Role of nano-SiO$_2$ on the performance of microporous foamed polystyrene polymer

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ABSTRACT

Microporous foamed polymers are widely used in numerous fields hence, research on these materials has great practical significance. In this work, the effects of nano-SiO$_2$ on the micromorphology of polystyrene (PS) microporous foamed materials were systematically studied by varying nano-SiO$_2$ content and foaming temperature. Relative to PS, the results show that addition of nano-SiO$_2$ significantly affects the foaming of PS materials. The use of nano-SiO$_2$ as a nucleating agent reduces the size and increases the density of the cells in the PS matrix. Moreover, the addition of nano-SiO$_2$ reduced the viscoelasticity and resistance in the foaming process, whereas the expansion ratio improved. A foamed material with minimum standard deviation of the cell diameter was obtained using PS with 3% nano-SiO$_2$ content. The addition of nano-SiO$_2$ led to an increase in the foaming temperature of the foaming material with the maximum expansion ratio as well as the one having the maximum cell diameter. Further, this addition also led to flattening of the expansion ratio curve in the region of the maximum expansion ratio.

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1. Introduction

Microporous foamed polymers materials, characterized by a small cell size (1–100 $\mu$m) and high density ($\geq 10^8$ cells/cm$^3$), have found widespread application due to outstanding properties such as high impact strength, low thermal conductivity, long fatigue life, and good thermal stability [1–6].

Polystyrene microporous foamed materials (PS-MCF) are widely used in various fields owing to the following advantageous properties: low density, low thermal conductivity, low water absorption, good electrical performance, heat insulation, shock absorption, and sound insulation [7–11]. However, PS-MCF suffers from several limitations, including poor permeability, low impact strength, slow recovery speed after high-strength impact, and susceptibility to cell cracking. The key properties, i.e. physical and chemical
properties, structure, rheology, and morphology of many microporous foamed materials have been studied in-depth [12]. Yokoyama et al. [13], for example, studied the foaming of PS/PFMA (polystyrene/perfluorooctylethyl methacrylate) composites. They discovered that the cell size is in the micron range when the foaming temperature is higher than the glass transition temperature. When the foaming temperature is below the glass transition temperature, the pore size is in the nanometer range. Goel and Beckman [14] used poly (methyl methacrylate) microporous foamed materials to study the influence of pressure on the distribution of cells. They found that cells were more compact and evenly distributed when foaming pressure is high. Yang et al. [15,16] conducted radiation treatment on foamed materials during the foaming process to study the effect on molecular weight, crystallinity, and cellular structure. With radiation cross-linking, the cell structure of polypropylene (PP) foam became more uniform and the foaming temperature window increased by 10°C. Similarly, the foaming window of isotactic PP increased by approximately 8°C. Rachtanapun et al. [17] found that micro-foamed materials with a high expansion ratio and a high-quality foam structure could be obtained by reducing the size of PP spherulites and destroying the regularity of PP crystals. The experimental results indicated that impact strength is strongly related to the cell morphology of foamed samples.

Addition of nanoparticles into foaming materials to change their microstructure has also been widely studied. Taki et al. [18] studied the foaming behavior of PP-based clay nanocomposites using an intermittent visual observation instrument. With increase of clay dosage, nucleation rate and density of bubbles both increased, and nucleation time decreased. Keshtkar et al. [19] investigated the foamability of polylactide containing 0–5 wt% of nano-clay. Cell density and expansion ratio were greatly promoted with increased clay content. Abadchi and Jalali-Arani [20] studied the synergistic effect of adding a combination of organoclay and nanoscale polybutadiene rubber power to foaming PP materials. Addition of organoclay containing 5 wt% of polybutadiene powder improved the mechanical properties of the foamed PP relative to adding either filler by itself. Wang et al. [21] used maleic anhydride-grafted polypropylene (PP-g-MAH) as a compatibilizer to study the effects of PP-g-MAH and nano-clay content on the crystallization and rheological properties of block-copolymerized polypropylene (B-PP). They found that adding nano-clay and PP-g-MAH decreased the melt strength and complex viscosity of B-PP.

