CELL MORPHOLOGY AND PHYSICO-MECHANICAL PROPERTIES OF HDPE/EVA/RICE HULL HYBRID FOAMED COMPOSITES

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In this research, the cell morphology and physico-mechanical properties of HDPE/EVA/Rice hull hybrid foamed composites were investigated. For this aim, composites were prepared via melt mixing and then foamed using a compression molding method. Mechanical properties such as tensile strength, tensile and flexural modulus, and density were measured. Morphology of the samples was also evaluated by scanning electron microscopy (SEM). Results indicated that the tensile strength, tensile and flexural modulus, and density increased with the increase of rice hull content. However, with addition of blowing agent content and EVA content, mechanical properties and density of foamed composites decreased. Rice hull fibers acted as nucleating agents that substantially reduced cell size and increased cell density. In addition, EVA played an important role in foaming process by increasing the melt viscosity of the polymer matrix, in a way that samples with higher content of EVA have the highest cell density and the lowest cell size.

Keywords: Hybrid foamed composites; Rice hull; EVA; Batch foaming method; Blowing agent; Cell morphology

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INTRODUCTION

Using natural fibers as a reinforcing agent of polymer materials has attracted considerable attention in recent years. Although wood plastic composites (WPC) have been commercialized, their potential for use in many industrial (mainly automotive and decking) applications has been limited because of their brittleness, lower impact resistance, and generally higher density compared to neat plastics (Matuana and Heiden 2004). The concept of creating foamed structures in the composites as a means to compensate for these shortcomings has successfully been demonstrated (Matuana *et al.* 1996). Foaming WPC by either chemical blowing agent (CBA) (Rizvi *et al.* 2002; Guo *et al.* 2007), physical blowing agents (PBA) (Matuana *et al.* 1998a; Zhang *et al.* 2004), or by stretching (Kim *et al.* 2004), can decrease the density, reduce material cost, and improve impact strength. Producing a uniform fine-celled structure in WPC has been demonstrated to be extremely effective at improving their performance (Matuana *et al.* 1998a,b). Due to these unique properties, plastic foams can be used in many industrial applications, including light-weight, high-strength parts for the automotive and aerospace industries, containers, sporting goods, and thermal and electrical insulators (Bledzki *et al.* 2005).

The field of WPC foam technology is in some ways in the early stages of research and development, not withstanding its relatively long history. The production of microcellular-foamed structures in wood fiber-PVC (poly-vinylchloride) composites through a batch-foaming using a physical blowing agent (Matuana *et al.* 2001a,b) was investigated. The results indicated that a batch-microcellular foaming process produces foamed plastic characterized by a cell-population density (i.e., the number of cells nucleated per unit volume of the original unfoamed polymer) in the range of $l0^9$ to 10^{15} cells/cm^3 and fully grown cells in range the range of 0.1 to 10 μm. Bledzki and Faruk (2006) studied the effects of chemical foaming agent content on cell morphology, density, and properties (tensile, flexural, and impact) injection molded WPC base on polypropylene. They found that mechanical properties (specific tensile strength, specific tensile modulus, specific flexural strength, and specific flexural modulus) decreased gradually with increasing CBA content, 2 to 5 wt%, and cells became bigger in size due to the availability of gas. Density was reduced with all foaming agent contents (to a maximum of 30%), and decreased to 0.739 g/cm^3 at a wood fiber content of 30 wt%. The microcellular foaming in an injection moulding process was also introduced containing natural fibre (flax fibre) as well as wood fibre (Bledzki and Faruk 2002, 2003; Bledzki *et al.* 2002). Rodrigue *et al.* (2006) investigated the effect of wood powder on the step of foaming of wood-low density PE composites in an extrusion process. They reported that wood particles acted as nucleating agents that substantially reduced cell size and increased cell density.

In foaming of thermoplastics, the solubility of gas in a polymer changes with pressure and temperature (Shim and Johnston 1991; Durril and Griskey 1966, 1969; Van Krevelen 1980; Park 2000). The fundamental factors in foam formation are bubble nucleation formation, bubble growth, and bubble stability. During foaming of natural fiber (wood fiber) reinforced polymer composites, because of the presence of solid natural fibers, there is a much higher potential for heterogeneous nucleation at the solid melt interfaces than for homogeneous nucleation. The heterogeneous nucleation can occur either as a result of an increase in the free energy of the system caused by reduced surface tension at the interface of the liquid polymer and the solid fiber, or because of entrapped gas in the micro-voids at the interfaces (Throne 1996). At higher processing temperatures, natural fiber (wood fiber) releases volatiles that affect the cell nucleation. These volatiles contain H_2O, CO_2, and other constituents (Mohanty and Misra 1995; Orfao *et al*. 1999; Tang and Bacon 1964; Degroot *et al*. 1988; Scheirs *et al*. 2001). The final step of microcellular foaming, that of control of the cell growth, is dependent on the following factors (Park *et al*. 1998): i) the use of an appropriate amount of blowing gas, ii) minimal diffusion of gas, and iii) the suppression of cell coalescence, cell coarsening, and cell collapse.

Recently, foams of polyethylene (PE) and ethylene vinyl acetate (EVA) blends are becoming more and more interesting because of their higher flexibility, toughness, impact resistance, controllable open cell contents, and lower thermal conductivity in comparison with PE foam (Riahinezhad *et al*. 2010).

Such foamed composites are gaining growing acceptance for structural applications, including a variety of building products, automotive, infrastructure, and other consumer/industrial applications, because of their unique structural properties, especially their low weight and higher impact strength (Riahinezhad 2009).

Unfortunately, only limited information dealing with the foaming in HDPE/EVA composites containing natural fiber is available.

