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Microcellular Wood Fiber Reinforced PP Composites: Cell Morphology, Surface Roughness, Impact, and Odor Properties*

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ABSTRACT: This article presents the investigations on the cell morphology, surface roughness, impact properties, and odor concentration of microcellular wood fiber reinforced PP composites in injection molding process with different chemical foaming agents. The chemical foaming agent and wood fiber content strongly affect the microcellular structures of wood-PP composites. Microcells' morphology (cell size, shape, and distribution) is investigated using optical light and scanning electron micrographs. Charpy impact strength, impact resistance, and damping index of the composites are influenced by different chemical foaming agent types and content. The odor concentration and surface roughness significantly improved due to microfoaming.

KEY WORDS: microcellular wood-PP composite, chemical foaming agent, injection molding process, cell morphology, surface roughness, impact, odor.

INTRODUCTION

The polymer-wood fiber composites, utilizing wood fibers as a reinforcing filler in the polymer matrix, are known to be

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advantageous over the neat polymers in terms of the materials' cost and some mechanical properties. These wood fiber composites are microcellular processed to create a new class of materials with unique properties. Recent studies have demonstrated the feasibility of developing microcellular structures in polymer-wood fiber composites.

Reinforced foamed polymeric materials offer a unique combination of dimensional stability, rigidity, and low specific gravity. In addition, they exhibit excellent strength-to-weight ratios. The reinforced foam will also have higher heat capabilities, greater flexural strength, and improved fatigue characteristics than a non-reinforced foam [1,2]. Incorporating the reinforcing filler actually improves the overall properties in two ways. The filler contributes to an increase in strength of the material and also acts as a nucleating agent to promote more uniform and complete cell formation.

Injection molding is one of the most commercially important fabrication processes for molding a broad spectrum of thermoplastics. The advantage of this injection molding processing technology is that in a conventional injection molding machine, shaped parts (sandwich structure) with compact outer skin and foamed cores and with different physical and mechanical characteristics can be prepared [3-8] using different chemical foaming agents (exothermic, endothermic, and endo/exothermic).

Researchers and foaming agents' manufacturers have shown that microfoaming gives products with a smooth surface compared to non-foamed products [9,10]. This is achieved by an outer non-foamed zone and a smaller surface structuring due to the microcellular foam structure. But there has been no practical investigation in the case of wood fiber microcellular composites.

The structure, type and content of fibers, matrix ductility, and microcells morphology of the composites are the main structural parameters, which affect the impact behavior [11].

Owing to the increasing emphasis on public health, environment, and quality of the product awareness, the emission behavior of building materials in indoor rooms and also in the automotive interior, becomes more and more important. Odors can come from the matrix, the wood fiber, or from a combination of both materials [12].

The objective of this study is to investigate the effect of different chemical foaming agents (CFAs) and the variation of their content on the cell morphology, surface roughness, impact properties, and odor concentration of microcellular wood fiber reinforced PP composites in injection molding process.

EXPERIMENTAL

Materials

Hard wood fiber (Lignocel HBS 150-500) with a particle size of 150–500 μm , was supplied by J. Rettenmaier & Söhne GmbH + Co., Germany.

Polypropylene (Stamylan P17M10) was provided as granules by Sabic Deutschland GmbH, Düsseldorf, Germany. Its melting temperature was 173°C and melting index was 10.5 g/10 min at 230°C. Its density at room temperature was 0.905 g/cm³.

To get foamed wood fiber reinforced composites, three types of chemical foaming agents were used, which were obtained from Clariant Masterbatch GmbH & Co. OHG, Ahrensburg, Germany. They are exothermic (Hydrocerol 530), endothermic (Hydrocerol BIH20E), and endo/exothermic (Hydrocerol AB40E) types of chemical foaming agents, described in Table 1.

A coupling agent, maleic anhydride–polypropylene copolymer (MAH–PP) was used and it was obtained from Clariant Corp., Frankfurt, Germany. It was used 5% by weight relative to the wood fiber content.