Despite the many investigations on preparation of foamed materials, the mechanism by which micromorphology affects the properties of microporous foamed materials is not fully understood. The aim of this study is to investigate the effect of nano-SiO$_2$ on the micromorphology of microporous foamed PS materials. In this study, PS-MCF was prepared with different nano-SiO$_2$ contents using supercritical CO$_2$. This was aimed at understanding the effect of nano-SiO$_2$ on the micromorphology of microporous foamed materials. The effect of nano-SiO$_2$ on expansion ratio, cell diameter, and cell density was measured to analyze its influence on the microcellular morphology of a microcellular foamed polymer. The microstructure of PS/SiO$_2$ was analyzed by varying the foaming temperature; further, the effect of nano-SiO$_2$ on the optimum foaming temperature was also studied.

2. Experimental

2.1. Reagents and instruments

General purpose, standard grade PS was purchased from Chi Mei Chemical Co. Ltd. (Jiangsu, China). Industrial grade CO$_2$ with purity greater than 99.9% was purchased
from Jiangsu Lianhai Biological Science limited company (Jiangsu, China). Hydrophilic nano-SiO$_2$ with purity greater than 99.94% was purchased from Huijing Sub-nanometer New Material Co. Ltd. (Shanghai, China).

The following instruments were used in the studies reported here. Single-screw extrusion granulator (HAAK, Thermo Fischer Scientific, USA). Electronic analytical balance (WY-10T, Shanghai Hengping Scientific Instrument Co. Ltd, China). Plate vulcanizing machine (HZ-7014B, Dongguan Lixian Instrument Technology Co. Ltd., China). Rotating rheometer (MARSIII, Thermo Fisher Scientific, USA). Scanning electron microscope (Quanta FEG 250, FEI Company, USA).

2.2. Material preparation

2.2.1. PS/SiO$_2$ composites matrix material preparation

PS/SiO$_2$ composites were prepared by the melt blending method. PS was dried for 10 h at 70 °C in an air blast drying box (PE-25BT, Huawei Fan Manufacturing Co. Ltd., China). After drying, the PS/SiO$_2$ materials with different nano-SiO$_2$ content were weighed and prepared. PS/SiO$_2$ composite in granular form was prepared by extruding in a single-screw extrusion granulator at a working temperature of 180 °C, screw rotation speed of 120 rpm, and a feed speed of 6 kg/h, and then dried. PS/SiO$_2$ granules were consolidated by pressing in a plate vulcanizing machine at an extrusion temperature of 170 °C and pressure of 5 MPa, with a preheat time of 3 min, extrusion time of 3 min, and cooldown time of 1 min.

2.2.2. PS/SiO$_2$-MCF preparation

The PS/SiO$_2$-MCF samples with different SiO$_2$ content were prepared in an autoclave by the lowering pressure method according to the test requirements. The materials were placed in the autoclave and sealed to ensure good air tightness of the device. Then, the autoclave chamber was evacuated for 2 min using a vacuum pump. Through the temperature control system, the tank body of the test device was heated to the required foaming temperature and held at this temperature for 10 min. CO$_2$ was injected into the test device so that the autoclave reached the set pressure, was fully saturated for a certain period, and a PS/CO$_2$/SiO$_2$ homogeneous phase system was formed. After the formation of a PS/CO$_2$/SiO$_2$ homogeneous system, the pressure was rapidly dropped to atmospheric pressure, and the samples were taken out to cool and set.

2.3. Testing and characterization

2.3.1. Measurement of apparent density

Apparent density refers to the density of the foamed sample. The apparent density of the samples was measured using an electronic analytical balance by the drainage method, according to Chinese Standard GB/T1033-1986.

