

Research Article

The Effect of Multiwalled Carbon Nanotubes on the Thermal Conductivity and Cellular Size of Polyurethane Foam

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Polyurethane (PU) foam is known as the popular material for the applications in many fields of industry and life. To improve the mechanical and thermal properties of this material, in this research, PU foam was reinforced with aniline-modified multiwalled carbon nanotubes (MWCNTs). Fourier transform infrared FTIR spectrum of modified MWCNTs showed the aniline was grafted on the surface of MWCNTs through the appearance of $-NH_2$ stretches. The effect of MWCNTs with and without modification on the density, porosity, compressive strength, and heat conductivity of PU/MWCNT foam nanocomposites was investigated. The dispersibility of MWCNTs in the PU matrix was enhanced after modification with aniline. Compressive strength of PU nanocomposite reached the highest value after adding 3 wt.% of modified MWCNTs into PU foam. Besides, the water uptake of PU nanocomposites using 3 wt.% of MWCNTs was decreased to 13.4% as compared to that using unmodified MWCNTs. The improvement in thermal conductivity of PU/aniline-modified MWCNT nanocomposite was observed due to the change in the cellular size of PU foam in the presence of MWCNTs as shown by SEM images.

1. Introduction

Unlike most plastics, polyurethane (PU) foam belongs to the cellular material that possesses unique properties in thermal porosity and mechanical properties. These properties can be changed in a wide range by adjusting the raw materials to manufacture PU foam such as polyol, isocyanate, and catalysts. Thanks to such flexible properties, PU foam has been applied in many different areas from aerospace components, ships, ballistic vests, and automobiles [1, 2]. However, the material has some drawbacks due to low mechanical and thermal properties that limit its applications [2]. Therefore, many researchers have focused on overcoming these disadvantages in order to improve the properties of PU foam [3, 4].

A great deal of studies have been reported on PU nanocomposite foams that reinforced with different types of nano-

particles such as nanoclay [4–6], titanium dioxide [7], and carbon nanofibers [8–10] to create a class of foam material with promising properties. In the research of Xu et al. [11], the addition of 2 phr of organoclay nanoparticles into PU foam led to the improvements in the tensile and compressive strengths of PU foam (110 and 152%, respectively). Saha et al. [2] used 1 wt.% of TiO_2 nanoparticles to reinforce PU foam. The obtained results showed a slight increase in Young's modulus, tensile strength, and compression strength of the PU/ TiO_2 nanocomposites about 14.6%, 5%, and 16.2%, respectively, as compared to PU foam. Carbon nanofibers (CNFs) were also studied as high-performance reinforcement additives for PU foam due to their excellent axial tensile strength, superior thermal and electrical properties, and thermal conductivity [12–14]. Guo et al. [15] reported that both the tensile modulus and tensile strength of the PU/MWCNT nanocomposites are remarkably enhanced by about 90% as

comparison with pure PU foam as the addition of 1 wt.% of MWCNTs into PU foam. Importantly, the elongation at break of PU/carbon nanotube (CNT) composite is greatly improved by about 500%, indicating that the toughness of neat PU is enhanced by adding CNTs into the matrix. Similar results were also reported by Sinaret for MWCNTs reinforced PU foam [3]. Compressive strength of PU/MWCNT composite with 0.5 wt.% of filler reached the peak at 1.162 MPa as compared to other foam composites. The energy absorption was increased from 22.89 J for PU matrix to 24.53 J for foam composites with 3 wt.% of MWCNTs.

Although previous papers showed that MWCNTs could significantly improve the properties of the PU foam nanocomposites, the number of papers regarding thermal conductivity of PU/MWCNT foam nanocomposites is somewhat scarce.

