

Utilization of Red Pepper Fruit Stem as Reinforcing Filler in Plastic Composites

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The effects of the amounts of flour from the red pepper (*Capsicum annum*) fruit stem (RPFS), together with coupling agent (CA), on the mechanical and physical properties of polypropylene (PP)-based composites were investigated. Pellets manufactured through single screw extruders were injection molded into composite samples. Density, mechanical property, and dimensional stability of manufactured composites were determined according to ASTM standards. Results were analyzed using central composite design (CCD). Statistical analyses showed that filler loading significantly affected the density, as well as mechanical and physical properties of thermoplastic composites. Density of the composites was increased with filler loading but not affected by coupling agent amounts. In the case of mechanical properties, tensile modulus, flexural strength, and flexural modulus were improved with increasing filler loading while the tensile strengths, elongation at break, and impact strength of the samples were decreased. The tensile strength of the thermoplastic composites was positively affected by CA contents, but other mechanical properties were not affected as much. In the case of physical properties, thickness swelling and water absorption of the composites were increased with increasing weight percent of RPFS flour. However, these properties were not significantly changed by CA addition. Overall results revealed that RPFS flour could be potentially suitable raw materials for thermoplastic composites.

Keywords: Waste pepper fruit stem; Polypropylene; Dimensional stability; Mechanical properties

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INTRODUCTION

Interest in using lignocellulosic materials in the production of thermoplastic composites has gained momentum in recent years (Mohanty *et al.* 2005). They are inexpensive and have a low density, nonabrasive nature, good thermal insulation, and mechanical properties (Clemons 2002; Mengelöglu and Matuana 2003; Panthapulakkal *et al.* 2006; Mengelöglu and Karakus 2008a). That is why the usage of several agricultural wastes in thermoplastic matrix as a filler and/or a reinforcer were investigated. In these studies, the potential of wheat straw (Mengelöglu and Karakus 2008b; Sain and Panthapulakkal 2006), rice husk (Yang *et al.* 2007), sunflower stalk and corn stalk (Ashori and Nourbakhsh 2009), and barley husk (Bledzki *et al.* 2010) were determined.

There is a growing demand for finding new raw materials as filler for wood plastic composites (WPC). Since agricultural wastes have been mostly either burned or landfilled and cause additional disposal costs and environmental pollution, there is a need to find new ways to consume these materials. The use of such fibrous materials is also motivated by environmental pressure groups and recycling legislation (Güntekin *et al.* 2008).

The stems attached to the fruits of red pepper (*Capsicum annuum*) can be considered as one of the candidate waste agricultural raw material. Red pepper is an important vegetable widely cultivated and used throughout the world (Chen *et al.* 2012). During red pepper spice production, approximately 20% of waste (pepper stems) is generated by mass. Approximately 2.8 million tons of red peppers are produced annually worldwide. The biggest red pepper producers in the world are India and China. Turkey also produces 38.275 tons/year red pepper and ranks 20th in the world relative to production (TUIK 2010). However, the stems are not utilized in any manufacturing process in Turkey and currently have no economical value. This study investigated the potential utilization of red pepper fruit stems in polymer composites. The effect of filler and coupling agent loadings on the dimensional stability and mechanical properties of the composites were evaluated using the central composite design (CCD).

MATERIALS AND METHODS

Materials

Red pepper (*Capsicum annuum*) fruit stem (RPFS) was supplied by a manufacturer of dry red pepper in Kahramanmaraş, Turkey. The stems were first air-dried, then ground with a high-speed rotary cutting mill and finally screened. The flour passing through a 40-mesh screen and retained on a 60 mesh-size screen (0.25 mm) were used for manufacturing. The flour was then oven-dried to 0 to 1% moisture content using a laboratory oven at 100 °C for 48 h. The polypropylene (Petoplen MH 418) by PETKIM and MAPP (Licomont AR 504) by Clariant were used as polymer matrix and coupling agent, respectively.

Compounding and Composite Manufacturing

The experimental design of the study is presented in Table 1. Polypropylene (PP), 60 mesh-size red peppers fruit stem (RPFS) flour, and coupling agent (MAPP) were dry-mixed in a high-intensity mixer to produce a homogeneous blend. This blend was then compounded in a laboratory-scale single screw extruder. Manufacturing conditions were presented in a previous publication (Mengeloglu and Karakuş 2012).

