

# Preparation and properties of polyurethane / functionalized multi-walled carbon nanotubes rigid foam nanocomposites

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**Abstract:** Surface modification of hydroxylated, multi-walled carbon nanotubes (OH-MWCNTs) was carried out by silanization with 3-aminopropyltriethoxysilane (APTS). Rigid polyurethane foam nanocomposites were prepared by silanized MWCNTs in different weight percents *via* a one shot method and the thermal, mechanical and morphological properties of the nanocomposites were investigated in detail. The obtained results from thermogravimetric analysis indicated that the thermal stability of the nanocomposites were increased by higher silanized MWCNTs contents. Morphological studies showed an increased cell density parallel to higher silanized MWCNTs contents in the polyurethane matrix. Furthermore, the tensile properties showed that the Young's moduli, as well as tensile strengths, were improved in comparison with pristine foam. The obtained data revealed reverse effects of 3 % functionalized MWCNT on the properties of respective nanocomposites due to agglomeration of MWCNTs.

**Keywords:** multi-walled carbon nanotubes, rigid polyurethane foams, nanocomposites, surface modification, 3-aminopropyltriethoxysilane.

## Otrzymywanie i właściwości nanokompozytów sztywnych pianek poliuretanowych z funkcjonalizowanymi wielościennymi nanorurkami węglowymi

**Streszczenie:** Przeprowadzono funkcjonalizację hydroksylowanych wielościennych nanorurek węglowych (OH-MWCNTs) w procesie silanizowania za pomocą 3-aminopropylotrietoksylanu (APTS). Nanokompozyty sztywnych pianek poliuretanowych wytwarzano przy użyciu różnej ilości silanizowanych MWCNTs (1,5, 2,0, 3,0 % mas.). Badano termiczne i mechaniczne właściwości uzyskanych pianek nanokompozytowych, a także ich morfologię. Wyniki otrzymane metodą analizy termogravimetrycznej wskazują, że stabilność termiczna kompozytów zwiększa się wraz ze wzrostem zawartości silanizowanych wielościennych nanorurek węglowych (Si-MWCNTs). Zwiększenie udziału zastosowanego napełniacza w matrycy poliuretanowej wpływa także na wzrost gęstości komórkowej pianki. Stwierdzono również poprawę modułu Younga oraz wytrzymałości przy rozciąganiu otrzymanych nanokompozytów. Dodatek do matrycy poliuretanowej już 3,0 % mas. silanizowanych wielościennych nanorurek węglowych powoduje pogorszenie omawianych właściwości nanokompozytów PUR/Si-MWCNTs, wynikające z obecności w matrycy poliuretanowej aglomeratów cząstek napełniacza.

**Słowa kluczowe:** wielościenne nanorurki węglowe, sztywne pianki poliuretanowe, nanokompozyty, modyfikacja powierzchni, 3-aminopropylotrietoksylan.

Currently, carbon nanotubes (CNTs) are a major topic of research due to their extraordinary mechanical, thermal, electrical, optical and electrical conductivity properties [1–4]. Carbon nanotubes are widely used as a reinforcing filler in polymer matrices to prepare high performance composites [5–8]. The agglomeration of the CNTs and poor interaction between the CNTs and matrix are two major problems that occur

during the preparation of CNT reinforced composites. Therefore, to overcome these problems, various methods and techniques have been devised. Chemical functionalization [9–12] and ultrasonic sonication [13, 14] are two effective methods that can improve the interfacial adhesion between the nanotubes and matrix. Silanization of the CNTs by silane coupling agents is another method that can be used to introduce reactive functional groups, as well as improve surface properties. Nowadays, silane coupling agents have been confirmed as effective and versatile surface treatment agents for nanoparticles, especially CNTs [15]. The general formula of silanes is given by Formula (I)

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where:  $n = 0-3$ ,  $\text{OR}'$  — the hydrolysable alkoxy group,  $\text{R}$  — the organic functional group. The organo functional group ( $\text{R}$ ) in the coupling agent causes the reaction with the polymer matrix.

Hydrolysis of the  $\text{OR}'$  groups leads to the trisilanol group, which can react with the hydroxyl groups on the surface carbon nanotubes that are already oxidized or reduced by various methods [16–18]. This method was used by Velasco *et al.* for the first time [19] where they modified the oxidized MWCNTs with 3-mercaptopropyltrimethoxysilane (MPTS). It was shown that silanization of CNTs with 3-mercaptopropyltrimethoxysilane is an effective way to lead to a better distribution of CNTs in the polymer matrix. Lee *et al.* investigated the effect of silanization of MWCNTs with 3-aminopropyltriethoxysilane (APTS) on the properties of ultra high molecular weight polyethylene (PE-UHMW) composites [20]. They showed that silanized MWCNTs have a better dispersion and interfacial adhesion in the polymer matrix. In another report, Ma and his coworkers studied the silanization of oxidized MWCNTs by using 3-glycidoxypropyltrimethoxysilane (GPTS), resulting in a good dispersion of MWCNTs in ethanol as a solvent [21].

