

Utilization of Sunflower Stalk in Manufacture of Thermoplastic Composite

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Abstract Dimensional stability and mechanical performance of polypropylene thermoplastic composites filled with sunflower stalk (SS) flour at 30, 40, 50, and 60 wt% contents of the SS flour were investigated. The thickness swelling and water absorption of the specimens increased with increasing SS flour content. The modulus in the flexural and tensile improved with increasing SS flour content while the tensile and flexural strengths of the specimens decreased. The use of maleic anhydride polypropylene (3 wt%) had a positive effect on the dimensional stability and mechanical properties of the polypropylene thermoplastic composites filled with SS flour. The melting temperature of polypropylene decreased with increasing content of the SS flour. The degree of crystallinity of filled polypropylene composites between fibre loading of 0–30 % by weight was higher than that of unfilled

polypropylene composites. However, further increment in the filler content decreased the degree of crystallinity. The obtained results showed that SS flour could be potentially suitable raw material in the manufacture of polypropylene composites.

Keywords Dimensional stability · Waste sunflower stalk · Lignocellulosic filled polypropylene composite · Mechanical testing

Introduction

As a result of a growing awareness of the interconnectivity of global environmental factors, principles of sustainability, industrial ecology, ecoefficiency, and green chemistry and engineering are being integrated into the development of the next generation of materials, products, and processes [1–8]. The depletion of petroleum resources coupled with increasing environmental regulations are acting synergistically to provide the impetus for new materials and products that are compatible with the environment and independent of fossil fuels. Natural organic fibres from renewable bioresources offer the potential to act as biodegradable reinforcing materials, alternative for the use of glass or carbon fibres and inorganic fillers [9–11]. Compared to these materials, natural fibres present lower density; less abrasiveness; lower cost and are renewable and biodegradable. Also most plastics by themselves are not suitable for load-bearing applications due to their lack of sufficient strength, stiffness, low thermal stability, high moisture absorption and low dimensional stability [12]. However, fibres possess high strength and stiffness but are difficult to use in load-bearing applications by themselves because of their fibrous structure. In fibre-reinforced composites, the fibres serve as

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reinforcement by giving strength and stiffness to the structure while the plastic matrix serves as the adhesive to hold the fibres in place so that suitable structural components can be made [13]. Moreover, the fact that it is obtained directly from natural resources makes it even more attractive in terms of sustainability and environmental awareness [14]. Numerous studies were conducted to manufacture thermoplastic composites utilizing wood flour, plant flour or fibre including hemp, flax, jute, sisal, bagasse, ramie and kapok [15–23].

Turkey faces the problem of lack of woody raw material since most of its forested areas are unproductive. However, approximately 700 ha field is being utilized for sunflower farming in Turkey generating roughly 3–3.5 million tons of waste sunflower stalks every year [24]. The utilization of such a raw material in combination with other woody species available in the country could benefit both environmentally and economically [25]. Although sunflower stalk has been a subject of some studies, most of them are relevant production of bioethanol, particleboard, fibreboard and paper [25–29]. Therefore very limited information is available on the utilization of waste sunflower stalk in manufacture of thermoplastic composite.

Experimental

Materials

The sunflower stalks (SS) were obtained from local farmers in Ankara, Turkey. Prior to use the SS were separated outer bark and inner bark. The outer bark of SS was dried in an oven at 60 °C for 10 h to moisture content of 20–30 % based on the oven-dry SS solid weight. Following the drying, the SS were then processed by a rotary grinder without adding additional water. Finally, the SS flour passing through a U.S. 35-mesh screen and was retained by a U.S. 80-mesh screen. The SS flour was then dried in a laboratory oven at 100 °C for 24 h to moisture content of 1–2 %. The chemical properties of SS samples used in the experimental studies is presented in Table 1 [4].

The polypropylene (MFI/230 °C/2.16 kg = 5.5 g/10 min) supplied by a Petrochemical Company in Ukraine, was used as the polymeric material. The maleic anhydride polypropylene (MAPP) (Optim[®]P-425, MFI/190 °C, 2.16 kg = 120 g/10 min) as the coupling agent was supplied by Pluss Polymers Pvt. Ltd. in India.