Carbon nanotubes (CNTs) were known as a sunlight absorber that has low cost, reusability, and excellent light-to-heat conversion properties [16]. Under sunlight, CNTs can absorb and scatter photons due to the strong interaction between CNTs and incident solar light; the generation of heat occurs from the surface of the CNTs where strong coupling occurs between the incident radiation and the electrons on the surface of the CNTs [17]. For PU foam composites, the generated heat on the surface of the CNTs could then transfer to the matrix and was stored in cell structure. Santiago-Calvo et al. [18] have modeled the thermal conductivity of PU/CNT foam through four heat conduction mechanisms: conduction along the cell walls and the struts of the solid polymer, conduction through the gas phase, thermal radiation, and convection within the cells. The authors indicated that the thermal conductivity of a PU foam could be improved by an addition of small amount of CNT nanoparticles (0.1–0.4 wt.% of CNT). However, the thermal conductivity of PU foam nanocomposites reinforced a large amount in the range of 1–5 wt.% of MWCNTs which has not been mentioned yet.

Therefore, this study will prepare the foam nanocomposites containing MWCNTs with high concentrations. The thermal conductivity, morphology, and compressive strength of foam nanocomposites will be also investigated and discussed in detail. Moreover, MWCNTs have been modified with aniline to improve the compatibility with PU matrix as well as the properties of PU foam nanocomposites.

2. Experiment

2.1. Material. Methyl diphenyl diisocyanate (MDI) and polypropylene polyethylene (PPG) were the products of Oriken chemical company, Malaysia. Multiwalled carbon nanotubes (MWCNTs) with a purity of 99% used in this study was supplied by Institute of Materials Science, Vietnam Academy of Science and Technology, Vietnam. The MWCNTs has an average diameter of 20 nm and a length of 50–200 microns. Aniline (purity of 99.5%) and nitric acid (concentration of 68%) were supplied by Xilong Chemical Company (China). Absolute alcohol was bought from Duc Giang Chemical Company (Vietnam).

2.2. Preparation of PU Foam and PU/MWCNT Foam Nanocomposites

2.2.1. Modification of MWCNTs with Aniline. First, MWCNTs were treated by concentrated nitric acid before being filtered and washed by distilled water to a pH of 7 [19, 20] and then dried at 80°C for 24 hours to obtain treated MWCNTs. After treatment, MWCNTs were modified with aniline as follows: 0.528 g MWCNTs were added into a mixture containing 120 ml of water and 20 ml of ethanol under sonication for 30 minutes at room temperature. Next, 6 g glyceride and aniline solution were added into a solution under stirring at 70°C for 24 hours. Finally, the MWCNTs were filtered and washed with distilled water before being dried at 80°C for 6 hours.

2.2.2. Fabrication of PU/MWCNT Foam Nanocomposites. The PU foam nanocomposites containing 0, 1, 3, and 5 wt.% of MWCNTs were prepared as follows: MWCNTs were firstly added to the polyol under mechanically stirring for 5 minutes at 1200 rpm. The mixture continued to be ultrasound by using T18 digital Ultra Turrax (IKA) for 30 minutes at 20 kHz of frequency. Finally, isocyanate was added to the MWCNT/polyol mixture using mechanical mixer with 1500 rpm for 10 seconds. After that, the mixture was quickly poured into a mould with dimensions 250 × 250 × 250 mm and left to foam freely in one direction for 24 h before removing from the mould. PU foam without MWCNTs was prepared under the same conditions.

2.3. Characterizations

2.3.1. Fourier Transform Infrared (FTIR) Spectra. Fourier transform infrared spectra (FTIR) were used to analyze samples with and without modification on a Fourier Nexus 670 spectrometer (USA), in the wave range of 4000–400 cm⁻¹ with a resolution of 4 cm⁻¹ and an average of 32 scans.

2.3.2. Density and Porosity. The density of PU foam nanocomposites is determined according to ASTM D1622-93, and the porosity of PU foam samples is determined according to ISO 5013-1985.

2.3.3. SEM Micrograph. Cell size of foam nanocomposites and MWCNT distribution were observed using a Field Emission Scanning Electron Microscope (FE-SEM) under a voltage of 80 kV and magnifications 30 and 50,000. Necessarily, samples were coated with silver to improve the qualification of SEM images at high magnification.

2.3.4. Compressive Strength. The compression tests were conducted according to ASTM D1621 at the crosshead speed of 10 mm/min using Zwick Z2.5 instrument (Germany).

