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Microcellular Injection Molded Wood Fiber-PP Composites: Part I – Effect of Chemical Foaming Agent Content on Cell Morphology and Physico-mechanical Properties

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ABSTRACT: Microcellular wood fiber reinforced polymer materials have important applications because of their ability to reduce the density of automotive components due to their structure–property advantages. This research investigates the effects of chemical foaming agent content on cell morphology, density, and mechanical (tensile, flexural, and impact) properties of microcellular injection molded wood fiber reinforced polypropylene (PP) composites. The effects of various concentrations (2–5 wt% of the composites) of the exothermic chemical foaming agent on the properties of the composites was studied with a view of establishing the concentration–structure–property relationships for these materials.

The results indicate that exothermic 2 wt% content shows finer microcellular structure than other contents, dependent on wood fiber content. Density was reduced with all foaming agent contents, to a maximum of 30%, and decreased up to 0.739 g/cm³ at wood fiber content of 30 wt% with coupling agent. Mechanical properties increased nearly 80% with the addition of coupling agent.

KEY WORDS: microcellular wood fiber-PP composites, chemical foaming agent, injection molding, cell morphology.

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Figures 1, 3, 4, and 8–10 appear in color online: <http://cel.sagepub.com>

INTRODUCTION

The worldwide competitive situation in the plastic processing industry makes it necessary to minimize production costs by improvements and innovations in the production process and to introduce new or improved products to the markets. The material 'microcellular foam' contributes to these demands by offering lower density and processing costs. It could be a new way to save materials, if solid polymers can be replaced by microcellular polymers with 10% or more reduction in material without significantly compromising the required properties [1].

Injection molding is one of the most commercially important fabrication processes for molding a broad spectrum of thermoplastics. An advantage of the injection molding process for microcellular composites is that injection pressure decreases due to lower viscosity from the presence of dissolved gas. The cycle time is also reduced due to the elimination of the 'hold and pack' time and also due to about 25% reduction in cooling time [2]. The microcellular composites in the injection molding process are more advantageous due to minimization of distortion/deformation and also clamp force reduction. Additionally, the injection molding processing technology uses a conventional injection molding machine allowing different chemical foaming agents (CFA) (exothermic, endothermic, and endo/exothermic), shaped parts (sandwich structure) with compact outer skin, and foamed cores with different physical and mechanical characteristics [3–6].

Foamed polymers can be produced by utilizing either a chemical or a physical foaming agent. The CFA are substances which decompose at processing temperatures, liberating gases like CO₂ and/or nitrogen. Solid organic and inorganic substances are used as CFA. The solid residues serve as nucleation centers, and this leads to a finer cell structure. There are several different types of CFAs, which differ mostly in the type of gas that is generated and the type of reaction that generates that gas. The reaction that produces the gas can either absorb energy (endothermic) or release energy (exothermic). Nowadays, a combination of exo- and endothermic CFA are also used for the foaming.

The effect of CFA [7,8] on processing and properties of wood-plastic composites gained interest due to their better properties, such as insulation values, shrinkage, and distortion. Stiffness can be positively influenced.

Chemical foaming agents (CFA) enable both commodity and engineering polymers to process more easily. Compatibility and chemical reactions with foaming agent residues are becoming more important

considering improvements in the molded part weight. New CFAs are also making dramatic strides in achieving reproducible cell size distributions in microcellular foaming.

This study will explore the effects of chemical foaming agent content on cell morphology and physico-mechanical properties of microcellular wood fiber-PP composites.

EXPERIMENTAL

Materials

Polymeric Matrix

Polypropylene (PP) (Stamylan P17M10) was provided as granules by DSM, Gelsenkirchen, 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.

Wood Fiber

Hard wood fiber (Lignocel HBS 150-500) with particle size 150–500 μm was supplied by J. Rettenmaier & Söhne GmbH+Co. Germany. The wood fibers are commercial products with a mixture of different wood types.