Production of Sunflower Stalk Flour Filled Polypropylene Composites

The SS flour, and PP and with MAPP and without MAPP granulates were processed in a 30 mm co-rotating

Table 1 Composition of sunflower stalk [4]

Sunflower stalk (SS) composition	Value (wt%)
Moisture	11.44
Hemicellulose	7.40
Cellulose	43.10
Lignin	9.7
Ash	14.56
Total organic carbon	52.96
C	46.40
H	6.90
O	40.80

Table 2 Comparison of the injected molded composite types

Composite type	Composite composition		
	Sunflower stalk (SS) flour (wt%)	Polypropylene (wt%)	Coupling agent (MAPP) content (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	0	100	–

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, 190, 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 passed through a water bath and was subsequently pelletized. These pellets were stored in a sealed container and then dried for about 3–4 h before being injection molded. The temperature used for injection molded specimens was 170–190 °C from feed zone to die zone. The specimens were injected at injection pressure between 5 and 6 MPa with cooling time about 30 s. Finally, the specimens were conditioned at a temperature of 23 °C and relative humidity of 50 % according to ASTM D 618 [30]. The composition of the polymer composites is presented in Table 2. Two different polymer composites groups were produced. The first group consists of PP and SS flour in varying proportions. Second group had PP, SS flour in different proportions and MAPP coupling agent in the formulation. The formulations of the composites are given in Table 2.

Property Testing

Determination of Physical and Mechanical Properties

The thickness swelling (TS) and water absorption (WA) tests were carried out according to ASTM D 570 [31] specifications. The test specimens were in the form of a disk 50.8 mm in diameter and 3.2 mm in thickness. The conditioned specimens were entirely immersed for 1-day, 7-days, and 28-days in a container of water at 23 ± 2 °C. At the end of each immersion time, the specimens were taken out from the water and all surface water was removed with a clean dry cloth. The specimens were weighed to the nearest 0.01 g and measured to the nearest 0.001 mm immediately.

The flexural tests were conducted in accordance with ASTM D 790 using a Lloyd testing machine at a rate of 1.3 mm/min crosshead speed. [32]. Dimensions of the test specimens were 3.5 mm × 13.2 mm × 128 mm. The tensile tests were conducted according to the ASTM D 638 [33]. Tensile specimens (dogbone shape (type III)) were tested with a crosshead speed of 5 mm/min in accordance with ASTM D638. Seven specimens were tested for the tensile and flexural properties of each composite formulation.

Differential Scanning Calorimetry (DSC) Analysis

Melting and crystallization behavior of the composites were studied in a heat-flux type differential scanning calorimeter (DSC, Perkin Elmer-DSC 8000) according to ASTM D3418 [34]. Temperature and heat flow calibration of the instrument were performed with high purity indium (In), tin (Sn) and zinc (Zn) metals. The test specimens weighing about 9–10 mg in an aluminum crucible were heated up to 200 °C with the heating rate of 10 °C/min and kept at this temperature for 2 min to remove thermal history. Then the specimens were cooled down to 0 °C with the cooling rate of 10 °C/min by an electrical cooling device to allow the sample crystallize dynamically and kept at this temperature for 2 min. Subsequently, the non-isothermally crystallized specimens were re-heated up to 200 °C with the heating rate of 10 °C/min. All heating-cooling runs in melting and crystallization studies were carried out under nitrogen (N₂) atmosphere at a flow rate of 50 ml/min to prevent oxidation of the specimens. Degree of crystallinity ($X_c\%$) was determined from the second melting enthalpy values using the following equation:

$$X_c = \frac{\Delta H_m}{(1 - \alpha)\Delta H_m^0} \times 100$$

where, ΔH_m is melting enthalpy of the specimens (J/g), ΔH_m^0 is the enthalpy value of melting of a 100 % crystalline form of polypropylene (209 J/g) and $(1-\alpha)$ is the weight fraction of polymer into the composite material.

Interfacial Morphological Analysis

The morphology of tensile fracture surfaces was studied by means of scanning electron microscopy (SEM, JEOL Neo Scope JSM-5000) under an acceleration voltage of 10 kV. The test specimens were attached to an aluminum stub and sputtered with gold to eliminate the electron charging effects. The objective was to get some information regarding the filler dispersion and bonding quality between filler and polymer matrix and to detect the presence of micro defects.

