MECHANICAL PROPERTIES OF INJECTION-MOLDED FOAMED WHEAT STRAW FILLED HDPE BIOCOMPOSITES: THE EFFECTS OF FILLER LOADING AND COUPLING AGENT CONTENTS

Fatih Mengeloglu,* and Kadir Karakuş

This study investigated the effect of filler loading and coupling agent contents on the densities and mechanical properties of injection-molded foamed biocomposites. Biocomposite pellets were manufactured using wheat straw flour, maleic anhydrite grafted polyethylene (MAPE), paraffin wax, and high-density polyethylene (HDPE) with an extrusion process. Pellets and the chemical foaming agent (azodicarbonamide) were dry-mixed and foamed in an injection-molding machine. Densities and mechanical properties of the foamed biocomposites samples were measured and analyzed using central composite design (CCD). The results showed that both filler loading and coupling agent contents affected the density and mechanical properties of foamed biocomposites. Densities in the range of 0.57 to 0.81 gr cm^{-3} were achieved. Best results were obtained when less than 20% wheat straw flour and 1% coupling agent content were used. The flexural modulus and tensile modulus of foamed biocomposites were improved with increasing filler loading. However, flexural strength, tensile strength, elongation at break, and impact strength values were diminished. The tensile strength of the biocomposites was positively affected by CA contents, but other mechanical properties were not affected by it. Overall, injection molded foamed biocomposites with moderate mechanical properties were produced.

Keywords: Biocomposites; Chemical foaming agent; Foaming; Coupling agent; High density polyethylene; Mechanical properties

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INTRODUCTION

Composite materials produced from natural fiber and petroleum-derived non-biodegradable polymers (polypropylene (PP), polyethylene (PE), epoxies, *etc.*) or biopolymers (poly(lactic acid) (PLA), polyhydroxyalkanoates (PHAs), *etc.*) are broadly defined as biocomposites (Mohanty *et al.* 2005). Recently, the manufacture of biocomposites utilizing natural fiber and petroleum-derived nonbiodegradable polymers have been drawing growing interest due to natural fibers' low densities, low cost, and nonabrasive nature (Clemons 2002; Mengeloglu and Matuana 2003; Panthapulakkal *et al.* 2006; Mengeloglu and Karakus 2008a). By the use of natural fiber, it is also possible to achieve high filler levels, low energy consumption, high specific properties, biodegradebility, and availability throughout the world (Abu-Sharkh *et al.* 2004; Matuana *et al.* 1998). This type of composite has numerous application areas including automobile

interior parts (Clemons 2002), siding, fencing, window framing, and decking (Clemons 2002; La Mantia *et al.* 2005; Youngquist 2005; Mengeloglu *et al.* 2007).

In the area of biocomposite manufacturing, several studies have been conducted using agricultural residues (Joseph *et al.* 1996; Hornsby *et al.* 1997; Gassan and Bledzki 1997; Chen *et al.* 1998; Vande Velde and Keikens 2001; Mwaikambo and Ansell 2002; Prasad and Sain 2003; Mengeloglu and Karakus 2008b; Cavdar *et al*. 2011) and various wood species (Li and Matuana 2003; Rachtanapun *et al*. 2003; Bengtsson and Oksman 2006; Kim *et al*. 2006; Mengeloglu and Karakus 2008a). Although agricultural plant residues and wood-fiber-filled biocomposites have been commercialized, their potential usage in many industrial (mainly automotive and deck) applications has been limited because of their brittleness, low impact resistance, and high densities. The concept of creating foamed structures in the composites as a means to improve these shortcomings has been successfully demonstrated (Matuana *et al*. 1998; Schut 2001; Faruk *et al*. 2007). Foaming, in general, reduces the amounts of material required, with the associated economic benefits. Because of the plasticising effects of gas, the foamed composites run at a lower temperature and at faster speeds than their unfoamed counterparts, and thus the production cost can be reduced (Schut 2001; Faruk *et al*. 2007; Matuana 2009).

