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The role of sonication time upon acoustic wave absorption efficiency, microstructure, and viscoelastic behavior of flexible polyurethane/CNT nanocomposite foam

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ABSTRACT

In the present work, acoustic wave absorber nanocomposites based on flexible polyurethane (PU) foam and sonicated multi walled carbon nanotube (CNT) has been fabricated by in-situ solution polymerization of toluene diisocyanate (TDI) and an ether based polyol in the presence of CNT particles. This study provides the influence of sonication time upon the acoustical and dynamic mechanical properties of the prepared composites. Flow resistivity measurement, scanning electron microscopy (SEM), dynamic mechanical thermal analysis (DMTA) and compressive mechanical measurement were performed on the prepared samples. The fabricated PU/CNT foamed nanocomposites were evaluated for their acoustic wave absorbency within a wide range of frequency (400–6300 Hz). Nanocomposites originated by highly sonicated CNT exhibited more acoustic absorbency. All CNT filled PU composites showed higher attenuation of the acoustic wave energy than the unfilled reference sample. Results revealed that sound absorbency of the foamed PU/CNT nanocomposites could be effectively enhanced by increasing the sonication time. Dynamic mechanical temperature sweep measurements showed decrease in mechanical loss tangent by enhancing the extent of CNT dispersion state. Results showed that improved sound absorbancy by CNT sonication time is mainly caused by increased CNT interacting surfaces rather than viscous response of the viscoelastic PU/CNT nanocomposites towards the applied dynamic field.

1. Introduction

Foams are two phase porous media which are composed of solid and fluid phases. Materials with porous structure have been shown to have a high sound absorption coefficient [1,2]. Alternation of the foam structural properties including flow resistivity, reticulation rate and cell sizes, allows to tailor the absorption coefficient of the foam for the attenuation of sound energy within a specific frequency range. Foamed polymer materials have been reported to have high sound absorption potential in high frequency regions, but exhibit low sound absorbancy within low frequency regions [3–6].

Flexible polyurethane foams have been shown to be efficient sound absorbent as the cells wall could be vibrated by the incident sound wave which results in attenuation of the wave amplitude [7,8]. The viscoelastic behavior of polyurethane foams can be controlled and optimized by various parameters such as chemical structure, pores size and density as well as inclusion of high aspect

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ratio nanofillers. Various types micro and nano fillers have been employed to improve the acoustic performance and sound wave energy dissipation of the foamed structure polymers materials [9,10].

Carbon nanotubes (CNT's) with high conductivity, strong mechanical strength, high aspect ratio as well as excellent acoustical properties have been utilized in the production of sound absorbing materials mainly polyurethane foams [11,12]. This nanofiller with high specific surface area is able to increase the potential of the polymer matrixes for the dissipation of sound energy as it provides extremely large interfacial areas for the interaction with the incident wave at a low filler concentration [13]. However, in spite of all extraordinary properties of CNT, dispersion of CNT tubes throughout the composite matrix has remained as a challenge. This is due to the strong van der walls attractive forces acting between the CNT particles which prevents their perfect dispersion.

Dispersion state and arrangement of carbon nanotubes play crucial role in engineering the composite microstructure for the desired properties. CNT particles are needed to be functionalized in order to enhance the polymers filler interaction, and also micro morphological stability. However, surface modification of CNT's with polar functional moieties such as acid groups would increase the Van der Walls attract active forces between the CNT particles. Therefore, the imposed mixing energy should be higher than the interacting energy forces, but lower than the fracture energy of individual CNT particles.

Conventional methods for the manufacturing of polymer /filler composites include either direct or master batch mixing of as received filler with the polymer matrix by subjecting the mix to mechanical shear forces. In many cases, high degree separation of CNT particles or clusters would not be achieved by simply mechanical mixing. Alternatively, ultrasonication of CNT has been shown as an efficient method for the breakdown of CNT physical networks as local shear stresses are transferred to the CNT bundles [14–16]. In this method, cavitation occurs in a low viscosity solution above a certain ultrasonic intensity in the low-pressure regions of the travelling wave. The collapse of cavitation bubbles creates high strain rates which leads to the peeling off the nanotubes. Extent of effectiveness of this process depends on the geometry of sonication, time, temperature, and solution viscosity. However, the imposed high energy during sonication might induce fracture of CNT particles which necessitates to control the process. Sonication time, and hence extent of CNT dispersion throughout the polymer matrix has important role in the final properties of corresponding nanocomposite, especially in fabrication of sound absorbent foamed nanocomposites. Kabir et al. [17] studied the effect of sonication time upon various properties of rigid PU foams loaded with multi walled carbon nanotubes (MWCNT). They observed that the probe sonicator is an effective method for appropriate dispersion of CNT, and sonication time depends on volume of the solution and power of sonicator. It is also suggested that by increasing the amounts of nanotubes, the time of sonication should be increased. Zhang et al. [18] have investigated the effect of time of mechanical stirring on the mechanical properties of the flexible PU foams loaded with various amounts of functionalized nanotubes, and reported that the time of mechanical stirrer has significant impacts upon the final properties of corresponding nanocomposites.