Rigid polyurethane (PUR) foams are a member of the family of polymer foams that have a variety of applications in everyday life due to their unique properties [22]. Unfilled and non-reinforced PUR, due to its poor mechanical and thermal properties, is unsuitable for many applications. Therefore, many researchers have attempted to solve this problem in order to enhance the properties of polyurethane composites [23–26]. For example, Saha *et al.* added different types of nanoparticles at 1 wt % loading into the polymer matrix. Their results showed that the mechanical properties of nanocomposites are dramatically increased and also the decomposition temperature was improved about 18 °C in comparison with pure samples [27]. Wang studied the thermal and mechanical properties of polyurethane/amine-terminated MWCNTs composites and indicated the performance of functionalized CNT in such properties of the final product [28].

In the present study, hydroxyl functionalized multi-wall carbon nanotubes (OH-MWCNTs) were functionalized by silanization processes using 3-aminopropyltriethoxysilane (APTS) and the effect of chemical functionalization of MWCNTs evaluated on the mechanical, thermal and morphological properties of Si-MWCNTs nanocomposites.

## EXPERIMENTAL PART

### Materials

— The hydroxyl multi-walled carbon nanotubes (OH-MWCNTs) used in this work were purchased from

the Neutrino Corporation Co. (Tehran, Iran). The OH-MWCNTs had a diameter range of 10–20 nm, lengths in the range 10–30  $\mu\text{m}$ , purity >95 %, Specific Surface Area (SSA) (200  $\text{m}^2/\text{g}$ ) and 3.06 wt % OH groups according to the manufacturer's data.

— DaltoFoam TA® 14066 is a commercial polyether polyol derived from the polymerization reaction between ethylene and propylene oxide and containing all of the required additives, namely amine catalysts, surfactants, cell stabilizer and water as the blowing agent. PMDI (Suprasec® 5005) for rigid PU foam formulations was supplied by Huntsman Company. Technical data are listed in Tables 1 and 2.

**Table 1. Polyol (DaltoFoamTA® 14066) technical data**

Appearance	Viscous yellow liquid
Viscosity	5.260 Pa · s at 25 °C
Specific gravity	1.06 $\text{g}/\text{cm}^3$ at 25 °C
Water content	2.3 %
pH	12
OH number	430 ± 20 mg KOH/g

**Table 2. PMDI (Suprasec® 5005) technical data**

Appearance	Dark brown liquid
Viscosity	0.220 Pa · s at 25 °C
Specific gravity	1.23 $\text{g}/\text{cm}^3$ at 25 °C
Flash point	233 °C
Fire point	245 °C

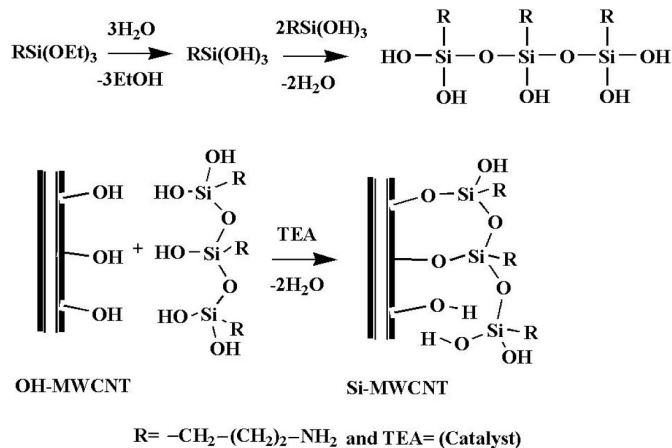
— 3-Aminopropyltriethoxysilane (wetting area 353  $\text{m}^2/\text{g}$ ), triethylamine (TEA), ethanol, acetone were supplied from Merck and used as received without further purification.

