Effect of processing conditions on the microstructure of microcellular PP/WF composites prepared by the continuous extrusion molding technology

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Abstract
The polypropylene (PP) and wood flour (WF) were mixed first and then extruded by a single-screw extruder foaming system to prepare the microcellular wood-polymer composites (WPCs) in this article. In addition to PP and WF, polyolefin elastomer (POE), talc powders and other fillers were also added to improve the cell structures of the samples. And two types of PP resins were introduced to the formulation of the composites to study the effect of the molecular chain structure on the rheological properties of the samples. Besides, the effects of processing conditions, such as die temperature and screw speed, on the properties of the samples were also investigated. The experimental results indicate that compared with PP1, the complex viscosity of PP2 decreased faster with the shear rate because of the linear molecular chain structure. Besides, the maximum impact strength of PP2/WF composites was 138.5 % higher than that of PP1/WF composites, while the tensile and flexural strength of PP2/WF composites were much lower. In addition, the best microcellular structure and the maximum impact strength were also obtained with the die temperature of 190 °C and the screw speed of 2 rpm.

1. Introduction

During the past 20 years, the wood-polymer composites (WPCs) become popular in automobile, packaging, construction and other fields because of the advantages in material costs, stiffness and so on [1, 2]. However, the addition of wood flour (WF) during the preparation of WPCs also led to the decreased ductility and the increased density of the samples [2, 3]. In order to overcome these disadvantages, a uniform microcellular structure was introduced into the products. The previous studies indicated that [2, 4–6] the small bubbles (<100 μm) in the samples could effectively passivate the crack tips and increase the energy used for crack growth during the notched impact test process. Compared with the conventional foamed polymers, polymers with the microcellular structure could achieve the higher impact strength, thermal stability and toughness [2, 3].

Moreover, the studies of microcellular WPCs mainly focus on polyvinyl chloride (PVC), polystyrene (PS) and polyethylene (PE) [1, 7–10]. The first microcellular WPCs were prepared by the batch foaming technology, based on the PVC/WF composites [7]. Compared with PVC, PS and PE, polypropylene (PP) was seldom investigated in the previous studies. On one hand, the melt strength of PP decreases quickly as the temperature exceeds the melt temperature of PP. On the other hand, the degradation temperature of WF is about 200 °C [11], which is very close to the processing temperature of PP. But, considering the lower density, higher toughness and better thermal stability of PP, the PP/WF composites have gradually become the focus of research.

Nowadays, the microcellular foaming technology mainly includes batch foaming [12, 13], injection foaming [10, 14, 15], moulding foaming and extrusion foaming [9, 16]. In this article, the composites were prepared by a single-screw extruder foaming system to prepare the microcellular WPCs. Besides, in order to avoid the effect of the incomplete dispersion of WF into the polymer, a cavity transfer mixer was mounted in the extruder foaming system to improve the distribution uniformity of WF in the foaming process. In addition, polyolefin elastomer (POE) was added before extrusion to alter the shear-thinning behavior of PP/WF composites [17, 18].
In this article, two kinds of PP were introduced to the formulation of the composites and the effect of the molecular chain structure on the rheological properties of materials was investigated. Besides, the effects of processing conditions, such as die temperature and screw speed, on the microstructure and the mechanical strength of the samples were also investigated. At last, the properties of composites prepared with different PP were compared to investigate the relationship between the molecular chain structure and the properties of the samples.

2. Experimental

2.1. Materials

PP1 (B1002W), with the density of 0.9 g cm$^{-3}$ and the melt flow index of 1.0 g/10 min at 230 °C/2.16 kg, was purchased from Sinopec Beijing Yanshan Petrochemical Company, China.

PP2 (B8101), with the density of 0.9 g cm$^{-3}$ and the melt flow index of 0.36 g/10 min at 230 °C/2.16 kg, was purchased from Sinopec Beijing Yanshan Petrochemical Company, China.

POE (DF605), with the density of 0.86 g cm$^{-3}$ and the melt flow index of 0.5 g/10 min at 190 °C/2.16 kg, was purchased from Mitsui Chemicals, Japan.

The poplar wood flour, with the size of 150 μm (100 mesh), was purchased from HC wooden Co., China.

Talc, with the particle size of 23 μm (600 mesh), was purchased from Haicheng Talc Powder Manufacturer, China.