Compatibilizer

A commercially available maleic anhydride-polypropylene copolymer (Licomont AR 504 FG) with acid number (37–43 mg KOH/g) was used as a compatibilizer for fiber treatment, and it was obtained from Clariant Corp., Frankfurt, Germany. Five percent by weight was used relative to the wood fiber content.

Chemical Foaming Agent

To get microcellular wood fiber reinforced composites, exothermic chemical foaming agent Hydrocerol 530 was used in this research work. It was obtained from Clariant Masterbatch GmbH & Co. OHG, Ahrensburg, Germany. The active ingredient was about 47–51% with the remainder as carrier polymer. The average gas yield was 120 mL/g. Exothermic CFA was used at levels of 2–5% (master batch) by weight relative to the wood fiber-PP composites.

Processing and Foaming

Hard wood fibers with PP were mixed by a high speed mixer (Henschel Mixer, type HM40 KM120) with and without the coupling agent. Hard wood fibers were dried at 80°C in an air circulating oven for 24 h (moisture content <1%) before mixing. Cold agglomerate granules were then mixed with CFA at the different levels. Before injection molding, the mixed granules were dried at 80°C for 24 h. The specimens (geometry: 200 × 90 × 4 mm in size) of hard wood fibers foamed composites were prepared by an injection molding process at melting temperature 150–180°C, mold temperature of 80–100°C and under an injection pressure of 20 kN/mm².

MEASUREMENTS

The tensile and flexural tests (Zwick Machine, UPM 1446) were conducted at a test speed of 2 mm/min according to EN ISO 527 and EN ISO 178 for hard wood fiber–PP composites, respectively. All tests were conducted at 23°C under a relative humidity of 50%. The densities of non-foamed and microfoamed specimens were measured according to DIN 53479. Fifteen replicates were conducted for each treatment. The microvoid content was calculated according to the standard ASTM D 2734-70 for foamed composites.

To measure the impact characteristic values (damping index), 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, and standard deviation (<10%) was used to measure damping index. Loss energy is a measure of dissipated energy and strain energy is a measure of the stored energy, and the damping index is the ratio of loss energy to strain energy [9]. For all experiments, the values within a standard deviation of <10% were used to calculate the mechanical properties.

SEM and Light Microscopy

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 scanning electron microscopy (SEM) (VEGA TESCAN) and a light microscope. Cross sections of sanded and polished surfaces were studied with the light

microscope. Fractured surfaces of flexural test samples were observed with SEM after coating with gold.

RESULTS AND DISCUSSION

Cell Morphology

The microcellular structures of foamed composites with wood fiber are influenced by the different types of CFA in the injection molding process [10,11]. It was observed that the influence of different CFA depends on their decomposition rate, type of decomposition gas, average gas yield, active components, and also on their carrier polymer. Cell morphology of the microcellular composites was strongly influenced by the CFA types and content, and the exothermic foaming agent showed better microcellular structure and finer cells compared to other foaming agents in all processes. For that reason, the influence of CFA content was investigated with exothermic CFA.

The effect of concentration of exothermic CFA on cell morphology for hard wood fiber-PP microcellular composites is summarized in Figure 1. In all cases, the wood fiber content is 30 wt%. It is seen that exothermic 2 wt% shows a finer microcellular structure than the others. With