2.3.5. Water Uptake. The water uptake of PU foam nanocomposites is determined according to ISO 5013-1985.

2.3.6. Volume Resistivity. Volume resistivity is determined on the Takeda TR8401 machine (Japan) with DC voltage of 100 V at 25°C and a humidity of 50%.

2.3.7. Thermal Conductivity. The thermal conductivity measurements of the foams were carried on a THB 500 (Linseis, Germany) with the range from 0.01 to 100 W/m·K⁻¹ at 20°C. Measurements were made under steady heat flow conditions through the test samples, in accordance with the UNE12667 method.

2.3.8. Heat of Absorption of Foam. The absorber thermometric system (as seen in Figure 1) is a vacuum chamber connected to 250 W infrared light inside. Samples were placed in the chamber with the distance of 390 mm from light source. The temperatures of irradiated surface, nonirradiated surface, and inside sample were collected to measure the heat absorption ratio.

3. Results and Discussion

3.1. FTIR Spectra of Original MWCNTs, Acid-Treated MWCNTs, and Aniline-Modified MWCNTs. FTIR spectra of original, acid, and aniline-modified MWCNTs are shown in Figure 2. For original MWCNTs, the peak at 1628 cm⁻¹ is characterized for C=C bonding, which related to the original structure of carbon nanotubes. After treatment with nitric acid, there is a new peak at 1717 cm⁻¹ corresponding to C=O stretching, indicating the existence of carboxyl groups in treated MWCNTs due to oxidation of HNO₃ acid [19].

The characteristic groups of aniline can be observed in the FT-IR spectrum of aniline-modified MWCNTs. Peaks at 3436 cm⁻¹ and 3368 cm⁻¹ are characterized for -NH₂ stretches. Weak peaks at 3037 cm⁻¹ and 2934 cm⁻¹ are assigned to the C-H bond of the benzene ring in aniline. C-N stretching vibration is appeared at 1262 cm⁻¹. Moreover, a slight shift from 1717 cm⁻¹ to 1728 cm⁻¹ of the C=O group vibration may be due to the interaction between COOH groups on the surface of acid-treated MWCNTs and -NH₂ groups of aniline. It could indicate that aniline was successfully attached to MWCNT.

3.2. Effect of MWCNT Contents on the Properties of PU/MWCNT Nanocomposites. The effects of MWCNT contents on the porosity, density, and compressive strength of the PU/MWCNT nanocomposites were studied and are reported in Table 1. In this table, the density of PU foam increased by the presence of MWCNTs. Neat PU has the density of 0.034 g/cm³ and increases to 0.055 g/cm³ for the nanocomposite containing 5 wt.% of MWCNTs. The change is consistent with the results of Sinar et al. [3], the density of PU foam/3 wt.% of MWCNT nanocomposites also increased by 11.2% as compared with PU foam. The reduction in the porosity was observed for the PU foam nanocomposites with increasing MWCNT contents. For instance, the porosity reached to 89.7% and 76.32% for PU foam and PU nanocomposites using 5 wt.% of MWCNTs, respectively. The result can be explained by the presence of MWCNTs in PU foam which led to the increase in the viscosity of precursor solution that prevented the formation and growth of bubble during foam preparation. Therefore, the higher MWCNT content was introduced in PU foam, the lower porosity was formed in its structure.



FIGURE 1: Absorber thermometric system.

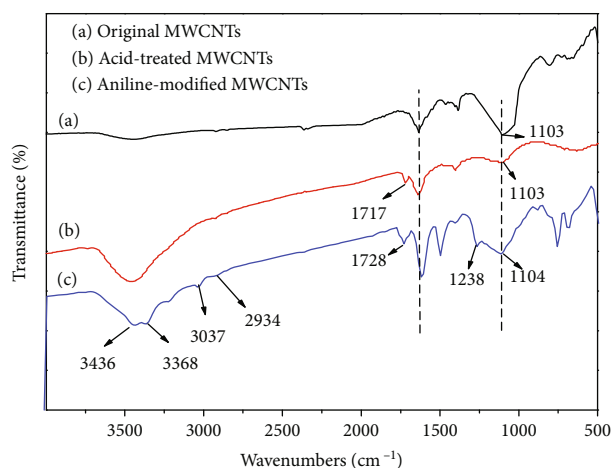


FIGURE 2: FT-IR spectra of original, acid-treated, and aniline-modified MWCNTs.