Processing and Foaming

The microcellular hard wood fiber–PP composites were prepared by the injection molding process. At first, hard wood fiber with PP was mixed by a high speed mixer (Henschel Mixer, type HM40 KM120) with and without coupling agent. The hard wood fiber was dried at 80°C in an air circulating oven for 24 h (moisture content <1%) before mixing. Then cold agglomerate granules were mixed with different types of chemical foaming agents, and before foaming in injection molding, the mixed granules were dried again (80°C, 24 h). The specimens (geometry: 200 × 90 × 4 mm³) of microcellular wood fiber–PP composites

Table 1. Different types of chemical foaming agents.

Reaction type	Decomposition temperature (°C)	Effective components (%)
Exothermic	170	47.5–51.5
Exo-/endothermic	160	38–42
Endothermic	150	20

were prepared by the injection molding process at a melting temperature of 150–180°C, a mold temperature of 80–100°C, and under an injection pressure of 20 kN/mm².

Measurements

The surface roughness of the wood fiber–PP composites was measured according to ISO 4287/1 with a laser sensor autofocus measurement system, which has a very good measuring ability on reflecting surfaces. It is able to handle a fast data acquisition (up to 1 kHz) and the possibility also to handle very small ($\approx 1 \mu\text{m}$ diameter) measuring points. The Charpy impact test was carried out according to EN ISO 179 with 10 unnotched samples (standard deviation <15%). To measure the impact characteristic values, the specimens were tested by using a low-velocity falling weight impact tester (EN ISO 6603-2) at room temperature in non-penetration mode. The impactor had a mass of 0.75 kg and the impact energy was 0.96 J. The odor measurements were performed with olfactometer T07 (ECOMA) as followed by standard method VDI 3881. The samples were stored for 30 min at 60°C in all cases. The reference quantity is the odorant concentration at the threshold, i.e., 1 OU/m³ (OU = odor unit).

SEM

The morphology of the wood fiber reinforced microcellular PP composites and the cell size, shape, and distribution of microcells in microcellular composites were investigated using a scanning electron microscope (SEM) (VEGA TESCAN). The fractured surfaces of flexural test samples were studied with an SEM after being sputter coated with gold.

Light Microscope

The investigation of the structure of the microcellular wood fiber reinforced composites was done with a Zeiss reflected light microscope (Type Axioplan) to determine the form and distribution of the microcells. Cross sections of sanded and polished surfaces were studied.

RESULTS AND DISCUSSION

The microcellular structure of hard wood–PP composites, strongly affected by different chemical foaming agents in the injection molding

process, is discussed in our previous papers [6,7]. It was seen that the exothermic foaming agents showed a finer microcellular structure. The effects of various concentrations (2, 3, and 5 wt%) of the exothermic chemical foaming agents on the cell morphology (cell size, shape, and distribution) of microcellular hard wood fiber–PP composites is studied with a view to establish the concentration–structure–property relationships for these materials.

Figure 1 illustrates the light micrographs of hard wood fiber–PP microcellular composites with a variation of exothermic CFA content. In all the cases, the wood fiber content is 30 wt%. It is clearly seen that exothermic 2 wt% shows a finer microcellular structure compared to others. With the increase of CFA content (2 wt% to 3 wt% to 5 wt%), the cells grow bigger in size. In this case, it can be said that exothermic CFA 2 wt% content is the optimum concentration to get the best microcellular structure.

Figure 2 shows the effect of an exothermic foaming agent content on the cell size of hard wood fiber–PP composites. The microcellular composites with an exothermic foaming agent content of 2 wt% shows a finer microcellular structure compared to other contents, and most of the cell sizes are below 100 μm . With the increasing of CFA content, the cells increase in size and become more oval in shape.