Biocomposites can be produced by means of extrusion molding, compression molding, or injection molding technologies. Although the majority of biocomposites (natural filler and thermoplastic mixture) are manufactured through an extrusion process, injection-molded products also have high marketing potentials (Matuana 2009). Several studies have been conducted to investigate the foaming of biocomposites. Most of these studies were on the extrusion foaming or batch process microcellular foaming (Matuana *et al* 1998; Rachtanapun *et al.* 2003; Mengeloglu and Matuana 2003; Faruk *et al.* 2007; Kord 2011). However, studies on the foaming of injection-molded parts are limited.

Injection molding is one of the most commercially important fabrication processes for molding a broad spectrum of thermoplastics (Faruk *et al.* 2007). Therefore, it is very important to study the foamability of biocomposites through injection molding technology and to characterize the foamed material properties. Previously, the effect of the chemical foaming agents (CFA), injection parameters, and melt flow index on the microstructure and mechanical properties of wood-fiber/polyproylene composites were studied (Bledzki and Faruk 2005a). The best performance with respect to the cell size, diameter, and distance was achieved when an exothermic chemical foaming agent was used. Some other studies investigated the effect of wood fiber size and wood fiber and CFA contents on the morphology and mechanical properties of injection-molded PP-based foamed biocomposites (Bledzki and Faruk 2005b,c, 2006a,b).

Although several studies have been conducted to investigate the morphology and mechanical properties of wood-fiber-filled PP-based injection-molded foamed biocomposites, there is limited information available on the mechanical properties of agricultural residue-filled high-density polyethylene-based injection molded foamed biocomposites. Since Turkey generates a great amount of agricultural residues, including roughly 17 million tons of wheat straw (Korucu and Mengeloglu 2007), it is important to study the potential usage of this material in injection-molded applications. In this study, the effect of filler loading and coupling agent contents on the mechanical properties of injection

molded foamed biocomposites were investigated using the central composite design (CCD).

EXPERIMENTAL

Materials

High-density polyethylene (HDPE) and wheat straw flours (WSF) were used as the polymer matrix and filler, respectively. Maleic anhydride-grafted polyethylene (MAPE) was used as a coupling agent, paraffin wax (K.130.1000) as a lubricant, and azidicarbonamide (Tracel DB170) as a chemical foaming agent. Wheat straw was collected from the local farmers and granulated into flour form using a Wiley mill. Produced flours, screened and retained on a 60 mesh-size screen (0.25 mm), were used for manufacturing.

Methods

Compounding and composite manufacturing

The experimental design of the study is presented in Table 1. High-density polyethylene (HDPE), 60 mesh-size wheat straw flour (WSF), and coupling agent (Maleic anhydride grafted polyethylene (MAPE)) 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 at 40 rpm screw speed in the temperatures (barrel to die) of 170-180-185-190-200°C. Extruded samples were collected, cooled, and granulated into pellets. Produced pellets were pre-mixed with 2% chemical foaming agents. These mixtures were then injection molded using an HDX-88 Injection Molding Machine (pressure: 100 bar; injection speed: 80mm/sec; screw speed: 40 rpm) to produce standard test samples.

Table 1. Experimental Design of the Study

Group ID	Point Type	HDPE Amount (%)	Wax Amount (%)	Natural Filler Loading (%)	MAPE Concentration (%)
A	Axial	82.50	2.50	15.00	0.00
B	Factorial	92.38	2.50	4.39	0.73
C	Factorial	71.16	2.50	25.61	0.73
D	Axial	95.00	2.50	0.00	2.50
E	Central	80.00	2.50	15.0	2.50
F	Axial	65.00	2.50	30.00	2.50
G	Factorial	88.84	2.50	4.39	4.27
H	Factorial	67.62	2.50	25.61	4.27
I	Axial	77.50	2.50	15.00	5.00
2% Azodicarbonamade (Tracel DP170) used as a foaming agent					

Property testing and Thermogravimetric analysis (TGA)

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. Flexural, tensile, and impact properties of all samples were determined according to ASTM D 790, ASTM D 638, and ASTM D 256, respectively. Ten samples for each group were tested. Flexural and tensile 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™.