TABLE 1: Density, porosity, and compressive strength of PU/MWCNT foam nanocomposites at various MWCNT contents.

MWCNT contents (wt.%)	Density (g/cm ³)	Porosity (%)	Compressive strength (kPa)
0	0.034	89.7	82.81
1	0.042	82.1	95.26
3	0.048	76.9	111.9
5	0.055	76.3	90.13

The compressive strength of PU foam was also yielded higher than introduction of MWCNTs causing the change in the microstructure of the foam. Neat PU foam has the lowest compressive strength at 82.81 kPa while the PU foam nanocomposite containing 3 wt.% of MWCNTs has the maximum compressive strength at 111.9 kPa. This is due to fine dispersion and good interaction of MWCNTs in PU matrix

at 3 wt.% content. However, the compressive strength of the nanocomposites tends to strongly decrease as MWCNT content exceeds 3 wt.% due to the agglomeration of MWCNTs on PU matrix [3, 20]. Thus, 3 wt.% of MWCNT content was selected for the next investigation.

3.3. The Effect of Modified MWCNTs on the Properties of PU/MWCNT Nanocomposites. To determine the effect of aniline modification, the properties of PU nanocomposites using 3 wt.% of MWCNTs with and without aniline were studied and are shown in Table 2. As seen in Table 2, the nanocomposites containing aniline-modified MWCNTs reveal the high density and low porosity in comparison with that containing unmodified MWCNTs. This may be due to the presence of aniline on the surface of MWCNTs that could enhance the dispersibility of MWCNTs and the viscosity of PU foam. The fine dispersion of modified MWCNTs in PU matrix also caused the slight increase in compressive strength (3%) and the decrease in water uptake (13.4%) as compared to the PU/unmodified MWCNT nanocomposites. On the one hand, the decrease in water uptake is attributed to hydrophobic surface of MWCNTs after modification with aniline. On the other hand, the reduction of porosity of the nanocomposites is also the reason for this decrease. It is worth noting that aniline improves the electrical conductivity that could also enhance thermal conductivity for PU/modified MWCNT nanocomposites as compared to PU/unmodified MWCNT nanocomposites [8].

3.4. Thermal Properties of PU Foam Nanocomposites

3.4.1. Heat of Absorption. The temperature at surface of samples when exposed under a constant light source has been used to determine the heat absorption capacity of the samples. Figure 3 reveals the temperature change as a function of the exposing time for the nanocomposites with different modified MWCNTs concentrations.

The heat absorption process can be assumed by the following equations [21, 22]:

$$\text{Absorption process : } Q = E \cdot S \cdot k_1,$$

$$\text{Deabsorption process : } P = \alpha(T_1 - T_0),$$

when the surface temperature remains constant : $Q = P$

$$\text{Lead to } \frac{\alpha(T_1 - T_0)}{E \cdot S},$$

$$\text{Therefore, } \frac{k_1}{k_0} = \frac{T_1 - T_0}{T_2 - T_0}, \quad (1)$$

where Q is the absorbing heat, P is the radiant heat, S is the surface area of sample, k_1 is the heat absorption constant of PU/MWCNT nanocomposite, k_2 is the heat absorption constant of PU reference, E is the radiant energy of light sources, α is the coefficient of heat absorption of the medium, T_0 is the room temperature ($^{\circ}\text{C}$), T_2 is the saturated temperature of PU reference ($^{\circ}\text{C}$), and T_1 is the saturated temperature of PU/MWCNT nanocomposite ($^{\circ}\text{C}$).

TABLE 2: The properties of PU nanocomposites using 3 wt.% MWCNTs with and without modification.