### Silanization of OH-MWCNTs with 3-aminopropyltriethoxysilane (APTS)

OH-MWCNT (1 g) was dispersed in ethanol (250  $\text{cm}^3$ ) with an ultra probe for 5 min. Then, distilled water (3  $\text{cm}^3$ ), APTS (3  $\text{cm}^3$ ) dispersed in ethanol (50  $\text{cm}^3$ ) and TEA (1  $\text{cm}^3$ ) were dropped into the suspension sequentially. The mixture was stirred at 900 rpm for 3 h. The final product was then filtered, washed with ethanol and acetone three times and dried in a vacuum desiccator at 80 °C for 24 h. A schematic illustration of the silanization process is shown in Scheme A

### Preparation of PUR foam nanocomposites

For the preparation of nanocomposites: first, different amounts of silanized MWNT — 1.5, 2.0 and 3.0 wt %, were added to the polyol matrix and sonicated in an ultrasonicator (IKA® T25 Digital) for 10 min to achieve



Scheme A

a uniform mixture. Then, the mixture was transferred into a paper cup with a volume of 300 cm<sup>3</sup> and open mold. In the next step, PMDI was added to the polyol blend and hand mixed. The foam reactivity parameters were recorded simultaneously using a digital timer as shown in Table 3. The neat PUR (polyurethane rigid) foam composite was also prepared using the same procedure for comparison purposes. The cream time of the nanocomposites was longer than pure PUR. This is due to the fact that -NCO groups of isocyanate quickly react with OH groups of polyol to form the PUR product. The addition of nanotubes to the reaction mixture reduces the effective concentration of the functional groups of components (hydroxyl or isocyanate), thus the rate of the reaction decreases and there is an increased reaction time.

Table 3. Reactivity parameters of PUR foam nanocomposites

Weight percent of Si-MWCNTs	Cream time, s	Rising time, s	String time, s	Tack free time, s
0.0	8	196	200	141
1.5	29	143	149	163
2.0	26	118	173	187
3.0	18	173	186	134

Furthermore, the higher amounts of Si-MWCNT in the PUR matrix, and larger numbers of functional groups on the nanotube surfaces, leads to an increase in the reaction rate between the amine functional groups (-NH<sub>2</sub>) on the MWCNT surface and PUR components, which led to increments in rising as well as stirring times. In other words, a decreasing trend in the cream time is seen from 1.0 to 3.0 wt % silanized MWCNT loading. After foaming, the test samples were cured at room temperature for 24 h. Finally, to prepare samples for mechanical and thermal testing, the skin layers of the molded foam block were cut

off to remove the influence of the non-uniform skin layers on the mechanical and thermal properties. The weight ratio of polyol to diisocyanate for all nanocomposites was 10 to 12.

### Methods of testing

– Fourier transform infrared spectrometer (FT-IR, Bruker Tensor 27) was used to evaluate the interfacial interaction between Si-MWCNT and PUR matrix, as well as to confirm the functionalization of OH-MWCNTs.

– Thermal stability of the nanocomposites was measured by a Thermogravimetry Analyzer (TGA, DuPont TGA951) from room temperature to 800 °C with a heating rate of 7 deg/min under N<sub>2</sub> (25 °C–600 °C) and O<sub>2</sub> (600 °C–800 °C) atmospheres.

– The morphology of the samples were examined under a Field Emission Scanning Electron Microscope (FE-SEM) (S4160 Hitachi, Japan) with an accelerating voltage of 20 kV. The fracture surfaces were coated with a thin layer of gold by the RF-sputtering method.

– Tensile tests were done using a Hegewald & Peschke measuring instrument according to ASTM D412. To ensure the reliability of the test results, at least four specimens of each sample were analyzed.

## RESULTS AND DISCUSSION

### FT-IR spectroscopy

The FT-IR spectra are shown in Fig. 1. In the OH-MWCNT spectrum, the bands at 3428, 2922, 2855, 1630, 1382 and 1056 cm<sup>-1</sup>, are attributed to O-H stretching, C-H stretching (Asym), C-H stretching (Sym), quinone groups, bending in alcoholic groups (C-OH) and C-O stretching, respectively. After silanization, the peak corresponding to C-OH bending almost disappeared and the peak intensity of O-H stretching was reduced, which are indications of successful silanation. The peak at around 1065–1090 cm<sup>-1</sup>, related to Si-O stretching, is not clearly seen due to overlap with other groups [19, 21, 29].