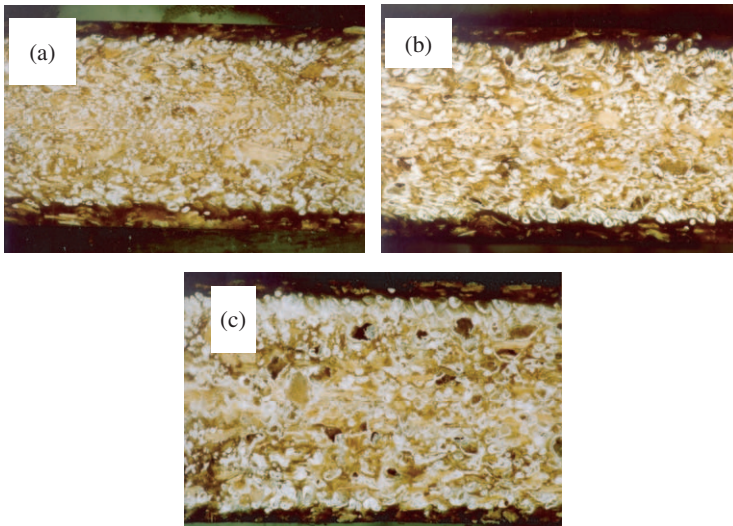


Figure 1. Influence of exothermic foaming agent content on the hard wood fiber–PP microcellular composites (exothermic foaming agent (a) 2 wt%; (b) 3 wt%; and (c) 5 wt%, wood fiber content 30 wt%).

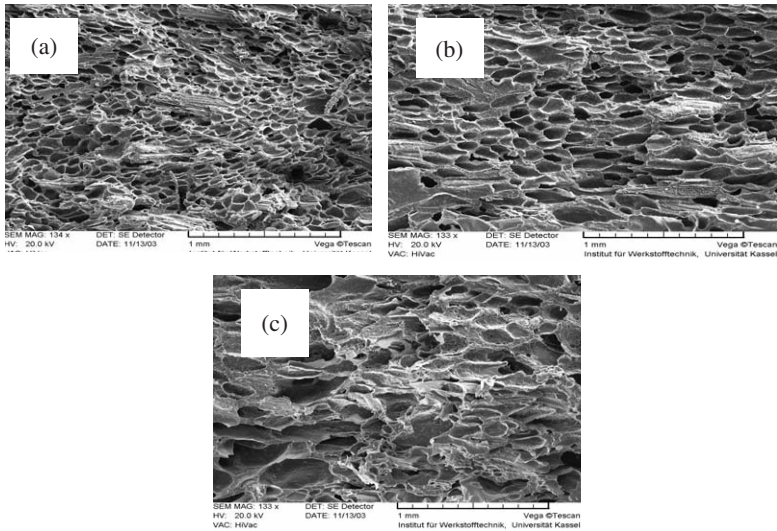


Figure 2. Effect of exothermic foaming agent content on the cell size of hard wood fiber–PP microcellular composites (exothermic foaming agent (a) 2 wt%; (b) 3 wt%; and (c) 5 wt%, wood fiber content 30 wt%).

The wood fiber content was increased to 50 wt% to observe its effect. Figure 3 illustrates that exothermic 2 wt% content creates a much lower number of cells. With the increase of CFA content, it can be seen that exothermic 5 wt% content creates an optimum large number of cells and the cells are not bigger in size.

In Figure 4, the laser sensor surface photographs of hard wood fiber–PP non-foamed and microcellular composites with 30 wt% wood fiber content are presented. The upper and lower peaks (number of peaks, area of the peaks) indicate the surface roughness of the composites, and from the figure it is clearly seen that microcellular composites with an endothermic foaming agent show lower surface roughness compared to non-foamed composites. These effects are due to the resulting internal pressure of the microfoam. Roughness irregularity and arithmetical roughness mean deviation of the microcellular composites are illustrated in Figure 5 and compared to non-foamed composites. The surface roughness of the composites is significantly reduced due to the microfoaming, and with an endothermic foaming agent, microcellular composites reduced the highest surface roughness by around 70% compared to non-foamed composites. This is due to the slow nucleation process of the endothermic foaming agent and due to the thickness of the outer zone of the composites.