Properties	Unit	Unmodified sample	Modified sample
Density	g/cm^3	0.048	0.050
Porosity	%	76.9	75.0
Compressive strength	kPa	111.9	115.3
Water uptake	%	68.8	59.6
Volume resistivity	$\Omega\cdot\text{m}$	0.75×10^{12}	1.67×10^9

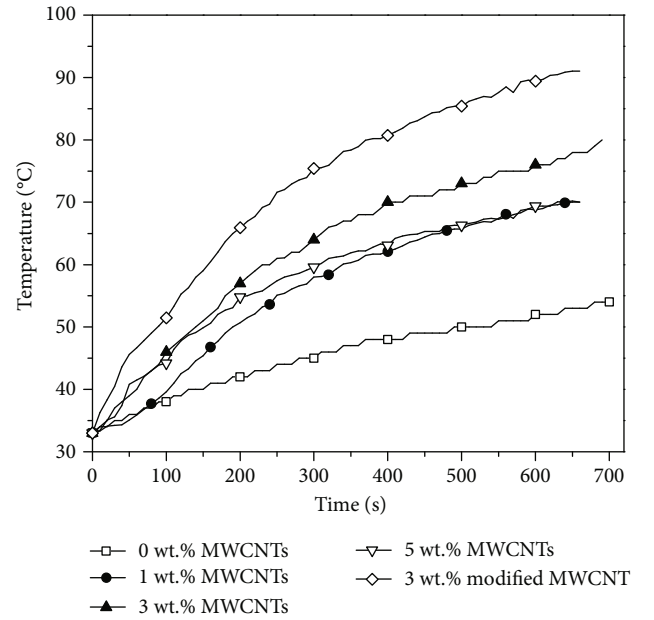


FIGURE 3: Temperature on the surface of the foam nanocomposites with different MWCNT contents.

As observation from Figure 3, the surface temperature of the samples increased rapidly in the first 400 seconds, then slowly reached a steady state over the last 300 seconds. Original PU foam showed the slow increase in temperature and reached the maximum value of 50°C after 700 seconds of testing. This is probably due to the high thermal insulation and less heat absorption of PU. In contrast, surface temperature of the foam nanocomposites has been dramatically changed with addition of 3 wt.% of modified MWCNTs and reached the maximum value of 90°C after 700 testing seconds. However, if the filler content is higher than 3 wt.%, the surface temperature of the foam nanocomposites is no longer enhanced. In this case, the addition of MWCNTs exceeds the fine dispersibility in PU matrix. Consequently, the heat-absorbing effect of the MWCNTs on the surface samples decreased. Thus, as MWCNTs were modified with aniline, the compatibility between dispersive phase and polymer matrix was improved. As a result, temperature at the surface of the nanocomposites containing 3 wt.% of modified MWCNTs is higher than that of unmodified samples.

From the obtained data of temperature on surface samples, the heat absorption ratio (as compared with PU foam)

TABLE 3: Heat absorption ratio and temperature deviation of PU/MWCNT nanocomposites with various MWCNT contents.

Properties	Foam nanocomposite				
	0 wt.% unmodified MWCNTs	1 wt.% unmodified MWCNTs	3 wt.% unmodified MWCNTs	3 wt.% modified MWCNTs	5 wt.% unmodified MWCNTs
Heat absorption ratio (k_1/k_0)	1.00	2.30	2.75	3.23	2.49
Temperature deviation (ΔT (°C))	31.4	27.5	24.8	16.9	20.8

and temperature deviation between irradiated and nonirradiated surfaces of samples were calculated and are shown in Table 3. The incorporation of MWCNTs into PU foam can enhance the heat absorption ratio from 2.30 to 2.75 as filler content changed from 1 wt.% to 3 wt.%, and then, it was decreased to 2.49 for the sample using 5 wt.% of modified MWCNTs. The higher heat absorption ratio was achieved for the PU/modified MWCNT samples meaning that the temperature deviation is the lowest for the PU/modified MWCNT nanocomposites as compared with PU matrix or PU/unmodified MWCNT nanocomposites. The obtained results demonstrated that MWCNTs could promote the heat conduction within the bulk of nanocomposites, especially modified MWCNTs.