To the best of our knowledge, the effects of sonication time and extent of CNT's destructurization upon various properties such as viscoelastic behavior, and sound energy absorbancy of flexible PU/CNT nanocomposites have not been deeply investigated. In the preset work, attempts have been made to prepare flexible PU/MWCNT foam samples by means of in-situ polymerization of isocyanate and ether based polyol in the presence of CNT particles. The role of CNT's sonication time upon the developed cellular microstructure (pores size and density) as well as viscoelastic characteristics and acoustic wave absorbency of the foamed nanocomposites have been studied.

2. Experimental

2.1. Materials

Toluene diisocyanate (TDI) with NCO content of 48.8% was provided by Karoon Petrochemical Company (Tehran, Iran), and ether based polyol supplied by Kumho Petrochemical Co. Ltd., (Seoul, Korea) with the commercial name of PPG3600 having OH and acid number equal to 46 and 0.1, respectively. The used silicone surfactant and amine catalyst were Dabco DC 2585 and Dabco 33LV (Air Products, Pennsylvania, United States of America) respectively. Tin catalyst with commercial name of U28 provided by Neostann Company (Osaka, Japan) was used for the production of foam samples. Purified water was employed as chemical blowing agent. Analytical grade methylene chloride (Samsung, Ulsan, South Korea) was used as physical blowing agent. The as received CNTs was unfunctionalized with the average diameter of 9.5 nm–1.5 μm was purchased from Nanocyl Company (Namur, Belgium) with the commercial name of NC7000.

2.2. Fabrication of foam samples

In order to compare the behavior and properties of the neat PU foam with those of CNT loaded nanocomposites, samples were fabricated according to the procedure described in Ref. [19], with predetermined structural parameters such as density and porosity ($\rho_D = 20\frac{kg}{m^3}$ and $\phi_D = 0.95$). Table 1 demonstrates the composition and weight percent of the constituents used for the preparation of

Table 1
Composition and part by weight of reactants for the production of neat PU foams.

	Polyol	Isocyanate	Distilled water	Amine catalyst	Silicone surfactant	Tin catalyst	Physical blowing agent	Glycerin
Part by weight	100	85.38	7	1.5	1.5	0.25	8	3.5

samples. The polyol was first mixed with water, silicone surfactant, amine catalysts, glycerin, and the mix subjected was subjected to intense mechanical mixing with the rate of 1300 rpm for 7 min. Dehydrated methylene chloride and tin catalyst were then fed into the mixture and mixing was continued for 30 s. Afterwards, the isocyanate solution was poured onto the prepared polyol mixture followed by 15 s mixing. The mixture was transferred as one shot procedure onto the cavity of the mold with the dimension of 25 cm × 25 cm × 10 cm and left at room temperature to obtain free rise PU foam. The molded foam samples were subjected to mechanical crushing after removal from the mold.

For the production of CNT based nanocomposite foams, the as received dried CNT with a fixed weight percent was incorporated into the vacuum dehydrated polyol and subjected to mechanical mixing for 7 min, and then sonication process was imposed by probe ultrasonic sonicator. The as prepared polyol/CNT solution was used to synthesize the nanocomposite foams similar to the procedure described for the neat or blank foam fabrication. In all prepared nanocomposites, the level of CNT was kept at 0.1 wt% based on the published works [9,11]. The used sonicator device was probe ultrasonic sonicator manufactured by BANDELIN Company (Berlin, Germany) with the commercial name of SONOPULS HD 3200 which works at frequency of 20 kHz with 200 W power.