Statistical Analysis

Analysis of variance (ANOVA) ($p < 0.01$) was used to determine the effect of SS content on selected mechanical and physical of the thermoplastic composites using SPSS statistical package program. Significant differences among the average values of the SS—PP thermoplastic composite types were determined using Duncan's multiple range tests.

Results and Discussion

Dimensional Stability

The TS and WA values of the SS reinforced polypropylene composites with and without the MAPP are presented in Table 3. The incorporation of the SS flour into the polypropylene resulted in high TS and WA values due to the hydrophilic property of SS, particularly above 50 wt% (Table 3). The specimens containing higher plastic content had less water absorption sites and thus decreased water absorption, as expected. However, the dimensional stability of the specimens was significantly improved by adding the compatibilizer MAPP. Because the coupling forms include covalent bonds, secondary bonding (such as hydrogen bonding and van der Waals' forces), polymer molecular entanglement, and mechanical interlocking [35]. It provided better adhesion and wettability between matrix and cellulosic fibre. Increase of interfacial bonds causes a reduction of the voids and limits penetration of the water molecules into fibre cell walls [36]. Significant differences ($p < 0.01$) between some group averages for the TS and WA values are shown in Table 3.

The rates of water absorption and thickness swelling were higher in case of higher loaded SS flour in the polypropylene composite. The reason behind this was the higher possibility of water absorption by hydrophilic nature of fibres, which proportionally increases the rate of water absorption with higher fibre loading. Polypropylene shows very negligible or no water absorption because of its being devoid of functional polar group and hence, it can be

Table 3 The physical properties of unfilled and sunflower stalk flour filled polypropylene composites

Composite type ¹	Physical properties						
	Density (kg/m ³)	Water absorption (%)			Thickness swelling (%)		
		1-day	7-days	28-days	1-day	7-days	28-days
A	980 (20) a	0.61 (0.05) ab ²	0.93 (0.05) ab	3.57 (0.13) ab	0.50 (0.08) a	1.03 (0.06) ab	3.02 (0.08) a
B	990 (10) a	0.67 (0.05) b	0.97 (0.02) bc	3.91 (0.21) bc	0.75 (0.09) bc	1.06 (0.08) abc	3.12 (1.46) a
C	1,000 (10) a	0.86 (0.06) cd	1.10 (0.05) d	4.03 (0.34) c	0.91 (0.07) cd	1.15 (0.06) c	3.18 (0.15) a
D	1,040 (20) a	0.94 (0.04) d	1.22 (0.06) e	4.09 (0.22) c	1.04 (0.13) d	1.28 (0.09) d	3.38 (0.49) a
E	990 (50) a	0.52 (0.12) a	0.86 (0.08) a	3.40 (0.28) a	0.42 (0.09) a	0.97 (0.08) a	2.97 (0.91) a
F	1,000 (10) a	0.61 (0.16) ab	0.93 (0.04) ab	3.78 (0.35) abc	0.65 (0.13) ab	1.01 (0.07) ab	3.09 (0.45) a
G	1,100 (20) a	0.80 (0.10) c	1.01 (0.05) c	3.93 (0.39) bc	0.88 (0.14) cd	1.09 (0.06) bc	3.15 (0.47) a
H	1,050 (40) a	0.89 (0.10) cd	1.09 (0.06) d	4.01 (0.33) c	1.00 (0.35) d	1.15 (0.04) c	3.29 (0.17) a
I	876 (10) b	0.06 (0.02) f	0.10 (0.01) f	0.18 (0.02) f	0.12 (0.01) e	0.14 (0.01) d	0.24 (0.01) b

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 Duncan's multiply range test

