

Critical Processing Temperature in the Manufacture of Fine-Celled Plastic/Wood-Fiber Composite Foams

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ABSTRACT: The main benefits of incorporating wood fibers (WF) in plastics are the increased stiffness and lowered cost of the resulting composites. However, these improvements are usually accompanied by a reduction in the ductility and impact resistance. These shortcomings can be removed by effectively foaming and incorporating a fine-celled structure in these composites. The volatiles released from WF during processing are known to deteriorate the cell structure. The maximum processing temperature, which affects the amount of volatiles released by the WF during extrusion of fine-celled plastic/WF composite, affects the cell morphology. This study was undertaken to identify the critical temperature above which the cellular structure of WF composite foams is significantly deteriorated. To clearly

identify the effects of the volatiles generated from WF on the cellular morphology, neither a chemical blowing agent nor a physical blowing agent was used in the foam processing. The experimental results show that regardless of the drying method, the highest processing temperature of plastic/WF composites should be minimized, preferably below 170°C, to avoid the adverse effects of the volatiles generated from the WF during processing. A method of estimating the emissions from WF during extrusion processing by using the TGA data is also proposed. © 2003 Wiley Periodicals, Inc. *J Appl Polym Sci* 91: 621–629, 2004

Key words: wood-fiber; composites; processing; extrusion; TGA

INTRODUCTION

Plastic composites using wood-fiber (WF) fillers have been gaining ever-increasing acceptance because of their superior advantages such as lower cost, improved stiffness, and better processibility compared to other fillers. These composites are increasingly competing with wood products and the large number of patents about them^{1–12} indicates the speed of commercialization of plastic/WF composite processing. However, the number of potential applications has been limited because of their low impact strength and high density compared to natural wood and certain plastics.^{13–16}

Incorporating a fine-celled structure into plastic/WF composites can significantly improve their impact strength and reduce their weight and cost.¹⁷ Because extrusion is one of the most cost-effective processes for production of plastics, the commercial utility of these composites would be further elevated, if these could be manufactured by using a continuous extrusion-based technology. The inherent moisture, present in the wood fiber, is released during the heating (plasticating) and subsequent stages of extrusion and is retained in the melt in a gaseous or liquid state until the time when the extrudate comes out of the die.

This leads to gross deterioration in the cell structure and surface quality of the extrudate.^{18,19} Consequently, a nonuniform cell distribution and a large average cell size usually characterize the obtained foams.

Dambauld²⁰ describes an effort of foaming polyolefin elastomer blends by using water as a blowing agent. He obtained density reductions of 10 to 70% while using 0.1 to 10 wt % of water. However, attempts to foam pure PE or PP with water were unsuccessful. Sahnoune²¹ and Snyder²² describe the use of water as a blowing agent for foaming TPEs. Matuana and Mengeloglou²³ used moisture as a foaming agent in the manufacture of rigid poly(vinyl chloride) (PVC)/wood-flour composite foams. Others^{6,8,9,11,12} talk about the importance of removing moisture for producing nonfoamed plastic/wood-fiber composite material in general terms. In general, there is a paucity of literature discussing the effects of moisture on the foaming of plastic/wood-fiber composite materials.

To ensure fine-celled morphology, the volatile contents of WF need to be reduced to a bare minimum, using any of the standard drying techniques, such as online devolatilization,^{18,24} oven drying, hot air convective drying, drying in K-mixer,²⁵ and the like. However, even the dried WF releases additional volatiles when further heated to the processing temperatures of the plastics, which is typically around 200°C. This phenomenon has been clearly demonstrated by thermogravimetric analysis (TGA) studies^{24,26} already

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published. These additional volatile emissions should be considered when fine-celled foaming of plastic/WF is undertaken.

As mentioned above, the fundamental problem in removing the volatiles from WF is that even the dried WF can gradually release volatiles, as long as it is being heated, depending upon its exposure time and temperature.^{24,26} When the quantity of these emissions from WF during extrusion foam processing is large, gross distortions may occur in the cell structure and it is extremely difficult to properly control the density of the extrudate.^{18,19} However, when the amount of these emissions is very low, the cellular structure does not seem to be affected severely.¹⁸

It is very difficult to remove all volatiles without causing degradation of WF. As the highest barrel temperature primarily governs the emissions from WF, this study was undertaken to determine this critical processing temperature, above which the volatile emissions prevent the formation of a uniformly distributed fine-celled structure. Different drying conditions were tried to minimize the volatile emissions from WF, either by changing the devolatilizing vent temperature or by varying the oven-drying conditions, to improve the cell morphology of plastic/WF composite foams. To clearly identify the effects of volatile emissions, no blowing agent, chemical or physical, was used in the processing, and thereby, the resultant foam structure was caused mainly by the additional emissions of WF.