To ensure the repeatability of foam production, each individual sample was produced by three times of molding under constant condition, and results were varianced using the following relationships.

$$\begin{aligned} Max\ Variations\ of\ Results &= Average\ of\ Results + \frac{Maximum\ of\ Results - Minimum\ of\ Results}{2 \times \sqrt{Count\ of\ Results}} \\ Min\ Variations\ of\ Results &= Average\ of\ Results - \frac{Maximum\ of\ Results - Minimum\ of\ Results}{2 \times \sqrt{Count\ of\ Results}} \end{aligned} \quad (1)$$

The collapse of the pores in the structure of PU foams has been known as a common phenomenon in the production of the foam samples. To resolve this problem, various instructions have been proposed in literature [20–22], including vacuum, feeding chemical components into the reaction, and crushing. In our present work, the collapse phenomena was controlled and prevented by using glycerin as cell opener together with applying mechanical crushing. However, MWCNT's behave as cell opener, which allows to lower the extent of mechanical crushing.

2.3. Samples preparation

In order to study the effect of CNT sonication time on the properties of PU foam nanocomposites, six sets of unfilled foams as control and nanocomposite foam samples comprising MWCNT's sonicated separately for 5, 10, 20 and 40 min were prepared. Moreover, as mentioned in Section 2.3, at least three similar samples were selected from each set. The characteristics of all nanocomposite foam specimens were compared with that of the neat or unfilled foamed samples.

2.4. Characterization

The bulk density (volume divided by mass) of the synthesized foam samples was evaluated according to the Standard of ASTM D3574. At least 30 test pieces were cut from each foam sample and density values were averaged. Flow resistivity of the prepared samples evaluated by using flow resistivity meter instrument (Shirley Development Ltd., Manchester, United Kingdom). To analyze the cellular structure, scanning electron microscope model AIS2100 from Seron Technologies Inc. (Gyeonggi-do, Korea) was performed on gold coated surfaces of samples. For this purpose, the foam samples were cryo-fractured under liquid nitrogen, and then sputtered with gold vapor under vacuum.

The Young modulus of different samples were measured according to the standard ISO 3386 using an Instron test machine (Instron Co., Massachusetts, United States of America). The test was carried out at the speed of 80 mm/min and samples were compressed to 70% of their original thickness. Also, dynamic viscoelastic properties including variation of loss modulus and loss factor versus frequency and temperature were examined according to the standard ASTM D4065-12 by utilizing the DMTA machine (Triton Tech. Ltd., Lincolnshire, UK). To evaluate the potential of the prepared nanocomposite foams to attenuate the sound wave energy, the acoustic absorption coefficient of each individual sample was measured according to the ISO-10534-2 standard method using SW422 series impedance tubes (BSWA Co., China).

3. Results and discussion

3.1. Structural properties

3.1.1. Density

Results of density measurements for both control and MWCNT loaded nanocomposite samples are presented in Table 2. The density and cellular structure of foamed PU nanocomposites are influenced by a number of factors including viscosity and ease of mobility of the PU segments during foam structurization. As seen, inclusion of non-sonicated CNT onto the PU foam composition leads to the increase in density compared with the blank foamed sample. This could be attributed to the increase of solution viscosity by the presence of CNT bundles which retards the expansion of the PU segments by the gas flow [23]. However upon sonication, the dispersion of CNT particles is improved, leading to the increase in pores nucleation, and hence increase in the number of pores which results in decrease of the foam bulk density. As the sonication is increased to 40 min, the breakdown of the CNT physical networks and hence enhancement of the CNT dispersion state results in the increase of rising time as a consequence of increase in polymer/CNT

Table 2
Comparison between bulk density foam ($\frac{kg}{m^3}$) of prepared CNT filled PU foams and blank PU foam.

Pure PU foam	23.87 ± 0.87
CNT 0 min	24.42 ± 0.57
CNT 5 min	23.37 ± 0.89
CNT 10 min	24.08 ± 1.08
CNT 20 min	25.60 ± 1.43
CNT 40 min	26.31 ± 1.27

interfaces. This leads to the formation of small size cells, and thus increase in the foam bulk density.

3.1.2. Microstructural characterization

For better understanding of the microstructure of the prepared PU foamed nanocomposites based on MWCNT's sonicated for various time intervals, scanning electron microscope (SEM) was performed on the samples. The obtained photo micrographs are illustrated in Fig. 1. It is seen that by increasing the sonication time, generally the average size of the cells decreases from 1252.4 μm to the 1184.3 μm, and reticulation rate of cells increases from 53% to 62% for the blank and nanocomposite foam samples evolved by 40 min sonication time. The measurement of the size and reticulation rate were done using ImageJ Software and results were averaged for several images of each samples.