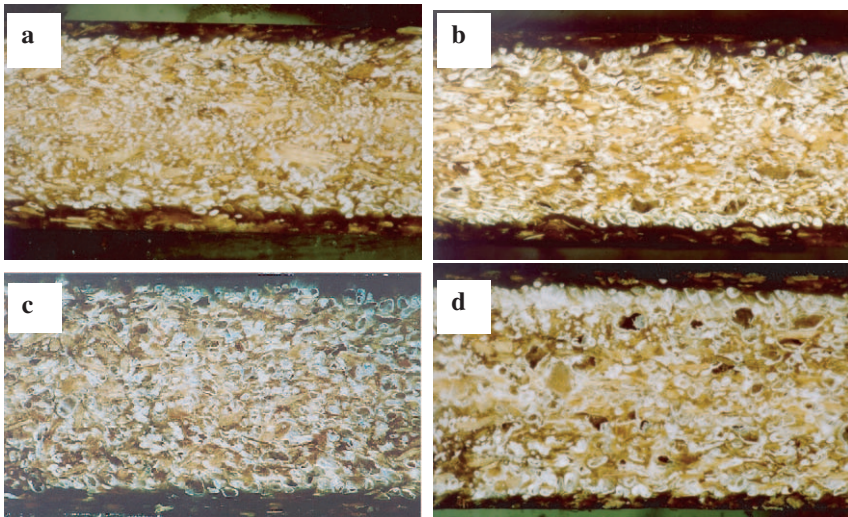


Figure 1. Light micrographs of hard wood fiber-PP microcellular composites (exothermic foaming agent, wood fiber content 30 wt%): (a) 2 wt%; (b) 3 wt%; (c) 4 wt%; and (d) 5 wt%).

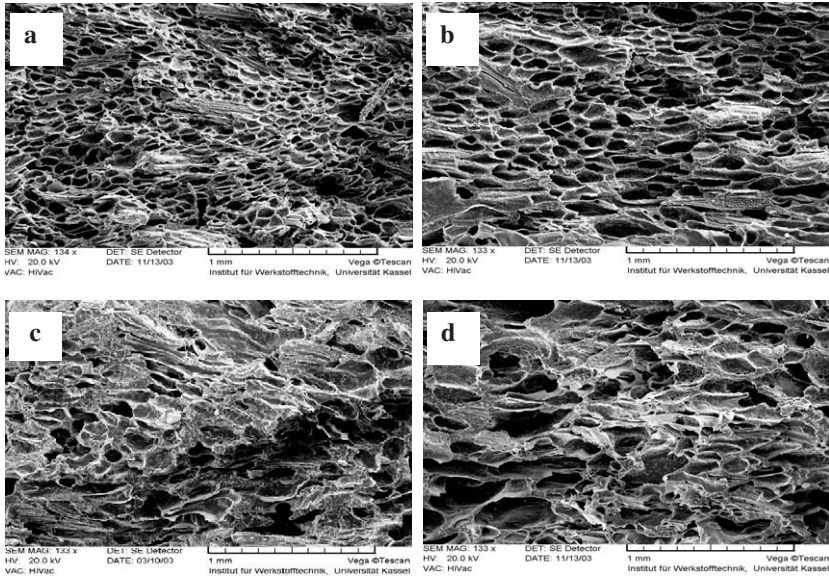


Figure 2. Effect of exothermic foaming agent content on the structure of hard wood fiber–PP microcellular composites (exothermic foaming agent, wood fiber content 30 wt%: (a) 2 wt%; (b) 3 wt%; (c) 4 wt%; and (d) 5 wt%).

increasing CFA content, 2–5 wt%, the cells become bigger in size due to the availability of gas. In this case, it can be said that exothermic CFA 2 wt% content is the optimum concentration to get the best microcellular structure.

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

To observe the effect of wood fiber content, the wood fiber was increased to 50 wt% content in the hard wood fiber–PP microcellular composites containing exothermic foaming agent with a variation of 2, 3, 4, and 5 wt% content (Figure 3). Figure 3 illustrates that 2 wt% creates very small number of cells. With increasing CFA content, it can be seen that 5 wt% loading creates the largest number of cells, and the cells are not larger in size.