Quantifying the acceptable amount of volatile that can be used by itself or tolerated in conjunction with another blowing agent for the formation of fine-celled foam structures is an extremely difficult task, because of the unknown thermal history of WF in the barrel. A TGA study of the weight loss from WF upon heating was carried out to estimate the amount of emissions from WF during the extrusion process.

FOAMING MECHANISMS IN PLASTIC/WOOD-FIBER COMPOSITES

The basic mechanisms involved in foaming of thermoplastics are that a blowing agent is dissolved or finely dispersed in the polymer, after which a large number of bubble nuclei are generated such that they are uniformly dispersed in the polymer matrix. This is followed by bubble growth due to the diffusion of the dissolved or evolved gas into these nuclei and finally by the stabilization of the grown bubbles.²⁷

In the extrusion foaming of plastic/WF composites with no blowing agent, the moisture and other volatiles released from WF during processing would cause foaming. Moisture is almost insoluble in plastic, and thus, when it is involved in foaming, it fails to dissolve in the plastic matrix and remains as a second phase in the composite. However, some volatiles given out by

WF are soluble in plastic, and therefore, the moisture-induced foaming mechanism would be quite different from that of a soluble gas-induced foaming. When a foam structure is developed with the moisture alone at the die exit, the volume expansion, attributed to moisture, occurs promptly because the second-phased moisture evaporates immediately at a lowered pressure. This immediate expansion at the die exit most likely would not significantly contribute to the final expansion ratio because the temperature of the rapidly expanded thin cell walls is still high, and therefore, through the hot thin walls, the vapor would diffuse and escape out to the air quickly.²⁸ The thin cell walls may be easily ruptured because of the WF, which can accelerate gas escape.²⁶ On the other hand, when the soluble gases, released from WF, are involved in foaming, they would play the same role as a regular blowing agent in the foaming process. They will first dissolve in the polymer matrix during extrusion and the dissolved gases will slowly diffuse into the nucleated cells at the die exit, which can cause them to grow and give rise to a cellular structure of a low density.

However, when both the insoluble moisture and the soluble gases are used in foaming, the undissolved moisture may cause the loss of even soluble gases. If the amount of moisture is large, the cell walls may possibly be easily ruptured, particularly because of the presence of WF in the thinned walls, by the rapidly expanding vapors upon exiting from the die as described above. This will cause the loss of the dissolved gases, resulting in very little or no reduction of density. Even if the cell walls are not ruptured, in the case of somewhat lower amounts of moisture, the gases, along with the vapors, can easily diffuse through these high-temperature thin walls, again resulting in a higher density.²⁸ For still lower amounts of moisture, gas loss may be reduced, but because of its presence in the undissolved state within the polymer/WF matrix, it again expands rapidly upon exiting the die so that the dissolved gases tend to diffuse into these large cavities. Even the gas from the smaller neighboring bubbles tends to diffuse into these large cells, causing them to grow even further and this results in a non-uniform cell structure with large cavities. Therefore, minimizing the nonsoluble moisture content will be desirable for production of fine-celled foams in WF composite processing. However, because purging of only moisture from the WF would be impossible, purging of all the volatiles would be required.