3.4.2. Study of Thermal Conductivity. Figure 4 shows the changes in thermal conductivity of the foam nanocomposites as a function of MWCNT contents. The thermal conductivity for the PU foam is 0.035 W/m·K. This value increased to 0.072 W/m·K for PU/unmodified MWCNT nanocomposites as MWCNT contents changed in the range of 0–5 wt.%. The change in thermal conductivity is in agreement with the results of Santiago-Calvo et al. [18]. The authors investigated the effects of different MWCNT contents (0.1–0.4 wt.% loading) and reported that thermal conductivity could remarkably improve as compared with PU foam at high contents of nanoparticles due to an increase of the heat conduction through the solid phase. The addition of nanoparticles enhanced the density of foam resulting in an enhancement in the thermal conductivity of the solid matrix. Regarding the thermal conductivity, Yan et al. [23] also explained the formation of an interconnected network of the fibers at the high content with polymer matrix could lead to an increase of the conductivity of the solid matrix. This suggests that good dispersion and compatibility between the matrix and the filler give a positive effect for the thermal conductivity of the material. As seen in Figure 4, the incorporation of modified MWCNTs with PU foam shows the significant improvement in thermal conductivity as compared with the unmodified nanoparticles. The obtained results showed that modification with aniline could promote the dispersibility of MWCNTs in PU matrix, leading to the improvement in the thermal conductivity of the foam nanocomposites.

3.5. Morphology of Foam Nanocomposites and MWCNT Distribution in the Foam. Microstructures of the PU foam and foam nanocomposites were determined by scanning electron microscopy analysis (SEM). The change in morphology of the foam nanocomposites at various MWCNT contents is shown in Figure 5 and summarized in Table 4. The micrographs of PU foam and its nanocomposites

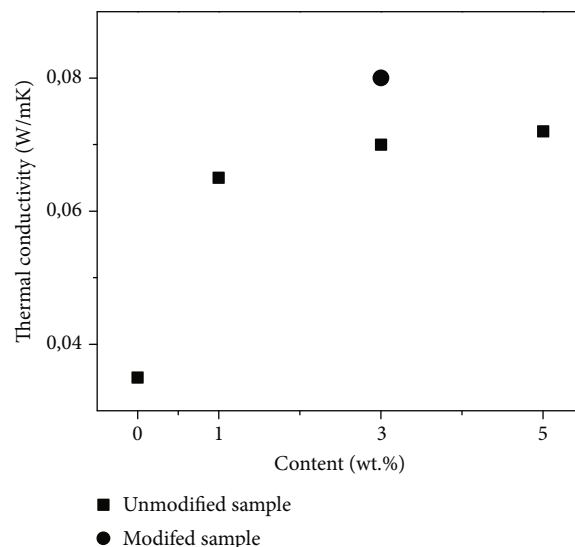


FIGURE 4: Thermal conductivity of modified 3 wt.% MWCNT nanocomposite and unmodified nanocomposites at various MWCNT contents.

showed a cellular structure with spherical and polyhedral shape. The cell distribution was somewhat uniform for PU foam and became less uniform after adding nanoparticles into PU foam. Average cell size calculated from micrograph of samples showed that the changes in cell size are obvious as shown in Table 4. The cell size of the pure PU foam is 714 μm , whereas the foam nanocomposites with 1 and 3 wt.% of unmodified MWCNTs has a cell size of 616 and 380 μm , respectively. However, the decrease in cell size was also recorded for sample containing 3 wt.% of modified MWCNTs, where cell size is 240 μm . It means that the cell size of the modified sample is finer than that of the unmodified sample at the same filler content.

In general, a cell size reduction of PU foam caused by the presence of nanoparticles has been reported previously [4]. MWCNTs played as a nucleation point for cell formation and growth of PU foam [24], leading to the creation of a larger number of cells; thus, the cell size became smaller. On the other hand, the viscosity of the foam nanocomposites seems to be higher than PU foam due to the incorporation of MWCNTs with PU matrix which limits the cell growth and results in smaller cell sizes when compared to the PU foam [4].