Table 1. Experimental Design of the Study

Group ID	Point Type	PP Amount (%)	Wax Amount (%)	Natural Filler Loading (%)	CA Concentration (%)
A	Axial	71.00	2.50	22.50	4.00
B	Factorial	73.00	2.50	22.50	2.00
C	Factorial	90.32	2.50	6.59	0.59
D	Axial	58.50	2.50	38.41	0.59
E	Central	55.68	2.50	38.41	3.41
F	Axial	95.50	2.50	0.00	2.00
G	Factorial	87.50	2.50	6.59	3.41
H	Factorial	50.50	2.50	45.00	2.00
I	Axial	75.00	2.50	22.50	0.00

Determination of Chemical Composition of RPFS

Red pepper fruit stems were milled and screened with a 40 to 60 mesh for the chemical composition analysis. The determination of sampling, moisture content, lignin (TAPPI T 222 om-88), and extractives soluble in ethanol/benzene (TAPPI T 204 om 88), hot water, (TAPPI T 207 om-88), and in 1% NaOH (TAPPI T 212 om-88) was performed using TAPPI standards. Furthermore, the holocellulose content was determined according to Wise's chlorite method, whereas the cellulose content was determined by Kürschner-Hoffner's nitric acid method.

Determination of Physical Properties

The thickness swelling (TSW) and water absorption (WA) tests were carried out according to ASTM D 570 specifications. The TS and WA tests were applied on the same specimen. The conditioned specimens were entirely immersed for 1-day, 7-days, and 21-days in a container of water at 23 ± 2 °C. At the end of each immersion time, the specimens were taken out from water and all surface water was removed with a dry cloth. The specimens for the WA test were weighed to the nearest 0.01 g. After the weight measurements, the thickness of the same specimens for the TS test was measured to the nearest 0.001 mm immediately. Fourteen specimens were tested for each composite formulation.

Determination of Mechanical Properties

Testing of the samples was conducted in a climate-controlled testing laboratory. Densities were measured by a water displacement technique according to the ASTM D 792 standard. Tensile, flexural, tensile, and impact properties of all samples were determined according to ASTM D 638, ASTM D 790, and ASTM D 256, respectively.

Seven samples for each group were tested. Tensile and flexural testing were performed on Zwick 10KN while a HIT5.5P by Zwick™ was used for impact property testing on notched samples. The notches were added using a Polytest notching cutter by RayRan™. All the mechanical properties were evaluated as a specific strength (strength/density) values.

Interfacial Morphological Analysis

Fractured surfaces of the samples were studied using a JEOL scanning electron microscope (SEM. Model JSM 5500 LV) at 10 kV accelerating voltage. First samples were dipped into liquid nitrogen and then broken in half to prepare the fractured surfaces. Finally, samples were mounted on the sample stub and were sputtered with gold to provide electrical conductivity.

Data Analysis

Design-Expert® Version 7.0.3 statistical software program was used for statistical analysis.

In this study, central composite design, one of the most popular response surface methods was used to analyze the effects of red pepper fruit stem flour and coupling agent content on the mechanical properties of manufactured wood plastic composites. This design includes factorial points, axial points, and center points (Fig. 1).

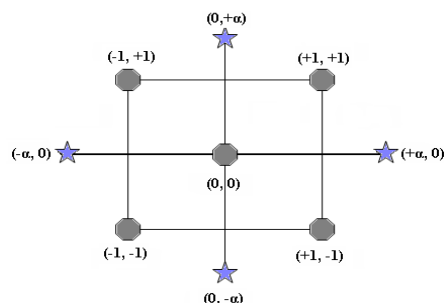


Fig. 1. Illustration of factorial, axial, and center points of the Central Composite Design (CCD)

RESULTS AND DISCUSSION

In this study, red peppers fruit stem (RPFS) flour was used as filler in polypropylene polymer matrix. Chemical composition of RPFS and, physical and mechanical properties of RPFS-filled composites were investigated. Discussions on these properties are presented as separate sections.

Chemical Composition of RPFS

The chemical composition of the RPFS is given in Table 2. The material was found to contain cellulose, hemicelluloses, and lignin, similar to wood. Chemical structure of the lignocellulosic materials, the hydroxyl groups in holocellulose and cellulose in particular, may play an important role on the physical properties of composites. RPFS flour was observed to have 49.63%, 28.60%, and 13.62% of holocellulose, cellulose, and lignin, respectively. The cellulose content of RPFS was lower than that of cotton stalks, hazelnut husk, flax, jute, softwood, and hardwood, as listed in Table 2. Solubility in hot water and in alcohol-benzene of RPFS was 38.09% and 14.61%, respectively.