EXPERIMENTAL

Materials

High-density polyethylene (HDPE 2710) supplied by Nova Chemicals (Calgary, Alberta, Canada) was utilized in the experiments. The 50 wt % WF used was

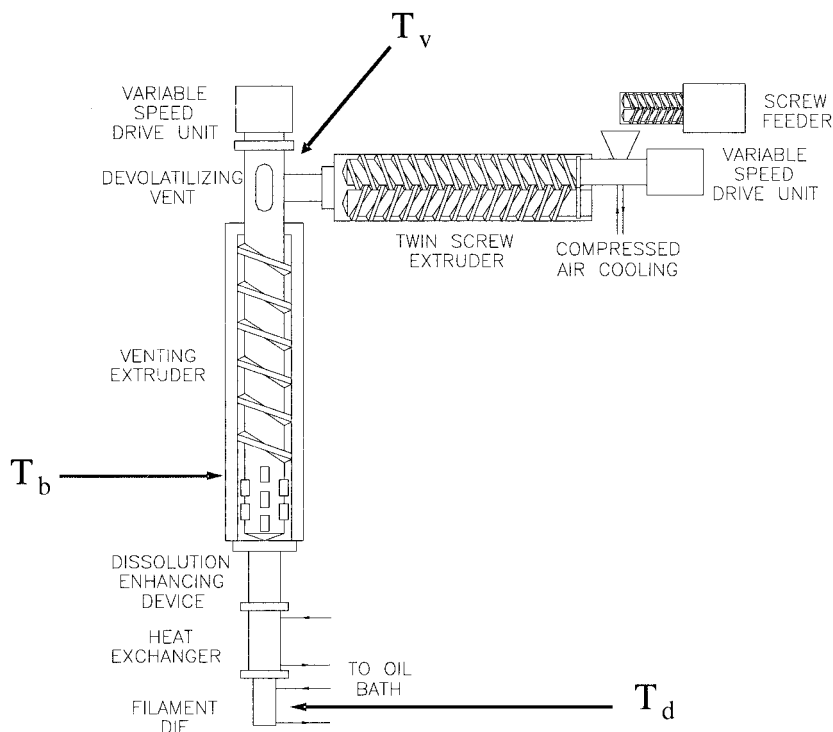


Figure 1 Schematic of tandem extrusion system.

standard softwood (pine) grade 12020, supplied by American Wood Fibers (Columbia, MD). Coupling agent (CA; 3 wt %), Fusabond adhesive resin E-MB-100D from DuPont Canada (Mississauga, Ontario, Canada), was used for improving the adhesion between the hydrophobic PE and the hydrophilic WF. All the materials were used as received.

Experimental setup and procedure

A schematic of the tandem extrusion system^{18,19} used for this study is shown in Figure 1. In the preparatory stage, WF was mixed and dry-blended with HDPE 2710 pellets, and the CA. For the predried WF experiments, the WF was dried in a vacuum oven at 105°C for 12 h and blended as described above, after they had cooled down to room temperature. The blended mixture was placed into the twin-screw feeder and fed into the counterrotating twin-screw extruder. The mixture was plasticated and uniformly mixed through the intensive counterrotation of the twin screws. The temperature in this extruder was maintained below 150°C throughout the extruder to prevent the degradation of WF, except at the vent where the minimum temperature was kept at 160°C to ensure smooth flow through the opening. The output of this extruder was fed into the second extruder. At the interconnection, which was open to atmosphere, all the vaporized water and other volatiles were purged out and the mois-

ture/volatile-free mixture was conveyed into the second extruder by the motion of the screw.

The temperatures in the second tandem extruder were maintained at low levels in zones 1 and 2. Additional volatiles were evolved from WF by elevating the mixture temperature in zone 3 and the released volatiles were dissolved/dispersed in the polymer matrix under the action of high shear at elevated temperature and pressure. The temperature of the melt was then lowered in a heat exchanger at the extruder exit. Finally, it was extruded out through the die where the surface temperature was further reduced to increase the stabilization of the foaming process.

The following procedures were adopted to produce HDPE/WF composite foams.

Experiments with varying barrel temperature

A set of experiments of HDPE mixed with WF and 3% coupling agent, without a blowing agent, were carried out to identify the relationship between processing temperatures and the cell structure, while fixing the drying (i.e., devolatilizing) conditions. This was done to find the lowest processing temperature at which the volatile generation from WF is minimized. In this set of experiments, three different devolatilizing temperatures at the vent were used and for each of these, the barrel temperature in the second extruder was varied

to identify its effect on the cell structure. The die temperature was maintained at 140°C.

Experiments with varying die temperature

Another set of experiments of HDPE mixed with WF and 3% coupling agent, without a blowing agent, were carried out to identify the relationship between die temperatures and the foam density at three different barrel temperatures, while fixing the drying (i.e., devolatilization) condition at 170°C. This was done to find the lowest density achievable when the volatiles generated from WF were utilized for foaming, while the amount of volatiles was minimal to produce a fine-celled structure, by maintaining the second extruder barrel temperature below 170°C. An experiment was also conducted at high second-extruder-barrel-temperature above 170°C for comparison purposes.

