

Effect of Graphene on the Sound Damping Properties of Flexible Polyurethane Foams

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Abstract: The sound absorption coefficient, airflow resistivity and cell size of polyurethane (PU) foam with graphene as a solid-type additive and tetramethylsilane (TEMS) as a liquid-type additive were investigated. The results show that the sound absorption coefficient and airflow resistivity are closely related, and that increase of the flow resistivity improves the sound absorption coefficient of the PU foam. For the PU foam/graphene (0.2 phr) composite, the values of the sound absorption coefficient and airflow resistivity were 0.52 (frequency range of 1600 to 2500 Hz) and 292,900 Ns/m⁴, respectively, which were the highest values among the investigated additive species and additive content. The sound absorption coefficient of the PU foam/graphene (0.2 phr) composite increased by 18.2% compared with that of the PU foam without graphene. The results of the sound absorption coefficient and airflow resistivity of the PU foam suggest that graphene is an effective additive in the formation of the PU foam to decrease the cell size and increase the tortuous paths of the foams, and this small cell size consequently increases the acoustic damping properties of the PU foam.

Keywords: polyurethane, foam, acoustic damping property, graphene, composite, morphology.

Introduction

Polyurethane (PU) foam is a unique polymer material having a wide range of physical and chemical properties. Major applications of PU foam are thermal insulation and structural materials such as building construction, liquefied natural gas (LNG) storage tank and refrigeration.¹⁻⁶ Another important application is the use in the design of automobile comfort to absorb sound by the acoustic damping abilities of the PU foam.⁷⁻¹¹ In a vehicle, there are several different frequency ranges, some that originate from the rotation of the engine at about 5-50 Hz, and some vibrations that emanate from the uneven surface of the road, and that are then transferred into the vehicle at about 500-3000 Hz.⁹

PU foam is a very important acoustic damping material and two major mechanisms to damp acoustic energy can be considered. First, sound energy may be absorbed by the friction between gas molecules of air oscillating inside the cells of the foam, which converts kinetic energy into heat. In this case, the cell morphology is mainly responsible for the amount of sound energy absorbed.^{12,13} Secondly, the cell wall material can absorb sound in what is known as intrinsic damping, where the sound waves propagate inside the material.^{9,12} In this case, the major factor influencing sound absorption in flexible PU

foam is the flow resistance based on the measurement of airflow resistivity.^{10,14}

Sound absorption capability of open-cell porous medium is largely affected by the microstructural parameters such as pore size and distribution.¹⁵ There were some studies to reduce the cell size through increased nucleation rate by reduced surface tension of polyol solutions using silicone-type surfactants or organoclay as a nucleating agent.¹⁶⁻¹⁸ Recently, the inclusion of carbon nanotube in PU foam has shown to improve its electrical and mechanical properties.¹⁹⁻²¹ However, studies of the improvement of acoustic damping properties for the use of carbon nanotube in flexible PU foam are very limited.^{8,11} Even these few studies reported the acoustic damping properties without correlation with the airflow resistivity of the flexible PU foam.

Silane compounds such as tetramethylsilane (TEMS) have been used as an emulsifying surfactant and also an efficient liquid-type nucleating agent in the preparation of PU foam as a thermal insulating material.^{17,18} In the improvement of the acoustic damping properties of flexible PU foam, however, studies of PU foam/graphene composite or PU foam/TEMS have not yet been reported. Therefore, the acoustic damping properties and airflow resistivity of flexible PU foam with solid-type additive such as graphene or liquid-type additive such as TEMS are worth studying, since PU foam research has so far mostly been limited to the thermal insulation or

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mechanical properties.

In this study, we synthesized flexible PU foam/graphene composite and PU foam/TEMS. In particular, we investigated the effects of graphene and TEMS additives on the acoustic damping properties and airflow resistivity of the flexible PU foam. The morphologies, sound absorption coefficients and airflow resistivities of the PU foam were measured by scanning electron microscopy (SEM), impedance tubes and flow resistivity meter, respectively.

Experimental

Materials. The materials used in this study were obtained from commercial sources. 4,4'-diphenylmethane diisocyanate (MDI) was supplied by BASF Korea Ltd. (Daejeon, Korea). The NCO content and viscosity of MDI were 32.1 wt% and 70.0 centipoise, respectively. The polyols used in this study were polyether type polyol (OH value=198 mg KOH/g), and the polyol (S-810W) was supplied from PIUSYS Co. (Paju, Korea). Distilled water was used as a blowing agent. Amine type catalyst (Dabco 2040) was supplied by Air Products and Chemicals, Inc. (Allentown, PA). Silicone-type surfactants of NIAX silicone L6900 by Momentive Co. (Waterford, NY) and Tegostab B8871 (Goldschmidt, Germany) were used in the manufacturing process of PUF, which should have an open-celled structure. Graphene was used as a solid-type additive and tetramethylsilane (TEMS) was used as a liquid-type additive. TEMS was supplied by Sigma Aldrich Korea (Yongin, Korea), and surface tension of the TEMS was 12.9 mN/m. The graphene nanoplates with thickness of 5-9 nm and average particle diameter of 3 μm were supplied by Hanwha Chemical Co. Ltd. (Seoul, Korea). Graphene was used as received, without any further treatment. Table I shows the chemical compositions of the materials used in the preparation of the polyurethane foams.