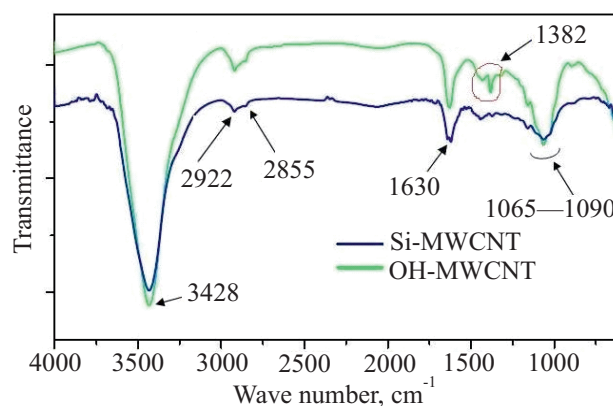


Fig. 1. FT-IR spectra of OH-MWCNT and Si-MWCNT



### Analysis of morphology

The cell density for both nanophased and neat PUR are given in Table 4. The cell density ( $N_f$ ) is calculated using the following equation [30]:

$$N_f = (n M^2 / A)^{3/2} \quad (1)$$

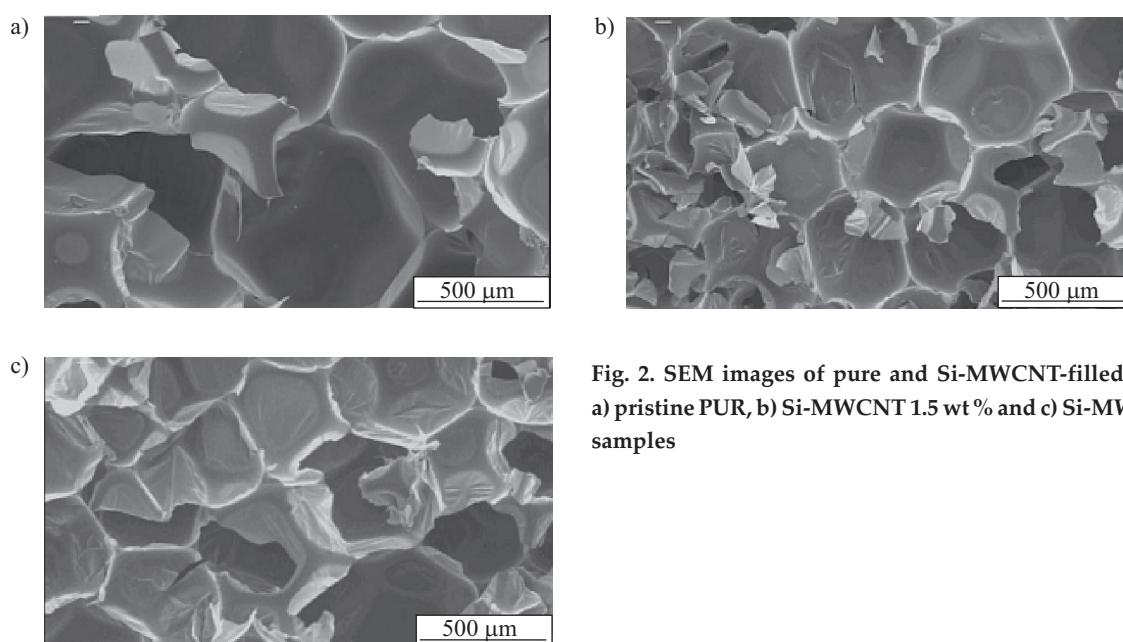
where:  $n$  – the number of cells,  $A$  – the area of the micrograph ( $\text{cm}^2$ ),  $M$  – the magnification factor.

SEM images of the samples are shown in Fig. 2 with a magnification factor of 60. With an increased number of MWCNT particles, the cell density increased and, as a result, the cell size decreased. This cell density increase is due to nucleation and growth phenomena during the

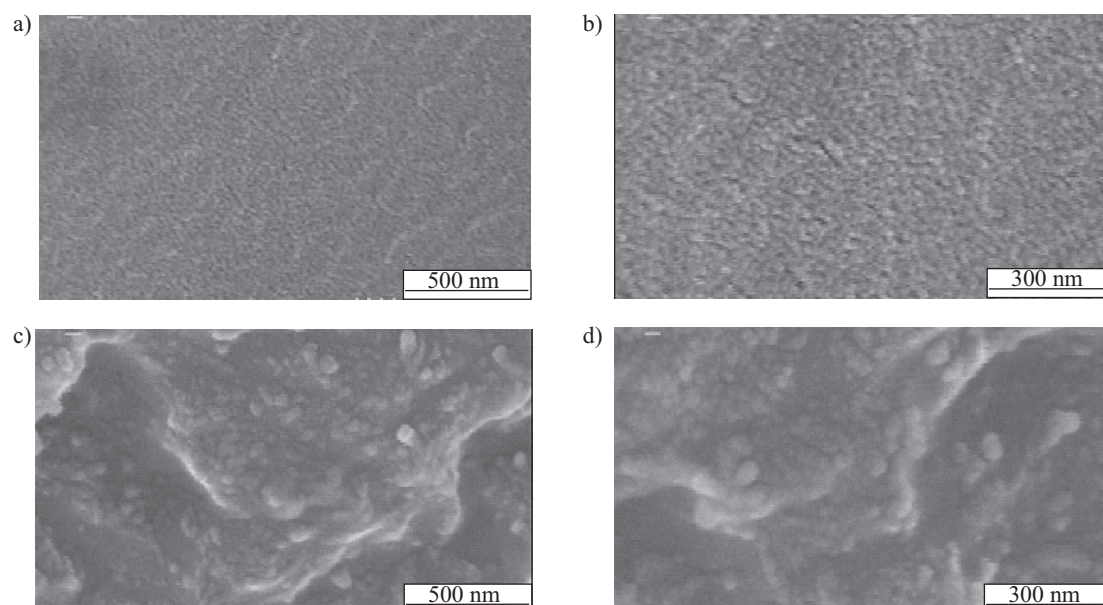
**Table 4.** Cell density of PUR foam nanocomposites