3.1.3. Flow resistivity

Flow resistivity is one of the main characteristics of porous media which is defined as the ratio of the pressure differential across the sample surfaces to the normal air flow velocity [24]. In another words, the higher value of flow resistivity is indicative of the lower air permeability. By measuring this parameter, the acoustical characteristic of foam samples could be analyzed. Table 3 illustrates the average values of the measured flow resistivity for various prepared nanocomposite foam samples.

It is observed that, the values of flow resistivity increases by increasing the CNT's sonication time, implying the retardation of the air flow velocity through the foam sample. This is explained to be due to the improved dispersion state of the CNT and enhanced surface to volume ratio by increasing the sonication time. This would result in increase of the cell nucleation within the composite matrix, and consequently reduction of the cell size and increase of the reticulation rate.

3.2. Mechanical properties

The extent of sound energy dissipation of polymer/filler nanocomposite is influenced by the dynamic of the polymer chain segments and consequently ease of dipoles polarization which is governed by the number of polymer/filler interfaces. Therefore, the mechanical properties of a nanostructured composite could be used as a tool to characterize its potential for sound energy attenuation. Compressive stress-strain curves for blank and CNT's filled PU foam samples are illustrated in Fig. 2.

As seen, there exist three regions including pseudo solid like or linear, rubbery like, and self-reinforcing which is the result of entropy reduction at high strains. Similar behavior has also been reported for flexible reinforced PU foams by other scientists [9,19]. The slope of the linear region represents the apparent compressive elastic modulus of the composite. However, after removal of the trapped air bubbles from the foam cells by the applied stress, the samples exhibit a high rubbery like strain behavior as a result of easily deformation of the foam structure. At high strain amplitude the molecular dynamic of the polymer segments is limited by the applied stress, and cells are almost totally collapsed, leading to the decrease of entropy, and hence self-reinforcing behavior by the samples.

Nanocomposites originated by higher sonicated CNT's exhibited higher mechanical reinforcement which is attributed to the increase of the PU/CNT interfaces as a result of improved CNT dispersion state. Moreover, the area under stress-strain diagram of nanocomposites based on highly sonicated CNT is increased, implying higher stored elastic energy which helps the sample to return back to its original dimensions after the removed of the stress.

Table 4 illustrates the Young elastic modulus of the prepared PU/CNT foamed samples measured from the slope of the linear region of their corresponding stress-strain curve. As can be seen, the Young modulus decreased by increasing the CNT sonication time. This is explained to be due to the change in the microcellular structure of the foam such as decrease in cells size and increase of density by increasing the sonication time, and hence less air trapped within the pores of the nanocomposite foam sample. This is consistent with the SEM photo micrographs of the foamed nanocomposites as illustrates in Fig. 1.

3.3. Dynamic mechanical thermal analysis (DMTA)

For better understanding of the molecular dynamic and viscoelastic characteristics of the nanostructured PU/CNT foam samples, dynamic mechanical thermal analysis was performed on prepared samples. In this experiment, variation of loss modulus as well as damping factor of the samples within a wide range of temperature (temperature sweep) was examined, and results are presented in Figs. 3 and 4, respectively.

As can be seen in this figure, incorporation of CNT onto the composition of the PU foams has led to the decrease of mechanical