The values in the parentheses are standard deviations

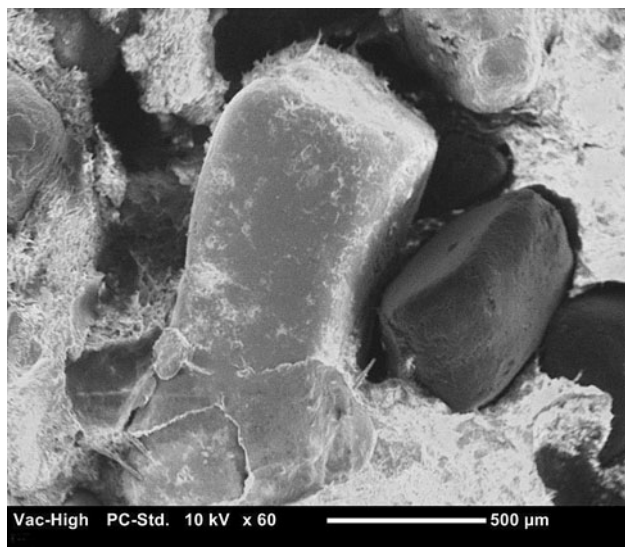


Fig. 1 SEM micrograph of the uncoupled polypropylene composite at 60 wt% loading of sunflower stalk flour (poor surface interaction)

assumed that 99.9 % of water is absorbed by SS flour. Furthermore, the SS were sufficiently encapsulated by increasing polypropylene matrix content. The formation of an interfacial layer over the surface of the dispersed particles can prevent water from penetrating its interior [11, 37]. 24 h WA of the neat polypropylene used in this study was found to be 0.06 % (Table 3). The WA of the composites was significantly affected by the presence of voids and defects mainly located in the filler/matrix interface. This was due to the poor dispersion of the filler in the polymer matrix. The SEM image of the fracture surfaces confirmed a loss of interfacial bonding between the filler and the polymer matrix (Fig. 1).

The dimensional stability of the specimens was significantly improved by adding the MAPP. This behavior can be attributed to the reaction of the hydrophilic–OH groups of the filler and the acid anhydride groups of the MAPP, thus forming ester linkages, as it has been proposed in the literature [37, 38]. The MAPP improves the interfacial adhesion between wood flour and polymer matrix, leading to less microvoids and fibre-polypropylene debondings in the interphase region. Due to the presence of polar hydroxyl groups in SS, the moisture absorption is high which leads to weak interfacial adhesion between the SS and the hydrophobic polymer matrix, which makes debonding. The chemical reaction (ester linkages) between the polypropylene and SS reduced the number of available hydrophilic groups [37, 38].

Adding the SS flour to the polypropylene increased its density. This was expected, because the cell density of the cellulose (average 1.3 g/cm³ after extrusion and injection molding) is much higher than injection molded polypropylene density (0.876 g/cm³) and is in accordance with previous reports [39, 40]. The densities of filled composites ranged from 0.980 to 0.110 g/cm³ while it was found to be 0.876 g/cm³ for the unfilled polypropylene. The densities of the coupled specimen were higher than those of the uncoupled specimens.

Flexural Properties

Table 4 summarizes the flexural properties of the thermoplastic composites. The flexural properties determined were flexural strength and flexural modulus. The coupled polypropylene composites showed significantly higher

Table 4 Mechanical properties of unfilled and sunflower stalk flour filled polypropylene composites

Composite type ¹	Mechanical properties				
	Flexural strength (MPa)	Flexural modulus (MPa)	Tensile strength (MPa)	Tensile modulus (MPa)	Elongation at break (%)
A	34.4 (2.82) ab ²	3.095 (174) a	19.7 (2.14) a	3.283 (394) a	1.61 (0.3) a
B	37.9 (1.91) b	3.819 (149) bc	23.4 (3.73) c	3.935 (317) ab	1.47 (0.09) a
C	35.1 (2.77) ab	4.460 (187) d	18.9 (2.39) a	4.207 (105) b	0.97 (0.16) b
D	30.1 (1.09) c	4.819 (360) de	13.2 (1.55) b	4.647 (177) b	0.73 (0.11) b
E	42.0 (2.02) d	3.485 (412) ab	22.1 (3.27) ac	3.964 (281) ab	1.64 (0.08) a
F	42.6 (3.49) d	4.290 (277) cd	27.7 (2.03) d	4.441 (525) b	1.57 (0.13) a
G	45.9 (1.05) e	4.534 (295) de	25.5 (2.73) cd	4.460 (489) b	1.34 (0.37) a
H	36.7 (3.96) a	5.077 (102) e	23.8 (2.32) c	5.730 (502) c	0.83 (0.09) b
I	28.3 (0.4) c	652 (73) f	27.8 (0.7) d	1.709 (92) d	8.59 (0.32) c

