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ORIGINAL PAPER

Foam rotational molding of hybrid polyethylene nanocomposites: synergistic effect of microtalc and nanoclay

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ABSTRACT

Rotational molding is a process used to produce seamless, one-piece, and hollow polymeric parts. Foam rotational molding has recently become an increasingly important process in the foam industry. However, foam rotational molding is still a challenging process to fabricate polymeric foams. The focus of this manuscript was to assess the effect of material parameters on the foam properties of samples produced by rotational molding. Rotational molding experiments were performed on a laboratory-scale two-axis rotational machine, designed and manufactured by the authors. The effects of microtalc as nucleating agent, nanoclay as reinforcing agent, and their synergetic effect were investigated on the cell density, cell size, and expansion ratio of hybrid microtalc/nanoclay polyethylene nanocomposites. The cell density was improved by 96% and 89% by addition of 1 wt% of microtalc and nanoclay, respectively, compared to pure polyethylene foams. The cell size was reduced by 20% and 17.5% in 1 wt% of microtalc and nanoclay, respectively. However, the synergetic effect of using both microtalc and nanoclay at 1 wt% was more significant compared to their individual effects. The cell density was enhanced by 313% and the cell size was decreased by 35% compared to pure samples. **Polyolefins J (2022) 9: 129-138**

Keywords: Foam rotational molding; microtalc; nanoclay; synergistic effect; cellular structure.

INTRODUCTION

Nowadays, rotational molding is taken into consideration due to its various applications such as the production of large and complex hollow shapes without welding lines. Polyethylene (PE), polycarbonate, polyamide, polyurethane, and polypropylene are among the various polymers used in the rotational molding industry. PE is one of the most applicable thermoplastics used in the rotational molding process due to its low cost, relatively low density, and broad processing window [1].

Foaming the polymeric parts not only reduces the consumed material, but also provides different properties such as high thermal insulation [2], high sound insulation [3], low electrical permittivity [4, 5], good piezoelectric performance [6], and proper oil absorbance [7]. Nanomaterials are appropriate candidates to improve the different properties of polymers [8]. These materials can also be

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useful for the foaming process because they act as nucleating agents [9-11]. Polymeric clay nanocomposites are of great importance because significant changes in their mechanical properties, thermal resistance, and flame resistance are achieved by adding small amounts of this type of filler [12].

In the rotational molding process, the mold rotates around two perpendicular axes and the process consists of four stages including charging, heating, cooling, and demolding. A schematic of this process is shown in Figure 1.

Although rotational molding has been developed for more than three decades, there have been few research studies compared to other methods of polymeric foam production. Rotational molding base developments began in the works of Crawford and co-workers [13-15]. Some research on rotational molding has also focused on the effect of processing parameters on the properties of polymeric parts by Antonio and Alfonso [16], Olink et al. [17], Spence and Crawford [18]. The mechanical properties of rotary molded parts have been extensively reviewed by Van et al. [19], and later by Pick and Harkin Jones [20] and Pick et al. [21]. As a breakthrough, Spence and Crawford [18] showed that a low viscosity material has fewer bubbles or surface pores than a high viscosity material.

Foam rotational molding has become an increasingly important process in the industry. The properties and behavior of polymeric nanocomposite foams are directly related to the geometry of the cell structure, the properties of the nanoparticles, and the polymer matrix. In addition to the polymer properties, dispersion of nanomaterials, cell size, cell density, and relative density are effective parameters on the different properties of polymeric nanocomposite foams [22-25]. In the field of rotational molding, several studies have been conducted on unreinforced polyethylene, most of which are related to the optimization and modeling of this process. However, the need for weight loss for some specific applications such as automotive, packaging, etc. has increased the need for polyolefin foams, a need that has been felt and increased for 30 years. To produce foam parts by rotational molding, a proper selection of the foaming agent, nucleating agents, and other additives must be carried out and the operating conditions must be checked. Pop-Iliev et al. [26] investigated the chemical rotational molding process of polyethylene foams at ambient pressure. Doroudiani et al. [27] studied the effect of crystallinity on the foam morphology structure in the rotational molding process. They changed the crystallization and morphology of the foam by controlling the cooling process. They predicted that polypropylene, polybutylene, and polyethylene terephthalate have higher crystallization at low cooling ratios. Archer et al. [28] reported that in linear low-density PE (LLDPE) foam rotational molding, lower-density foams can be produced using an exothermic blowing agent compared to an endothermic agent. Liu et al. [29] conducted a study on LLDPE foams produced in rotational molding and concluded that the amount of blowing agent, the heating time, and the operating temperature were important factors in determining the morphology of the final cells. Several studies [30-33] proved that adding nanoclay improves different properties of polymeric and polymeric foam parts in various processing methods.