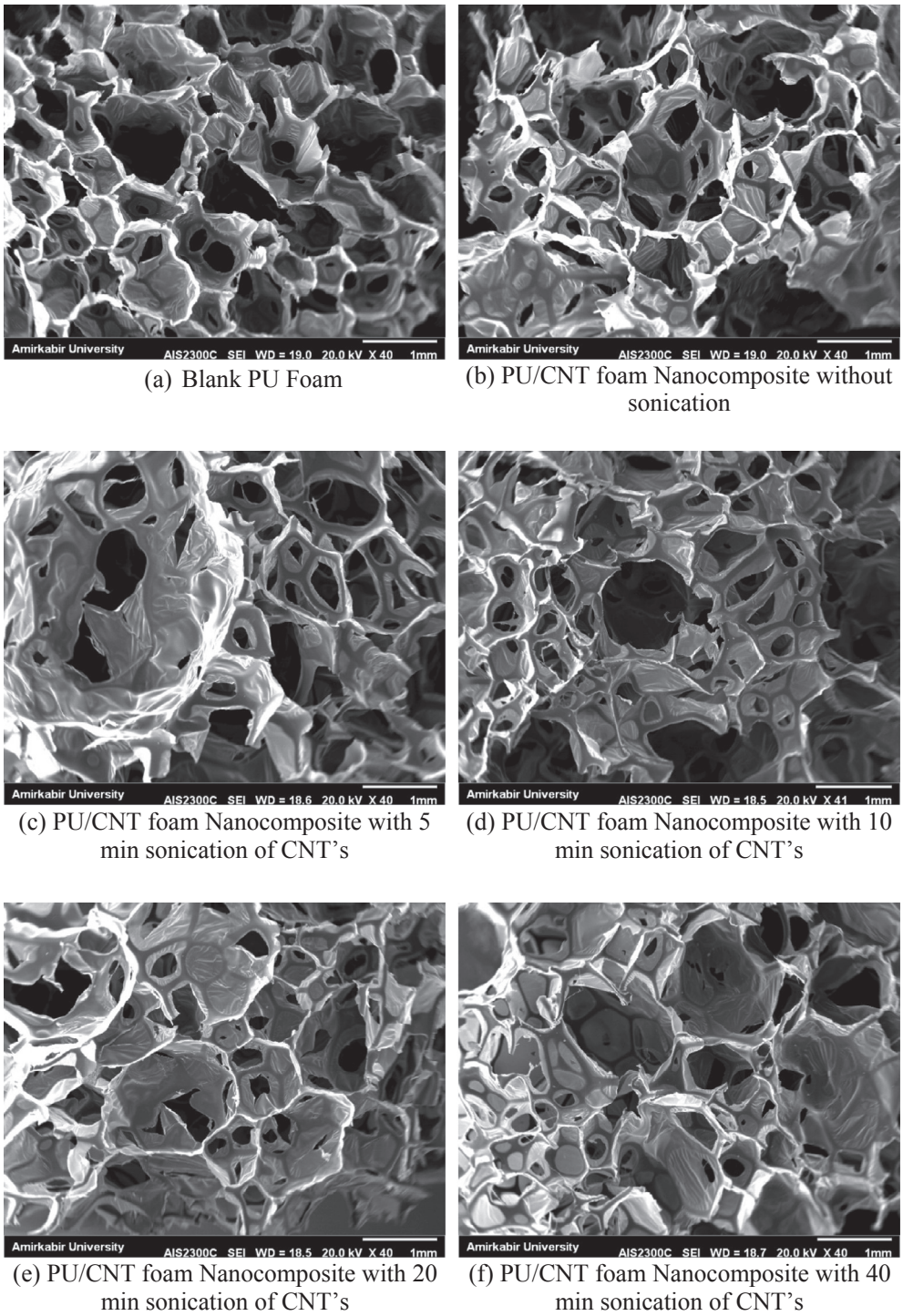

Fig. 1. SEM images of (a) blank PU foam, (b) PU/CNT foam nanocomposite without sonication of CNT's, (c) PU/CNT foam nanocomposite with 5 min sonication of CNT's, (d) PU/CNT foam nanocomposite with 10 min sonication of CNT's, (e) PU/CNT foam nanocomposite with 20 min sonication of CNT's and (f) PU/CNT foam nanocomposite with 40 min sonication of CNT's.

damping behavior compared to the blank PU foam sample especially in ambient temperature region, indicating the reduction of the viscous motions of the PU segments as a result of enhanced interactions with CNT particles. However, by increasing the sonication time to 20 min, the viscous characteristics decreases which is explained to be due to the reduction of pore sizes as well as better dispersion of the CNT particles. Nevertheless, the PU/CNT foam sample originated from 40 min sonicated CNT exhibits higher damping feature which could be attributed to the increase of the cells number as a result of higher CNT dispersion, and hence more

Table 3
Average values of flow resistivity measurement for the fabricated PU foam samples.

Sample	$\left(\frac{N \cdot s}{m^4}\right)$
Pure PU foam	4650.42 ± 322.83
Nanocomposite foam with without sonication	4965.89 ± 378.86
Nanocomposite foam with 5 min sonication	4981.81 ± 304.09
Nanocomposite foam with 10 min sonication	5204.12 ± 353.77
Nanocomposite foam with 20 min sonication	5360.68 ± 471.57
Nanocomposite foam with 40 min sonication	5716.84 ± 740.44

voids nucleation. This is consistent with the increase of the density measured for the foam composites by increasing the sonication time to 40 min (Table 2).

Moreover, when the cells number increases the retardation time (λ) spectrum would become wider, leading to the higher time dependency for the PU segments to respond to the applied dynamic field.