Table 2. Chemical Composition Values of Some Agricultural Wastes and Wood (Güntekin *et al.* 2008)

Source	Holocellulose (%)	Cellulose (%)	Lignin (%)
Pepper stalks	95	60.51	4.89
Cereal straw	64-71	35-39	12-17
Hazelnut husk	55	34.50	35.10
Softwood	63-70	29-47	25.35
Hardwood	70-78	38-50	30.35
Cotton	97	95	0.90
Cotton stalks	76.80	51.80	10.70
Flax	81	65	2.50
Jute	18-21	58	21-26
Sunflower stalk	50.50	43.10	9.70
Walnut shell	47.78	26.51	49.18

Physical Properties of RPFS Filled Composites

The effect of filler and coupling agent (CA) amount on the density, thickness swelling (TSW), and water absorption (WA) of the composites were studied. Mean density values are given in Table 3 and a contour graph is presented in Fig. 2. In this graph, changes in density with RPFS loading and coupling agent amount are presented on the x

and y axis, respectively. Statistical analysis showed that RPFS loading had a significant effect on density ($P < 0.0001$) but coupling agent did not ($P = 0.2662$). It can also be seen on the contour graph that the density of the composites was increased with increasing RPFS loading but was not changed with coupling agent. This result was expected because the cell wall density of lignocellulosic materials (average 1.5 g cm^{-3}) is higher than the density of polypropylene (0.905 g cm^{-3} provided by PETKIM). Density of the resulting composites is expected to be higher than polymer density due to the rule of mixtures. Similar results were reported by other researchers (Soucy 2007; Steckel *et al.* 2007).

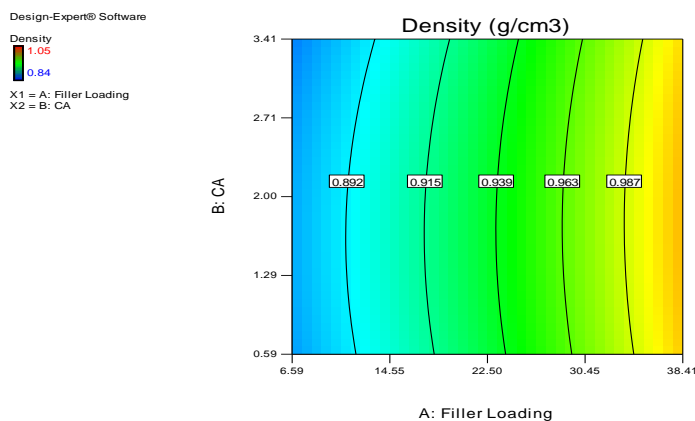


Fig. 2. Contour graph for mechanical properties of RPFS-filled composites

Table 3. Physical Properties of RPFS Flour Filled Composites

Composite type*	Density (kg/m^3)	Thickness swelling (TSW) (%)			Water absorption (WA) (%)		
		1-day	7-day	21-day	1-day	7-day	21-day
A	0.92(0.02)	0.72(0.17)	0.82(0.15)	1.56(0.30)	0.40 (0.11)	0.94 (0.12)	1.54 (0.33)
B	0.94(0.01)	0.75(0.14)	0.90(0.09)	1.45(0.16)	0.43 (0.12)	0.97 (0.16)	1.61 (0.25)
C	0.89(0.01)	0.60(0.05)	0.86(0.11)	1.43(0.20)	0.39 (0.05)	0.85 (0.06)	1.50 (0.22)
D	1.01(0.02)	0.79(0.06)	1.04(0.07)	1.69(0.09)	0.45 (0.09)	1.02 (0.10)	1.79 (0.24)
E	1.00(0.01)	0.77(0.06)	1.02(0.07)	1.59(0.08)	0.43 (0.08)	1.01 (0.10)	1.77 (0.14)
F	0.85(0.00)	0.06(0.02)	0.11(0.02)	0.24(0.06)	0.08 (0.03)	0.11 (0.02)	0.16 (0.03)
G	0.87(0.01)	0.58(0.07)	0.78(0.11)	1.32(0.20)	0.38 (0.07)	0.83 (0.10)	1.43 (0.15)
H	1.05(0.01)	0.87(0.15)	1.08(0.09)	1.77(0.13)	0.49 (0.10)	1.07 (0.21)	1.83 (0.25)
I	0.92(0.01)	0.78(0.16)	0.95(0.17)	1.60(0.25)	0.44 (0.10)	0.98 (0.21)	1.62 (0.28)