2.3.2. Calculation of expansion ratio

The expansion ratio, also known as the volume expansion ratio, is used to calculate the volume ratio of foamed to pre-foamed sample and is obtained from Equation (1).
\[
\varphi = \frac{V_f}{V_0} = \frac{\rho_f}{\rho_0} = \frac{1}{\rho_0} \rho_f
\] (1)

where \(V_f\) is the volume of the sample after foaming in cm\(^3\), \(V_0\) is the volume of the sample before foaming in cm\(^3\), \(\rho_0\) is the density of the sample before foaming in g/cm\(^3\), and \(\rho_f\) is the density of the sample after foaming in g/cm\(^3\).

2.3.3. Calculation of cell size and cell diameter distribution

The cell shape and cell size of the foamed samples were evaluated from the material fracture surface morphology. To ensure the integrity of the fracture surface morphology, brittle fracture treatment of the sample is necessary. The samples were examined by freeze fracture electron microscopy; i.e. treated with liquid nitrogen and freeze-fractured, then sputtered with gold and observed under a scanning electron microscope. The SEM images of the PS/SiO\(_2\)-MCF nanocomposite samples were analyzed using the software ImageJ. To ensure accuracy and reproducibility of the results, the total number of cells in the image should exceed 100. The cell size is usually expressed as the average cell diameter \(\bar{D}\) which can be obtained from Equation (2). The cell diameter distribution is evaluated using the standard deviation to analyze the fluctuation in cell size, which is calculated using Equation (3).

\[
\bar{D} = \frac{\sum_{i=1}^{n} D_i}{n}
\] (2)

\[
S_d = \sqrt{\frac{1}{n} \sum_{i=1}^{n} (D_i - \bar{D})^2}
\] (3)

Here, \(n\) is the number of cells in an SEM image, \(D_i\) is the diameter of the cell (calculated to scale) in an SEM image in micrometers (\(\mu m\)), \(\bar{D}\) is the average cell diameter in micrometers (\(\mu m\)), and \(S_d\) is the standard deviation of cell diameter.

2.3.4. Calculation of cell density

The cell density \((N_f)\) of the foamed material refers to the number of cells in a unit volume (1 cm\(^3\)) and is calculated from Equation (4).

\[
N_f = \left( \frac{N_0}{A} \right)^{\frac{3}{2}} \varphi
\] (4)

where \(N_f\) is the cell density of the foamed material in cells/cm\(^3\), \(N_0\) is the number of cells in an SEM image, \(A\) is the area of an SEM image in cm\(^2\), and \(\varphi\) is the expansion ratio.

3. Results and discussion

3.1. Matrix characterization and rheological analysis

3.1.1. Analysis of section microstructure

The microsection microstructures of the PS/SiO\(_2\)-MCF nanocomposites were observed by scanning electron microscopy. Figure 1(a–c) shows SEM images of PS/SiO\(_2\)-MCF sections with nano-SiO\(_2\) contents of 1, 3, and 5%, respectively. The nano-SiO\(_2\) particles in the PS/SiO\(_2\)-1% and PS/SiO\(_2\)-3% samples were evenly distributed with slight agglomeration,
which met the characteristics of nanoparticle dispersion uniformity and ultra-fineness. The nano-SiO₂ particles in PS/SiO₂-5% show significant agglomeration and their dispersion is not ideal. Nanoparticles are prone to agglomeration due to high surface effect.

### 3.1.2. Analysis of rheological properties

Dynamic frequency scanning of the composites was performed at a test temperature of 180°C, using a rotating rheometer in the frequency range 100–0.1 rad/s with 1% strain. Test specimens were in the form of a wafer with 20 mm diameter and 1 mm thickness. The influence of nano-SiO₂ on the viscoelasticity of the PS/SiO₂-MCF nanocomposites was analyzed using the variation of energy storage modulus (G’), loss modulus (G’’), and complex viscosity (|\(\eta^*\)|) with frequency (\(\omega\)). The results for the PS-MCF materials with 0–5% nano-silica are shown in Figure 2.