Thermogravimetric analysis (TGA) of the samples was done in a Shimadzu TGA-50 thermal analyzer using a scanning rate of 10°C min^{-1} heating rate under nitrogen with 20 mL min^{-1} flow rate, from room temperature to 800°C.

Scanning electron microscope (SEM) study

Fractured surfaces of the samples were studied using a JEOL scanning electron microscope (SEM, Model JSM 5500LV) at 15 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 wheat straw flour and coupling agent content on the mechanical properties of manufactured foamed composites. This design includes factorial points, axial points, and center points (Fig. 1).

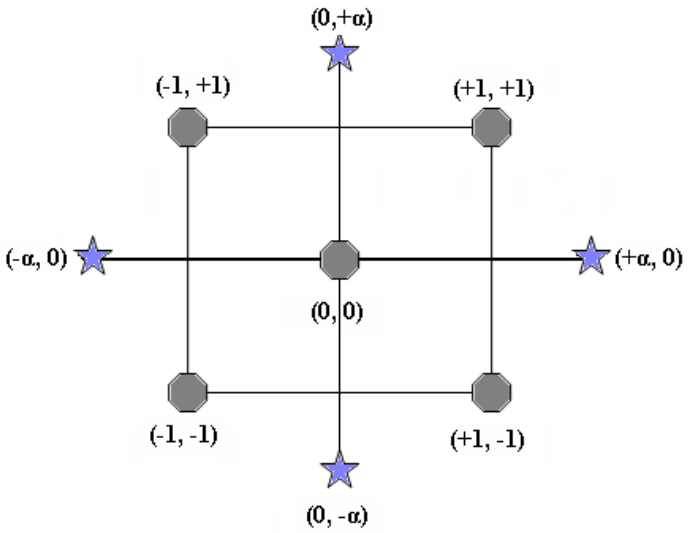


Fig. 1. Illustration of factorial, axial, and center points of the Central Composite Design (CCD)

RESULTS AND DISCUSSION

Thermogravimetric Analysis (TGA) of Chemical Foaming Agent (CFA)

Manufacturing of foamed biocomposites was accomplished in a two-step process, which consisted of pellet manufacturing through extrusion and foaming in an injection-molding machine. For both processes, melting temperature of the polymer and degradation temperature of the filler have great importance. On the other hand, decomposition temperature of the chemical foaming agent (CFA) is only important for foaming in injection molding.

Differential scanning calorimetry (DSC) analysis is utilized to determine the melting temperature, while TGA is used to designate degradation and decomposition temperatures of the materials. Mengeloglu and Karakus (2008a) previously reported that high-density polyethylene (HDPE)-based composite should be extruded over a temperature of 129°C to provide melting of the polymer matrix. It is also reported that extruder temperature should be less than 220°C to prevent the lignocellulosic material from degrading. Based on these findings, extruder temperatures were selected in the range of 180°C to 200°C.

The decomposition temperature of CFA is very important, since gas necessary for foaming is produced during its decomposition. Thermogravimetric analysis (TGA) was performed on CFA samples to determine decomposition temperatures. TGA and DTGA curves are shown in Fig. 2. The initial and maximum decomposition temperatures of CFA were around 175°C and 220°C, respectively. Consequently, temperatures for the injection molding machine were selected around 200°C to provide enough gas for foaming.