Weight percent of Si-MWCNTs	0.0	1.5	3.0
Cell density $\times 10^4$ , cell/ $\text{cm}^3$	1.10	4.45	4.90

foaming process. A small amount of well-dispersed nanoparticles in the polymer may serve as nucleation sites to facilitate the bubble nucleation process [31]. Figure 3 shows the distribution of the MWNTs in PUR foam nanocomposites. It can be observed from Fig. 3c and d that the PUR containing 3.0 wt % Si-MWCNT was severely agglomerated. However, the high surface energy between nanoparticles leads to agglomeration. These agglomera-



**Fig. 2.** SEM images of pure and Si-MWCNT-filled PUR foams: a) pristine PUR, b) Si-MWCNT 1.5 wt % and c) Si-MWCNT 3 wt % samples



**Fig. 3.** SEM microphotographs of PUR foam nanocomposites: a) Si-MWCNT 1.5 wt %, b) is a magnified image of (a) (at  $\times 100\,000$ ), c) Si-MWCNT 3 wt % and d) is a magnified image of (c) (at  $\times 100\,000$ )

ted nanoparticles act as defects and can have a detrimental effect on polymer performances [14].

### TGA analysis

Figure 4 shows the TGA curves for OH-MWCNT and Si-MWCNT, respectively. The thermogram of OH-MWCNT shows a weight loss of about 3.0 % (in agreement with the manufacturer's data) in the temperature range of 512–714 °C, which is attributed to the number of OH groups on the surface of the MWCNTs. Ther-

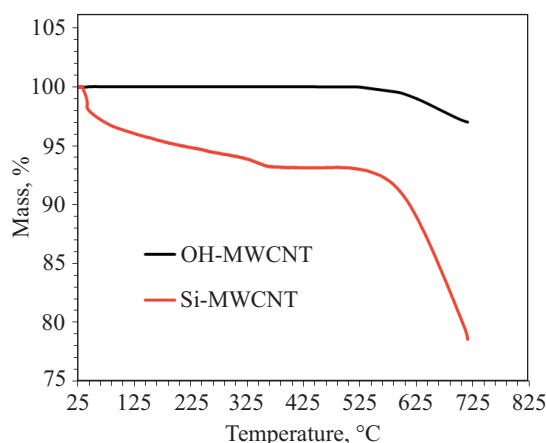


Fig. 4. TGA thermograms of OH-MWCNT and Si-MWCNT

mal degradation, in the case of PUR nanocomposites containing OH-MWCNTs after silanization by using APTS, were carried out in three steps. The first step was between 25 and 40.9 °C with a weight loss of 1.8 %, which is attributed to the evaporation of adsorbed water on the Si-MWCNT surfaces [32] and the second step occurred between 40.9 and 383 °C with a weight loss of about 5 %, which is proportional to the amount of coupling agent that coated the MWCNTs. Therefore, the surface coating of the OH-MWCNTs was confirmed by the TGA as used

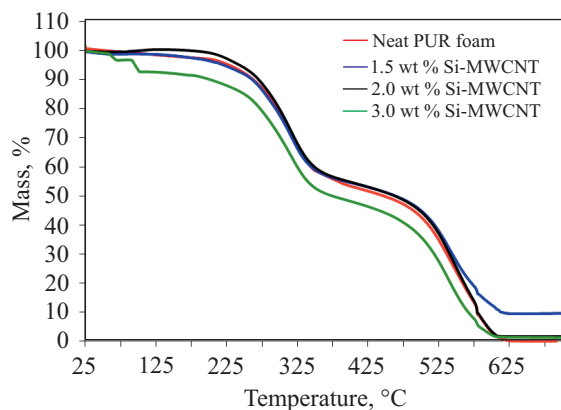


Fig. 5. TGA thermograms of neat PUR and its nanocomposites containing 1.5, 2.0 and 3.0 wt % of Si-MWNT

for the reinforcement of PUR foam matrix. The final stage was in the temperature range of 484–716 °C at which the MWCNTs structurally decomposed.