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 Duncan's multiply range test. The values in the parentheses are standard deviations

flexural strength and flexural modulus than the uncoupled composites. This was good consistent with previous studies [37, 41–43]. It is very well known that the performance of composites, for example the mechanical properties, depends on the properties of the individual components and their interfacial compatibility [43]. One of the most typical types of failure in composite materials is interfacial debonding [35, 36, 44, 45]. The MAPP provides higher mechanical interlocking between the filler and polymer matrix. A higher mechanical interlocking can increase the interfacial frictional sliding. This process can absorb significant quantities of energy and can affect the modulus and toughness of the reinforced polypropylene composite. If the interface is weak, effective load distribution is not achieved and the mechanical properties of the composites are impaired. The uncoupled composites showed a rough morphology with the presence of voids between the wood filler particles and the polymer matrix, clearly indicating the poor interaction between them (Fig. 1). The MAPP reduces the voids sizes and turns the surface more homogeneous confirming its effect on promoting adhesion in the interfacial region. The improvement in the interfacial adhesion and enhanced stress transfer from the polymer matrix to the stiffer cellulosic flour caused a significant increase in the MOE of the specimens [37, 41–44].

One of the most popular usage areas of thermoplastic composites is in decking boards. For polyolefin-based plastic lumber decking boards, ASTM D 6662 [46] standard requires the minimum flexural strength of 6.9 MPa. All filled polypropylene composites with and without MAPP in this study provided flexural strength values that are well over the requirement by the relevant ASTM standard [47].

Tensile Properties

The results of the tensile strength and tensile modulus of the SS flour filled polypropylene composites are given in Table 4. Tensile strength of the polypropylene decreased with the incorporation of SS flour. The tensile strength values of the filled polypropylene composites increased from 19.7 to 23.4 MPa as the amount of SS flour increased from 30 to 40 wt%. However, further increment in the filler content decreased the tensile strength of the composites. The MAPP coupled composites with a high content of the SS flour had better the tensile modulus and strength than the non-coupled ones (Figs. 2, 3). For example, at the constant content of the SS flour (40 wt%), the tensile strength value of the coupled composites was found to be 27.7 MPa while it was found to be 23.4 MPa for the uncoupled composites. The decrease in the tensile strength was associated with the poor dispersion and adhesion of the SS flour in the polymer matrix as the filler content was beyond 40 wt%. The poor adhesion between the SS flour and polymer matrix creates weak interfacial regions (Fig. 1). The decrement in the tensile strength was also due to the decrease in the binding level between the lignocellulosic filler and polymer matrix. Since the filler particles are very small, a high interfacial surface exists between the polar filler and the nonpolar polymer matrix. The deterioration of the tensile strength due to high loading of the filler beyond 40 wt% indicates the incapability of the filler particles to support the transfer of stress from the polymer matrix to the filler.

The results of the tensile modulus test are similar to the results of the MOE test; the composites with high wood flour content and treated with the MAPP had better MOE

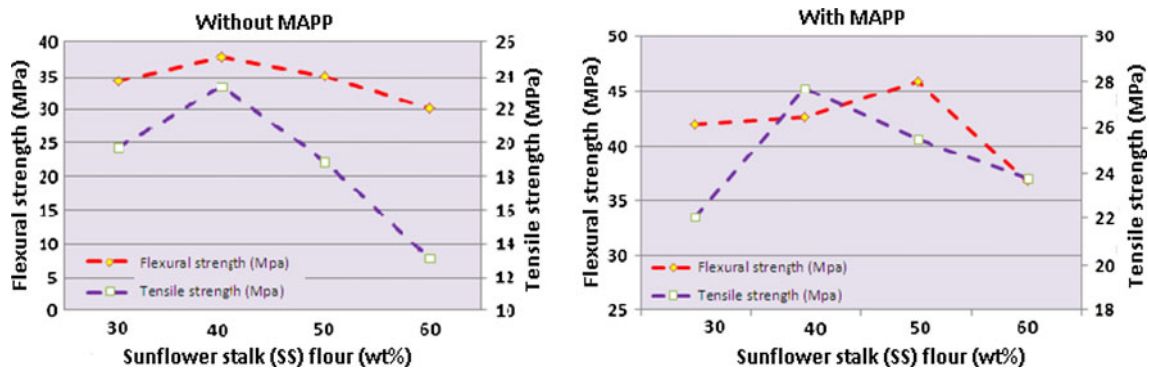


Fig. 2 The variation of flexural and tensile strength of polypropylene composites without and with MAPP as a function of the SS flour content