*See Table 1 for composite formulation

The thickness swelling (TSW) and water absorption (WA) values of the manufactured composites are summarized in Table 3. As expected, TSW and WA values of the specimens increased with increasing immersion time. Similar results were reported by others (Rowell *et al.* 1997; Rana *et al.* 1998; Yadav *et al.* 1999; Simonsen *et al.* 1998). The interaction graphs showing the effect of filler and CA amounts on the TSW and WA after 21 days immersion time are also presented in Fig. 3. In this graph, filler loading is given on the x axis while TSW and WA are presented on the y axis. Lower CA content is shown with a darker line, while the higher CA content is shown with a light color line. Based on the statistical analysis, filler loading significantly affected the TSW ($P < 0.0001$) and WA ($P < 0.0001$) of the composites. These results can be explained by higher water absorption

potential of the hydrophilic fibers. An increase of filler loading might proportionally increase the rate of water absorption. It is well known that WA of wood plastic composites (WPCs) increases with the rising concentration of cellulose in the composites (Klysov 2007). The WA values of PP-based WPC having 30 to 40% wood flour amount is reported to be around 5% (Klysov 2007). Lower TSW and WA values in our study might be due to the lower cellulose content of RPFS (28%) compared to the wood flour's cellulose content (40 to 44%, Haygreen and Bowyer 1994). Polypropylene shows very negligible or no water absorption due to its being lack of functional polar group and hence, it can be assumed that almost all of water is absorbed by RPFS fibers. Similar results for different fillers were reported by Ayrılmış *et al.* (2013).

In the case of CA amount, TSW and WA values (light color lines in Fig. 3) were slightly reduced with increasing CA content. However, this reduction was not found to be statistically significant for TSW (P 0.5166) and WA (P 0.4515) values. This might be due to the lower TSW and WA values in these composites. Since the maximum TSW and WA values were less than 2%, it might be difficult to detect the effect of CA on the TSW and WA in these samples.

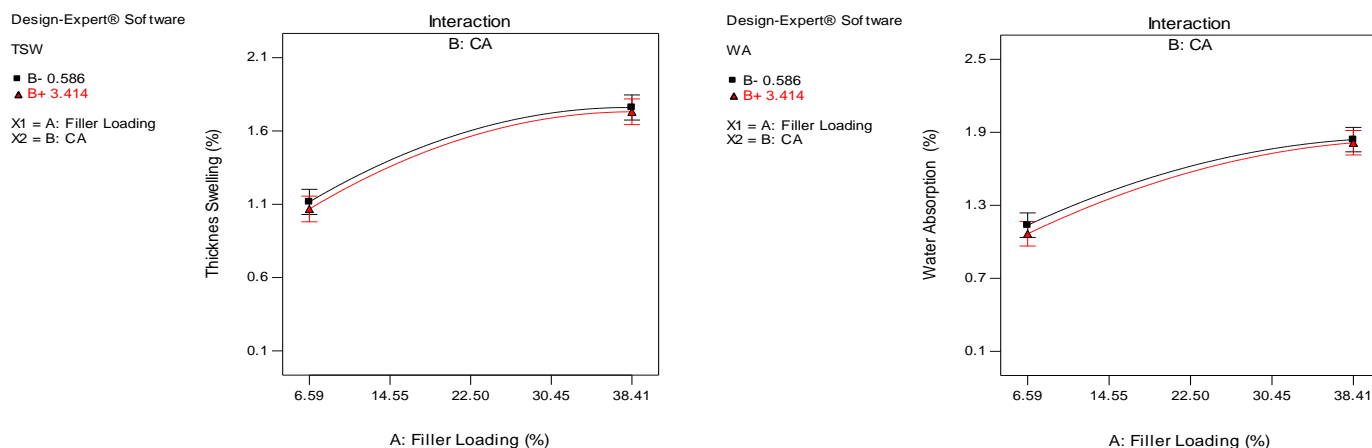


Fig. 3. Interaction graphs of the filler and CA amount on the physical properties (TSW and WA)

Mechanical Properties of RPFS Filled Composites

The effects of filler loading and coupling agent amount on the specific mechanical properties of the composites were studied. The effects of filler loading and coupling agent (CA) contents on the mechanical properties of the manufactured thermoplastic composites were investigated utilizing central composite design (CCD). Nine different groups of plastic composites were produced, and flexural, tensile, elongation at break, and impact properties were determined. Some of the mechanical properties were summarized in Table 4 and 3D graphs are presented in Fig 4.