Figure 5 shows the decomposition process of neat PUR and Si-MWCNT nanocomposites. The thermal degradation of samples took place in two stages, which corresponded to the soft and hard segments, respectively [27].

The  $T_{50}$  for neat PUR matrix is observed around 447 °C that was improved to approximately 464 and 463 °C at 1.5 and 2.0 wt % Si-MWNT filled PUR nanocomposites, respectively. This indicates that the thermal stability of the PUR foam nanocomposites is improved by the addition of the Si-MWCNT to the polymer matrix and interfacial interactions between MWCNT and the PUR matrix [33]. The thermal decomposition of the nanocomposite containing 3.0 % wt Si-MWCNT indicated a weak thermal stability compared to neat PUR foam due to agglomeration of nanoparticles. The agglomeration of Si-CNTs also resulted in poor interfacial adhesion with the PUR matrix. However, due to the poor dispersion of the Si-MWCNTs in the PUR matrix, strong van der Waals forces between CNTs caused the formation of aggregates in the matrix and the thermal stability of the PUR nanocomposite was reduced [34].

### Tensile characteristics

The typical stress–strain curves for the pure PUR and PUR/Si-MWCNT nanocomposites are shown in Fig. 6. All samples showed a linear elastic behavior in the low stress region and plastic deformation in the high stress region. The tensile strength and modulus were improved in comparison with neat PUR by the incorporation of Si-MWNT into the PUR matrix. The mechanical properties of the nanocomposites are given in Table 5. The modulus and tensile strength of the 2 wt % Si-MWNT nanocomposites were 5.25 MPa and 0.53 MPa respectively, which are enhanced by about 41.5 % and 32.5 %, respectively, compared to the neat PUR.

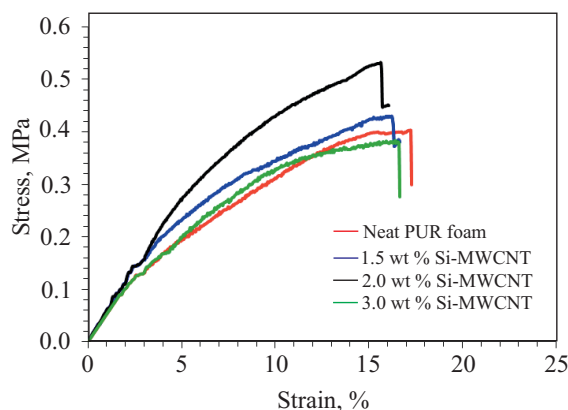


Fig. 6. Stress–strain curves of neat PUR and its nanocomposites with 1.5, 2.0 and 3.0 wt % Si-MWNT loadings

**Table 5. The mechanical properties of neat PUR and its nanocomposites**

Weight percent of Si-MWCNTs	Apparent density g/cm <sup>3</sup>	Young's modulus MPa	Tensile strength MPa	Elongation at break, %
0.0	4.447×10 <sup>-2</sup>	3.71	0.40	17.25
1.5	4.255× 10 <sup>-2</sup>	4.46	0.43	15.93
2.0	4.363× 10 <sup>-2</sup>	5.25	0.53	15.20
3.0	3.673× 10 <sup>-2</sup>	3.82	0.38	16.50

Enhancement of the mechanical properties of the composites is attributed to the uniform distribution of MWCNTs in the polyurethane matrix, and the strong interfacial interaction between the MWCNTs and the polyurethane matrix [23]. It can be seen from Fig. 3 that the sample containing 3.0 wt % Si-MWCNTs formed many clusters in the polyurethane matrix. So, due to an uneven distribution of Si-MWCNTs in the matrix, the mechanical properties of nanocomposites are degraded. A good distribution of CNTs in the matrix increases the interfacial interaction between the nanotubes and the polymer chains, which leads to the molecular restrictions of the polymer chains and results in the elongation at the break of the composites in comparison with pristine samples [35]. In addition, the increase of Si-MWCNTs in the polyurethane matrix due to the formation of nucleation sites increases the cross-linking density and so decreases the apparent density of the nanocomposites.