Sample preparation and characterization

The foam samples were randomly collected at each barrel and die temperature when a stabilized processing condition, in terms of the pressure and melt flow rate, was obtained. This took about 10–20 min after a new temperature profile was set. The samples were characterized for foam density and cell morphology. The foam density (ρ_f) was calculated as

$$\rho_f = \frac{4M}{\pi d^2 l} \quad (1)$$

where M is the mass of foam sample, g; d is the average diameter of foam sample, cm; and l is the length of foam sample, cm.

For cellular morphology, each sample was first dipped in liquid nitrogen and then fractured. The fractured surface was then gold-coated, using a sputter coater (E 50000C PS3), and the microstructure was examined by using a scanning electron microscope (SEM, Hitachi 510).

Thermogravimetric analysis of wood fibers

TGA was conducted to study thermal devolatilization of moisture contained in wood fibers, using TGA model SDT 2060 from TA Instruments. The wood fibers were initially heated isothermally for 4 min at 75°C, to achieve similar drying states for all samples, and then the temperature was suddenly raised to the desired isothermal conditions for the remaining duration of the experiment (the time required to reach from 75 to 100°C was about 1 min, and from 75 to 225°C was about 1.25 min). A typical amount of material used for the TGA experiments was 10 mg. The sample fibers

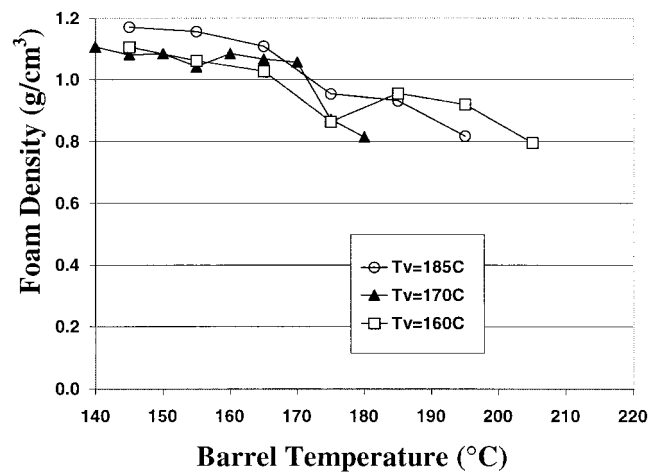


Figure 2 Foam density of HDPE/WF composites at different barrel temperatures. $T_d = 140^\circ\text{C}$.

were not oven-dried. The oven was continuously purged with nitrogen during the experiment to remove the released volatiles and maintain dry conditions. The purging also prevented the degradation of wood fibers.

RESULTS AND DISCUSSION

Effect of the barrel temperature

Figure 2 shows the effect of the barrel temperature on the foam density of WF composites for various devolatilization temperatures while the die temperature was fixed at 140°C. Overall, the foam density decreased as the barrel temperature in the second extruder increased. When the barrel temperature was in the range of 145°C–170°C, the foam density was around 1.1 g/cm³. Above 170°C, the foam density decreased quickly to 0.85 g/cm³. This implies that plenty of volatiles were released from WF above 170°C and resulted in significant foaming.

At the same devolatilizing temperature of 160°C, when the barrel temperature increased from 145, 165, to 175°C, the corresponding foam density was 1.1, 1.0, and 0.87 g/cm³, respectively. Figure 3 shows the typical cell morphologies of these HDPE/WF composite foam samples. In the composite produced with the barrel temperature of 145°C, there was almost no foaming. As the barrel temperature was increased to 165°C, some small-sized bubbles and voids appeared. When the barrel temperature was further increased to 175°C, it was obvious that more volatiles were released from WF, resulting in significant foaming with a clearly visible irregular cellular structure.