Variation in storage modulus G’ with frequency is shown in Figure 2(a). All curves show the same trend. G’ increases with frequency, signifying that PS molecular chains cannot move at the same rate as the applied deformation, i.e. the molecular chains unwind too slowly at high shear frequencies and the chain segments are effectively ‘frozen’. The material becomes stiffer, hence G’ increases. At high frequency, the nanocomposite G’ values are close to that of PS. At low shear frequencies and less than 3% nano-SiO₂, G’ increases with increasing SiO₂ content. Therefore, adding nano-SiO₂ at this level increasingly restricts polymer chain movements. Possible mechanisms include increasing the degree of entanglement and filling voids between polymer molecules in the composite. With 5% nano-SiO₂, G’ was lower than that of pure PS. At the 5% level, nano-SiO₂ agglomerates form in the PS matrix. These destroy the molecular chain structure of PS and weaken the interaction between nano-SiO₂ and PS. G’ decreases as a result. Figure 2(b) shows that the behavior of loss modulus G’’ with frequency and nano-SiO₂ is similar to that of G’. However, G’’ does not increase as rapidly with frequency, and the maxima are lower. Therefore, proportionately less viscous energy must be lost by heat dissipation as the material becomes more rigid at higher frequencies. Adding nano-SiO₂ below 3% increases G’’ at low frequency, indicating that the frictional forces of intermolecular motion also increase. The opposite effect was observed with 5% nano-SiO₂, which is consistent with G’. Figure 2(c) shows the variation of complex viscosity |\(\eta^*\)| with frequency. PS and the PS/SiO₂-MCF nanocomposites both exhibit the rheological characteristics of pseudoplastic fluids, i.e. |\(\eta^*\)| decreases with increase in frequency. High shear breaks the interlocking of molecular chains, resulting in lower viscosity. At low frequency and less than 3% nano-SiO₂, |\(\eta^*\)| increases slightly with addition of nano-SiO₂. This trend is reflective of G’ and G’’; i.e. when nano-SiO₂ is added in this range, more energy is required for molecular chain movement, leading to an increase in |\(\eta^*\)|. At 5% nano-SiO₂ content, |\(\eta^*\)| was lower than the other samples. In the low-frequency region, the difference between each sample is obvious. With increasing frequency,
the $|\eta^*|$ curve of each PS/SiO$_2$-MCF nanocomposite sample tends to converge with the PS curve. It indicates that PS controls the material properties at high frequency.

From the data in Figure 2 (obtained at 180°C), it can be concluded that, at less than 3% nano-SiO$_2$ content, the viscosity of the PS/SiO$_2$-MCF matrix increases with increasing nano-SiO$_2$ content. At 5% nano-SiO$_2$ content, nano-SiO$_2$ agglomerates form in the PS/SiO$_2$-MCF matrix and destroy the molecular chain structure of PS. Thus, the viscosity of the composite material decreases.

### 3.2. Influence of nano-SiO$_2$ on the microstructure

Samples of PS, PS/SiO$_2$-1%, PS/SiO$_2$-3%, and PS/SiO$_2$-5% were selected to study the influence of nano-SiO$_2$ on the microstructure of the PS/SiO$_2$-MCF nanocomposites. The
samples were prepared under the following conditions: 110 °C (foaming temperature), 16 MPa (foaming pressure), and 3 h (foaming time).

Figure 3 shows the SEM images of the brittle sections of the PS/SiO2-MCF samples. The number of cells increases up to 3% nano-SiO2 content, and then decreases. Thus, the cell wall initially becomes thinner. Nano-SiO2 plays the role of nucleating agent in PS foam, but the agglomeration and uneven distribution of nano-SiO2 limits homogeneous nucleation. Some cells in the 3% nano-SiO2 sample ruptured and merged.

Figure 4 shows the cell size distribution of the brittle sections of PS/SiO2 microporous foamed samples with different nano-SiO2 content. The standard deviation (S_d) of the cell diameters are 5.64, 5.46, 3.43, and 4.80 μm, respectively, for 0, 1, 3, and 5% nano-SiO2. The smallest standard deviation and most evenly distributed cells were observed with 3% nano-SiO2 content. At 5% nano-SiO2 content, the uniformity of the cells decreased. Thus, it can be inferred that addition of an appropriate amount of nano-SiO2 to the PS matrix can yield high quality, uniform cells. When the nano-SiO2/PS ratio is too high, the number and uniformity of cells decrease.