Despite some advances in the rotational molding process of polymeric foams, few experimental studies have been conducted on the properties and structure of PE foams produced by rotational molding. In particular, studies investigating the effects of material compound on the PE foam properties are rare. This study is performed using a laboratory-size rotational molding machine to investigate the effects of material composition on the morphology and density of PE foams. Different samples are produced with different compositions and their

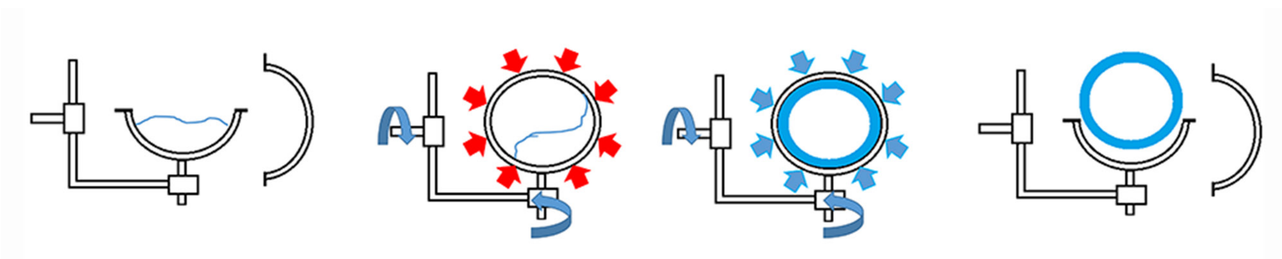


Figure 1. Schematic of the rotational molding process.

morphology and foam structure are investigated. The effects of addition of microtalc and nanoclay and their synergetic effect are studied on the structure of PE nanocomposite foams produced by rotational molding.

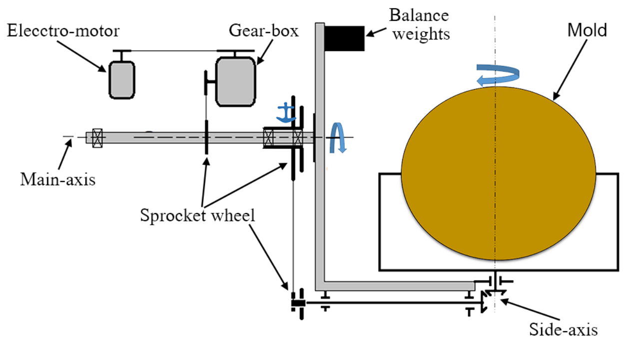
EXPERIMENTAL

Materials

The PE used in this study was manufactured by Tabriz Petrochemical Company with the grade of HD3840UA suitable for rotational molding process with a melt flow index of 4 g/10 min (2.16 kg & 190°C). Its density was 0.938 gr/cm^3 (according to ASTM D-1505 standard). Azodicarbonamide (ACA) purchased from Dupont microfilm, USA, was used as a chemical blowing agent. This grade of ACA has an average particle size of 3 mm with a decomposition temperature of 192°C and produces 228 ml/g of gas at atmospheric pressure. Merk KGaA's German-made microtalc was added to PE as a nucleating agent. The average microtalc size used was 5 µm and its density was 2.7 g/cm^3. Modified montmorillonite nanoparticles under the brand name Montmorillonite K-10 brand and manufactured by the American company SIGMA-ALDRICH were used as reinforcement agent. The average density of nanoclay was 0.6 g/cm3 and had an average particle size of 1.5 nm. PE grafted by maleic anhydride (PE-g-MA) was utilized as compatibilizer. PE-g-MA produced by Aria Polymer Pishgam, had a density of 0.945 g/cm^3. Prior to testing, the polymer powder, foaming agent, compatibilizer, microtalc, and nanoclay were first mixed using a dry mixer.

The samples were manufactured using a rotary molding machine, designed and made by the authors in laboratory size. Its schematic is presented in Figure 2-a. The mold was able to rotate in two directions by the machine during production and its speed could be controlled. The used mold (presented in Figure 2-b) is proportional to the size of the ASTM-D638 Type I standard made of stainless steel with a thickness of 1 mm. 630 g of compounded powders were required for molding to have the desired thickness required for the standard.

A 0.02 mm thick aluminum sheet was used to easily remove the workpiece from the mold without applying stress to it. The sheet was mounted on the inner surface of the mold. Then, a layer of WD-40 separator oil by WEICON was sprayed on the sheet surface. The mold was pre-heated for 10 min. The compounded material was poured into the mold and then, the mold was placed inside an oven at 200 °C. The mold was then rotated in the oven for 20 min. The rotational speed of the main axis was 24 rpm and the ratio of the rotational speed of the sub-axis to the main axis was 2:3. After heating process, the mold was removed from the oven and cooled with water. The cooling time was defined as the time required for the indoor air temperature to drop to 30°C.