Effect of the die temperature

Figure 4 shows the effect of die temperature on the foam density at various barrel temperatures while the



Figure 3 Effects of barrel temperature on the cell morphology of HDPE/WF foams. $T_v = 160^\circ\text{C}$, $T_d = 140^\circ\text{C}$. (a) $T_b = 145^\circ\text{C}$; (b) $T_b = 165^\circ\text{C}$; (c) $T_b = 175^\circ\text{C}$.

devolatilization temperature was fixed at 170°C . At a low die temperature, the loss of the volatiles would be reduced and most generated volatiles would contribute to the density reduction.²⁹ For example, when the barrel temperature was 175°C , the amount of volatiles generated was large, and therefore, the foam density decreased significantly (to 0.3 g/cm^3) as the die temperature decreased. However, because of the excessively released volatiles, irregular-shaped, big-sized cells were observed and the foam structure was very nonuniform. At a lower barrel temperature of 160°C , the foam density could be lowered to about 0.75 g/cm^3 , by decreasing the die temperature below 140°C , which indicates that the emissions from the WF were suppressed to a great extent. The foam structure was very fine because of the small amount of volatiles generated at this low barrel temperature. For a barrel temperature of 145°C , no foaming could be observed. In fact, the final density was always more than the density of the individual compo-

nents, indicating that the high extrusion pressures compacted the composite because of the compaction of WF.^{30,31} This also indicates that the lower processing temperature successfully suppressed the volatile emission from the WF which has been dried (i.e., devolatilized) at a higher temperature.

From the foregoing results, it can be concluded that when the barrel temperature was very high, the amount of generated volatiles was significant, and thereby, the cell structure was deteriorated. Therefore, to minimize the effects of the volatiles, especially the moisture, on foaming, the maximum processing temperature in the second extruder should be lower than 175°C , preferably below 170°C .

Effect of the devolatilizing temperature

Figure 2 also shows that the foam density of composites generally increased as the devolatilizing temperature was increased. This indicates that more moisture and other volatiles were removed when the devolatilizing temperature was higher, and that the devolatilizing vent was effective in removing the volatiles generated from WF. Another interesting observation from this figure is that the foam density decreased only slightly as the barrel temperature increased while the barrel temperature was lower than the devolatilizing temperature ($T_b < T_v$). However, when the barrel temperature was greater than the devolatilizing temperature ($T_b > T_v$), the foam density decreased sharply as the barrel temperature increased. This shows that WF further released the volatiles even after the devolatilization at the vent, when the barrel temperature was higher than the drying (i.e., devolatilizing) temperature.

Figure 5 shows the cell morphology of foams obtained at different devolatilizing temperatures of 160 , 170 , and 185°C for the same barrel temperature $T_b = 165^\circ\text{C}$. As the vent temperature increased, the foam-

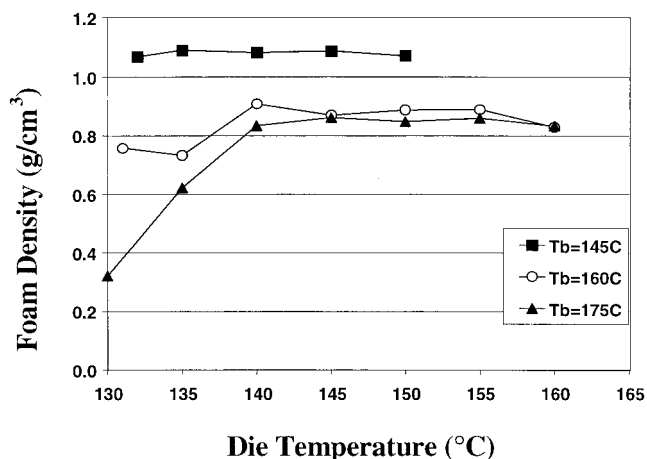


Figure 4 Foam density of HDPE/WF at various die temperatures. $T_v = 170^\circ\text{C}$.