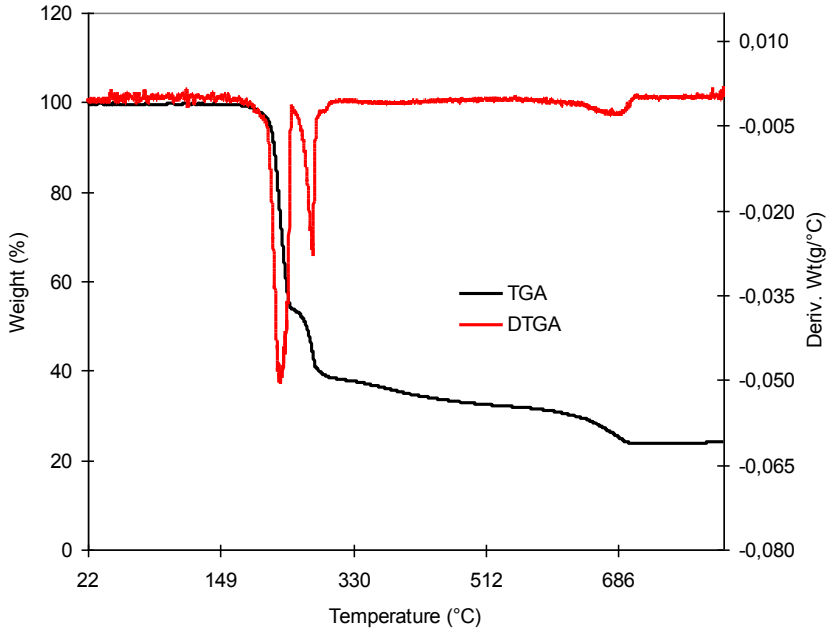


Fig. 2. TGA and DTGA thermographs of chemical foaming agent

Density of Foamed Biocomposites

Densities of manufactured foamed biocomposites were determined using a water displacement technique and are summarized in Table 2. Foamed biocomposites in the density range of 0.57 to 0.81 were produced. Measured density results were analyzed utilizing central composite design (CCD) and presented in a contour graph in Fig. 3. The contour plot is a two-dimensional representation of the response for selected factors. The number on each line shows the estimated densities underneath.

Table 2. Density of Manufactured Foamed Biocomposites

Group ID	Point Type	Density of foamed biocomposites (g/cm^{-3})
A	Axial	0.66 (0.01)*
B	Factorial	0.65 (0.02)
C	Factorial	0.71 (0.02)
D	Axial	0.72 (0.08)
E	Central	0.67 (0.02)
F	Axial	0.71 (0.01)
G	Factorial	0.69 (0.02)
H	Factorial	0.69 (0.04)
I	Axial	0.72 (0.07)
*The numerical value in the parenthesis is standard deviation		