The main objective of this work is to investigate the cell morphology and physico-mechanical properties of HDPE/EVA/Rice hull hybrid foamed composites. The novelty of the present study concerns the use of EVA, which has the important role of impact modifier of wood plastic composites and influences the foaming process by increasing the melt viscosity of the polymer matrix. In addition, adding EVA leads to increasing the flexibility, as well as the capacity for matrix branching. Thus, when EVA content increases, the matrix branching as well as the cell density of foam increase too (Riahinezhad 2009).

Cell morphology was evaluated using cell size and cell density. The result could be used as a preliminary baseline data for study the feasibility of using rice hulls as filler for micro-foaming in polymer.

EXPERIMENTAL

Materials

High-density polyethylene (EA 6070), supplied by Iran Petrochemical Commercial Co., was used in this study. It has a density of 0.956 g/cm^3 and a melt flow index of 6.8 g/10min. Ethylene vinyl acetate with 18%wt VA content (EVA, MFI = 2 g/10min, density = 0.93 g/cm^3), from Samsung, Korea was utilized in the experiment. The commercial type of Rice Hull (RH) (100-mesh size) provided by North Shaltouk was used as the filler. Maleic anhydride-g-HDPE (MAPE, MFI = 5 g/10min, density = 0.93 g/cm^3) from Gity Pasand Co, Iran was utilized for improving the adhesion between the hydrophobic HDPE and the hydrophilic RH as a coupling agent (CA). Azodicarbonamide (ADC, density = 1.65g/cm^3, decomposition temperature in air = 205-215˚C, average gas yield = 220 mL/g, Anhui Huishang Group, China) as a chemical blowing agent, zinc oxide (ZnO, density = 5.606 g/cm^3, melting point=1975˚C, boiling point = 2360˚C, Rangine Pars, Iran) as an ADC activator, and stearic acid (density = 0.85 g/cm^3, melting point = 69.6˚C, boiling point = 383˚C, Palmac, Malaysia) as an external lubricant were also used.

Sample Preparation

Formulation of the samples is depicted in Table 1. The written numbers in front of the letters indicate the weight percentage of the materials. The effects of the three parameters blowing agent content (at three levels of 2, 4, and 6 wt.%), EVA content (at two levels of 5 and 10 wt.%), and rice hull fiber (RH) content (at three levels of 30, 40, and 50 wt.%) have been considered in this work. The amount of MAPE, ZnO, and stearic acid were kept constant at 2, 1.5, and 1 per hundred parts with respect to polymers, respectively.

TABLE 1. Formulation of the Samples

Sample	Rice Hull (RH) (wt.%)	HDPE (wt.%)	EVA (wt.%)	MAPE (wt.%)	ADC (wt.%)
RH 30/ EVA 5/ ADC 2	30	61	5	2	2
RH 30/ EVA 10/ ADC2	30	56	10	2	2
RH 40/ EVA 5/ ADC 2	40	51	5	2	2
RH 40/ EVA 10/ ADC2	40	46	10	2	2
RH 50/ EVA 5/ ADC 2	50	41	5	2	2
RH 50/ EVA 10/ ADC2	50	36	10	2	2
RH 30/ EVA 5/ ADC 4	30	59	5	2	4
RH 30/ EVA 10/ ADC4	30	54	10	2	4
RH 40/ EVA 5/ ADC 4	40	49	5	2	4
RH 40/ EVA 10/ ADC4	40	44	10	2	4
RH 50/ EVA 5/ ADC 4	50	39	5	2	4
RH 50/ EVA 10/ ADC4	50	34	10	2	4
RH 30/ EVA 5/ ADC 6	30	57	5	2	6
RH 30/ EVA 10/ ADC6	30	52	10	2	6
RH 40/ EVA 5/ ADC 6	40	47	5	2	6
RH 40/ EVA 10/ ADC6	40	42	10	2	6
RH 50/ EVA 5/ ADC 6	50	37	5	2	6
RH 50/ EVA 10/ ADC6	50	32	10	2	6

Composite Preparation

The preparation of HDPE/EVA/rice hull hybrid composites was conducted in an internal mixer (HAAKE SYS90, USA) at a constant temperature of 120˚C and rotor speed of 60 rpm. Tangential Banbury-type rotors were used with the mixing volume chamber of 300 cm^3. The procedure for mixing was as follows (Riahinezhad *et al*. 2010): (i) HDPE, stearic acid, EVA, and HDPE-g-MA (MAPE) were added into the mixer and allowed to melt. The melting behavior was followed by monitoring the variation of torque versus time; (ii) After the melting of polymeric ingredients, the air-circulating oven-dried RH fiber (at 85˚C for 24 h) was added, and then mixing continued up to 10 min; and (iii) then ADC and ZnO were incorporated into the mixture and allowed to mix for 2min. Finally, compounding was completed at 15 min.

The low mixing temperature of 120˚C was chosen to avoid the premature decomposition of blowing agent.

Foam Preparation

Composites were foamed via a compression molding method. The dimensions of the mold used were 136×111×6 mm. Samples were placed in a hot press machine (model Davenport 25, English) at a temperature of 180˚C and a pressure of 50 bars. The dimensions of the plates of press were 254×254 mm. After 15 min, samples were cooled with cold water circulation for 3 min, and then pressure was removed and the samples were allowed to expand.

Foam Property Characterization

Density test

The density measurements of the foamed (P_f) samples were performed using a Kern ALJ 220-4 NM density tester and determined by averaging the mass/volume measurement results of six specimens per sample, following the procedure described in ASTM D 1622-03 standard.