(a)

(b)

Figure 2. (a) schematic of the designed rotational molding machine and (b) the mold used in this study.

A SEM-ProX, Phenom Co. testing device was utilized for scanning electron microscopy (SEM) tests. Before SEM tests, the samples were frozen in a liquid nitrogen tank for maintaining the morphological structure in the broking step. Image-Pro Express Software (Media Cybernetics) was utilized for analyzing the SEM micrographs to obtain cell size and cell density. At least, one hundred cells were measured for each test. Cell density (N) was defined as the cell numbers per cm^3 of the foamed samples volume by assuming spherical cells. The samples were cut to the specified dimensions and measured with a weighing scale of 0.001 g. Then, the density of the samples was determined by the Archimedes densitometry method, in which double-distilled water was used. Finally, expansion ratio, an important characteristic indicating the foaming degree, was obtained as the ratio of the solid density to the foam density.

Table 1 shows the conditions and composition of the materials used in each of the tests. Test 1 was conducted to observe the properties of pure polymeric foam produced by the rotational molding. Tests 2 and 3 indicate the effects of microtalc (as nucleating agent) and nanoclay (as reinforcement agent) on the properties of foamed samples produced by the rotational molding. Test 4 reveals the synergetic effect of microtalc and nanoclay on the foam properties. Tests 5 and 6 indicate the synergetic effect of these materials at higher concentrations.

Table 1. Design of experiments.

Test	Base material	Nanoclay (wt%)	Microtalc (wt%)	ACA (wt%)	Solid density (g/cm^3)
1	PE	0	0	1	0.938
2	PE	0	1	1	0.963
3	PE	1	0	1	0.933
4	PE	1	1	1	0.939
5	PE	1	3	1	0.951
6	PE	3	1	1	0.929

RESULTS AND DISCUSSION

The representative SEM pictures are presented in Figure 3. The quantitative results of the cell density, cell size, and expansion ratio are presented in Table 2. By analyzing these data it is possible to investigate the effects of addition of microtalc, nanoclay, and their synergetic influence on the foam properties of samples produced by the rotational molding.

The results indicate that the pure PE sample foamed by rotational molding has a relatively high expansion ratio of 2.4, cell size of 553 µm, and cell density of 6.88×10^3 cell/cm^3.

To have a better perspective of the comparison of different material parameters, some graphical charts are plotted. Figure 4 demonstrates the effect of using microtalc as a nucleation agent on the properties of the obtained foam sample.

As it is observed in Figure 4, by comparing the results of experiment (1) with experiment (2), the effects of microtalc are determined as a nucleating agent. The findings reveal that the cell density increases and the cell size decreases by adding microtalc. The enhancement of the cell density is by 96% and the reduction of the cell size is by 20% by addition of microtalc com-