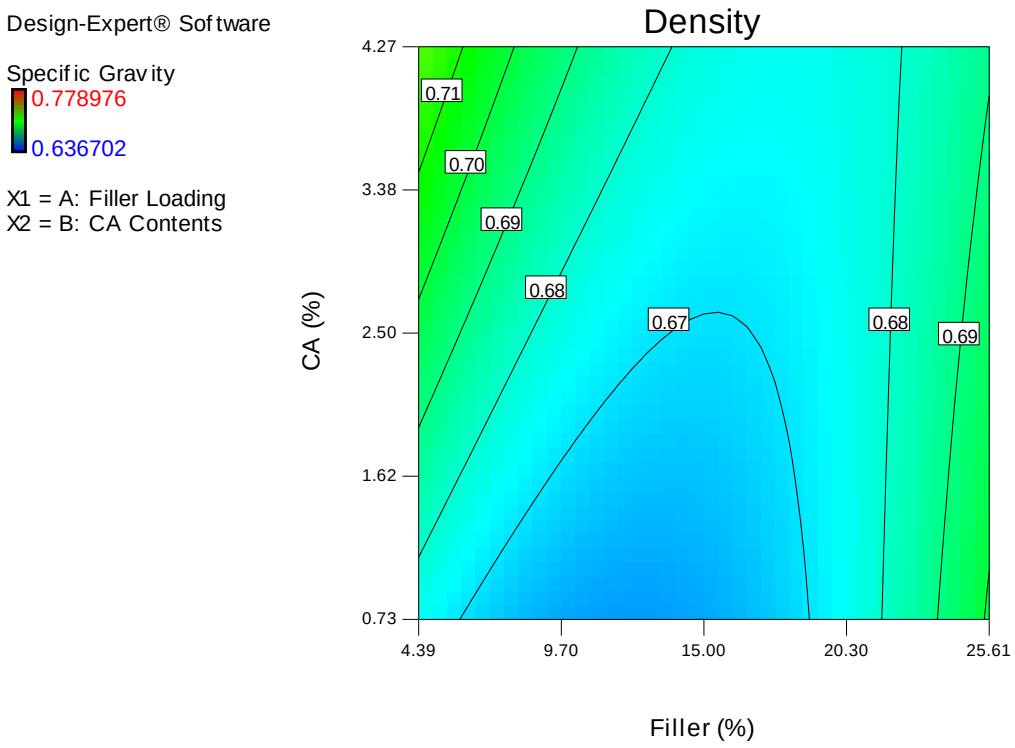
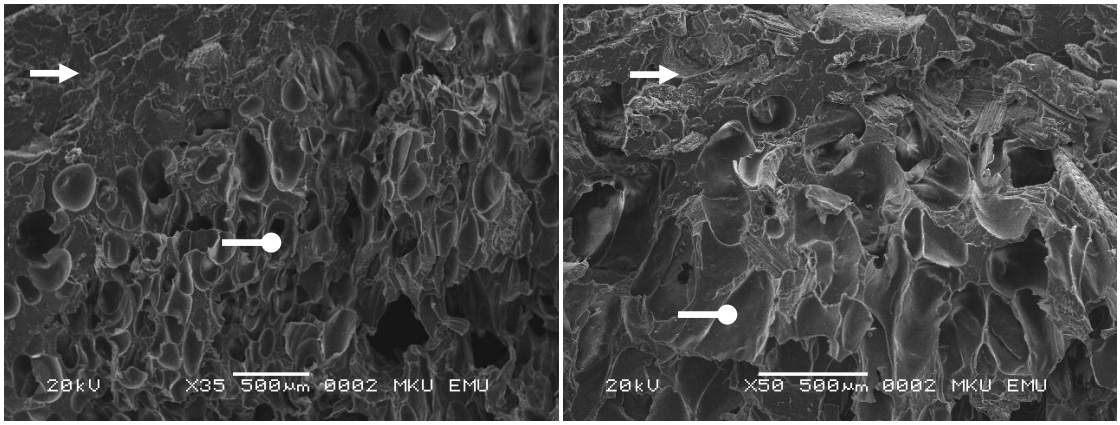


Fig. 3. Contour graph for density of foamed biocomposites

It can be seen in Fig. 3 that lower percentages of filler and CA contents resulted in a better foaming with lower densities. The effect of filler loading and CA contents on the morphology of foamed biocomposites was analyzed by scanning electron microscopy (SEM) and presented in Figs. 4 and 5, respectively. Figure 4 compares the SEM images of foamed composites produced with 15% and 30% filler loading at the same (2.5%) CA contents.



