly attributed to the presence of voids and defects mainly located in the filler/matrix interface.

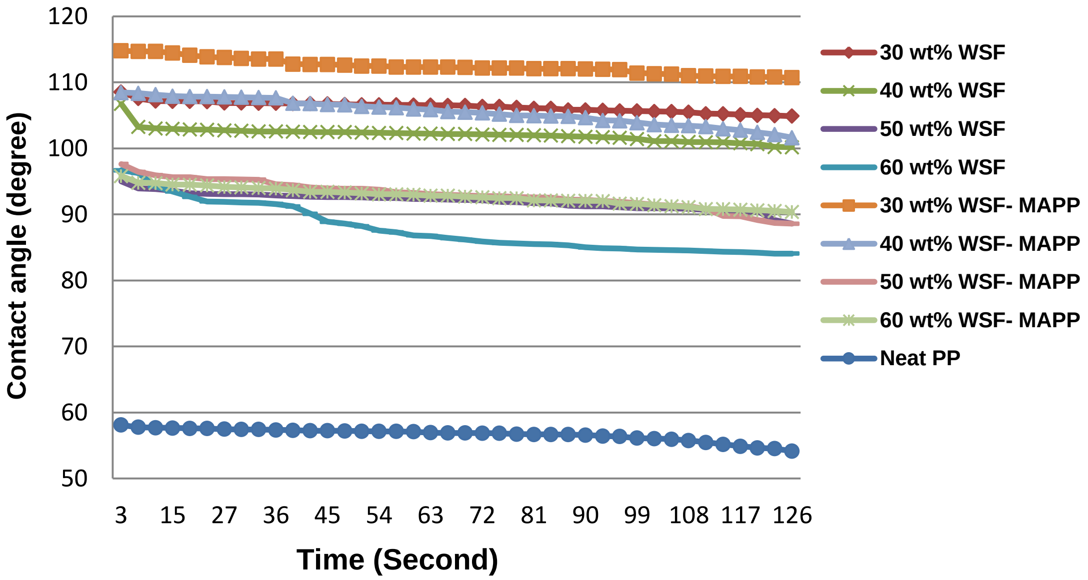

Fig. 2. Effects of WSF content and coupling agent (MAPP) on the contact angle of the PP composite (WSF: waste sunflower stalk flour)

Hardness

Scratch hardness

The scratch hardness values of the PP composites filled with the WSF are presented in Table 4. The hardness values of the PP composites increased with increasing WSF content, except for 30 wt%. Composite type A had the lowest scratch hardness value (2.3 N), while the highest scratch hardness value was found for composite type H (7.6 N). The scratch hardness values of the PP composites with and without MAPP increased with increasing WSF content (Fig. 3). The scratch hardness of the composites without MAPP increased by 226% as the WSF increased from 30 to 60 wt%. The incorporation of the coupling agent in the composites increased the scratch hardness of the specimens. For example, at the content of 30 wt% WSF, the scratch hardness of the PP composites increased by 13% as 3 wt% MAPP was incorporated into the composite. The improvement in the scratch hardness through coupling agent can be attributed to the improved interfacial adhesion between the WSF and polymer matrix, which led to less micro-voids and filler-polypropylene debondings in the interphase region.

Another explanation for improving scratch hardness was related to formation of ester bonds between the anhydride carbonyl groups of MAPP and hydroxyl groups of the natural fibers. Upon esterification, the exposed polyolefin chains can diffuse into the PP matrix phase and entangle with PP chains during the molding process. These changes create chemical bonds at the interface between the WSF filler and the PP matrix and thereby improve the compatibility between the WSF filler and PP matrix, which in turn, can enhance the scratch hardness (Ayrilmis 2013; Clemons 2002).

Table 4. The Variations in the Values of Scratch Hardness of Unfilled and Filled PP composites

Composite Type [1]	Scratch Hardness (N)
A	2.3 (0.22)a[2]
B	5.6 (0.41)b
C	6.9 (0.65)c
D	7.5 (0.67)c
E	2.6 (0.40)a
F	5.9 (0.62)b
G	7.1 (0.41)c
H	7.6 (0.56)c
I	4.1 (0.15)d

[1]See Table 2 for composite formulation.
[2]Groups with same letters in column indicate that there is no statistical difference ($p<0.01$) between the specimens according to Duncan's multiple range test. The values in the parentheses are standard deviations.

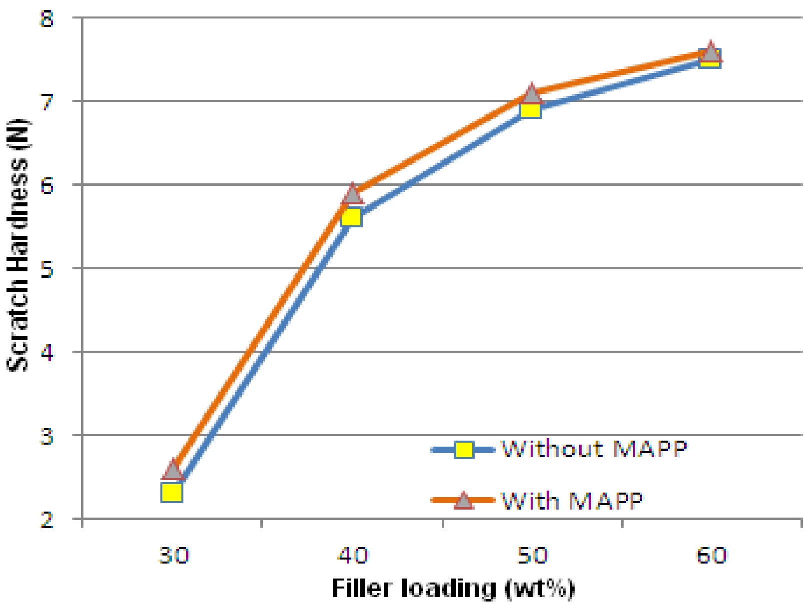

Fig. 3. Effects of WSF content and coupling agent (MAPP) on the scratch hardness of the PP composite (WSF: waste sunflower stalk flour)