Maleic anhydride grafted polypropylene (MAH-g-PP, OREVAC CA100), with the melt flow index of 10 g/10 min at 190 °C/0.325 kg, was purchased from ARKEMA Co., Ltd, France.

The blowing agent Azodicarbonamide (AC), with the gas production of 220 mL g$^{-1}$ and the decomposition temperature of 160 °C, was purchased from Selon Industrial Co., China.

The blowing activator zinc oxide (ZnO, Z111843) was purchased from Aladdin Industrial Co., America.

2.2. Composites preparation

First, the PP particles were blended with the POE particles at the ratio of 6/4 in a high-speed mixer for 4 min, which was used to improve the viscosity of the composites. Then, the mixtures were melt-extruded by a co-rotating twin-screw extruder with the diameter of 20 mm and the L/D of 40 (Kunshan Kesun, Rubber & Plastic Machinery Co., China). Besides, the pellets of polymer blends with uniform size were prepared by a pelletizer.

Second, the WF, which was dried at 80 °C for 24 h, was blended with the pellets of polymer blends, talc and MAH-g-PP in the mixer for 30 min. The formulations of PP/POE/wood-flour composites are listed in table 1.

Maleic anhydride grafted polypropylene (MAH-g-PP, OREVAC CA100), with the melt flow index of 10 g/10 min at 190 °C/0.325 kg, was purchased from ARKEMA Co., Ltd, France.

The blowing agent Azodicarbonamide (AC), with the gas production of 220 mL g$^{-1}$ and the decomposition temperature of 160 °C, was purchased from Selon Industrial Co., China.

The blowing activator zinc oxide (ZnO, Z111843) was purchased from Aladdin Industrial Co., America.

2.3. Extrusion foaming

As shown in figure 1, the microcellular PP/WF composites were prepared by a single-screw extruder foaming system. The samples with an 80 mm × 8 mm rectangular cross-section were also prepared for the next tests.

Before the extrusion process, the mixtures of the prepared pellets, AC and ZnO with the mass ratio of 100:1.0:0.5 were blended in the mixer for 3 min. Besides, the liquid paraffin was added to increase the adhesion.

| Table 1. The formulation of PP/POE/wood-flour composites.  
| Sample | Weight (%) | PP1 | PP2 | POE | WF | MAH-g-PP | Talc | AC | ZnO |
|--------|------------|-----|-----|-----|----|----------|------|----|-----|
| 1      |            | 42  | 28  | 30  | 3.5| 10       | 1    | 0.5|
| 2      |            | 42  | 28  | 30  | 3.5| 10       | 1    | 0.5|

| Table 2. The process conditions of the extruders.  
| Extruder | Temperature (°C, from hopper to die) | Speed (rpm) |
|----------|---------------------------------------|-------------|
|          | Zone 1 | Zone 2 | Zone 3 | Zone 4 | Zone 5 |            |
| Twin-screw extruder | 130    | 150    | 170    | 185    | 170    | 150         |
| Single-screw extruder | 140    | 160    | 175    | 180    | 180–210 | 1–3         |
between the powders and the pellets, which helped to improve the dispersion of materials. During the extrusion process, the mixtures were processed into a single-phase polymer solution under the strong shear action of screw and the cavity transfer mixer. With the extrusion of screw in barrel, the increased temperature of materials caused the decomposition of the foaming agent and the produced gases dissolved in the single-phase solution. And the rapid pressure drop of the melt in the die reduced the solubility of gases in the solution, which led to the nucleation and growth of bubbles. According to the decomposition temperature of the blowing agent and the melting temperature of the composites, the zone temperatures and other process parameters of the single-extruder are listed in table 2.

2.4. Characterization

2.4.1. Rheological property testing
In this article, the rheological properties of the samples were measured by a parallel-plate rheometer (HAAKE MARS III, Thermo Fisher Scientific Inc., Waltham, MA, USA). The tests were conducted in the dynamic mode between parallel plates (diameter 25 mm, gap 1 mm) at 190 °C in nitrogen atmosphere. Besides, in the process of testing, frequency sweeps between 0.01 and 100 rad s\(^{-1}\) were carried out at strains within the linear viscoelastic range and the scan amplitude was 1%.