Morphology

The cellular morphology of foams was characterized by scanning electron microscopy (SEM, Philips XL 30). Samples were first frozen in liquid nitrogen (-196˚C) and fractured to ensure that the microstructure remained clean and intact, and then coated with a gold layer to provide electrical conductivity. Cell size, which is characterized by the average diameter of the cells, was determined by analyzing SEM micrographs. Cell density, which is the number of cells per unit volume of the original unfoamed polymer (N_0), was calculated using the following equations (Lee *et al*. 2003; Baldwin *et al*. 1996a,b),

$$V_f = \left(1 - \left(\frac{P_f}{P_p}\right) \right) \tag{1}$$

$$N_f = \left(\frac{nM^2}{A}\right)^{3/2} \left[\frac{1}{1 - V_f}\right] \tag{2}$$

where V_f is the void fraction, P_f is the density of the foamed samples, P_p is the density of the unfoamed samples, N_f is the cell density, n is the number of cells in the micrograph, and A and M are the area and the magnification factor of the micrograph, respectively.

Mechanical property tests

The tensile and flexural tests were conducted with an Instron universal testing machine (model 4486) with a cross-head speed of 5 mm/min according to the ASTM standards D638 and D790, respectively, for the HDPE/EVA/rice hull hybrid composite foams. The dimensions of the samples used for flexural tests were 115×13×6 mm. samples with a dimension of 115×19×6 mm type IV were cut for tensile testing.

All tests were performed at room temperature (23˚C) at a relative humidity of 50%; and three to five samples were tested for each treatment.

RESULTS AND DISCUSSION

Composite Foam Morphology

The morphology of the composite foams was analyzed in terms of cell size and cell density. SEM micrographs of HDPE/EVA/rice hull hybrid composites with different levels of rice hull contents and blowing agent contents are shown in Figs. 1 and 2.

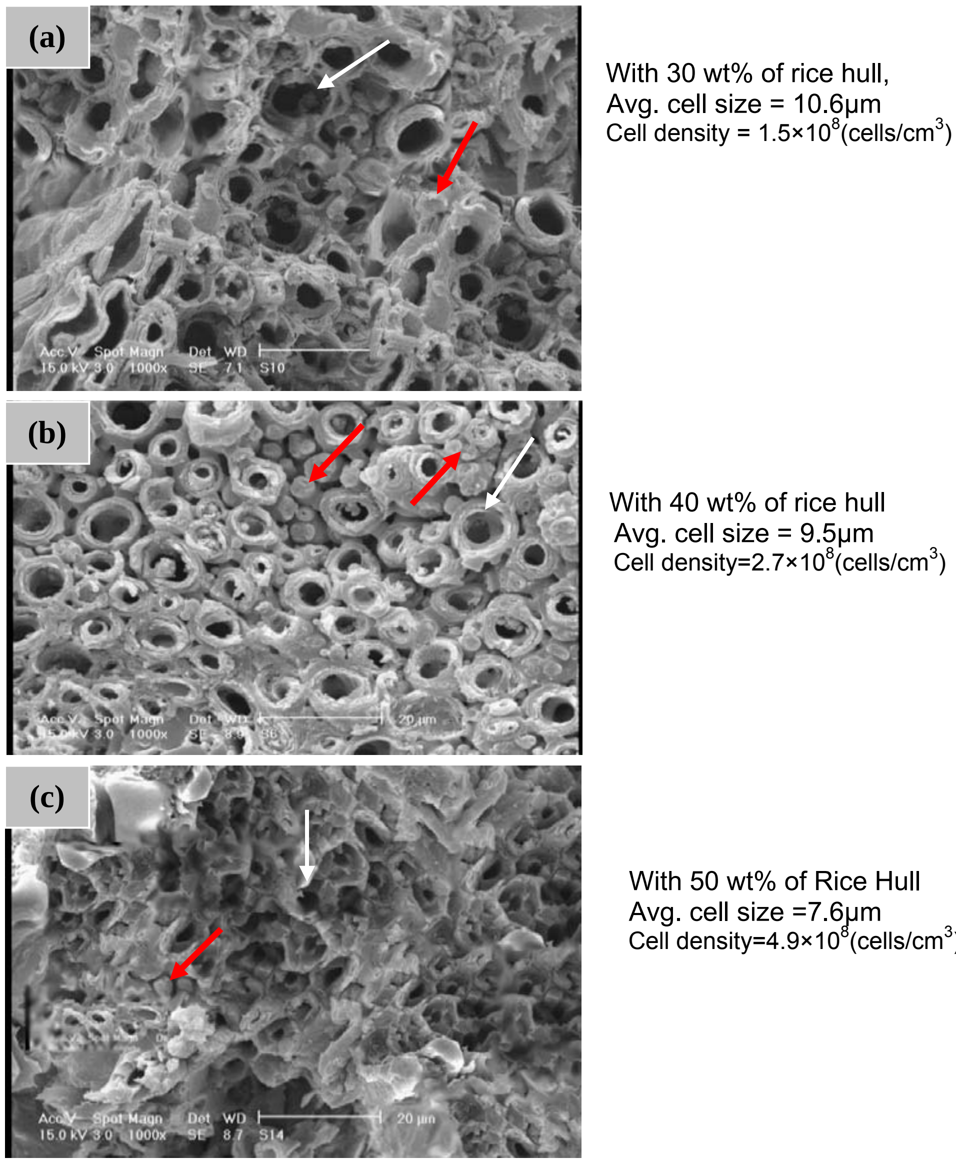

Fig. 1. SEM micrographs of foamed HDPE/EVA/rice hull composites with different levels of rice hull contents: a) RH 30/ EVA 10/ ADC 6, b) RH 40 / EVA 10/ ADC 6, c) RH 50/ EVA 10/ ADC 6

From these figures it is clear that rice hull fiber content and blowing agent content had a definite effect on cell morphology. It seems that the shown filled circles on the SEM micrographs using red arrows indicate the rice hull fiber (Fig. 1). The image magnification used was 1000x.