Brinell hardness

The Brinell hardness values of the PP composites filled with WSF are presented in Table 5. The Brinell hardness values of the composites increased significantly with increasing WSF content. As shown in Table 5, the Brinell hardness of the filled PP composites was higher than that of the neat polypropylene composite, which was 48.8 N/mm^2. This result was consistent with previous studies (Kord 2011; Radojević *et al.* 2006). At similar filler loading, the PP composites with MAPP had lower Brinell hardness value than those without MAPP. For example, at the constant content of the WSF flour (60 wt%), the Brinell hardness value of the PP composites with MAPP was found to be 164.3 N/mm^2, while it was found to be 189.9 N/mm^2 for those without MAPP (Fig. 4).

Table 5. Variations in the Values of Brinell Hardness of Unfilled and Filled PP Composites

Composite Type[1]	Brinell Hardness (N/mm^2)
A	97.5 (15.6)a[2]
B	106.5 (24.9)ab
C	143.6 (12.4)c
D	189.9 (32.1)d
E	94.2 (16.7)a
F	100.9 (13.5)a
G	138.1 (15.3)bc
H	164.3 (20.6)cd
I	48.8 (10.3)a

[1]See Table 2 for composite formulation.
[2]Groups with same letters in column indicate that there is no statistical difference ($p<0.01$) between the specimens according to Duncan's multiple range test. The values in the parentheses are standard deviations.

As shown in Fig. 4, the modification of the PP composites with the MAPP decreased the Brinell hardness of the filled PP composites.

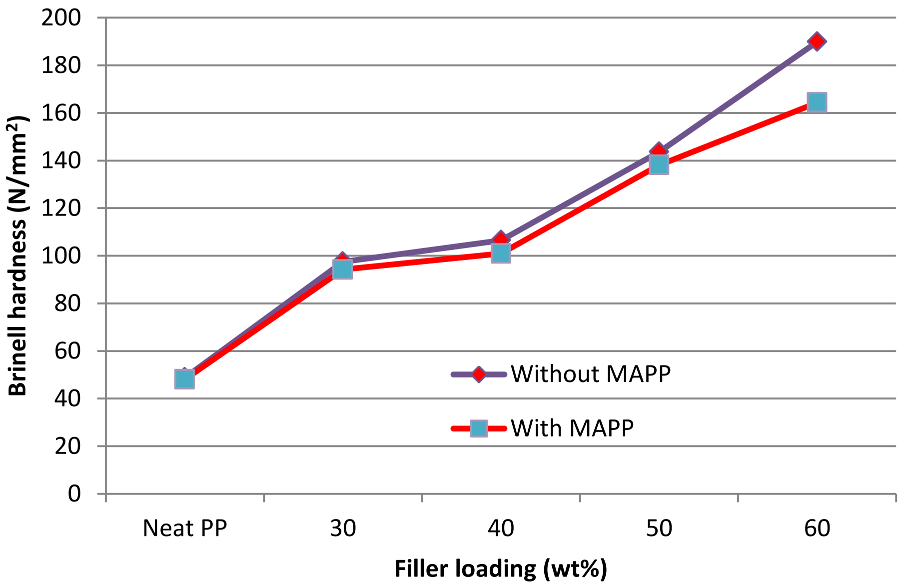

Fig. 4. Effects of WSF content and coupling agent (MAPP) on the Brinell hardness of the PP composite (WSF: waste sunflower stalk flour)

This decrease in Brinell hardness at high filler loadings with the addition of MAPP was mainly attributed to the thermoplastic character of the MAPP as compared to the lignocellulosic filler. In particular, the difference in the values of Brinell hardness of the coupled and uncoupled PP composites increased as the amount of the filler was beyond 50 wt% (Fig. 4).

CONCLUSIONS

The following conclusions were drawn from the results of the present study:

1. The surface roughness and wettability of the PP composites significantly increased with increasing content of the WSF.
2. The PP composite formulations without MAPP were found to have higher surface roughness but higher wettability. As the WSF content increased, the surface roughness values of the PP composites with and without MAPP increased. The surface roughness of the samples increased by 36% as the WSF increased from 30 to 60 wt%, where the wettability decreased by 11%. The PP composites with the MAPP showed lower surface roughness and higher contact angle values compared with those of without the MAPP.
3. The scratch hardness of the PP composites significantly increased with increasing content of the WSF. The scratch hardness of the composites without MAPP increased by 226% as the WSF increased from 30 to 60 wt%. The incorporation of the coupling agent in the composites increased the scratch hardness of the specimens. The Brinell hardness of the PP composites increased with increasing filler loading. At similar filler loading, the PP composites with MAPP had lower Brinell hardness value than the composites without MAPP.
4. The optimum surface properties and hardness for the composites were found for 50/47/3 formulation of WSF, PP, and MAPP.

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Article submitted: October 11, 2012; Peer review completed: November 1, 2012; Revised version received and accepted: December 7, 2012; Published: December 10, 2012.