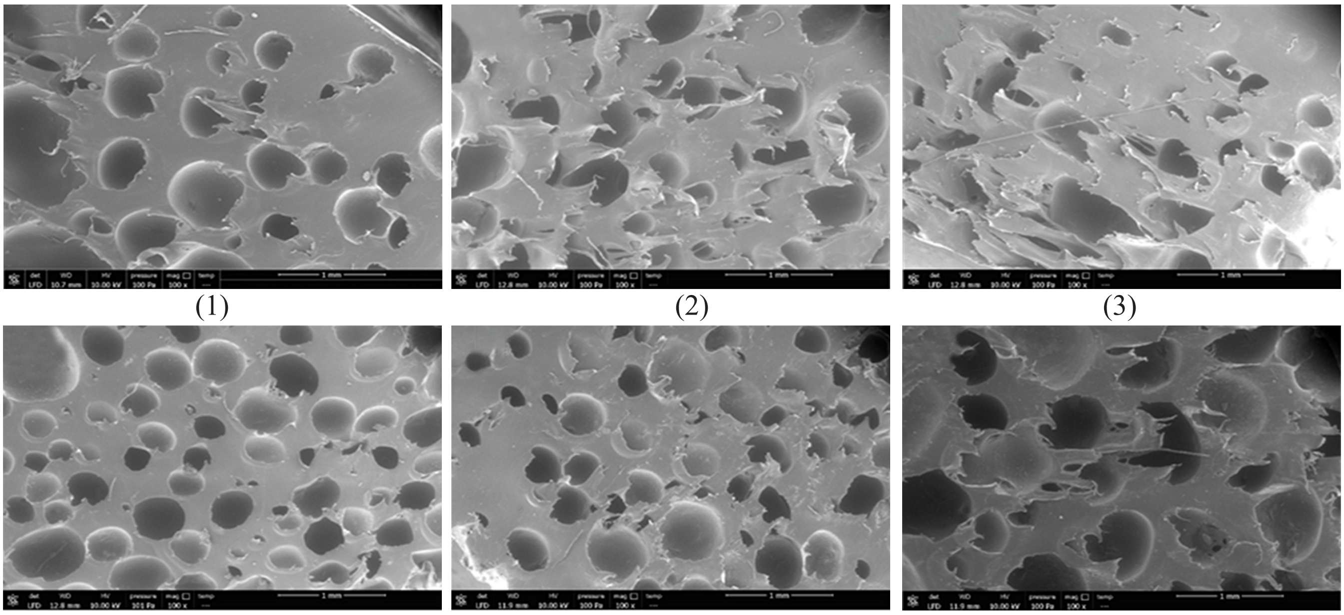


Figure 3. Representative SEM results.

Table 2. Results of cell density, cell size, and expansion ratio.

Test	Cell density (cells/cm³)	Cell size (μm)	Expansion ratio
1	6.88×10^{3}	553.5	2.3567
2	1.35×10^{4}	443.6	1.8771
3	1.30×10^{4}	456.7	1.5295
4	2.84×10^{4}	360.6	2.0502
5	1.77×10^{4}	416.6	2.8993
6	1.26×10^{4}	542.2	1.7332

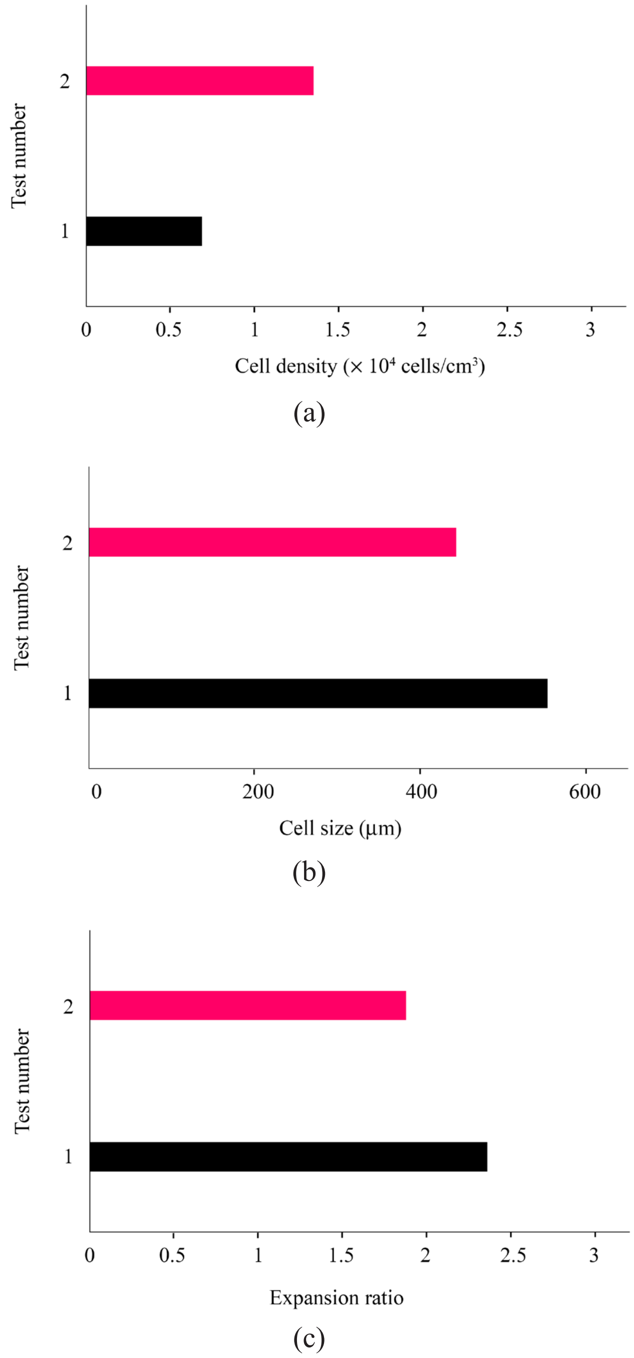


Figure 4. Effects of microtalc addition on the foam properties: (a) cell density, (b) cell size, and (c) expansion ratio.

pared to the pure sample. This significant effect is due to the nucleating role of microtalc [34-36]. Utilizing the nucleating agents has been reported as an efficient approach for enhancing the nucleation rate in polymeric foams [37-39]. The heterogeneous nucleation is activated and the critical work required for the cell growth to the critical radius is decreased in this case. As a consequence, more cells can reach the critical radius and spontaneous growth. Hence, the nucleation rate and cell density are boosted and consequently, the cell size is reduced. A 20% reduction is observed in the samples produced with microtalc compared to pure samples.