The effect of rice hull fiber content on cell size and cell density is reported in Table 2. The results indicate that as the rice hull fiber content increased, the average cell size decreased and cell density increased. This can be explained by two effects: first, the melt viscosity of the matrix increases with rice hull content, generating higher resistance to cell growth in the foaming process (Matuana *et al.* 1998a). Second, improvement in cell morphology of composite foams can be attributed to the dispersed natural fiber (in this work rice hull) in the polymer matrix. These particles are believed to act like nucleating agents, thus promoting heterogeneous nucleation (Rodrigue *et al.* 2001). At a constant blowing agent concentration, the amount of gas available for foaming is constant. Increasing the number of nucleation sites through a heterogeneous mechanism leads to smaller sizes (Zhang *et al.* 2005). In other words, a larger amount of cells more rapidly consumed the available gas and limited cell growth. As a result, cell size decreased and its distribution was narrower (Rodrigue and Gasselin 2003). Thus, as expected, the highest and the lowest cell size were found in the composite foams with 30 wt% of rice hull and 50 wt% of rice hull, respectively.

Table 2. Effect of Rice Hull Fiber Content on Cell Size and Cell Density of Foamed HDPE/EVA/Rice Hull Composites

Sample	Rice hull contents (wt.%)	Cell size (μm)	Cell density (Cells/Cm^3)
RH 30/ EVA / ADC	30	9.0	3.2×10^8
RH 40/ EVA / ADC	40	7.6	4.2×10^8
RH 50/ EVA / ADC	50	6.2	5.5×10^8

Table 3 shows the effect of blowing agent content on the cell size and cell density. From Fig. 2 and Table 3, it is found that there is a strong dependence of cell size and cell density on the blowing agent content. With the increase of blowing agent content, average cell size increases and cell density decreases. This was expected, because that as higher blowing agent content were used, more gas was subsequently generated, it migrates preferentially to the existing bubbles rather than nucleating new bubbles, leading to the growth of large irregular bubbles (Klempner and Frisch 1991). As reported by Matuana et al (1997, 2001b), once the cells were nucleated, they continue to grow as long as there was enough gas for diffusion into the nucleated cells.

Table 3. Effect of Blowing Agent Content on Cell Size and Cell Density of Foamed HDPE/EVA/Rice Hull Composites

Sample	Blowing agent contents(wt.%)	Cell size (μm)	Cell density (Cells/Cm^3)
RH / EVA / 2ADC	2	3.5	5.0×10^8
RH / EVA /4 ADC	4	9.4	4.4×10^8
RH / EVA /6 ADC	6	9.9	3.2×10^8

Based on these observations, the smaller cell density of foamed samples may be attributed to the decrease of the thickness of the cell walls. The thickness of the walls will decrease as the void fraction and cell size increase. Thereby, these results could be caused by the cell growth mechanism, which depends on the amount of gas dissolving in the polymer matrix during the foaming process (Matuana *et al.* 1997).

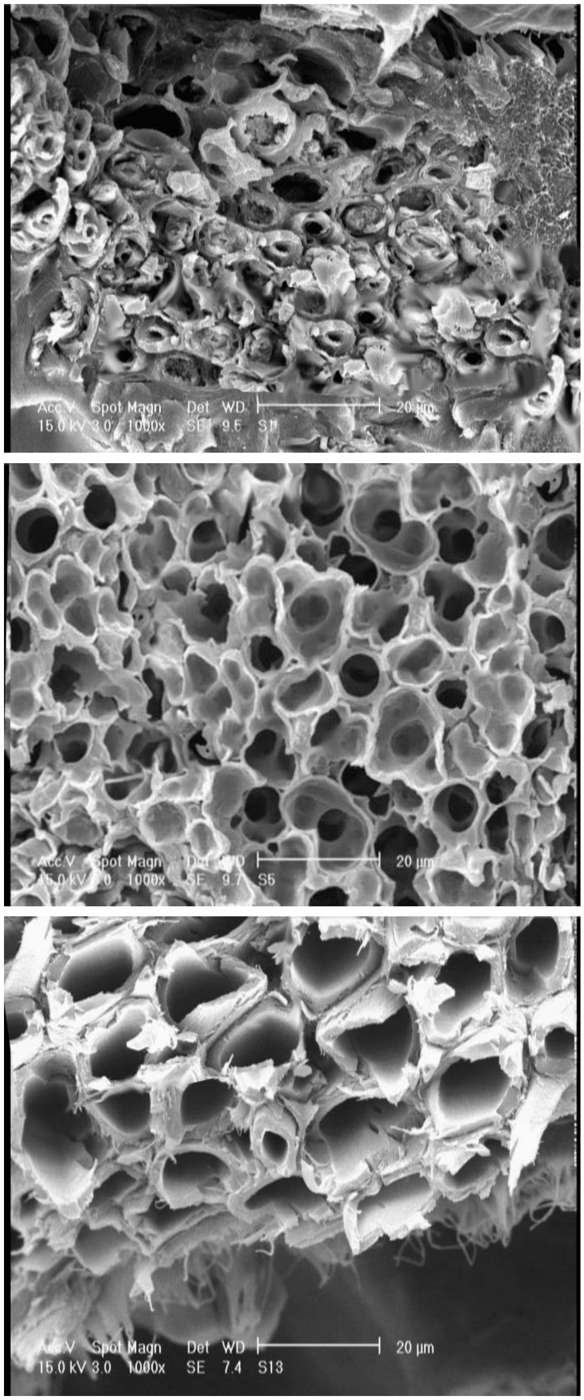

Fig. 2. SEM micrographs of foamed HDPE/EVA/rice hull composites with different levels of blowing agent contents: a) RH 40/ EVA 5/ ADC 2, b) RH 40/ EVA 5/ ADC 4, c) RH 40/ EVA 5/ ADC 6

Figure 3 presents the dependence of the cell morphology on the EVA content for foamed HDPE/EVA/rice hull composites. As shown in Fig. 3 (and Table 4), as the EVA content increases, the average cell size decreases and cell density increases. This may be attributed to increase of melt viscosity, which provided high resistance to the cell growth in the polymer, resulting in a reduction in the rate of cell growth during foaming and thus, cell size decreases.