2.4.2. Morphological analysis
The microstructures of the samples were observed by a scanning electron microscope (Model S-4700, Japan Hitachi Company, Japan). In order to avoid damaging the surface morphology, each sample was fractured after cooling in liquid nitrogen for about 10 min and sputter-coated with gold before observation. Then the SEM photographs were analyzed by image analysis software (Image Pro Plus 6.0) to obtain the average cell size and cell density of the samples.

The number average diameter of all cells in one micrograph, \(\bar{d}\), was calculated as follows:

\[
\bar{d} = \frac{\sum_{i=1}^{n} d_i}{n}
\]

where \(d_i\) is the diameter of a single cell, and \(n\) is the number of counted cells. At least 100 cells were selected randomly from the SEM graph of each sample to evaluate the average diameter.

Besides, the cell density \(N_0\), defined as the number of cells per unit volume of the polymer, was calculated as follows:

\[
N_0 = \left[ \frac{nM^2}{A} \right]^{1/3} \frac{\rho_u}{\rho_f}
\]

where \(A\) is the area of an SEM graph (cm\(^2\)), \(n\) is the number of cells in the micrograph, \(M\) is the magnification factor, \(\rho_u\) is the density of the unfoamed sample and \(\rho_f\) is the density of the foamed sample.

In addition, the apparent density of the samples was measured by a porosity and density tester (AR-300VP, Dongguan DahoMeter Instrument Co., Ltd, China) with an accuracy of 0.0001 g cm\(^{-3}\), according to ASTM D792.
2.4.3. Mechanical property testing
The tensile strength and flexural strength of the samples were tested by an universal tester with a load cell of 5 kN (KXWW, Chengde Taiding Testing Machine Manufacturing Co., Ltd, China). Besides, the tensile strength of the samples was measured with a draw speed of 10 mm min\(^{-1}\), according to ISO 8256-2005. The three-point flexural tests were conducted with a cross-head speed of 10 mm min\(^{-1}\), according to ISO 178-2010.

Moreover, the notched impact strength of samples was tested by an impact tester (KBANM-II, Chengde Taiding Testing Machine Manufacturing Co., Ltd, China). A side-edge notch with a depth of 2 mm was machined on each specimen before testing, according to ISO 8256-2005. In this article, at least 5 samples were tested for each data point.

2.4.4. Fourier transform infrared analysis
The Fourier transform infrared (FTIR) spectra of the samples were recorded by a FTIR spectrometer (NEXUS 670, NECO INDUSTRIES INC., America) in the range of 400–4000 cm\(^{-1}\) wave numbers. All spectra were averaged over 32 scans with a resolution of 4 cm\(^{-1}\).

3. Results and discussions
3.1. Rheological properties of PP and the composites
As shown in figure 2, blending with POE and other fillers could effectively improve the rheological properties of materials, such as the storage modulus (\(G’\)), loss modulus (\(G''\)), complex viscosity (\(\eta\)) and loss tangent (\(\tan\delta\)). According to the previous studies, the increased rheological properties could be contributed to the following reasons. On one hand, with the addition of POE, numerous branched chains of POE could entangle with PP molecular chains and form physical interlocks, which resulted in the increase of the oscillating shear modulus and complex viscosity of the composites [18]. On the other hand, the addition of MAH-g-PP could form the...
ester bonds and physical interlocks with PP and WF respectively, which was demonstrated by the new peak at 1714 cm⁻¹ in figure 3 and helped to improve the adhesion between resin and WF.

According to the data in figure 2, with the increase of the shear rate, the curves of PP2 and PP2/WF composites in figures 2(a) and (b) increased slower than that of PP1 and PP1/WF composites. Besides, as the shear rate increased, the complex viscosity of PP2 and PP2/WF composites also decreased faster than PP1 and PP1/WF composites. On one hand, PP1 was a kind of high-melt-strength polypropylene with long branched chain structure, while PP2 was a kind of random copolymer polypropylene with linear molecular chain structure [19]. On the other hand, the polydispersity of PP1 was 10 and higher than that of PP2, which was judged by the intersection position of the curves of the storage modulus ($G'$) and loss modulus ($G''$) [20]. As a result, the molecular structure led to the strain hardening behavior of PP1 during the shear process, which made the viscosity and the loss tangent of PP1 and PP1/WF composites less affected by the shear rate.