(a) **(b)**

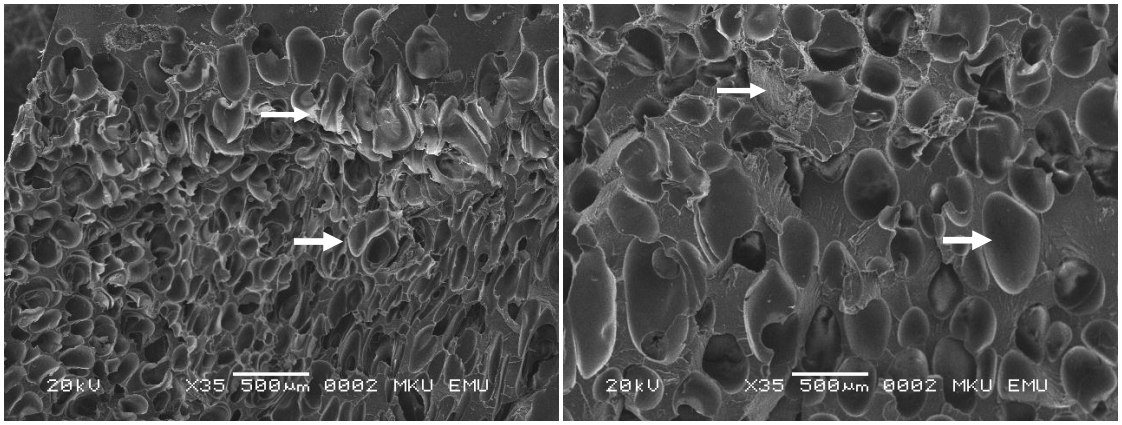
Fig. 4. SEM images of foamed biocomposites with 2,5% CA contens: a) 15% filer loading and b) 30% filler loading

It can easily be seen that fewer and larger foam bubbles were produced when high filler contents were used. On the other hand, at low filler loading, there were more, smaller, and more uniform (similar in size) bubbles produced. The arrow with a circle head shows foamed area (core of the samples), while a regular arrow shows the skin of the sample. It is believed that high filler loading may hinder foam generation due to increased stiffness of the matrix. It should also be noted that the success of foaming is closely related to the melt strength, foaming conditions, and filler type (Matuana and Mengeloglu 2001; Mengeloglu and Matuana 2001).

In Fig. 5, SEM images of samples having the same filler loading (4.39%) but two different CA contents (0.73% vs 4.27%) are presented. It can be seen from this graph that when 0.73% CA was used, a higher number of bubbles having smaller size were produced, resulting in lower density values. On the other hand, high (4.27%) CA content provided fewer and larger bubbles.

The melt behavior is important for foaming. When wood filler was added into the polymer matrix, melt viscosity of the matrix increased significantly. Similarly, melt viscosity of the HDPE matrix were increased from 15 to 20% by the addition of CA into the polymer matrix. It is believed that, similar to filler loading, higher CA content may also hinder foamability of biocomposites due to the increased viscosity. Bledzki and Faruk (2006b) studied the effect of CA contents (0% vs 5%) on the density reduction of injection-molded PP composites. For exothermic CFA, little effect of CA content on density reduction was reported. In another study, positive effect of CA contents on the foamability was reported for solid-state microcellular foaming of PVC-based composites

(Matuana and Mengeloglu 2001). It should be noted that solid-state microcellular foaming was performed on compression-molded composite samples. Improved adhesion between polymer matrix and lignocellulosic filler might help foaming by reducing the gas loss from the samples. In contrast to microcellular foaming, the polymer matrix is in a molten state during foaming in the course of an injection molding process. Thus, it is difficult to talk about adhesion between filler and matrix at this point. The effect of CA on the foamality of lignocellulosic filled biocomposites should be investigated with follow-up studies.



(a) **(b)**

Fig. 5. SEM images of foamed biocomposites with 4.39% filler loading: a) 0.73% CA content and b) 4.27% CA content