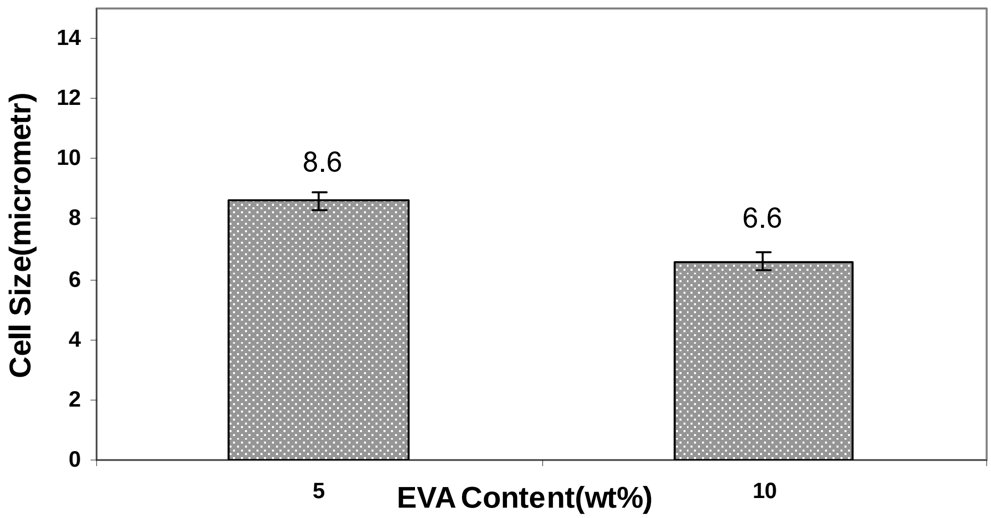

Fig. 3. Effect of EVA content on the cell size for foamed HDPE/EVA/ rice hull composites

Table 4. Effect of EVA Content on Cell Size and Cell Density of Foamed HDPE/EVA/Rice Hull Composites

Sample	EVA contents (wt.%)	Cell size (μm)	Cell density (Cells/Cm^3)
RH /5 EVA / ADC	5	8.6	4.0×10^8
RH / 10EVA / ADC	10	6.6	4.6×10^8

Foam Density

One important objective in foaming composites is density reduction, which is related to the void content achieved in the polymer matrix. In this study, the effects of the blowing agent content, EVA content, and rice hull fiber (RH) content on the density of the foamed composites were investigated. Figure 4 shows the variation of density versus RH concentration at different levels of ADC. As seen, the density of the composite decreased with increase of blowing agent content. This is because as greater concentrations of blowing agent were used, more gas was subsequently generated, reducing the relative foam density. In other words, the higher blowing agent concentrations shorten the growth time of the foam, thus restricting the gas from escaping through the foam surface, allowing the foam to expand more, and consequently, producing foam with a lower relative density. Another interesting point in Fig. 4 is that with the increase of rice hull fiber content, density also increased in all cases due to the different density of polymers (HDPE and EVA) and rice hull fiber. The high pressures used in polymer processing tend to compact the material to its maximum density of around 1300 to 1400 kg/m^3 (English *et al.* 1996). The other reason may be due to the increased viscosity of mixture when the rice hull content increases. The increased viscosity would inhibit the foam from rising, and therefore the density of the foam would increase. Also the density of rice hull itself was higher than that of the foams.

Park and co-workers (2006) also studied the extrusion foaming of rice hull–PP composites with a physical foaming agent. It was found that the desired foam density of 0.6 to 0.8 g/cm^3 was successfully achieved with rice hull as filler. The effect of foaming on the density of wood plastic composite with varying concentrations of microspheres and wood flour has been investigated by Ahmad (2004). He concluded that the density of WPC increases by approximately 21 percent with wood filler contents up to 50 percent. An optimum concentration of microspheres approximately 3 percent and wood contents in the range 20 to 30 percent provided the best density reduction of 38 percent in the WPC samples compared to the similar unfoamed samples. Similar results have been reported in the literature by several authors (Zhang *et al.* 2005; Bledzki and Faruk 2002, 2006; Rodrigue *et al.* 2006).

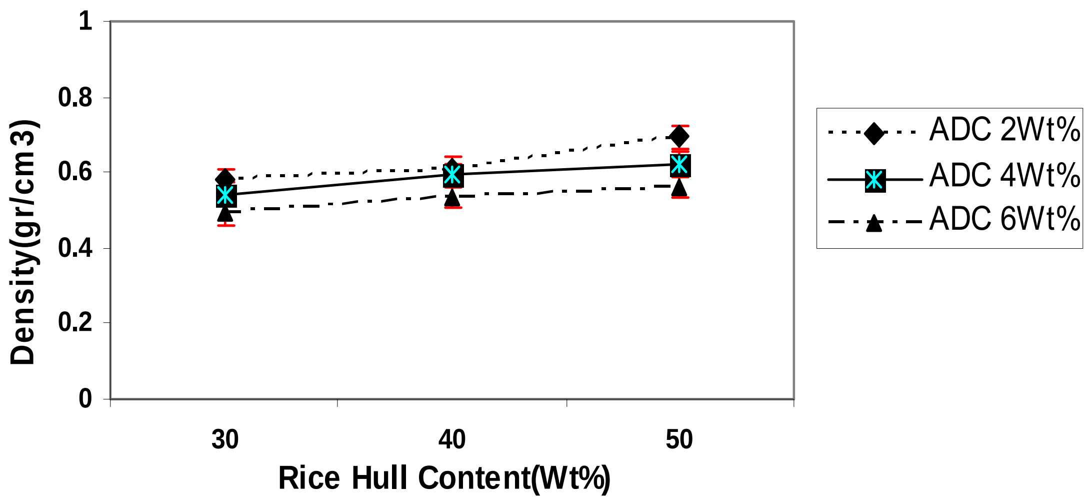

Fig. 4. Effect of rice hull (RH) content and blowing agent content on the density of foamed HDPE/EVA/rice hull composites

Figure 5 presents the effect of EVA content on the density of the foamed composites. As can be seen, the density decreased with increase of EVA content. This may be caused by an increased the melt viscosity of the matrix when the EVA content increases. A high melt viscosity and a high stiffness offer a large resistance to the polymer matrix for the growth of cells. As a result, the rate of cell growth during foaming would be reduced and a higher and more stable number of cells per unit volume would be produced. Therefore the density of the foamed composites is decreased.