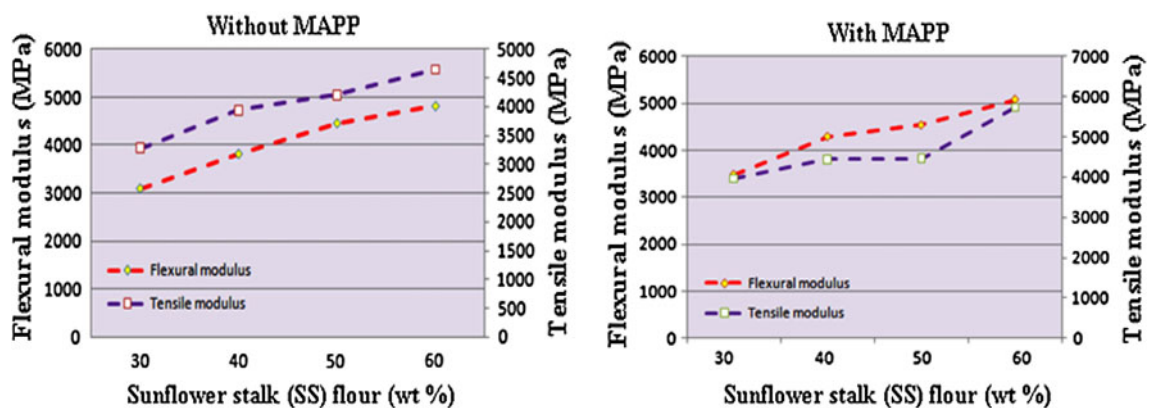


Fig. 3 The variation of flexural and tensile modulus of polypropylene composites without and with MAPP as a function of the SS flour content

than the untreated ones. The MAPP coupled composites showed the highest tensile strength, tensile modulus and elongation at break for all the composites tested. The observed increase in the tensile strength, tensile modulus and elongation at break is attributed to the improved interfacial bonding between the SS and the PP matrix as well as the modification of individual components. The improvements with the coupling agent are believed to be due to the formation of ester bonds between the anhydride carbonyl groups of MAPP and hydroxyl groups of the natural fibres. Upon esterification, the exposed polyolefin chains diffused into the PP matrix phase and entangled with PP chains during molding process. These changes created chemical bonds at the interface between filler and the PP matrix and thereby improved the compatibility between filler and PP matrix, which in turn, enhanced the mechanical properties [48–50].

The tensile elongation at break values declined significantly with the addition of the SS flour (Table 4). The tensile elongation at break of the polypropylene was found

to be 8.59 %. It decreased from 1.61 to 0.73 % as the SS flour content increased from 30 to 60 wt%. The elongation dropped significantly between fibre loading of 0–30 wt%; after this the decrease was slightly. This was because the composite became stiffer when the amount of lignocellulosic filler was increased. Our results were good consistent with previous studies [51, 52, 54]. Although the coupling agent had no significant effect on the elongation at break, the coupled composites showed higher elongation at break than ones without MAPP. This could be explained by the good dispersion of the SS flour in the polymer matrix. In general, the literature reports improvements in tensile strength and elongation at break when maleic anhydride grafted matrices are used as compatibilizers (coupling agent) [53].

DSC Analysis

The increment in filler content decreased the melting temperature of polypropylene, representing that less energy

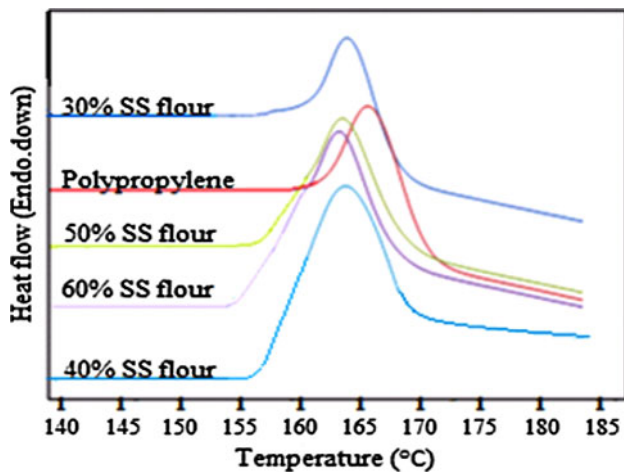


Fig. 4 Second melting (*peak*) temperatures of unfilled and SS flour filled polypropylene composites