### CONCLUSIONS

The hydroxyl multi-walled carbon nanotubes (OH-MWCNTs) were functionalized *via* a silanization process using 3-aminopropyltriethoxysilane (APTS) in order to introduce amine functional groups (-NH<sub>2</sub>) on the MWCNT surface. Nanocomposites with different amounts of silanized MWCNT (1.5, 2.0 and 3.0 wt %) were prepared to investigate the effects of amine functionalized MWCNT on the properties of PUR foam nanocomposites. The results of FT-IR and TGA analysis confirmed the functionalization of OH-MWCNTs with 3-aminopropyltriethoxysilane. The obtained data from SEM analysis indicated that the cell density increases with increase MWCNT content because of heterogeneous nucleation of MWCNT in the PUR matrix. In addition, the Young's modulus of the PUR foam nanocomposites increased by about 20.2 and 41.5 % with 1.5 and 2.0 wt % silanized MWCNT loading, respectively, according to tensile tests. Finally, compared to neat PUR foam, thermogravimetric analysis revealed that the degradation temperature (at a 50 % weight loss) of the PUR foam nanocomposites increased by about 16.8 °C and 15.5 °C with the incorporation of 1.5 and 2.0 wt % silanized MWCNT loading, respectively. In all cases, 3.0 wt % nanocomposites indicated poor performance due to their uneven dispersion.

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### REFERENCES

- [1] Iijima S.: *Physica B* **2002**, 323, 1. [http://dx.doi.org/10.1016/S0921-4526\(02\)00869-4](http://dx.doi.org/10.1016/S0921-4526(02)00869-4)
- [2] Iijima S.: *Nature* **1991**, 354, 56. <http://dx.doi.org/10.1038/354056a0>
- [3] Popov V.N.: *Mater. Sci. Eng., R* **2004**, 43, 61. <http://dx.doi.org/10.1016/j.mser.2003.10.001>
- [4] Dresselhaus M.S., Dresselhaus G., Saito R.: *Carbon* **1995**, 33, 883. [http://dx.doi.org/10.1016/0008-6223\(95\)00017-8](http://dx.doi.org/10.1016/0008-6223(95)00017-8)
- [5] Spitalsky Z., Tasis D., Papagelis K., Galiotis C.: *Prog. Polym. Sci.* **2010**, 35, 357. <http://dx.doi.org/10.1016/j.progpolymsci.2009.09.003>
- [6] Coleman J.N., Khan U., Blau W.J., Gun'ko Y.K.: *Carbon* **2006**, 44, 1624. <http://dx.doi.org/10.1016/j.carbon.2006.02.038>
- [7] Thostenson E.T., Ren Z., Chou T.W.: *Compos. Sci. Technol.* **2001**, 61, 1899. [http://dx.doi.org/10.1016/S0266-3538\(01\)00094-X](http://dx.doi.org/10.1016/S0266-3538(01)00094-X)
- [8] Ajayan P.M., Schadler L.S., Giannaris C., Rubio A.: *Adv. Mater.* **2000**, 12, 750. [http://dx.doi.org/10.1002/\(sici\)1521-4095\(200005\)12:10<750::aid-adma750>3.0.co;2-6](http://dx.doi.org/10.1002/(sici)1521-4095(200005)12:10<750::aid-adma750>3.0.co;2-6)
- [9] Wang Y., Iqbal Z., Malhotra S.V.: *Chem. Phys. Lett.* **2005**, 402, 96. <http://dx.doi.org/10.1016/j.cplett.2004.11.099>
- [10] Zhu Z.Z., Wang Z., Li H.L.: *Appl. Surf. Sci.* **2008**, 254, 2934. <http://dx.doi.org/10.1016/j.apsusc.2007.10.033>
- [11] Gójny F.H., Nastalczyk J., Roslaniec Z., Schulte K.: *Chem. Phys. Lett.* **2003**, 370, 820. [http://dx.doi.org/10.1016/S0009-2614\(03\)00187-8](http://dx.doi.org/10.1016/S0009-2614(03)00187-8)
- [12] Ma P.C., Siddiqui N.A., Marom G., Kim J.K.: *Composites, A* **2010**, 41, 1345. <http://dx.doi.org/10.1016/j.compositesa.2010.07.003>
- [13] Ryszkowska J., Jurczyk-Kowalska M., Szyborski T., Kurzydłowski K.J.: *Physica, E* **2007**, 39, 124. <http://dx.doi.org/10.1016/j.physe.2007.02.003>
- [14] Kabir M.E., Saha M.C., Jeelani S.: *Mater. Sci. Eng., A* **2007**, 459, 111. <http://dx.doi.org/10.1016/j.msea.2007.01.031>
- [15] Bledzki A.K., Gassan J.: *Prog. Polym. Sci.* **1999**, 24, 221. [http://dx.doi.org/10.1016/S0079-6700\(98\)00018-5](http://dx.doi.org/10.1016/S0079-6700(98)00018-5)
- [16] Scheibe B., Borowiak-Palen E., Kalenczuk R.J.: *Mater. Charact.* **2010**, 61, 185. <http://dx.doi.org/10.1016/j.matchar.2009.11.008>
- [17] Datsyuk V., Kalyva M., Papagelis K., Parthenios J., Tasis D., Siokou A., Kallitsis I., Galiotis C.: *Carbon* **2008**, 46, 833. <http://dx.doi.org/10.1016/j.carbon.2008.02.012>
- [18] Avilés F., Cauich-Rodríguez J.V., Moo-Tah L., May-Pat A., Vargas-Coronado R.: *Carbon* **2009**, 47, 2970. <http://dx.doi.org/10.1016/j.carbon.2009.06.044>
- [19] Velasco-Santos C., Martínez-Hernández A.L., Lozada-Casou M., Alvarez-Castillo A., Castano V.M.: *Nanotechnology* **2002**, 13, 495. <http://dx.doi.org/10.1088/0957-4484/13/4/311>
- [20] Lee J.H., Kathi J., Rhee K.Y., Lee J.H.: *Polym. Eng. Sci.* **2010**, 50, 1433. <http://dx.doi.org/10.1002/pen.21682>