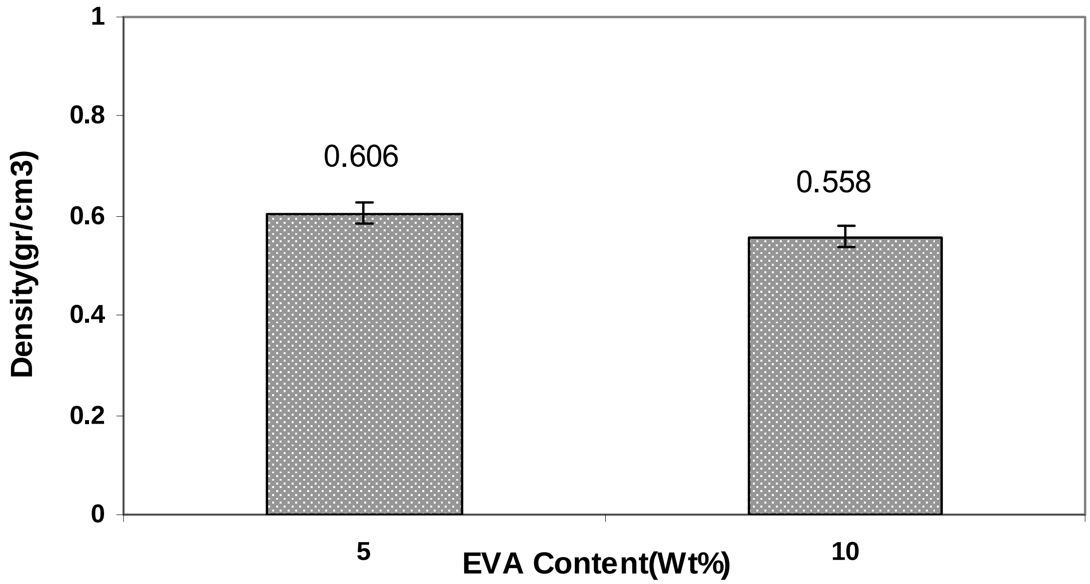

Fig. 5. Effect of EVA content on the density for foamed HDPE/EVA/rice hull composites

Mechanical Properties

Tensile properties of foamed HDPE/EVA/rice hull composites

The dependency of the tensile strength and tensile modulus on the rice hull content and blowing agent content for foamed HDPE/EVA/rice hull composites is illustrated in Figs. 6 and 7, respectively. The results show that the tensile strength and tensile modulus increased with the increase of rice hull content.

Since mechanical properties are related to the morphology of the foam, the relation between cell size and tensile properties should be considered. As previously mentioned, rice hull fibers can act as heterogeneous nucleating agents and definitely affect cell nucleation and cell growth. Therefore, as the greater content of rice hull was used, smaller cell size was subsequently produced and cell density was increased, improving the tensile properties. Another reason could be also that the more fibers meant smaller spaces in them. Other researchers also reported that foams with very fine cells generally exhibit better mechanical properties (Matuana *et al.* 1998b; Ahmad 2004; Bledzki *et al.* 2005).

Another interesting point in Fig. 6 and 7 is that as the blowing agent content increased, the tensile strength and tensile modulus decreased due to their larger cell size and a very nonuniform cell size distribution. The results are in agreement with those reported by Mengeloglu and Matuana (2001, 2003). It seems that larger cells present in the some samples produced in this study acted as cracks or defects and led to premature failure, resulting in strength and stiffness reduction in the samples.

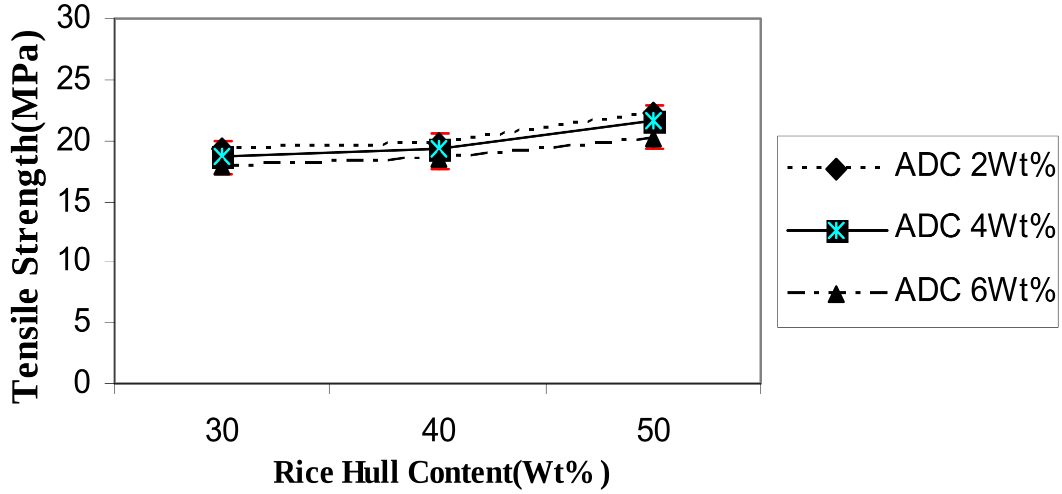

Fig. 6. Dependence of tensile strength on the rice hull content and blowing agent content for foamed HDPE/EVA/rice hull composites