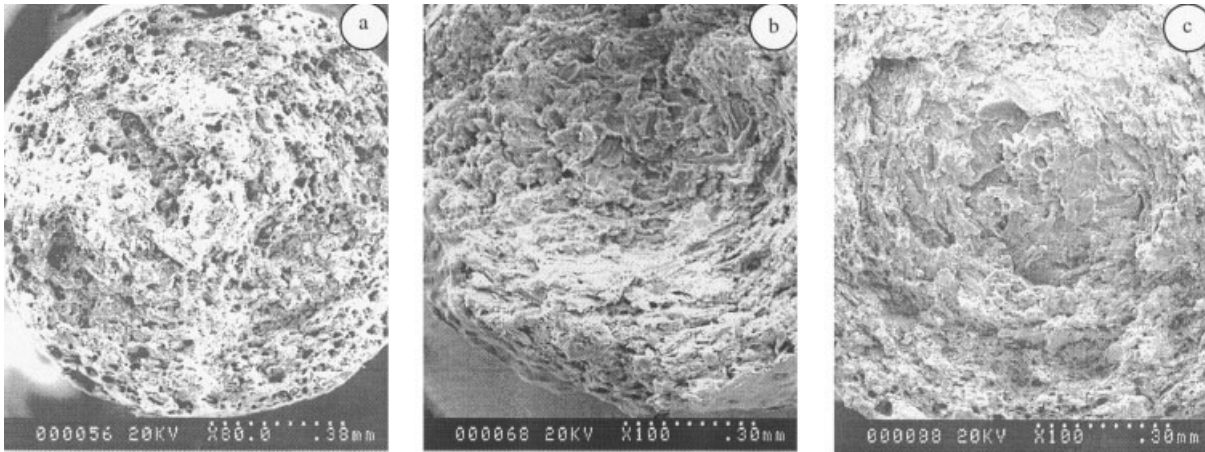


Figure 5 Effects of devolatilizing temperature on the cell morphology of HDPE/WF composites. $T_b = 165^\circ\text{C}$. (a) $T_v = 160^\circ\text{C}$; (b) $T_v = 170^\circ\text{C}$; (c) $T_v = 185^\circ\text{C}$.

ing effect of volatiles became less obvious. This confirms that the devolatilizing temperature (i.e., the drying temperature) should be higher than the maximum processing temperature to minimize the effects of emissions from WF.

This finding has special significance to the use of blowing agents for foaming of plastic/WF composites in a tandem extrusion system. When a chemical blowing agent (CBA) is used, the vent temperature should not be higher than the decomposition temperature of the CBA. The CBA has to be decomposed by raising the processing temperature in the second extruder, which implies that significant moisture and other volatiles will always be generated during this process. So instead of devolatilization, predrying of WF at a

temperature higher than the decomposition temperature of CBA may have to be used in CBA processing. Also, we can hypothesize that the use of a physical blowing agent (PBA) may be more suitable for generating a fine-celled structure in plastic/WF composites, as the melt temperature does not have to be increased in the second extruder, such as in CBA processing, and thereby, reducing the adverse effects of the additional volatile emissions generated from WF.

Weight loss behavior of WF and estimation of the amount of volatiles used in foaming

Figure 6 shows the TGA results of the devolatilization of WF to various isothermal conditions. As the WF

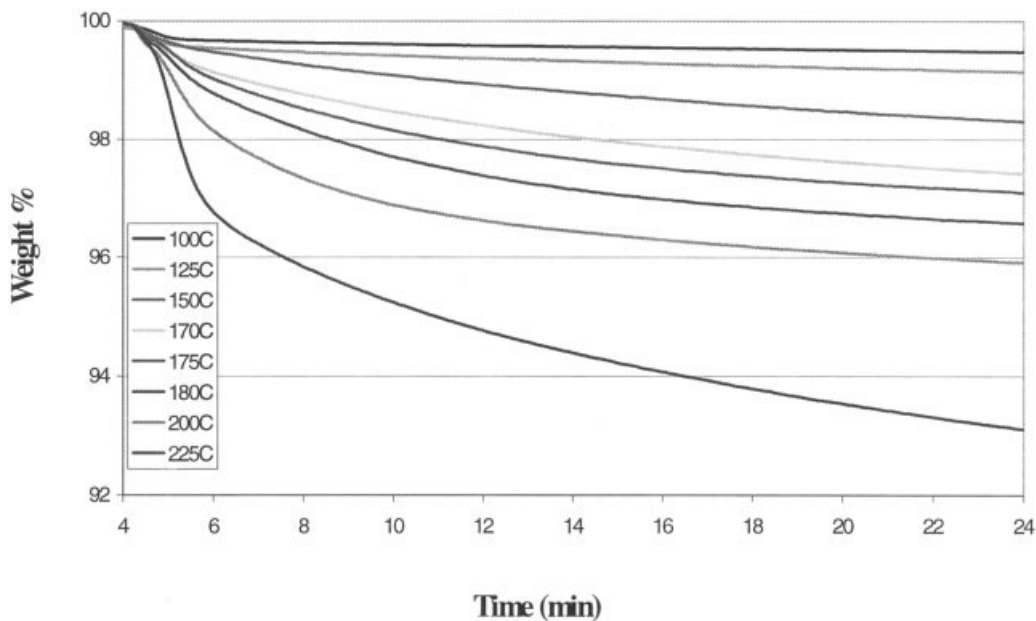


Figure 6 TGA thermograms of WF at isothermal temperatures.