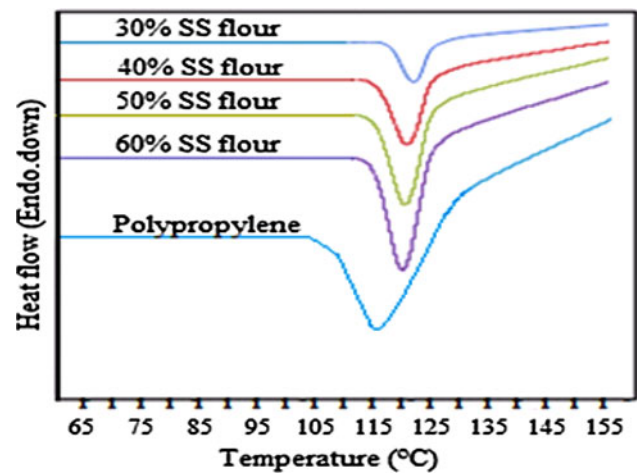


Fig. 5 Crystallization peak temperatures of unfilled and SS flour filled polypropylene composites

Table 5 The results of differential scanning calorimetry (DSC) analysis

Composite type ¹	First melting peak temperature (°C)	Crystallization peak temperature (°C)	Second melting enthalpy (J/g)	Second melting temperature (peak) (°C)	Crystallization enthalpy (J/g)	X _c Degree of crystallinity (%)
A	167.8	123.3	58.8	165.8	66.0	40.2
B	166.4	122.5	46.1	164.4	58.3	36.8
C	166.0	122.0	37.2	164.1	50.5	35.6
D	165.7	121.8	28.8	163.8	47.5	34.4
I	167.2	115.2	78.8	166.9	91.8	37.7

¹ See Table 2 for composite formulation

was needed to melt the composites (Fig. 4). Unfilled polypropylene composite had the highest melting point (166.9 °C for second melting temperature), while the polypropylene composites filled with 60 wt% SS flour had the highest melting temperature (163.8 °C) (Table 5). The melting temperature of the polypropylene composites decreased very slightly between fibre loading of 3–60 wt%.

The crystallization enthalpy of the polypropylene composites decreased significantly with increasing content of the SS flour. As compared to the unfilled polypropylene composite (91.8 J/g), the crystallization enthalpy of the filled composites decreased from 66.0 to 47.5 J/g. This result indicates that SS flour absorbed more heat energy in the melting of the composites. The crystallization enthalpy decreased considerably between fibre loading of 0–30 wt%. The crystalline peak temperature of the SS flour filler polypropylene composites was higher than that of unfilled ones (Fig. 5). The crystalline peak temperature of the unfilled polypropylene composites was found to 115.2 °C. It decreased from 123.3 to 121.8 °C as the filler content increased from 30 to 60 wt%. The crystallinity (X_c) of the neat polypropylene (37.7 %) increased by 40.2 % as the

amount of SS filler increased by 10 wt% but further increment in the filler content decreased the X_c (34.4 %) of the filled polypropylene composites.

Conclusions

Based on the results of this investigation, sunflower stalk flour and PP can be successfully used to produce stable and strong thermoplastic composites. In the results of this study, mechanical properties of the composites decreased by the increase of fibre loading except for the moduli. The WA and TS of the composites increased slightly as the filler loading increased. The main disadvantage of natural fibres is their hydrophilic nature, which lowers the compatibility with hydrophobic polymer matrices during composite production. Adding the MAPP significantly improved the physical and mechanical properties of the polypropylene composites. Satisfactory results were obtained with 40 wt% of the SS. The melting temperature of polypropylene decreased with increasing content of the SS flour. The degree of crystallinity of filled polypropylene

composites between fibre loading of 0–30 % by weight was higher than that of unfilled polypropylene composites. However, further increment in the filler content decreased the degree of crystallinity. It appears that SS can be potentially suitable raw material in the manufacture of polypropylene composites.

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