3.4. Acoustic properties

Acoustic absorption coefficient versus frequency (400–1600 Hz) for the 3 cm thick blank PU foam and corresponding nanocomposites originated by 0.1 wt% of CNT sonicated for various time durations are illustrated in Fig. 5.

All blank and PU/CNT nanocomposites, exhibit increasing in absorption coefficients increases by frequency with a nonlinear feature and the maximum absorption occurs within 1000–1300 Hz which is consistent with the results reported by others for flexible PU foams [11]. This is explained to be due to the increase in time limitation for the viscous responses by the PU segments as frequency is increased. In another words, by increasing the wave frequency, the occurrence of viscous motions by the PU matrix with viscoelastic feature becomes more out of phase with the sound wave, leading to the more dissipation of the wave energy. However, at high frequencies, the time dependent viscous motions by the PU segments will hardly happen which leads to the decrease of the wave energy dissipation and hence reduction of the absorption coefficient. It should be noticed that the PU matrix with viscoelastic characteristics has a defined relaxation time which represents the response time of the polymer, therefore the ease of time dependent viscous responses is governed by the frequency or time duration of the sound as a dynamic field. It is generally seen that by increasing the CNT sonication time the absorption coefficient increases to higher values compared to that of blank PU foam sample. This is believed to be attributed to the combined effects of pores size reduction and hence increase in tortuous paths [24], flow resistivity, and also number of PU/CNT interfaces as a result of improved CNT dispersion state. The increase of absorption coefficient by 35% for the nanocomposite based on 40 min sonicated CNT compared to the unfilled PU foam specimen is noticeable in this figure, which implies the role of CNT sonication time as a tool to optimize the acoustic absorption potential of PU/CNT foam nanocomposites. Generally speaking, the inclusion of sonicated CNT particles into the PU matrix leads to the increase of absorption coefficient. However, as PU foam nanocomposites based on sonicated CNT's exhibited lower mechano-dynamic loss factor, it seems that improved in acoustic energy absorption efficiency by CNT's sonication time is more likely caused by both increase in number and reduction in the size of the micro voids. In another words, increase in CNT's sonication time would results in increasing the number of PU-CNT interfaces which limits the number of viscous motions by the PU segments in the presence of the sound wave. Therefore the

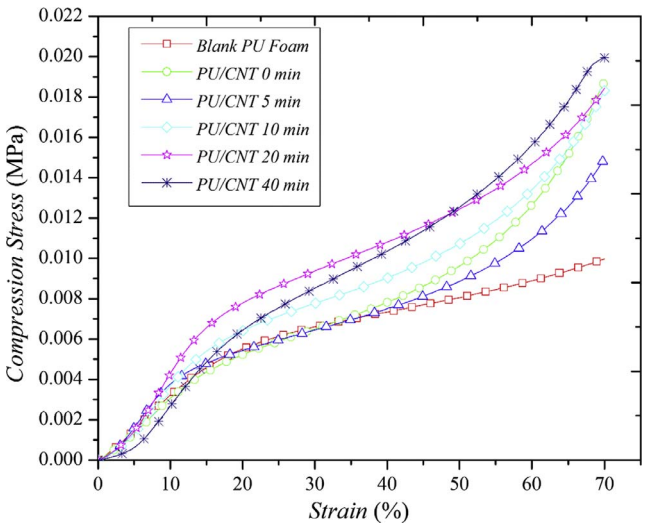

Fig. 2. Effects of sonication time upon the compression stress-strain behavior of the PU/CNT foamed nanocomposites.

Table 4
Young Elastic Modulus of blank and PU foam nanocomposites loaded with 0.1 wt% CNT's sonicated for different time duration.

Sample	Sonication time (min)	Modulus (h Pa)
Blank PU foam	0	392.153 ± 59.154
PU/CNT nanocomposite	0	379.051 ± 37.148
PU/CNT nanocomposite	5	335.843 ± 58.980
PU/CNT nanocomposite	10	319.206 ± 62.858
PU/CNT nanocomposite	20	290.219 ± 40.995
PU/CNT nanocomposite	40	278.865 ± 55.791