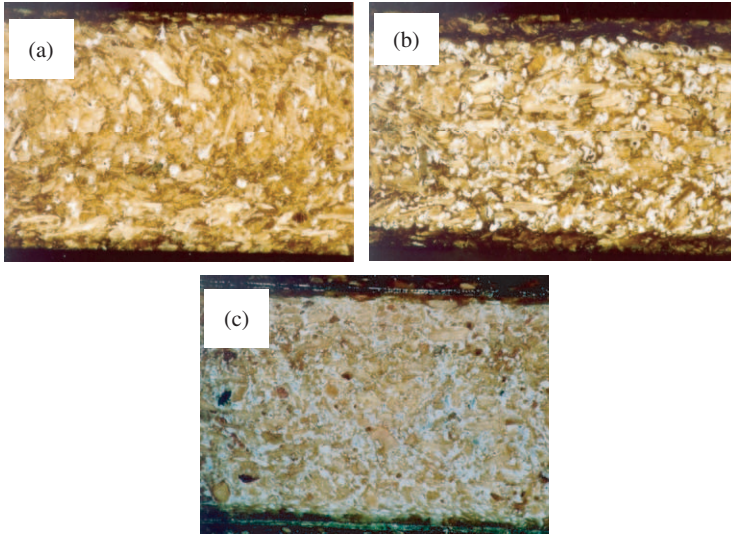


Figure 3. Influence of wood fiber content on the structure of hard wood fiber-PP microcellular composites (exothermic foaming agent (a) 2 wt%; (b) 3 wt%; and (c) 5 wt%, wood fiber content 50 wt%).

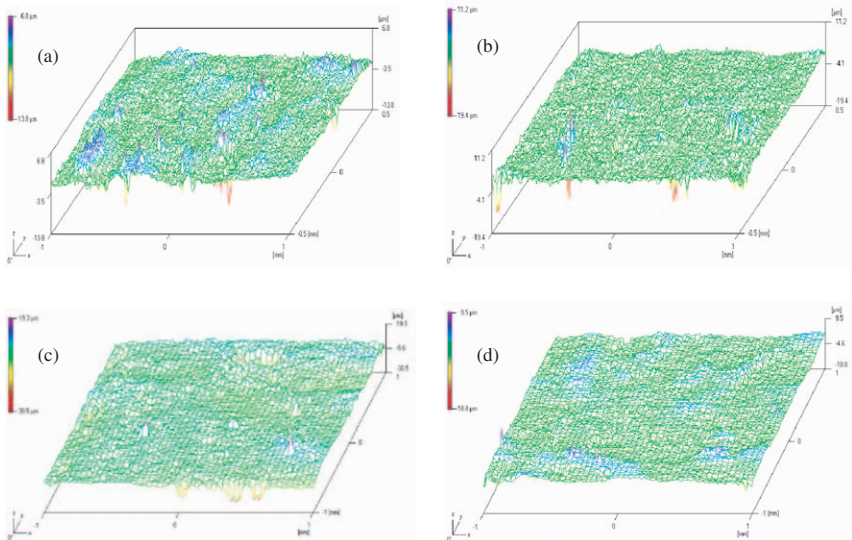


Figure 4. Surface roughness (laser sensor photographs) of non-foamed and microcellular composites ((a) non-foamed; (b) endothermic foaming agent; (c) exothermic; and (d) endo/exothermic, foaming agent content 4 wt%, wood fiber content 30 wt%).

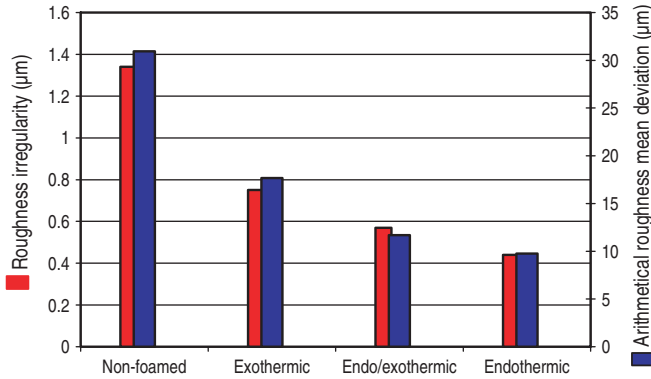


Figure 5. Roughness irregularity and arithmetical roughness mean deviation of hard wood fiber-PP composites (CFA content 4 wt% and wood fiber content 30 wt%).