The distribution of MWCNTs with and without aniline modification in the PU matrix can be observed from SEM images at the higher magnification. It can be seen from Figure 6(a) that a uniform dispersion of modified MWCNTs

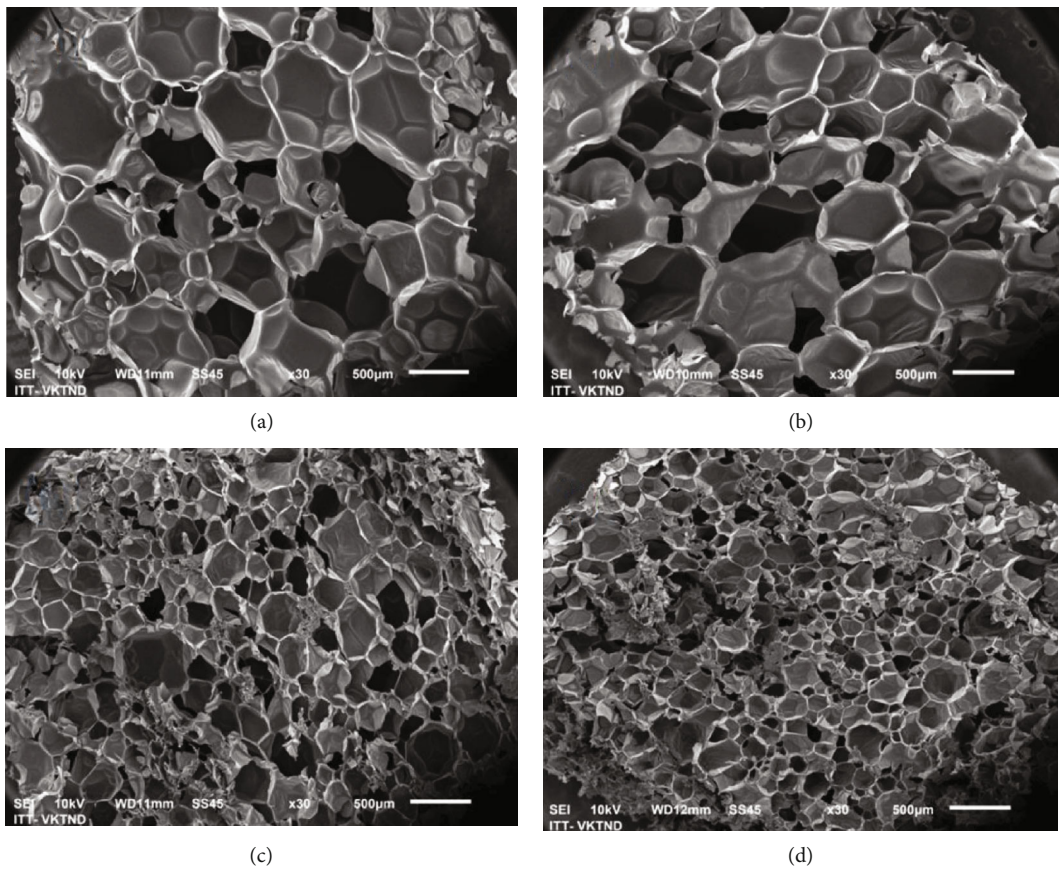


FIGURE 5: SEM micrographs of (a) original PU foam and foam nanocomposites containing (b) 1 wt.% MWCNTs, (c) 3 wt.% MWCNTs, and (d) 3 wt.% modified MWCNTs with low magnification (30 times).

TABLE 4: Microstructural results of foam nanocomposites.