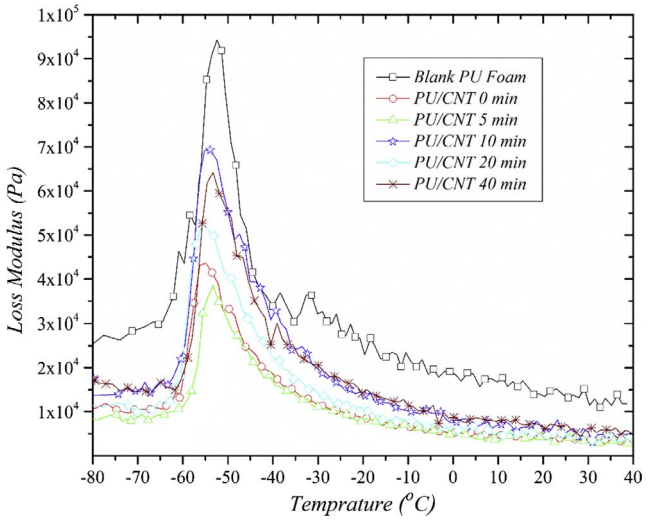

Fig. 3. Mechanical loss modulus vs temperature of blank PU foam and nanocomposite originated by CNT sonicated for various times.

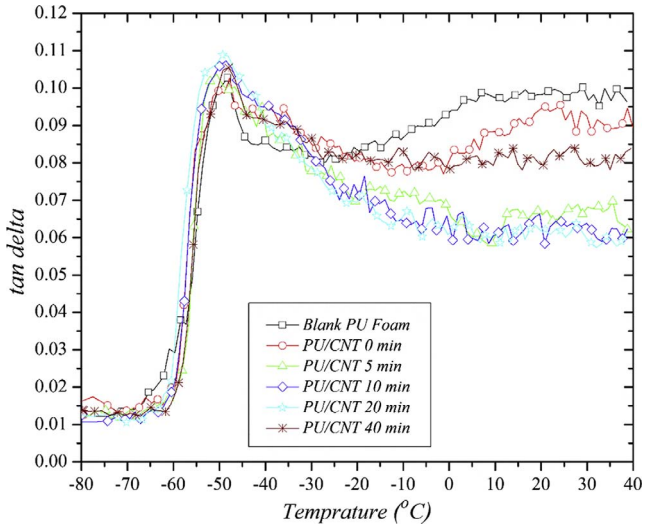

Fig. 4. Mechanical loss tangent vs temperature for blank PU foam and corresponding nanocomposites.

change in micro cellular structure of the foam by CNT sonication time is predominant in enhancing the acoustic energy dissipation. Similar results have been published for flexible PU foam nanocomposites loaded by functionalized CNT's by Bandarian et al. [9].

Fig. 6 illustrates the upper and lower limit variations of absorption coefficient versus frequency for blank PU foam and PU/CNT nanocomposite originated by 40 min sonicated CNT. As can be seen inclusion of 0.1 wt% of 40 min sonicated CNT onto the foamed PU matrix leads to remarkable improvements, the sound absorption ability within frequency range of 400–1200 Hz. This is also ascribed to the improved CNT dispersion state, and hence increase of the PU/CNT interfaces which not only results in more time limitation for the viscous responses of the PU segments, but increase in the number of voids and reduction in their sizes.

Variation of the absorption coefficient versus sound frequency within high frequency region is presented in Fig. 7. It is observed

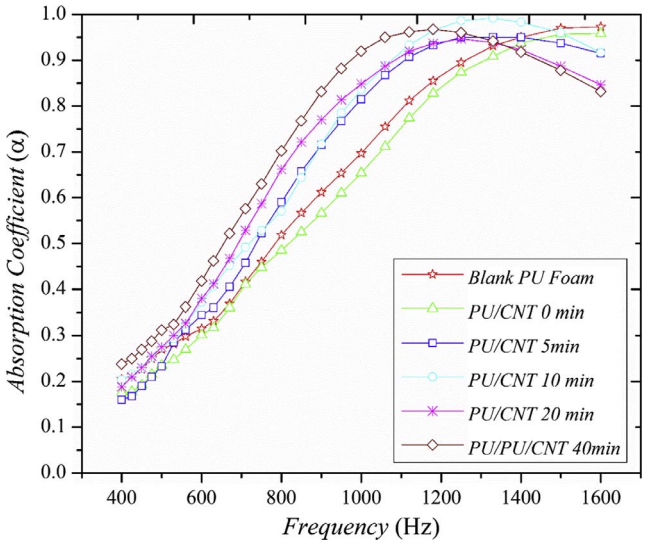

Fig. 5. Sound absorption coefficient versus frequency for the 3 cm thick blank PU foam and various PU/CNT foam nanocomposites over frequency range of 400–1600 Hz.