- [21] Ma P.C., Kim J.K., Tang B.Z.: *Carbon* **2006**, *44*, 3232. <http://dx.doi.org/10.1016/j.carbon.2006.06.032>
- [22] Klemptner D., Frisch K.C.: "Handbook of Polymeric Foams and Foam Technology" (Eds. Klemptner D., Frisch K.C.), Hanser Gardner Publications, Munich 1991, p. 454.
- [23] Sahoo N.G., Jung Y.C., Yoo H.J., Cho J.W.: *Compos. Sci. Technol.* **2007**, *67*, 1920. <http://dx.doi.org/10.1016/j.compscitech.2006.10.013>
- [24] Kuan H.C., Ma C.-C.M., Chang W.P., Yuen S.M., Wu H.H., Lee T.M.: *Compos. Sci. Technol.* **2005**, *65*, 1703. <http://dx.doi.org/10.1016/j.compscitech.2005.02.017>
- [25] Buffa F., Abraham G.A., Grady B.P., Resasco D.: *J. Polym. Sci., Part B.* **2007**, *45*, 490. <http://dx.doi.org/10.1002/polb.21069>
- [26] Koerner H., Liu W., Alexander M., Mirau P., Dowty H., Vaia R.A.: *Polymer* **2005**, *46*, 4405. <http://dx.doi.org/10.1016/j.polymer.2005.02.025>
- [27] Saha M.C., Kabir M.E., Jeelani S.: *Mater. Sci. Eng., A* **2008**, *479*, 213. <http://dx.doi.org/10.1016/j.msea.2007.06.060>
- [28] Xiong J., Zheng Z., Qin X., Li M., Li H., Wang X.: *Carbon* **2006**, *44*, 2701. <http://dx.doi.org/10.1016/j.carbon.2006.04.005>
- [29] Avilés F., Sierra-Chi C.A., Nistal A., May-Pat A., Rubio F., Rubio J.: *Carbon* **2013**, *57*, 520. <http://dx.doi.org/10.1016/j.carbon.2013.02.031>
- [30] Han X., Zeng C., Lee L.J., Koelling K.W., Tomasko D.L.: *Polym. Eng. Sci.* **2003**, *43*, 1261. <http://dx.doi.org/10.1002/pen.10107>
- [31] Modesti M., Lorenzetti A., Besco S.: *Polym. Eng. Sci.* **2007**, *47*, 1351. <http://dx.doi.org/10.1002/pen.20819>
- [32] Schönfelder R., Avilés F., Bachmatiuk A., Cauch-Rodriguez J.V., Knapfer M., Büchner B., Rummeli M.H.: *Appl. Phys., A* **2012**, *106*, 843. <http://dx.doi.org/10.1007/s00339-012-6787-8>
- [33] Xia H., Song M.: *J. Mater. Chem.* **2006**, *16*, 1843. <http://dx.doi.org/10.1039/b601152g>
- [34] Chen W., Tao X.: *Macromol. Rapid Commun.* **2005**, *26*, 1763. <http://dx.doi.org/10.1002/marc.200500531>
- [35] Deka H., Karak N., Kalita R.D., Buragohain A.K.: *Carbon* **2010**, *48*, 2013. <http://dx.doi.org/10.1016/j.carbon.2010.02.009>

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