Figure 5 shows the effects of using nanoclay as a nucleation agent on the properties of the obtained foam sample.

By comparing the results of experiment (1) with experiment (3), the effects of nanoclay are determined. Nanoclay, in addition to the nucleation role, causes local stresses and decreases the critical radius. The addition of nanomaterials into the polymer matrix results in a large number of small cells. This effect increases cell density by up to 88%, decreases cell size by up to 17.5%, and reduces the expansion ratio by up to 35%.

In Figure 6, the effects of using nanoclay and microtalc as nucleation agents are plotted to have a better comparison.

The results of experiment (2) and experiment (3) are compared to determine which nucleating agent acts better, microtalc or nanoclay? If microtalc is used as a nucleation agent, cell density is 4% higher, cell size is 13% lower, and the expansion ratio is 18% higher. Microtalc is several times larger than nanoclay and a microtalc particle is capable of causing much local stress than a particle of nanoclay. Hence, the nucleation rate is higher in the case of microtalc and the cell size and cell density are improved. However, the number of nanoclay contained in the unit of volume is much higher than that of microtalc, and stress occurs in many places, making cell collapse more likely due to the proximity of the points. Therefore, it is more possible to deteriorate the expansion ratio in the case of nanoclay.

Figure 7 demonstrates the synergistic effect of using both microtalc and nanoclay on the properties of the obtained foam sample.

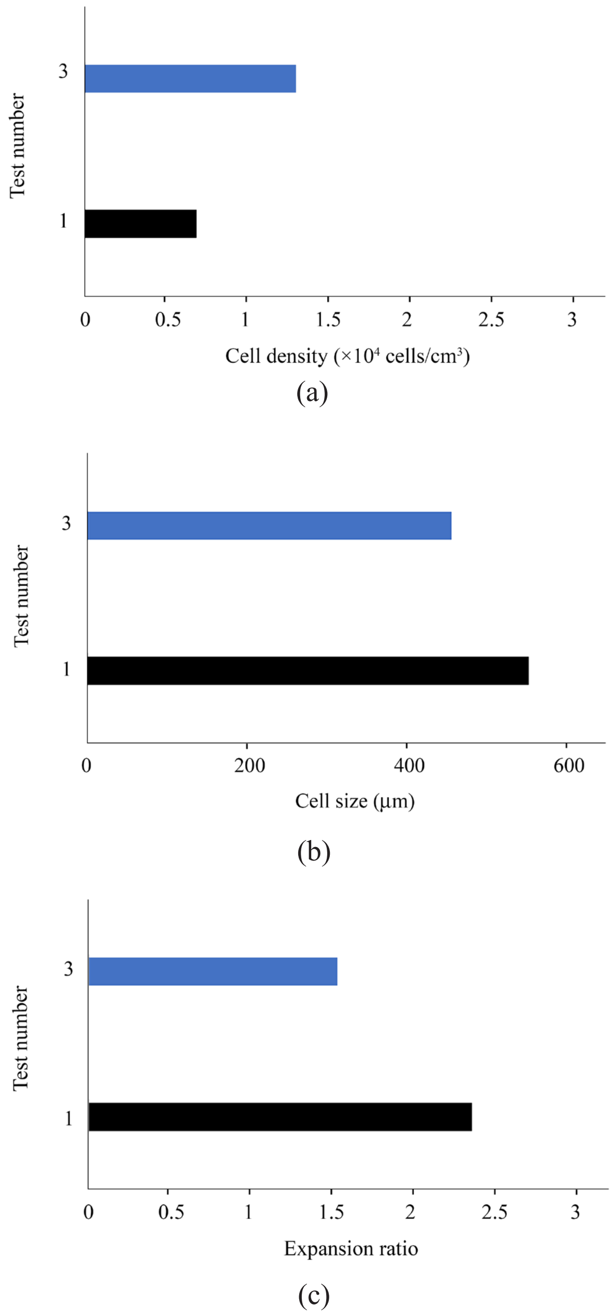


Figure 5. Effects of nanoclay addition on the foam properties: (a) cell density, (b) cell size, and (c) expansion ratio.