Table 4. Some Mechanical Properties of RPFS Filled Composites

ID	A	B	C	D	E	F	G	H	I
Elongation at Break (%)	5.89 (0.29)*	6.78 (1.31)	11.78 (3.99)	4.09 (1.09)	4.45 (1.19)	122.7 5.29	10.74 (2.72)	3.56 (0.19)	7.04 (0.54)
Impact Strength (J/m)	19.27 (4.92)	19.63 (2.88)	18.22 (3.12)	18.29 (2.15)	17.05 (2.66)	28.02 (5.73)	22.01 (2.30)	15.13 (2.33)	19.31 (3.23)

*The numerical value in the parenthesis is standard deviation.

The mean tensile strength values ranged from 20.44 MPa to 33.09 MPa. Tensile strength of the manufactured composites was significantly reduced by the increase of filler loading ($P < 0.0001$). Addition of the lignocellulosic filler into the polymer matrix weakened the resulting composites due to the dissimilarities between the hydrophilic filler and hydrophobic polymer matrix. With the introduction of MAPP coupling agent into the polymer matrix, tensile strength of the composites was significantly improved ($P < 0.0001$). However, this increase was not linear with CA concentration (Fig. 4). There was a significant interaction between filler loading and CA amount ($P < 0.0001$), indicating that the effect of CA was more pronounced at higher filler loadings. SEM images of the manufactured composites with 45% RPFS filler and 2.5% MAPP are presented in Fig. 5. On these micrographs, pulled out natural fillers (white arrows) and embedded fiber into polymer matrix can be seen. This indicates that MAPP had improved compatibility between filler and polymer matrix to some extent, but there was still room for improvement because pulled out filler still could be observed in the matrix. Positive effects of CA on tensile strength have also been reported by others (Clemons 2002; Li and Matuana 2003; Lu *et al.* 2005; Mengeloglu and Karakus 2008a). It is believed that MAPP coupling agent rendered the solubility characteristics to be more similar between the red pepper fruit stem flour and polymer matrix, resulting in an improved bonding between them.

With respect to the tensile modulus of the composites, the mean tensile modulus values ranged from 565.5 MPa to 817.1 MPa. While filler loading had a significant effect ($P < 0.0001$), CA contents had no notable effect ($P = 0.4671$) on tensile modulus. Tensile modulus of thermoplastic composites was significantly increased with a rising concentration of filler. Natural fillers have higher modulus when compared to polymer matrix; as a result, their mixture produces modulus values higher than the polymer itself. This is one of the advantages of the use of natural filler (Mengeloglu and Karakus 2008a). CA content had no noticeable effect on tensile modulus. The mean elongation at break of the composites values ranged from 3.56% to 122.7%. They were significantly reduced with the increased filler loading ($P < 0.0001$) due to increased stiffness of composites. On the other hand, CA amounts did not significantly affect the elongation at break values ($P = 0.9696$).

The mean flexural strength values ranged from 41.49 MPa to 50.42 MPa. Statistical analysis showed that flexural strength was significantly improved by increasing filler loading ($P < 0.0001$). Flexural strength of the extruded composite samples were reduced usually by the lignocellulosic filler amounts (Mengeloglu *et al.* 2007; Mengeloglu and Karakus 2008a,b). However, for injection molded small samples, sometimes flexural strength was improved, depending on the alignment and aspect ratio of the fillers. For injection molded samples, the surface was usually covered with a thin layer of polymer, which may inhibit the failure at the surface. For extruded samples, there is some filler present at the surface, and this leads to the failure due to the lack of adhesion polymer and filler. The effect of CA content in this study was not statistically significant ($P = 0.1580$).

In the case of flexural modulus, the mean values ranged from 1232 MPa to 2044 MPa. Even though filler loading significantly improved flexural modulus ($P < 0.0001$), CA content had no significant effect on it ($P = 0.5890$). This increase can be explained by the rule of mixture. Similar results for composite produced with various wood flour were reported (Li and Matuana 2003; Wang *et al.* 2003). The mean impact strength values ranged from 15.13 to 28.02 J/m. Similar to elongation at break values, the impact strength of plastic composites was also significantly reduced with the increased filler loading ($P < 0.0001$), but not significantly affected by CA contents ($P = 0.7766$).