Figure 5(a) shows the variation of expansion ratio with nano-SiO2 content for the foamed samples. With an increase in nano-SiO2 content, the expansion ratio of the foamed sample first decreased and then increased. In particular, the expansion ratio was lowest (19.5) for the PS/SiO2-3% sample; and highest (40.9) for the PS/SiO2-5% microporous foamed sample. Based on the analysis of the rheological properties of PS and PS/SiO2 composites, it can be concluded that at 5% nano-SiO2 content, the viscoelasticity of the
PS/SiO$_2$-5% composite decreases, which is conducive to the growth of foam cells. In contrast, the PS/SiO$_2$-3% composite had the highest viscoelasticity, which correlates with a low expansion ratio and limits the growth of cells. Figure 5(b) shows the variations in cell diameter and cell density of the PS-MCF samples with varying nano-SiO$_2$ content. Cell diameter decreases with nano-SiO$_2$ addition up to 3%, and then increases. PS/SiO$_2$-3% cell diameter is smallest (17.3 $\mu$m). Relative to the PS-MCF sample (30 $\mu$m), the cell diameter of PS/SiO$_2$-3% decreased by 12 $\mu$m (40%). This is related to the viscoelastic properties of the composite materials. Contrary to the change in cell diameter, the cell density of the PS-MCF samples increased with nano-SiO$_2$ addition up to 3%, and then decreased. Hence, the diameter and density of microporous foams are mutually exclusive. The expansion density of the PS/SiO$_2$-3% foam sample is 8.07 $\times$ 10$^9$ cells/cm$^3$, which is approximately three times that of the pure PS-MCF sample. Nano-SiO$_2$ was added to the PS matrix as a nucleating agent; these results demonstrate that it performed this function, particularly in the PS/SiO$_2$-3% nanocomposite. The expansion cell density of the PS/SiO$_2$-5% sample is 1.5 $\times$ 10$^9$ cells/cm$^3$ (approximately 1.6 times that of PS-MCF), indicating less optimal nucleation relative to PS/SiO$_2$-3%. On the basis of these results, the correct ratio and homogeneous dispersion of nano-SiO$_2$ are necessary to optimize its function as a nucleation agent.

### 3.3. Effect of SiO$_2$ on optimum foaming temperature

PS and PS/SiO$_2$-1% were used to study the effect of nano-SiO$_2$ on the optimum foaming temperature of PS/SiO$_2$ microporous foamed materials. The most appropriate experimental conditions were selected for the investigation. Samples were prepared under the following conditions: 15 MPa (foaming pressure) and 3.5 h (foaming time). According to standard foaming temperatures for pure PS, the foaming temperatures for the samples were set at: 110, 120, 130, 135, 140, and 150 $^\circ$C.

Figure 6 is a photo of PS-MCF prepared at different foaming temperatures. The volume of the foamed sample increased with an increase in foaming temperature up to 135 $^\circ$C, and then decreased. At foaming temperatures of 110 $^\circ$C and 120 $^\circ$C, the foam samples had large volumes. At 140 $^\circ$C, the foam sample was flat owing to a reduction in melt strength of the PS, leading to the collapse of the foam sample. Figure 7 shows the SEM images of PS/SiO$_2$-1%
microporous foamed material samples at different foaming temperatures. As the cell size increases in Figure 7(a–f), the number of cells decreases and the cell wall becomes thinner.