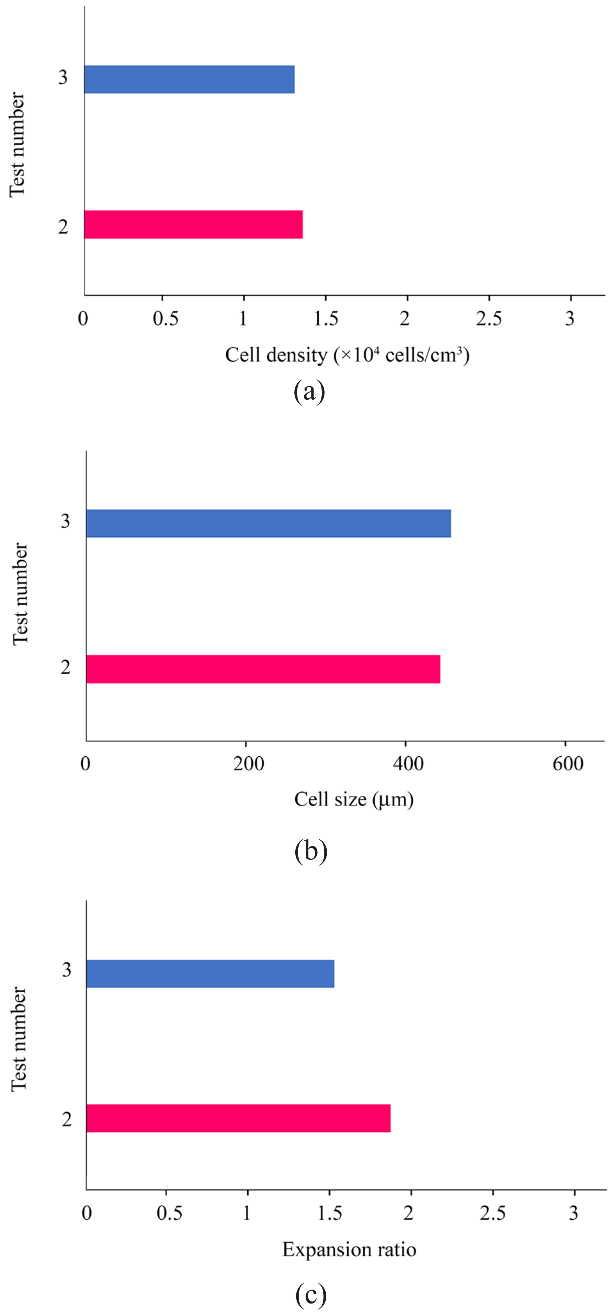


Figure 6. Comparing the effects of microtalc and nanoclay on the foam properties: (a) cell density, (b) cell size, and (c) expansion ratio.

The synergistic effect of microtalc and nanoclay can be revealed by comparing experiment (4) with experiment (1). Cell density is increased by 312%, cell size is decreased by 54%, and expansion ratio is decreased by 13% in the case of synergetic effect of nanoclay and microtalc compared to the pure sample. This remarkable effect is due to the simultaneous local stresses of microtalc and nanoclay, which dramatically reduces the critical radius, decreases cell size, and increases cell density. The synergetic effect of microtalc and nanoclay causes local stresses of different sizes in the polymer matrix. For this reason, the final properties of experiment (4) compared to experiments (2) and (3) are increased, respectively, by 110% and 120% in cell density, 18.5% and 21% in cell size, and 10% and 34% in expansion ratio.

Figure 8 compares the synergistic effect of using both nucleation agents to their individual use.