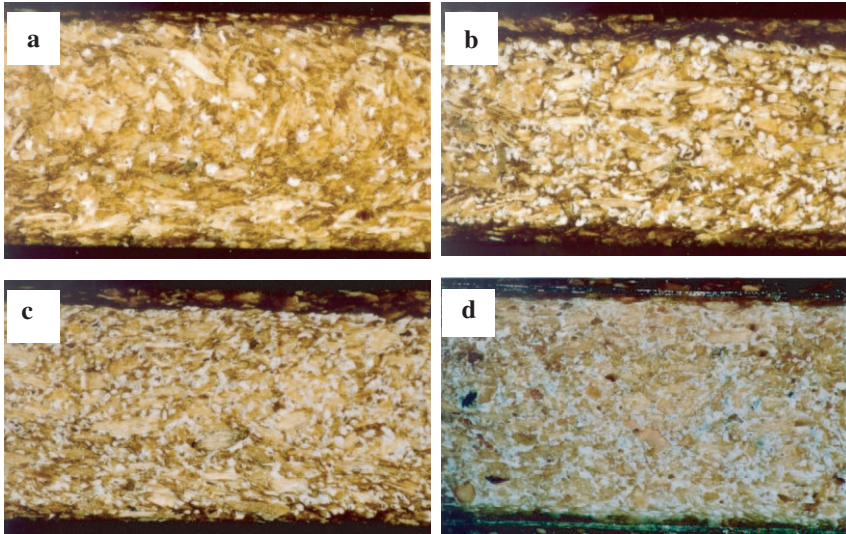


Figure 3. Influence of wood fiber content on the structure of hard wood fiber-PP microcellular composites (exothermic foaming agent, wood fiber content 30 wt%: (a) 2 wt%; (b) 3 wt%; (c) 4 wt%; and (d) 5 wt%).

Density

Figure 4 describes the effect of exothermic CFA content on the density of microcellular composites. Figure 4 illustrates that the density is reduced with increasing exothermic CFA content. It is also seen that, with 30 wt% wood fiber, density is reduced very little with increasing CFA content. In the foaming process, at first the cells nucleate and then they continue to grow as long as there is enough gas for diffusion into the nucleated cells and the polymer can be extended. The end effect of this diffusion of gas is the density of the foamed composites which decrease with increasing CFA content due to cell growth. With further increase in CFA content, cell coalescence and/or collapse may take place when the nucleated cells are fully grown. This phenomenon was described as the gas containment limit [12–15]. But with wood fiber content 50 wt%, density reduced gradually with increasing CFA content. This means that gas containment limit did not take place due to the higher amount of wood fibers. With the addition of a coupling agent (MAH-PP5%), the density was further reduced in all contents, with a maximum density reduction of 30%, and decreased up to 0.739 g/cm^3 at 30 wt% wood fiber content.

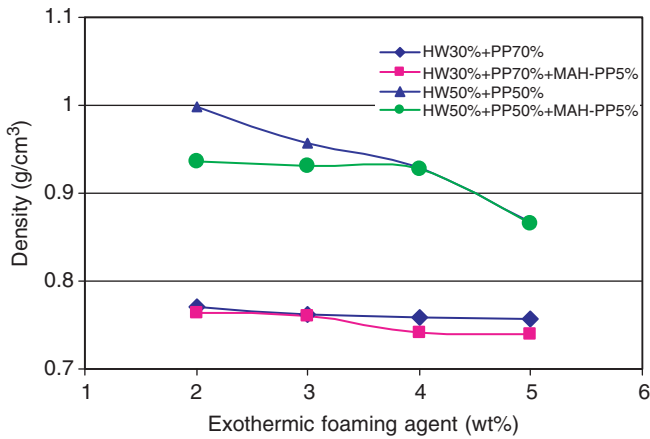


Figure 4. Effect of exothermic CFA content on the density of hard wood fiber-PP microcellular composites.

Mechanical Properties

In order to characterize the influence of CFA content on the hard wood fiber-PP microcellular composites, the tensile, flexural, and impact properties (damping index) were measured. These mechanical characteristics were normalized to the density.