3.2. Effect of the die temperature on the properties of composites

3.2.1. Cell morphology

In order to investigate the effect of the die temperature on the cell structure of composites, the samples prepared with different temperatures were observed and the photographs taken by the scanning electron microscope were shown in figure 4. Besides, the average cell size and cell density of each sample were calculated and listed in figure 5. In addition, the density of the foaming samples prepared with different die temperature were listed in figure 6.

According to the data in figure 5, the cell size of both PP1/WF composites and PP2/WF composites increased with the die temperature, while the cell density decreased with the temperature. On one hand, the increased die temperature could reduce the solubility of gas prepared by the blowing agent in the melt, which increased the amount of gas used for bubble nucleation and bubble growth. On the other hand, the increased die temperature could also enhance the thermal motion of the molecular chains and reduce the melt strength of the composites, which resulted in the merging of the adjacent bubbles to form larger ones. In addition, a part of WF would degrade as the temperature increased, which could further weaken the melt strength of the composites and destroy the microporous structure of the samples [18]. Affected by the microstructure, the density of PP2/WF composites also decreased with die temperature, which was shown in figure 6. Compared with PP2/WF composites, the density of PP1/WF composites changed little with the increase of die temperature. Besides, the data listed in figure 5 also indicated that the cell size of PP1/WF composites was usually smaller than that of PP2/WF composites at the same die temperature, and the density of PP1/WF composites was also slightly higher than that of PP2/WF composites. According to the curves of the composites’ complex viscosity in figure 2(c), the viscosity of PP2/WF composites decreased faster than that of PP1/WF composites. Considering the fact that the shear rate of the screw was larger than the rheometer, the melt strength of PP2/WF composites was lower than that of PP1/WF composites in the extrusion foaming process, which resulted in the larger cell size and the smaller cell density of PP2/WF composites.
Figure 4. Cell morphology and cell diameter distribution of the foaming samples with different die temperature: (a) 180 °C-PP1/WF composites, (b) 190 °C-PP1/WF composites, (c) 200 °C-PP1/WF composites, (d) 210 °C-PP1/WF composites, (e) 190 °C-PP2/WF composites, (f) 200 °C-PP2/WF composites, (g) 210 °C-PP2/WF composites.
3.2.2. Mechanical properties

Affected by the microstructure, the mechanical strength of the samples also changed with the die temperature, which was listed in figure 7 respectively. According to the data in figure 7, with the raise of the die temperature, the impact strength, tensile strength and flexural strength of PP1/WF composites increased by 7.2 %, 18.6 % and 1.1 % respectively.

However, as the die temperature exceeded 190 °C, the mechanical strength of both PP1/WF composites and PP2/WF composites all decreased with the raise of the die temperature. On one hand, the increased temperature resulted the larger cell size and lower cell density, which reduced the effective forced area of the samples and the ability to passivate the crack tip. And the effective forced area was the area on the cross section of the sample with removal of all bubbles, where actually bore the load during the test process. On the other hand, the increased temperature could result in the degradation of WF, which also accounted for the decrease of the samples’ strength.

Besides, compared with PP1/WF composites, the impact strength of PP2/WF composites was much higher, while the tensile and flexural strength were relatively lower. The maximum impact strength of PP2/WF composites was 15.5 kJ m\(^{-2}\), which was 138.5 % higher than that of PP1/WF composites. But the tensile and flexural strength of PP2/WF composites were 7.0 MPa and 15.6 MPa, which were 16.7 % and 15.7 % lower than that of PP1/WF composites. Compared with PP1, PP2 was a random ethylene-propylene copolymer and the ethylene sequences in the molecular chain structure could reduce the crystallization rate and the crystallinity of PP2 [19], which caused the increased ductility and the decreased tensile strength of PP2.
3.3. Effect of the screw speed on the properties of composites

3.3.1. Cell morphology

In the extrusion process, the cell structures were affected not only by the die temperature, but also by the melt pressure. But compared with the die temperature, the melt pressure in the die was hard to adjust directly. Therefore, the screw speed was changed to adjust the melt pressure indirectly. As the screw speed increased, the melt conveying efficiency was also improved, which caused the more melts accumulated and compressed reciprocally in the die \[21-23\]. As a result, the melt pressure in the die essentially increased with the screw speed.

The pressure sensor indicated that as the screw speed increased from 1 rpm to 3 rpm, the melt pressure also increased from 0.75 MPa to 2.0 MPa. The electron microscope photographs of the samples prepared with different screw speed were listed in figure 8. Besides, the average cell size and cell density of each sample were calculated and listed in figure 9. In addition, the density of the foaming samples prepared with different screw speed were listed in figure 10.