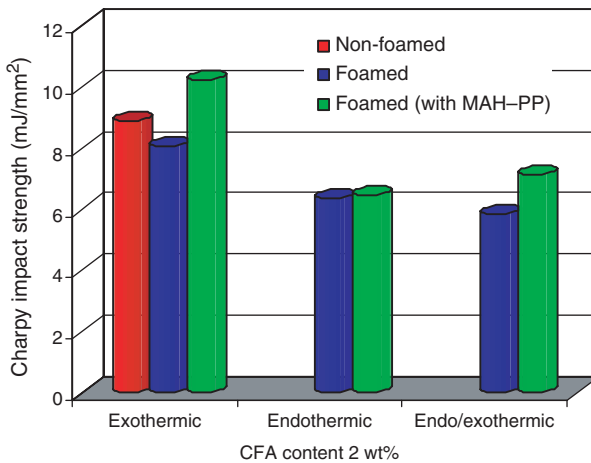


Figure 6. Effect of CFA type on the Charpy impact strength of hard wood fiber-PP microcellular composites (wood fiber content 30 wt%).

The specific Charpy impact strength of non-foamed and microcellular composites with different CFA types are presented in Figure 6. Specific Charpy impact strength followed the same tendency as tensile and flexural properties. In comparison among different CFA types, it is seen that exothermic CFA shows the highest values and with MAH-PP also, which indicates that finer microcellular structures increase the impact strength compared to non-foamed composites.

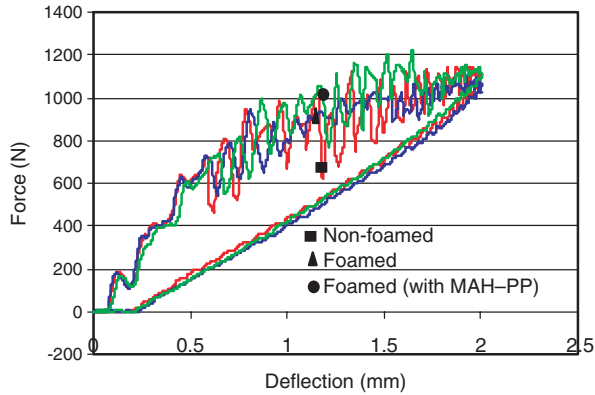


Figure 7. Impact resistance (maximum force) of hard wood fiber-PP microcellular composites (exothermic 2 wt% and wood fiber content 30 wt%).

Impact resistance is the ability of a material and its structure to survive impact-induced damages during an impact. The force-deflection curve as measured in a drop weight impact test refers to associate damage initiation by the first significant change in the slope of the curve [11]. The impact resistance of hard wood fiber-PP non-foamed and microcellular composites is presented in Figure 7. This figure illustrates that the microcellular composites show lower values with non-foamed composites and with MAH-PP, microcellular composites show higher values compared to non-foamed composites. But the values are not significantly different.

The damping index was calculated by taking the ratio of dissipated energy (loss energy) to the stored energy (strain energy) to measure the impact characteristic values. Figure 8 shows the damping index for hard wood fiber-PP non-foamed and microcellular composites with a variation of different CFA types. Microcellular composites show a higher damping index than non-foamed composites compared to CFAs, exothermic CFA shows comparatively lower damping index; with MAH-PP, exothermic CFA shows the lowest value, which is lower than that of non-foamed composites.

Figure 9 represents the damping index of the hard wood fiber-PP composites with the variation of exothermic CFA content from 0 wt% (non-foamed) to 5 wt%. It is observed that damping index increases with the increase of CFA content. But the remarkable thing is that at CFA 2 wt% content, the damping index value is very close to that of non-foamed composites, and after that the damping index increases very significantly. This means, when the composites are more completely

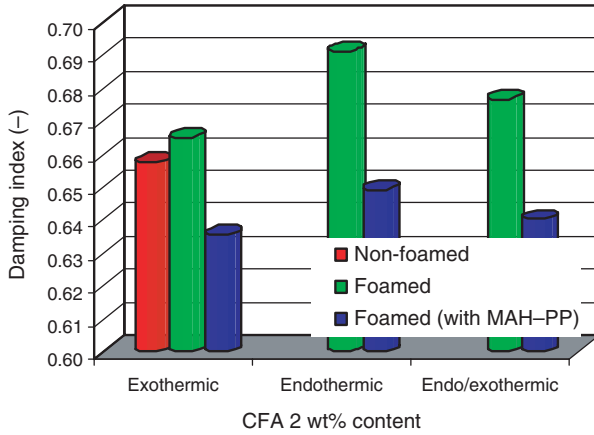


Figure 8. Effect of CFA type on the damping index of hard wood fiber-PP microcellular composites (wood fiber content 30 wt%).