Type of property	Neat PU	1 wt.% unmodified MWCNTs	3 wt.% unmodified MWCNTs	3 wt.% modified MWCNTs
Cell type	Closed	Closed	Closed	Closed
Symmetry of structure	Asymmetric	Asymmetric	Asymmetric	Asymmetric
Cell size (µm)	714	616	380	240

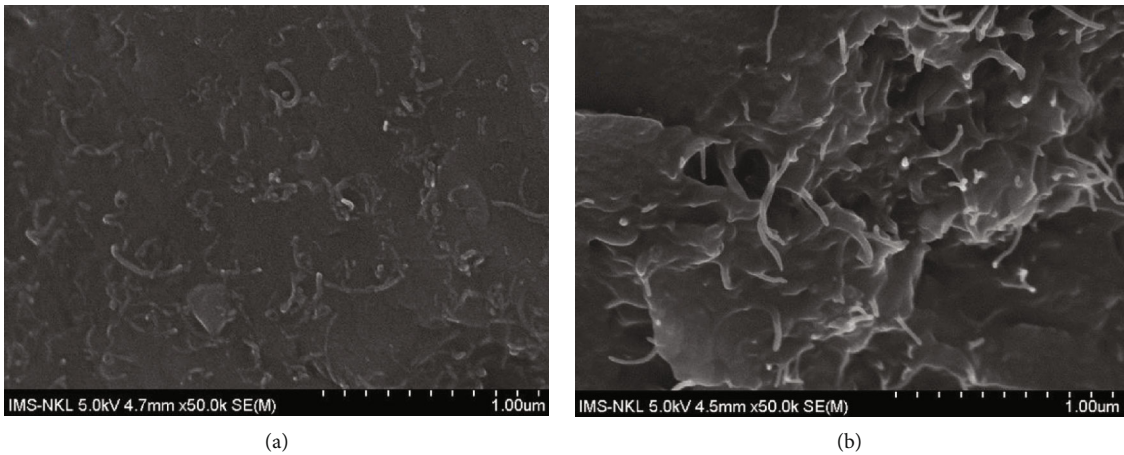


FIGURE 6: High magnification SEM micrograph of foam containing (a) 3 wt.% MWCNTs and (b) 3 wt.% modified MWCNTs.

was observed on the PU cell edges (not detected on the cell walls). However, the phase separation between the filler and matrix is quite clear due to the differences in the hydrophilic properties of PU and modified MWCNTs. The presence of aniline on the surface of MWCNTs improved the compatibility with PU matrix, which led to the generation of smaller cells as compared to the foam containing unmodified MWCNTs (Figure 6(b)).

4. Conclusion

The MWCNTs were successfully modified with aniline through the interaction between anilines with carboxylated MWCNTs. PU foam nanocomposites reinforced with unmodified and modified MWCNTs with different concentrations were prepared by a moulding process. The changes in properties of PU foam as introducing MWCNTs showed that the presence of MWCNTs led to an increase in density and a reduction on porosity due to the nanoparticle addition enhances the nucleation point for cell formation and growth of PU foam. The compressive strength of foam nanocomposite achieved the maximum value for the sample containing 3 wt.% of unmodified MWCNTs. As compared to unmodified samples, the foam nanocomposites containing 3 wt.% of modified MWCNTs show better results. The compressive strength of the PU/modified MWCNT nanocomposites increased from 111.9 kPa to 115.3 kPa, while water uptake significantly decreased from 68.8% to 59.6% as compared to the unmodified sample. A similar improvement in thermal conductivity was also seen for the nanocomposites containing modified MWCNTs with an increase from 0.072 W/m·K to 0.08 W/m·K. Moreover, SEM micrograph showed that the addition of modified MWCNTs affected the cellular size of foam more clearly than the unmodified filler. The average cell size of the foam decreased with increasing nanoparticle content, and the lowest value was 240 μm for the nanocomposite containing 3 wt.% of modified MWCNTs. A possible explanation of this result is that modified MWCNTs could be better dispersed in the PU matrix than the unmodified filler due to better chemical interactions of the modified nanoparticles with the PU matrix. It is worth noting that high temperature could be achieved at surface for the foam nanocomposites under light source opening up possibilities for drying agricultural products.

Data Availability

The data used to support this study can be available upon request to the corresponding author.

Conflicts of Interest

The authors declare that they have no conflicts of interest

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