According to the data in figure 9, with the screw speed increased from 1 rpm to 2 rpm, the cell size of PP1/WF composites decreased from 113.7 μm to 87.6 μm, and the cell size of PP2/WF composites also decreased from 104.6 μm to 81.5 μm. But as the screw speed increased from 2 rpm to 3 rpm, the cell size of PP1/WF composites and PP2/WF composites increased by 4.9% and 8.2% respectively. Similar with the cell size, the maximum cell densities of both PP1/WF composites and PP2/WF composites were obtained at the speed of 2 rpm. On one hand, the high melt pressure caused by the increased screw speed provided a higher ambient pressure \[24\]. To overcome the ambient pressure and the interfacial energy, a higher driving force of bubble nucleation was needed, which led to the decrease of the cell density. But the melt pressure obtained at the screw speed of 1 rpm was not sufficient to prevent the merging of the neighbouring bubbles, which caused the higher cell size and lower cell density. On the other hand, the higher melt pressure could increase the solubility of gas in the melt, which reduced the amount of gas used for bubble nucleation and bubble growth \[25\]. Besides, affected by the microstructure, the density of the foaming samples for both PP1/WF composites and PP2/WF composites all increased with the screw speed, which was shown in figure 10.
3.3.2. Mechanical properties

The increase of the melt pressure affected not the cell structure but also the mechanical strength of the samples. The impact strength, tensile strength and flexural strength of the samples prepared with different screw speeds were listed in figure 11. According to the data in figure 11, with the increase of the screw speed, the impact strength of both PP1/WF composites and PP2/WF composites increased first and then decreased and the maximum value was obtained at the speed of 2 rpm, which were 16.1 % and 14.8 % higher than that of 1 rpm respectively. The previous studies indicated that [4–6] during the notched impact test process, the bubbles in the samples with the microcellular structure could effectively passivate the crack tips and increase the energy used for crack growth, which contributed to suppress the growth of the cracks and improve the ductility of the samples. And the best microporous structure was obtained at the speed of 2 rpm, which resulted in the maximum impact strength of the composites.

![Figure 8. Cell morphology and cell diameter distribution of the foaming samples with different die temperature: (a) 1 rpm-PP1/WF composites, (b) 2 rpm-PP1/WF composites, (c) 3 rpm-PP1/WF composites, (d) 1 rpm-PP2/WF composites, (e) 2 rpm-PP2/WF composites, (f) 3 rpm-PP2/WF composites.](image)
However, compared with the impact strength, the tensile strength and flexural strength of both PP1/WF composites and PP2/WF composites all increased with the screw speed. As the screw speed increased to 3 rpm, the tensile and flexural strength of PP1/WF composites increased by 35.7% and 43.1% respectively. Besides, the tensile and flexural strength of PP2/WF composites increased by 41.4% and 47.3% respectively. As mentioned above, the increased pressure could not only reduce the amount of gas but also increase the difficulty of bubble nucleation, which both decreased the number of bubbles in the samples and increased the effective forced area of the samples.

4. Conclusions

In this article, the rheological properties of two types of PP and its composites blending with WF were investigated. Besides, the effects of die temperature and screw speed on the cell structures and mechanical strength of the samples were also investigated. Some conclusions were summarized as follows:

(1) Compared with pure PP, the addition of WF, POE and other fillers could effectively improve the melt strength of the composites. But due to the different structures of the molecular chains, the complex viscosity of PP2/WF composites decreased faster than PP1/WF composites.

(2) The increased die temperature could not only increase the amount of gas used for bubble nucleation and growth, but also enhance the thermal motion of the molecular chains, which caused great damage to the
microporous structure of the samples. Besides, the increased temperature also led to the degradation of WF, which resulted in the decrease of the composites’ mechanical strength.

(3) The increased screw speed contributed to increase the melt pressure in the die. But the low pressure was not sufficient to prevent the mergence of bubbles, while the high pressure could increase the ambient pressure and the interfacial energy for bubble nucleation. As a result, the best microporous structure of the samples were obtained at the speed of 2 rpm.

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Figure 11. Mechanical strength of the samples with different die temperature: (a) notched impact strength, (b) tensile strength, (c) flexural strength.
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