Figure 8(a) shows the variation in expansion ratio of PS-MCF and PS/SiO$_2$-1% samples with foaming temperature. The expansion ratio of the PS-MCF sample increased with the increasing foaming temperature. The expansion ratio of PS-MCF increased initially from 9.57 to 48.21 and then stabilized. The expansion ratio of PS-MCF was higher in the range of 120°C to 135°C. However, when the foaming temperature was 140°C, the expansion rate of the sample suddenly dropped to 2.87. In the foaming temperature range of 110°C to 140°C, the expansion ratio of the PS/SiO$_2$-1% sample increased in the initial stage from 9.78 to 48.21 and then stabilized. When the foaming temperature reached 150°C, the expansion ratio of the PS/SiO$_2$-1% sample suddenly dropped to 12.39. Thus, the expansion ratios of PS-MCF and PS/SiO$_2$-1% microporous foamed samples increase in the initial stage and stabilize with an increase in foaming temperature. As the foaming temperature increases, the viscoelasticity of the PS matrix decreases, enabling cell expansion. However, when the foaming temperature is higher than a certain value, the solubility of CO$_2$ (blowing agent) decreases, and the diffusion coefficient increases, resulting in easy diffusion of CO$_2$ out of the foaming melt; thus, the amount of CO$_2$ involved in cell growth decreases. Therefore, both the expansion ratio and cell diameter decrease, with the expansion ratio showing a sudden drop.

Figure 8(b) shows the variation in cell diameters of the PS-MCF and PS/SiO$_2$-1% microporous foamed samples at different foaming temperatures. The results are consistent with the expansion ratio data of Figure 8(a). The cell diameters of the PS and PS/SiO$_2$-1% samples increase initially, and then decrease at high foaming temperature. This is because temperature affects the viscoelasticity and melt strength of the PS matrices and PS/SiO$_2$-1% matrices. When the foaming temperature increases, the viscoelasticity of the PS matrix and resistance to cell growth decrease, and the cell diameter increases. When the foaming temperature is greater than 130°C, the melt strength of the foam substrate reduces, and the CO$_2$ coating ability diminishes; this leads to a decrease in the cell diameter. As shown
in Figure 8, the foaming temperatures at which the PS and PS/SiO$_2$-1% samples had the largest cell diameters were 130°C and 135°C, respectively. When PS-MFC is above 130°C, the melt strength of the foam substrate decreases, and CO$_2$ blowing ability diminishes, leading to a decrease in cell diameter. This stage is reached at 135°C with PS/SiO$_2$-1%. This is because the foaming temperature affects the viscoelasticity of the PS material and the PS/SiO$_2$-1% composites. At lower foaming temperatures, PS/SiO$_2$-1% is higher in viscoelasticity than PS-MCF, which limits the increase of cell diameter. The expansion ratio and cell diameter of PS-MCF and PS/SiO$_2$-1% samples exhibit the same trend with change in foaming temperature. However, based on Figure 8(a,b), the temperature of the PS/SiO$_2$-1% sample with a larger expansion ratio or a larger cell diameter is higher than

Figure 7. SEM images of PS/SiO$_2$-1% microporous foamed material samples at different foaming temperatures: (a) 110°C; (b) 120°C; (c) 130°C; (d) 135°C; (e) 140°C; (f) 150°C.

Figure 8. Variation in microscopic parameters of PS-MCF and PS/SiO$_2$-1% samples with foaming temperature: (a) expansion ratio; (b) cell diameter.
that of the PS foamed material. This is due to the high viscoelasticity of PS/SiO₂-1% com-
posite material, which requires a higher foaming temperature to overcome the resistance
to cell growth.

4. Conclusions
In this paper, the effects of nano-SiO₂ on the microstructure of PS-MCF/SiO₂ were
studied. Samples were prepared by the supercritical CO₂ intermittent foaming method.
The formation mechanism of PS/SiO₂ was analyzed based on the cell growth process and
related properties of the matrix. The main conclusions are as follows:

1. Nano-SiO₂ has two main effects on the matrix materials: acting as a nucleating agent
and changing their viscoelastic properties. Foamed materials with small cell size, high
cell density, and uniform cell distribution can be obtained by the addition of nano-
SiO₂ to the PS matrix.
2. The use of nano-SiO₂ in the matrix materials changes their optimum foaming tem-
perature. With an increase in foaming temperature, the expansion ratio and cell
diameter of the foamed materials increased in the initial stage and then decreased.

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