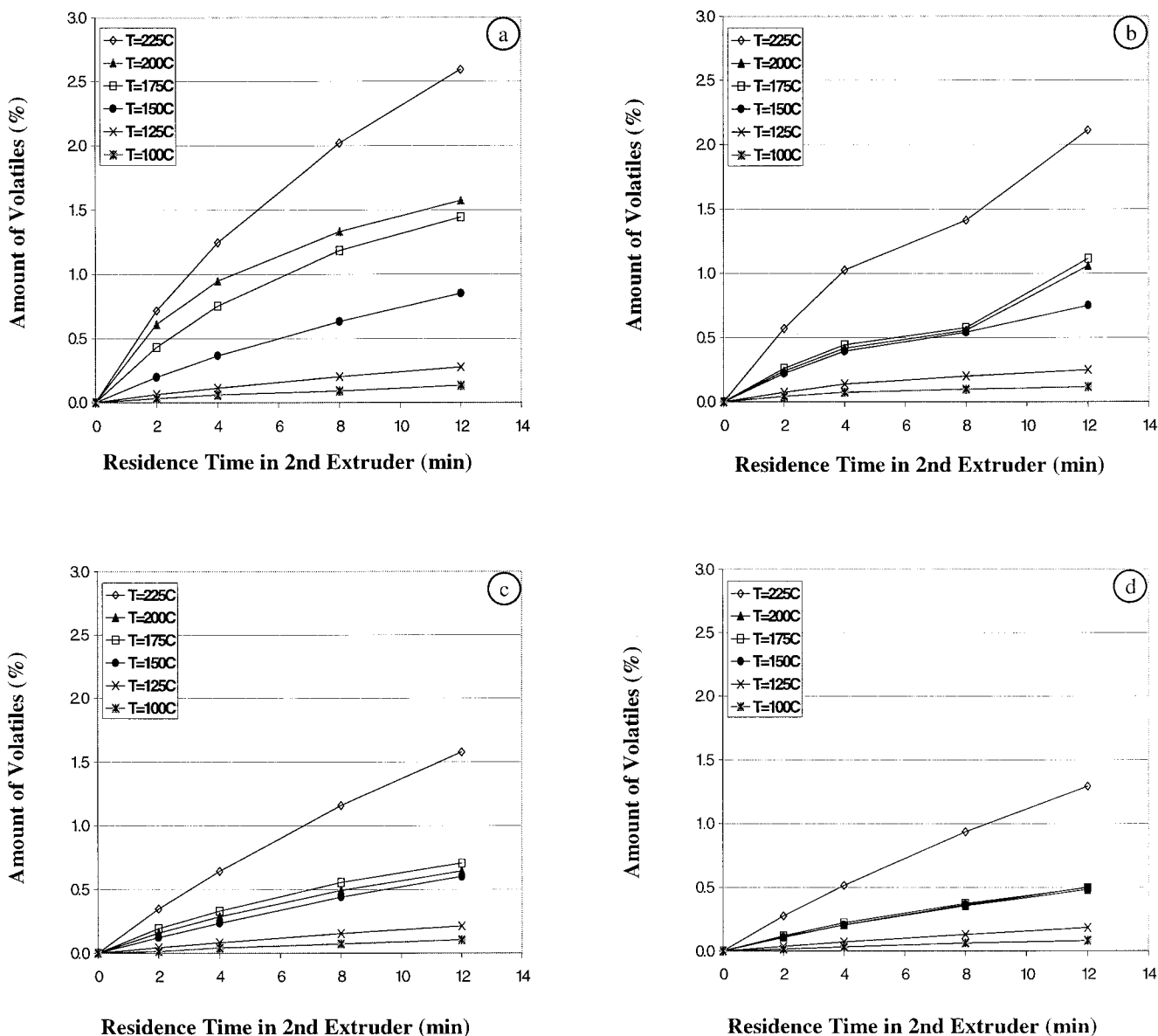


Figure 7 Estimated amount of volatiles used for foaming based on TGA data. The time in minutes indicates the residence time spent in the second extruder. (a) After a residence time of 2 min in first extruder; (b) after a residence time of 4 min in first extruder; (c) after a residence time of 8 min in first extruder; (d) after a residence time of 12 min in first extruder.