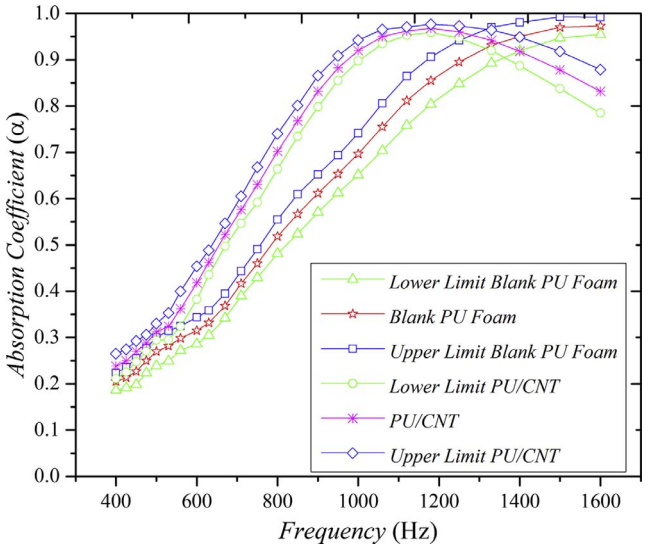

Fig. 6. Comparison between upper and lower limits variation of absorption coefficient versus frequency for 3 cm thick blank PU foam and PU/CNT nanocomposite originated by 40 min sonicated time.

that above 3000 Hz frequency, blank and PU/CNT nanocomposite exhibit similar behavior and the absorption coefficient frequency curves show plateau behavior, implying pseudo solid like characteristic by both blank and nanocomposite PU foam samples [9]. This is due to the inhibition of viscous responses by the PU segments due to the high time limitation, and hence all samples show pseudo-solid like material.

4. Conclusions

Flexible PU foams loaded with MWCNT's sonicated for various time intervals were successfully fabricated using free rising process. SEM examination showed increase in the cells nucleation within the PU microcellular structure by increasing the CNT sonication time, leading to the reduction of voids sizes, and increase in reticulation rate. Therefore, the value of flow resistivity increased by increasing the CNT sonication time. Compressive Young modulus of nanocomposite foams decreases by increasing the sonication time. Nanocomposites originated by higher sonicated CNT's exhibit higher mechanical behavior which is attributed to the increase of the PU/CNT interfaces as a result of improved CNT dispersion state. In addition, the inclusion of CNT onto the composition of the PU foams has led to the decrease of mechanical damping behavior compared to the blank PU foam samples especially within ambient temperature region, indicating the reduction of the viscous motions of the PU segments upon interactions

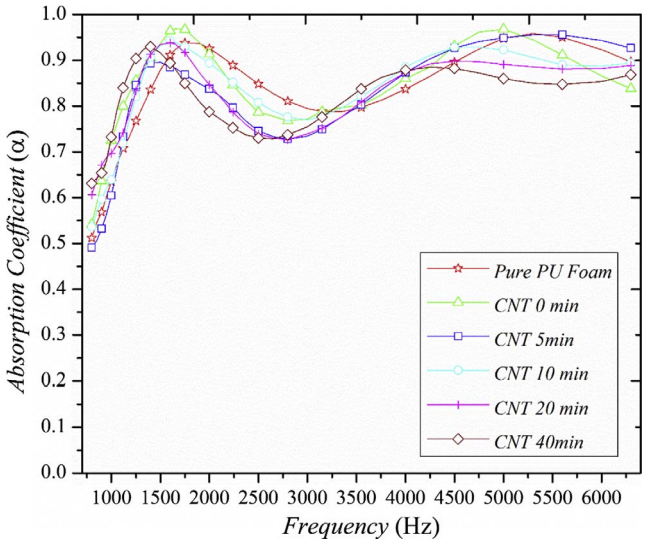

Fig. 7. Sound absorption coefficient versus frequency for 3 cm thick samples of blank PU foam and corresponding nanocomposites within frequency range of 800–6300 Hz.

with CNT particles. Acoustic absorption coefficient of PU foam nanocomposites was effectively improved by CNT sonication time, so that the foam sample based on 40 min CNT sonication time showed around 35% improvement in acoustic absorbancy compared with the blank PU sample. All obtained results led to the conclusion that increase in number and reduction in sizes of the cells by increasing the sonication time play as predominant factors for the attenuation of the sound energy which is attributed to the increase in CNT interacting surfaces rather than viscous characteristics of the PU foam nanocomposites.

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