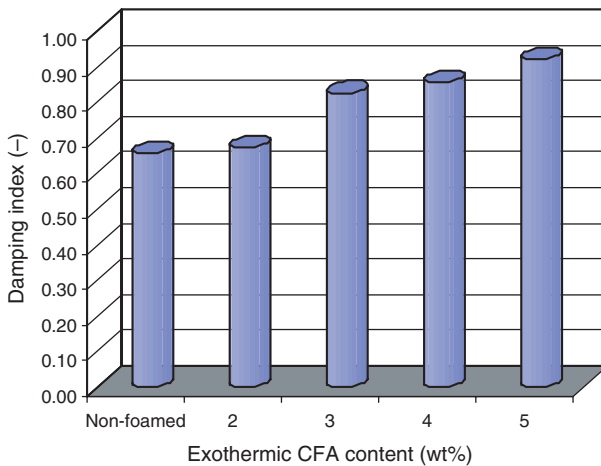


Figure 9. Effect of CFA content on the damping index of hard wood fiber-PP microcellular composites (wood fiber content 30 wt%).

foamed, the damping shows a better performance. The previous figure also showed that with MAH-PP, the composites show a lower damping index. In the cell morphology section, it is discussed that at exothermic CFA 2 wt% content, composites showed finer microcellular structures compared to higher CFA content composites. In our previous study [8], it was seen that MAH-PP has a positive influence on the cell morphology of the microcellular hard wood fiber-PP composites.

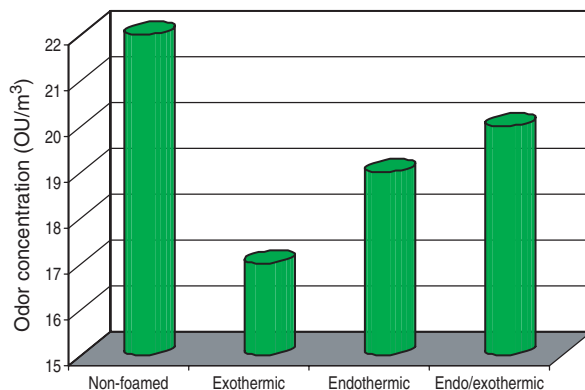


Figure 10. Odor concentration of hard wood fiber–PP microcellular composites (CFA 2 wt% and wood fiber content 30 wt%).

The odor concentration of microcellular hard wood fiber–PP composites is presented in Figure 10 and compared with non-foamed composites. From the figure, it is seen that due to the foaming, the microcellular composites show lower odor concentration compared to non-foamed composites. In the foaming process, the chemical foaming agent decomposes into gas, and some portion of the gas comes out of the composites. At that time, the odor of the composites also comes out with the gas. Comparing different CFA types, the exothermic foaming agent shows a lower odor concentration than other CFAs. It seems to be dependent on the decomposition rate and the nature of the decomposition gas.

CONCLUSIONS

This study examined the effects of different chemical foaming agents and concentrations, as well as the effect of the addition of a coupling agent on the cell morphology, surface roughness, odor, and impact properties of microcellular wood fiber–PP composites in the injection molding process. The following conclusions can be made:

- Light and SEM micrographs showed that exothermic 2 wt% content shows finer cellular structure compared to other contents at the same wood fiber content.
- An optimum CFA content varied with variation of wood fiber content.
- The microfoaming process provides products with a smoother surface compared to non-foamed composites. With the use of an endothermic

foaming agent, the surface roughness is reduced nearly 70% compared to non-foamed composites.

- Owing to the microfoaming, it is observed that the odor concentration of the wood fiber reinforced composites can be reduced.
- Different CFA types and contents have an effect on the impact properties, and with MAH-PP, damping index is reduced around 60% using an exothermic foaming agent.

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