Mechanical Properties of Foamed Biocomposites

The effects of filler loading and coupling agent (CA) contents on the mechanical properties of the manufactured biocomposites were investigated utilising central composite design (CCD). Nine different groups of biocomposites were produced, and flexural, tensile, and impact properties were determined (Table 3). The mean flexural strength values ranged from 14.43 MPa to 18.87 MPa. Statistical analysis showed that flexural strength was significantly reduced by increasing filler loading ($P < 0.0001$). Since a hydrophobic polymer and hydrophilic filler are not compatible with one another, adhesion between two components might be weakened with increased filler loading. The effect of CA content in this study was not statistically significant ($P = 0.2680$). In some other studies, positive effects on flexural strength were observed for unfoamed samples (Mengeloglu *et al.* 2007; Mengeloglu and Karakus 2008a, 2008b). It is believed that the foamed structure of biocomposites influences mechanical properties more than adhesion between polymer matrix and filler loading. ASTM D 6662 (2001) standard requires the minimum flexural strength of 6.9 N mm^{-2} (1,000 psi) for polyolefin-based plastic lumber decking boards. Foamed biocomposites produced in this study provided flexural strength values (14 to 19 N mm^{-2}) that are well over the requirement of the standard.

In the case of flexural modulus, the mean values ranged from 338.20 MPa to 519.23 MPa. Even though filler loading significantly improved flexural modulus ($P < 0.0001$), CA content had no significant effect on it ($P = 0.7575$). In other studies, similar trends were reported for composite produced with various wood flours (Li and Matuana

2003; Wang *et al.* 2003). ASTM D 6662 (2001) standard requires the minimum flexural modulus of 340 N mm^{-2} (50,000 psi) for polyolefin-based plastic lumber decking boards. Foamed biocomposites produced in this study provided flexural modulus values (340 to 560 N mm^{-2}) that are within the range of standard requirements.

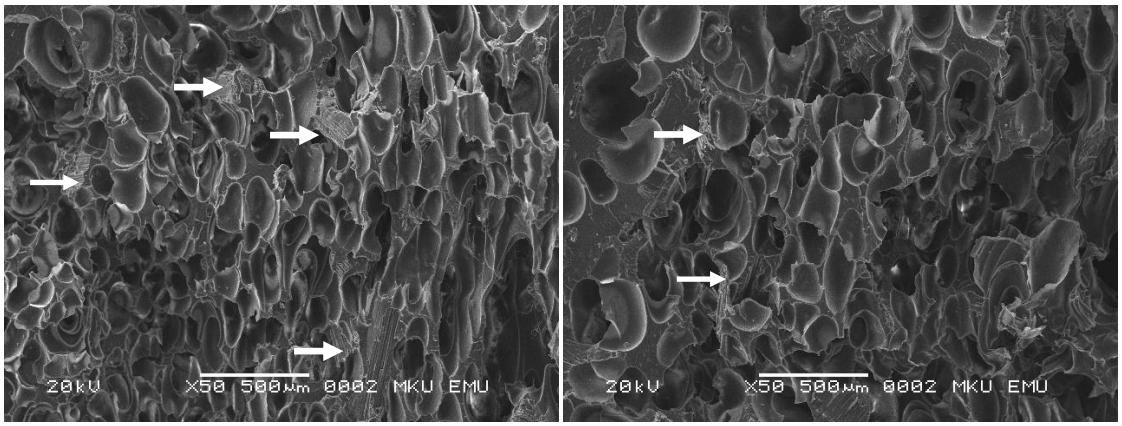
Table 3. Mechanical Properties of Manufactured Foamed Biocomposites