The specific tensile strengths of hard wood fiber-PP microcellular composites with exothermic foaming agent are presented in Figure 5. It was found that specific tensile strength decreased gradually with increasing CFA content and also increasing wood fiber content. With the addition of MAH-PP5%, specific tensile strength increases in all cases, and it was at most 80% at 30 wt% wood fiber content.

Figure 6 describes the specific tensile modulus with the variation of exothermic foaming agent content. With increasing exothermic foaming agent content, specific tensile modulus gradually decreases, and with the increasing wood fiber content it increases. It is also notable that MAH-PP5% significantly influenced specific tensile modulus in the case of 50 wt% wood fiber content, whereas in the case of 30 wt% wood fiber content, the properties increased slightly. There appears to be different density reduction with wood fiber content.

The specific flexural strength for hard wood fiber-PP microcellular composites with the variation of exothermic foaming agent content are summarized in Figure 7. With CFA, specific flexural strength decreases

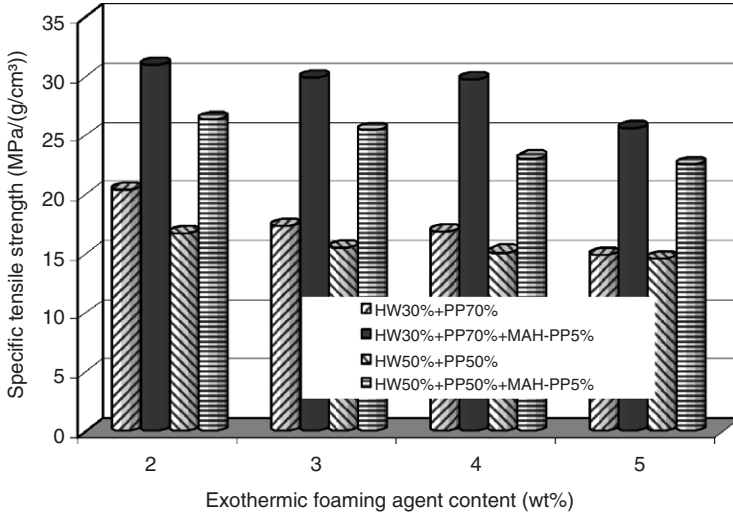


Figure 5. Effect of exothermic CFA content on the specific tensile strength of hard wood fiber-PP microcellular composites.

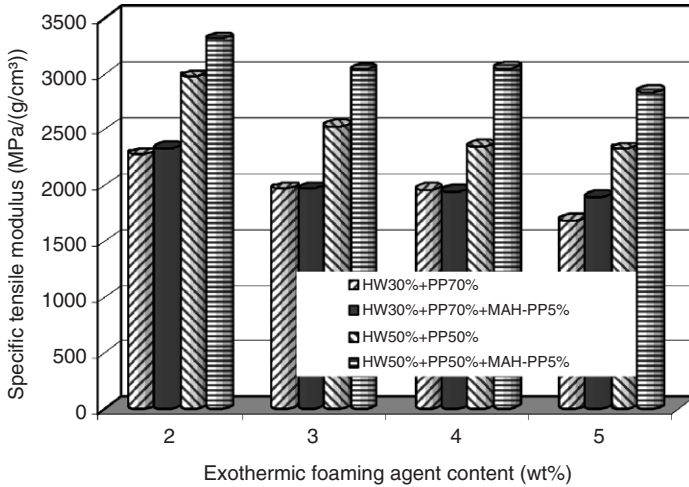


Figure 6. Effect of exothermic CFA content on the specific tensile modulus of hard wood fiber-PP microcellular composites.

proportionately with increasing CFA content and also increasing wood fiber content. As before, MAH-PP5% increases the specific flexural strength in all cases. Specific flexural modulus followed the same trend like specific tensile strength in all cases.