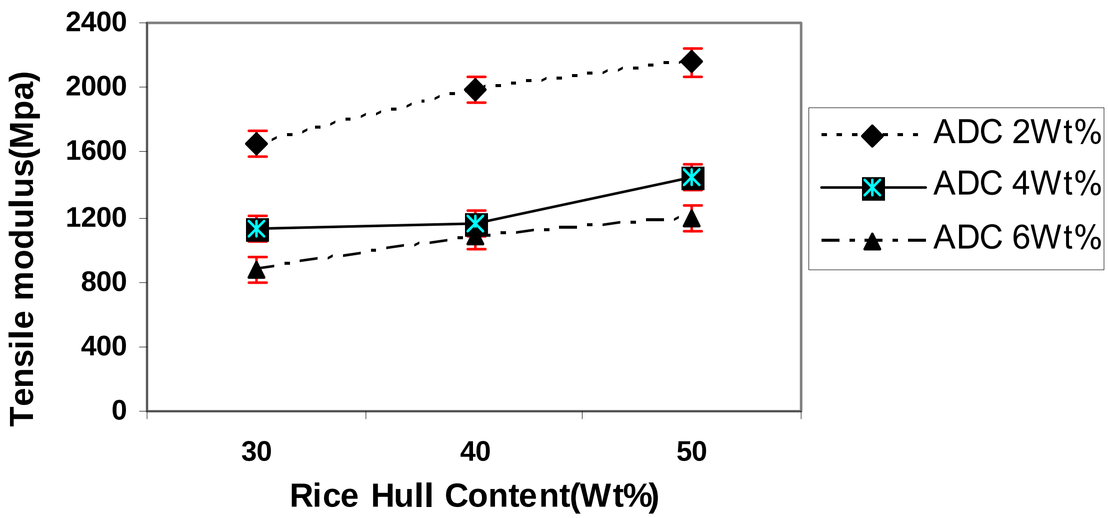

Fig. 7. Dependence of tensile modulus on the rice hull content and blowing agent content for foamed HDPE/EVA/rice hull composites

The effect of EVA content on the tensile strength and tensile modulus of the foamed composites is presented in Figs. 8 and 9, respectively. As can be seen, the tensile properties decreased with the increase of EVA content. As the EVA has lower modulus than HDPE matrix, therefore this effect can be explained by the decreased modulus of the matrix due to the incorporation of EVA. Low modulus leads to unstable cells that are easy to break up. Thus the tensile properties would be decreased.

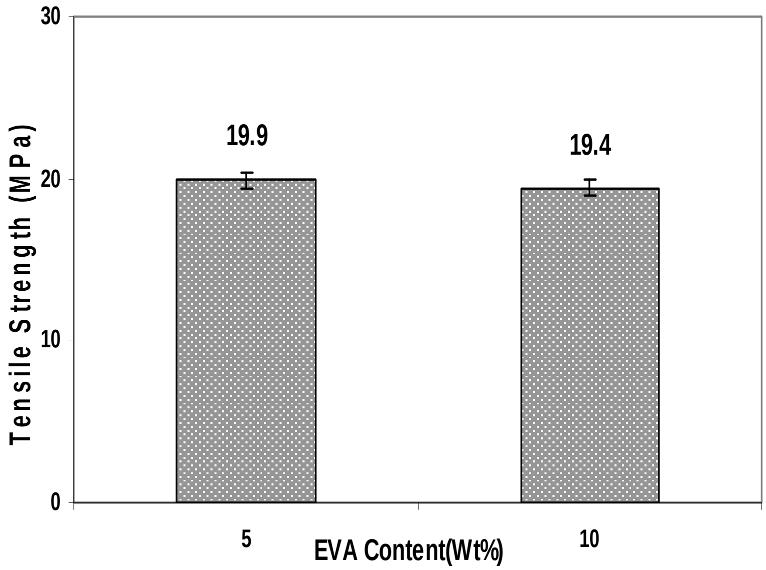

Fig. 8. Effect of EVA content on the tensile strength for foamed HDPE/EVA/rice hull composites

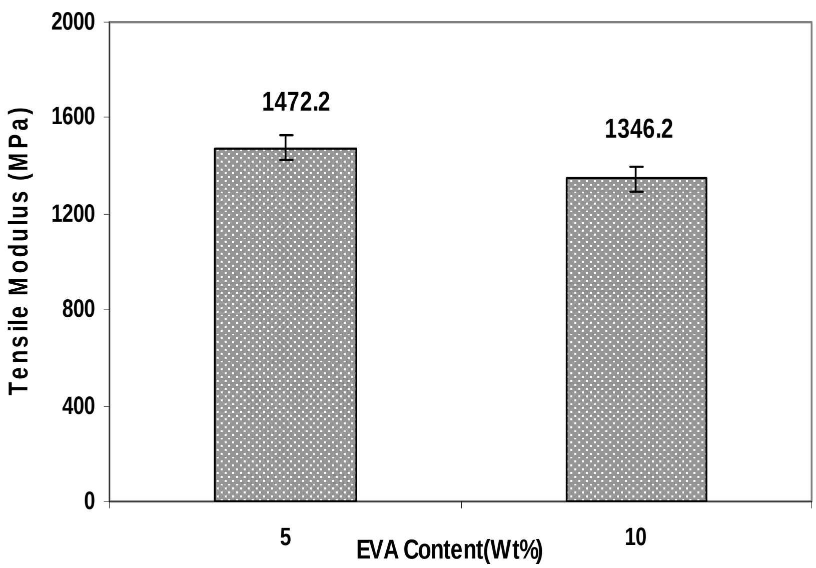

Fig. 9. Effect of EVA content on the tensile modulus for foamed HDPE/EVA/rice hull composites

Flexural Modulus of Foamed HDPE/EVA/Rice Hull Composites

The changes in flexural modulus for foamed composites with different rice hull content and different blowing agent content are illustrated in Fig. 10. Again, the flexural modulus increased as the rice hull content increased from 30% to 50%, and with the increase of blowing agent content, the flexural modulus decreased. The results are in agreement with those reported by Wang *et al.* (2008). When blowing agent content was 2wt%, flexural modulus was the highest; when the content was 6wt%, flexural modulus was the lowest.