was dried at 75°C for 4 min, before being subjected to heating, most of the adsorbed water was purged out. Further weight loss occurred during isothermal heating either because of the loss of moisture, which was chemically bonded to the WF, or because of other emissions, which are primarily the vaporization of the volatile components of the “extractives” contained in the WF.²⁴ The figure clearly shows that the initial rate of weight loss was quite high and gradually decreased with the passage of time.

As this TGA gives the time-dependent rate of weight loss at each isothermal condition, it can also be used to predict the amount of volatiles that may be released from the WF during processing in the tandem

extrusion system. The rationale is that the WF undergoes heating in the first extruder and the volatiles evolved during the residence period in this extruder will all be lost to atmosphere during the passage of the extrudate via the vent at the interconnection of the two extruders. The WF will again encounter high temperature in the second extruder and will further release volatiles, which will contribute to the foaming process. The rate of volatile emission in the second extruder will depend on the amount already lost in the first extruder.

To use the TGA data to predict the volatile contribution to foaming, it is assumed that the same processing temperature is used in the entire tandem sys-

tem, and the volatiles liberated in the first extruder, in addition to the adsorbed moisture, are all purged out at the devolatilizing vent or through the hopper inlet. It is also assumed that most of the volatiles liberated in the second extruder are trapped in the melt and contribute to the formation of the foam structure.

Figure 7 shows the estimated amount of volatiles used in foaming based on the TGA results, which is essentially the same as the weight loss of WF (or volatiles released) in the second extruder. Comparing Figures 7(a–d), it can be seen that as the residence time in the first extruder is increased, the volatile emissions in the second extruder are decreased. Second, the lower the residence time in the second extruder, the less the emissions from the WF are. This indicates that if the WF is exposed to a high temperature for a long time in the first extruder, the amount of the volatiles generated from WF in the second extruder can be very small even at higher temperatures such as above 175°C. This strategy may possibly be used to make fine-celled foams from WF composites with a high melting-temperature material. However, this would mean severe degradation of WF to a certain degree in the first extruder. Therefore, it would be desirable to process the WF materials at a temperature as low as possible, preferably below 170°C, as discussed before.

CONCLUSION

Based on the experiments of HDPE/WF composites foamed with the volatiles of WF only, the highest critical processing temperature was estimated to be 170°C, below which the volatile emissions from the WF were suppressed significantly, provided the drying (or devolatilizing) temperature was also in the same range. Also, the effects of the volatiles released from WF at the different processing temperatures on the cell morphology and foam density were illustrated. The following conclusions can be drawn from the extrusion experiments for production of HDPE/WF composite foams.

- The effects of volatiles because of WF degradation cannot be suppressed completely in extrusion processing of plastic/WF composites.
- To minimize the foaming effects of the volatiles generated from WF during extrusion of plastic/WF composite foams, the highest processing temperature should be below 170°C.
- Even when the drying (or vent) temperature is greater than the barrel temperature, the foaming effects of the volatiles become significant when the barrel temperature is greater than 170°C.

In a tandem extrusion system, the devolatilizing vent at the interconnection of two cascading extruders was effective in removing moisture and other volatiles.

However, when the devolatilizing temperature was lower than the downstream barrel temperature, further volatiles were released in the second barrel, which resulted in the undesired irregular foamed structures. Therefore, the devolatilizing temperature at the vent should, preferably, be the highest processing temperature.

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NOMENCLATURE

T_b	Barrel temperature, °C
T_v	Devolatilizing temperature, °C
T_d	Die temperature, °C
ρ_f	Foam density, g/cm ³
M	Mass of foam sample, g
d	Diameter of foam sample, cm
l	Length of foam sample, cm

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