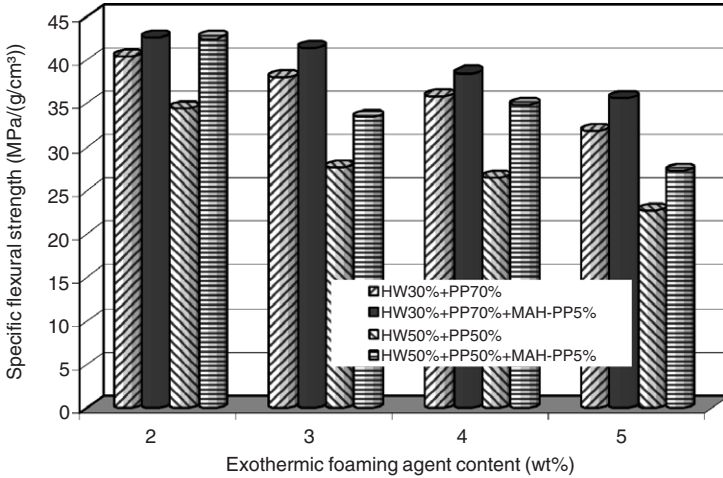


Figure 7. Effect of exothermic CFA content on the specific flexural strength of hard wood fiber-PP microcellular composites.

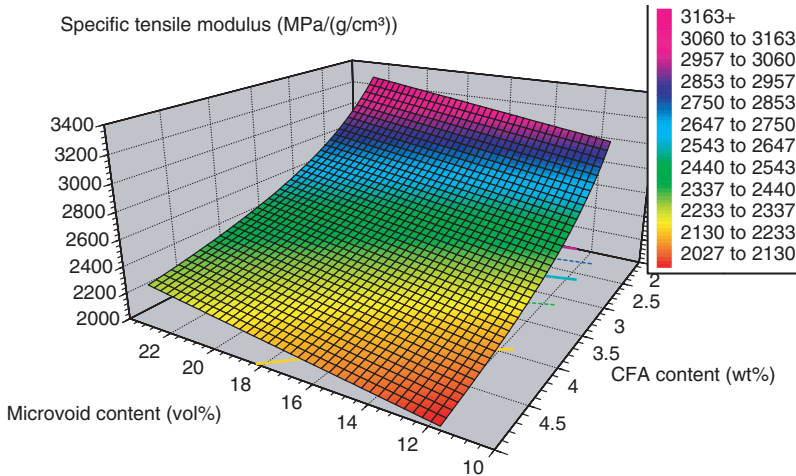


Figure 8. Three-dimensional diagram of specific tensile modulus with the effect of CFA content in injection molding process (exothermic foaming agent, wood fiber content 30 wt%).

The three-dimensional diagram of optimization of mechanical properties of hard wood fiber-PP microcellular composites was determined by the RESINT program [16,17]. The influence of exothermic foaming agent and microvoid content on the specific tensile modulus is presented in Figure 8. With the increasing of foaming agent content, specific