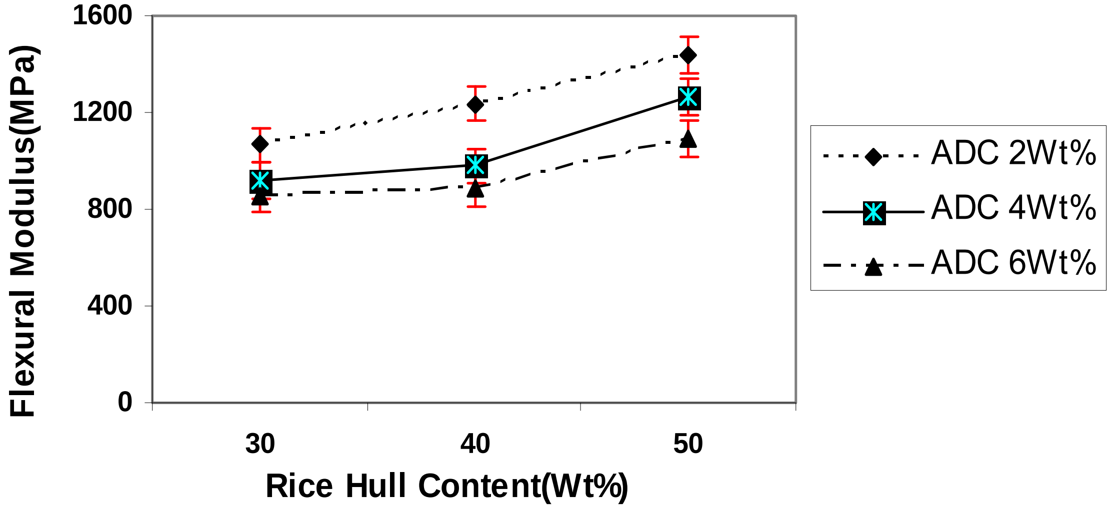

Fig. 10. Dependence of flexural modulus on the rice hull content and blowing agent content for foamed HDPE/EVA/rice hull composites

Figure 11 shows the dependence of flexural modulus on the EVA content for foamed composites. As the EVA content increased from 5 wt% to 10 wt%, the flexural modulus decreased. As mentioned earlier, this could be attributed to decrease of the modulus of the matrix, due to the lower modulus of EVA, which leads to unstable cells. The other possible reason may be due to the higher melt viscosity of these samples among the others, which restricts cell growth and decreases flexural modulus of the foamed composites.

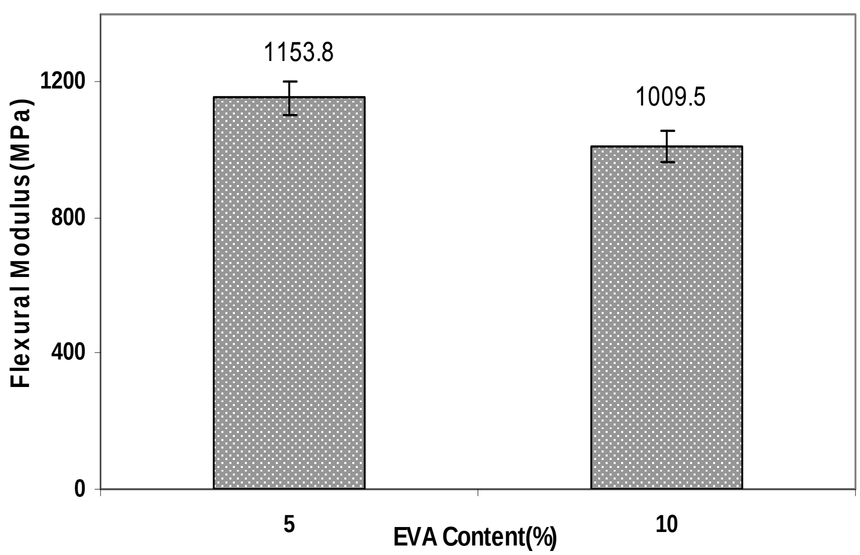

Fig. 11. Effect of EVA content on the flexural modulus for foamed HDPE/EVA/rice hull composites

CONCLUSIONS

This research work presents the analysis of the cell morphology, density, and mechanical properties of HDPE/EVA/ rice hull composite foams. Composites were prepared by melt mixing in an internal mixer, and then single-stage batch foaming was carried out by using ADC as a chemical blowing agent and zinc oxide (ZnO) as an ADC activator in a hot plate press. Morphology and physico-mechanical properties of samples were studied, and based on the experimental results, the following conclusions were drawn:

1) Tensile strength, tensile modulus, flexural modulus, and density of foamed composites increased with addition of rice hull content.
2) With the increase of blowing agent content and EVA content, tensile strength, tensile modulus, flexural modulus, and density of foamed composites decreased.
3) A strong dependence of cell morphology (including cell size and cell density) on the rice hull content, blowing agent content, and EVA content was observed for the foamed composites. Increasing rice hull content and EVA content substantially decreased the average cell size and increased cell density of the foams. However, as the blowing agent content increased, cell size increased and cell density decreased.

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