As it was explained, the synergetic effect of micro-

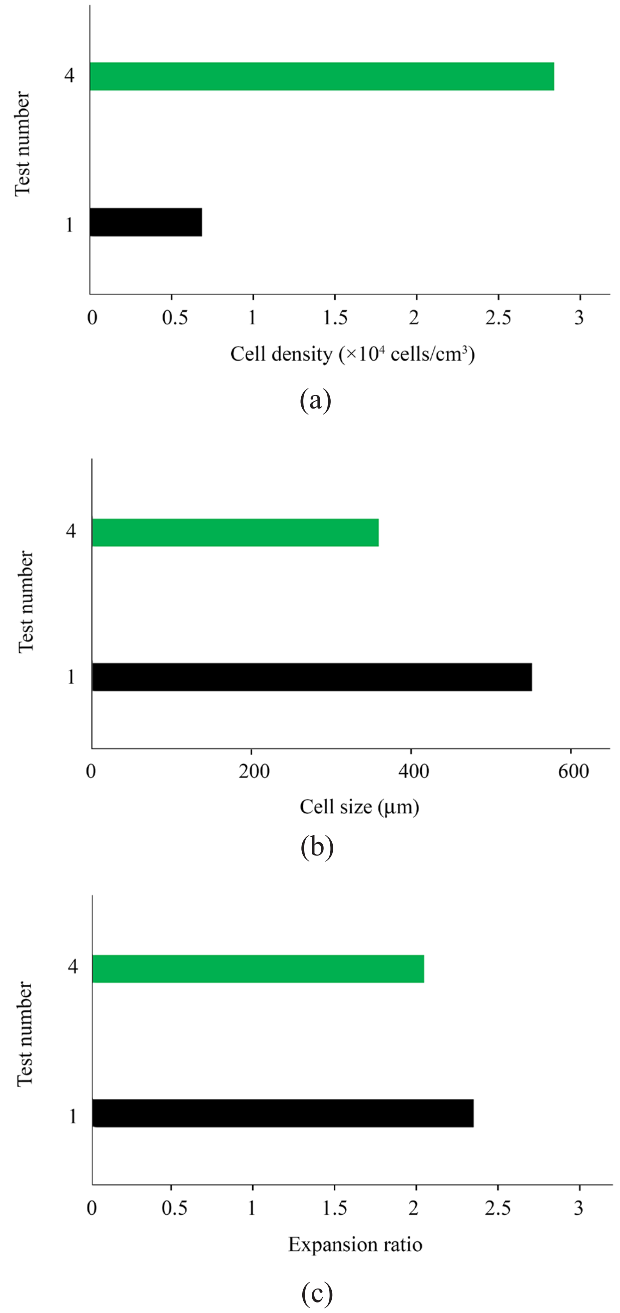


Figure 7. Synergistic effect of microtalc and nanoclay on the foam properties: (a) cell density, (b) cell size, and (c) expansion ratio.

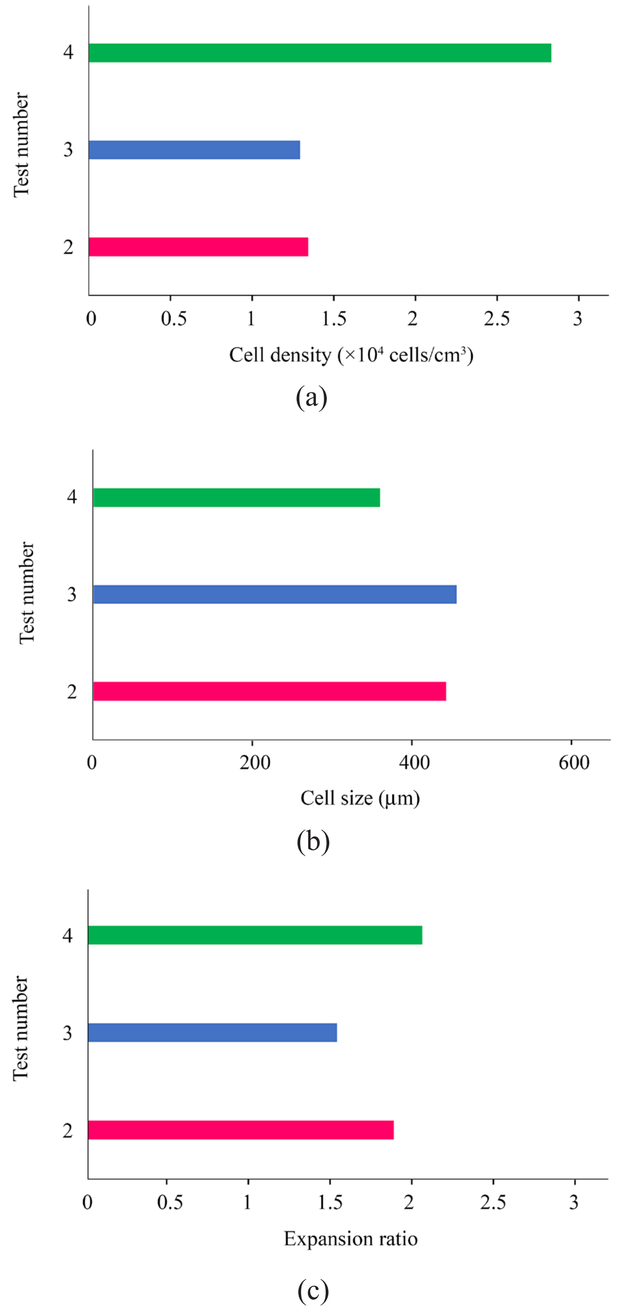


Figure 8. Comparing the synergistic effect of microtalc and nanoclay to their individual effect on the foam properties: (a) cell density, (b) cell size, and (c) expansion ratio.

talc and nanoclay causes local stresses of different sizes in the polymer matrix. For this reason, the final properties of experiment (4) compared to experiments (2) and (3) are increased, respectively, by 110% and 120% in cell density, 18.5% and 21% in cell size, and 10% and 34% in expansion ratio. This reveals that using two different nucleation agents together can help to improve the desired properties of foam samples. However, the important point here is choosing the correct composition of materials in a way that two different particles act in synergism. For this purpose, different compositions are also examined in the following.