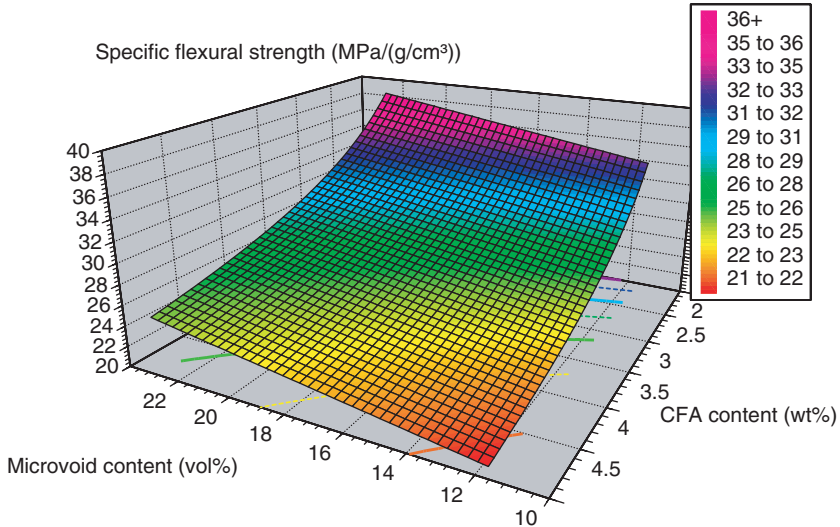


Figure 9. Three-dimensional diagram of specific flexural strength with the effect of CFA content in injection molding process (exothermic foaming agent, wood fiber content 30 wt%).

tensile modulus decreases gradually. But with the increasing of microvoid content at a specific CFA content, the specific tensile modulus increases, which indicate that finer foam structure increases the properties.

The three-dimensional diagram of specific flexural strength with a variation of exothermic foaming agent content and microvoid content for hard wood fiber-PP microcellular composites is presented in Figure 9. With increasing CFA content and microvoid content, specific flexural strength decreases gradually similar to specific tensile modulus.

Damping index of the microcellular hard wood fiber-PP composites with the variation of exothermic CFA content (2–5 wt%) and also non-foamed composites are illustrated in Figure 10. Damping index increased with increasing CFA content. But it is remarkable that at 2 wt% CFA content, the damping index value is very close to that of the non-foamed composites, and after that, the damping index increases very significantly. That means, finer microcellular composites show better damping performance. In the cell morphology part, it was

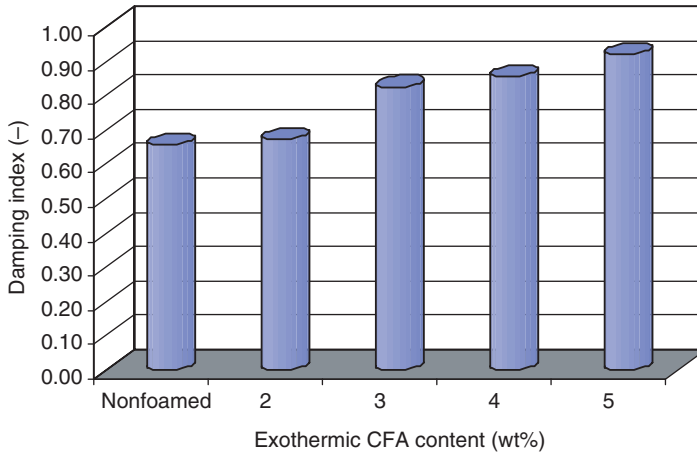


Figure 10. Effect of CFA content on the damping index of hard wood fiber-PP microcellular composites.

discussed that at exothermic CFA 2 wt%, composites showed finer microcellular structures compared to higher CFA content composites.

CONCLUSIONS

Microcellular wood fiber reinforced PP composites were prepared in an injection molding process and their physico-mechanical properties were studied with a variation of exothermic chemical foaming agent (CFA) content from 2 to 5 wt%. The following conclusions can be made:

- (1) The microscopic observations (light and SEM micrographs) showed that foam prepared with exothermic 2 wt% content had finer cellular structure compared to the other contents at the same wood fiber content. It is also notable that an optimum CFA content varied with the wood fiber content.
- (2) Due to the microfoaming, density was reduced at all foaming agent levels, with a maximum density reduction of 30% and a decrease up to 0.739 g/cm^3 at 30 wt% wood fiber content with MAH-PP5%.
- (3) With the addition of MAH-PP, physico-mechanical properties of the microcellular wood fiber-PP composites improved up to 80% at 30 wt% wood fiber content.
- (4) The mechanical properties of the microcellular composites can be optimized with three-dimensional diagrams (RESINT program) in

which a specific physical property is plotted as a function of both CFA and microvoid content. The largest effect on physical property is the variation of CFA.

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