ID	Tensile Strength (MPa)	Tensile Modulus (MPa)	Elongation at Break (%)	Flexural Strength (MPa)	Flexural Modulus (MPa)	Impact Strength (J/m)
A	10.4 (0.2)*	20.0 (1.5)	43.5 (3.1)	15.0 (0.4)	413.8 (20.6)	59.4 (2.9)
B	9.8 (0.2)	18.6 (0.9)	46.3 (2.5)	15.0 (0.4)	364.0 (21.2)	90.3 (8.4)
C	10.5 (0.7)	19.9 (5.0)	31.5 (4.0)	15.2 (1.7)	518.3 (121.8)	41.9 (6.2)
D	12.5 (0.9)	38.5 (9.2)	300.0 (21.8)	18.9 (1.5)	519.2 (60.5)	193.1 (25.1)
E	11.0 (0.3)	16.2 (1.5)	44.7 (5.1)	16.5 (0.6)	451.8 (29.9)	50.8 (5.1)
F	10.8 (0.3)	19.9 (2.9)	31.0 (1.8)	14.5 (0.9)	496.0 (69.8)	34.1 (4.8)
G	10.2 (0.2)	21.9 (2.0)	53.3 (6.6)	14.4 (1.1)	338.2 (42.2)	96.5 (31.3)
H	10.5 (0.1)	14.8 (1.4)	32.5 (1.8)	15.3 (0.4)	483.2 (27.2)	38.9 (5.0)
I	11.2 (0.3)	22.5 (4.5)	38.1 (3.8)	16.0 (0.7)	429.4 (24.4)	44.9 (2.1)
*The numerical value in the parenthesis is standard deviation.						

With respect to the tensile strength of the foamed biocomposites, the mean values ranged from 9.83 MPa to 12.50 MPa. Statistical analysis showed that both filler loading ($P = 0.0092$) and CA contents ($P = 0.0330$) had significant effects on tensile strength. Tensile strength might be reduced due to the poor adhesion between the polar natural filler and nonpolar plastic matrix. The addition, MAPE coupling agent improved the tensile strength of the composites. However, this increase was not linear with CA concentration. Best results were recorded with the use of 2.5 to 3.0% CA concentrations. 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 MAPE coupling agent improved the similarities in solubility characteristics between the wheat straw flour and polymer matrix, resulting in good bonding between them. Figure 6 shows the SEM image of the foamed biocomposites with 15% filler loading and two different CA contents (0% and 2.5%). On these micrographs, pulled out natural fillers are shown with white arrows indicating poor adhesion. It is obvious in this figure that the number of pulled out fillers was greater in the samples produced without CAs.

The mean tensile modulus values ranged from 14.75 MPa to 38.48 MPa. While filler loading had a significant effect ($P < 0.0001$), CA contents had no notable effect ($P = 0.6717$) on tensile modulus. Tensile modulus of foamed biocomposites was significantly reduced with a rising concentration of filler. For unfoamed samples, addition of the lignocellulosic filler improves tensile modulus of the thermoplastic composites and usually could simply be explained by the rule of mixtures (Matuana *et al.* 1998). In the case of foamed composites, tensile modulus could be more prone to irregular foam structure than 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.

In the case of elongation at break values, the mean values ranged from 31.03% to 300%. They were significantly reduced with the increased filler loading ($P < 0.0001$) but not significantly affected by CA contents ($P = 0.2104$). It is believed that elongation at break values were usually reduced with the increased modulus values for biocomposites filled with lignocellulosics. The mean impact strength values were in the range of 34.08 J/m to 193.11 J/m. Similar to elongation at break values, impact strength of foamed biocomposites were also significantly reduced with the increased filler loading ($P < 0.0001$), but not significantly affected by CA contents ($P = 0.3787$).



(a) **(b)**

Fig. 6. SEM images of foamed biocomposites with 15% filler loading: a) 0% CA content and b) 2.5% CA content

CONCLUSIONS

1. Foamed biocomposites in the density range of 0.57 to 0.81 g cm^{-3} were manufactured utilizing wheat straw flour as a natural filler and high-density polyethylene as a polymer matrix. The lowest density values were achieved when less than 20% filler loading and 1% CA contents were used in the formulations.
2. Flexural and tensile modulus values were improved with increased filler loading. However, flexural strength, tensile strength, elongation at break, and impact strength of foamed biocomposites were reduced.
3. The use of a coupling agent improved only the tensile strength but either did not affect or reduced the other mechanical properties of foamed biocomposites.
4. It is demonstrated that mechanical properties of foamed biocomposites were more prone to cellular structure than dissimilarities between polymer matrix and fillers.

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