To investigate the effect of higher percentages of these nucleation agents, Figure 9 is plotted.

Experiments (5) and (6) were conducted using microtalc and nanoclay compositions with a high percentage of microtalc and nanoclay, respectively. Compared to experiment (1), cell density is decreased

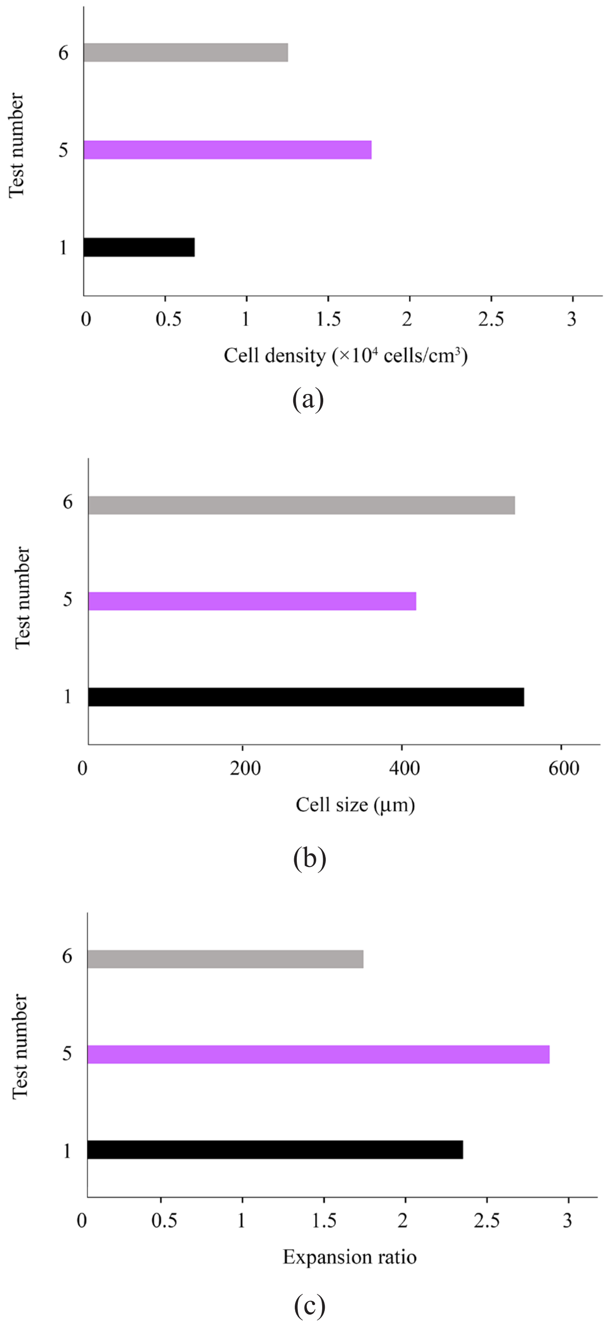


Figure 9. Effect of addition of higher concentration of microtalc and nanoclay on the foam properties: (a) cell density, (b) cell size, and (c) expansion ratio.

and cell size is increased in both experiments. The reduction of cell density is 38% and 55.6% in the case of experiments (5) and (6), respectively. The cell size is increased by 15.5% and 50% in experiments (5) and (6), respectively. The agglomeration of microtalc and nanoclay particles is a possible phenomenon at higher concentrations. This agglomeration causes a drastic increment in local stresses. The cell-to-cell interactions are increased by significantly reducing the critical radius and therefore deteriorating phenomena such as cell coalescence are increased. Therefore, the foam properties deteriorate at high concentrations.

CONCLUSIONS

In this study, PE was used as a base material and ACA was used as a foaming agent. Microtalc and nanoclay powders were used as additives to improve the final properties of the foam produced by rotational molding. Samples were produced under different compounding conditions using a laboratory rotational molding machine. The effects of different weight percentages of additives and their synergistic effect were investigated in detail. The main conclusions can be summarized as follows:

- Adding microtalc and nanoclay led to increased cell density and decreased cell size due to the nucleating role of these materials. However, the nucleating role of microtalc was more significant compared to nanoclay because the local stresses created by larger particles are more remarkable.
- The synergetic effect of microtalc and nanoclay was more significant than the individual effect of these particles. The highest cell density and smallest cell size were obtained at 1 wt% of synergistic of microtalc/nanoclay.
- The cell size was increased and the cell density was decreased at higher concentrations of microtalc and nanoclay due to the possible agglomeration of the particles.

CONFLICTS OF INTEREST

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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