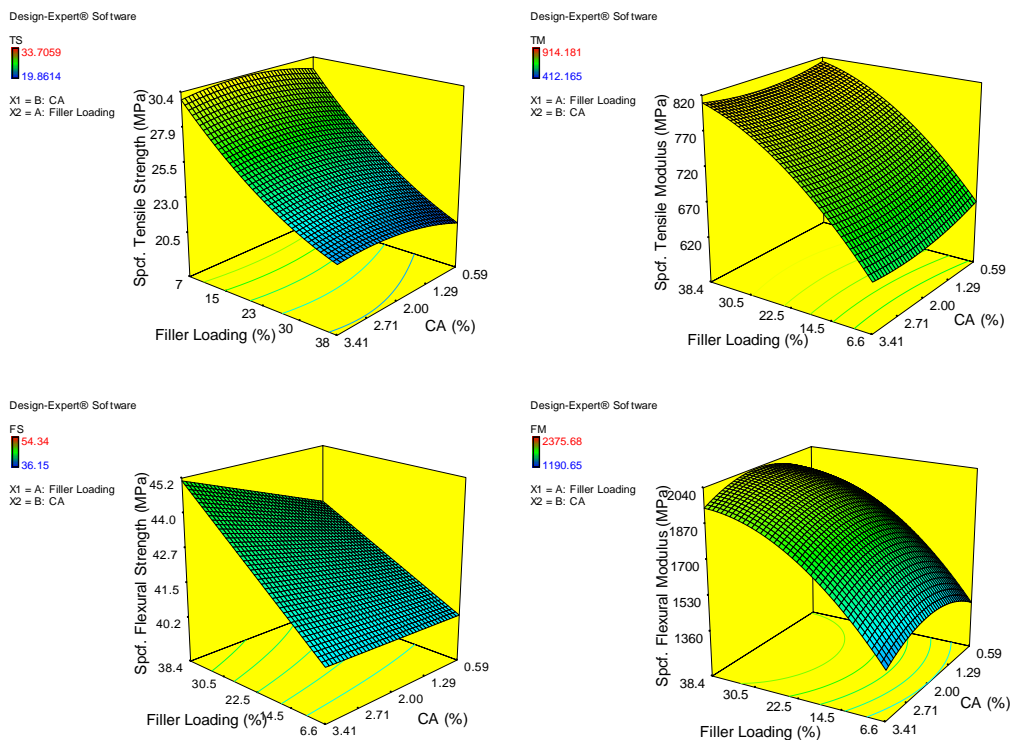


Fig. 4. Contour graph for mechanical properties of PP composites filled with RPFS flour

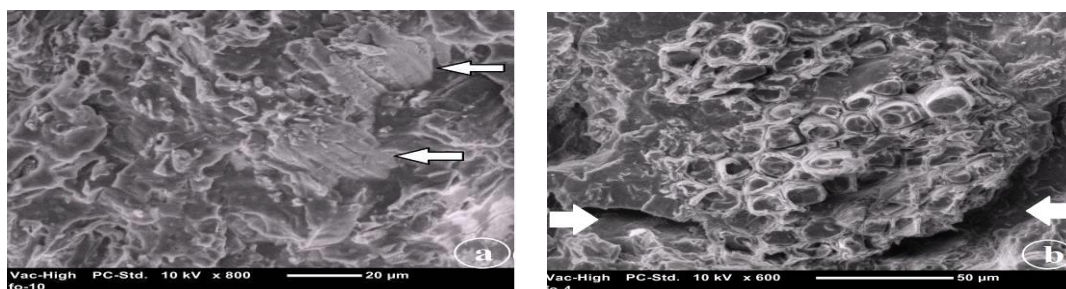


Fig. 5. SEM images of composites with 45% RPFS

CONCLUSIONS

1. Chemical constituents of the red pepper fruit stem (RPFS) were determined.
2. Polypropylene-based composites in the density range of 0.85 to 1.05 g cm⁻³ were manufactured utilizing RPFS flour as natural filler.
3. RPFS-filled composites had less than 3% thickness swelling and water absorption.
4. Specific tensile modulus, flexural strength, and flexural modulus values were improved with increased filler loading. However, tensile strength, elongation at break, and impact strength of polypropylene composites filled with RPFS were reduced.
5. The use of coupling agent improved the tensile strength and flexural strength but did not significantly affect other mechanical properties of RPFS-filled polypropylene based composites.

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