Preparation of Polyurethane Foams. First, polyols, catalyst, surfactant and liquid additive were put into a reactor, and mixed for 120 s. The rotating speed started at 300 rpm, and gradually increased to 1000 rpm. Low speed at the initial

stage of mixing is desirable for homogeneous mixing of highly viscous polyols and additives of low viscosity. The amounts of liquid additive (TEMS) added were 0.1, 0.2, 0.5, 0.7, and 1.0 parts per hundred resins (MDI and polyols) by weight (phr). After mixing, distilled water as a blowing agent was put into the polyol mixture and mixed again for 60 seconds using brushless type at 3000 rpm. Next, MDI was added into the polyol mixture, and all the reactants were mixed for 7 seconds using a brushless type stirrer at 7500 rpm. Finally, the reactants were poured into an open mold ($250 \times 250 \times 250 \text{ mm}^3$), to produce free-rise foams. The PU foam was cured for 1 day at room temperature, to prevent deterioration of the mechanical properties. For the solid-type additive (graphene) was added into the MDI, the reactants were mixed at 500 rpm, and the speed was gradually increased to 3000 rpm for 60 s. When the flexible PU foam sample is synthesized at high speed, the graphenes do not disperse homogeneously, and the graphenes aggregate with each other. The amounts of graphenes added were 0.05, 0.1, 0.2, 0.3, and 0.4 phr. Next, MDI/graphene mixture was added into the polyol solution, and all the reactants were mixed for 7 s using a brushless type stirrer at 7500 rpm. Finally, the reactants were poured into an open mold ($250 \times 250 \times 250 \text{ mm}^3$), to produce free-rise foams. The density of the PU foams prepared in this study was about 19 kg/m^3 .

Morphology. The morphologies of the PU foam with graphene or TEMS were studied by field emission scanning electron microscopy (FE-SEM) (Hitachi Model S-4300SE, Tokyo, Japan). The samples were cryogenically fractured, and the surface was coated with gold before scanning. The accelerating voltage was 15 kV. For calculation of the average size of the cells, we counted 50 cells from the obtained SEM pictures, and used image analysis software (image J) to determine the cell size of the sample.³

Surface Tension Measurements. Surface tensions of the polyol solution with or without liquid-type additive (TEMS) mixtures were measured using contact angle analyzer (model GonioStar 150, Surfactech Co., Korea). Droplets of the solutions were dropped on the surface of glass plate. After that, contact angle between droplet and glass plate was measured. The surface tension is calculated using eq. (1):²²

$$\cos \theta = 2\varnothing \left(\frac{\gamma_s}{\gamma_L} \right)^{\frac{1}{2}} - 1 \quad (1)$$

where θ is the contact angle, \varnothing the ratio of molar volume of liquid and solid which is approximated to 1.0, γ_s the surface energy of solid which is glass plate (24.8 mN/m), and γ_L the surface tension of liquid.

Acoustic Properties. The sound absorption coefficients of the PU foam with graphene or TEMS were measured using two-microphone impedance tubes (Model Two MIC 4206, Bruel & Kjaer Co., Denmark) according to ISO-10534-2 and ASTM E1050-10. The larger tube of 10 cm diameter was used to measure the absorption coefficient in the range 16-800 Hz,

Table I. Compositions of the Materials Used in the Preparation of Polyurethane Foam with Graphene and Tetramethylsilane

Chemicals	Description	Weight (g)
MDI	4,4'-Diphenylmethane diisocyanate	150.0
Polyol	Polyether Type	100.0
Surfactant	Silicone Type (NIAX L6900/B8871)	2.0 (1/1)
Blowing Agent	Water	6.0
Catalyst	Amine Type	1.5
Additive	Tetramethylsilane	0.25, 0.50, 1.25, 1.75, 2.50
	Graphene	0.12, 0.25, 0.50, 0.75, 1.00

and the smaller tube of 2.9 cm diameter was used to measure the absorption coefficient in the range 800–6,300 Hz.¹¹ The PU foam samples were cut from the prepared foams with a round cutter mounted on a milling machine. The cylinders with diameters of 10 and 2.9 cm were sliced into samples of 2.0 cm height. Five specimens per sample were measured and averaged for each sound absorption test.

Properties of Airflow Resistivity. The airflow resistivities of the PU foam with graphene or TEMS were measured using a flow resistivity meter (AFR, Autonuem, Swiss), according to ISO 9053-1991. The PUF samples were cut into cylindrical shapes of 10.0 cm diameter and 2.0 cm height. Five specimens per sample were measured and averaged for each airflow resistance test.

Results and Discussion

Cell Morphology of Polyurethane Foams. Figure 1(a)–(f) shows cell morphology of the cryogenically fractured cross-sectional surfaces of the PU foam with graphene content of 0.0, 0.05, 0.1, 0.2, 0.3, and 0.4 phr, respectively. The cell morphology of the PU foam samples were observed using scanning electron microscope. Figure 1 shows that the average cell size of the PU foam/graphene composite reached a minimum at a graphene content of 0.2 phr; and when the

graphene content was 0.0, 0.05, 0.1, 0.2, 0.3, and 0.4 phr, the cell size of the PU foam was found to be 583, 540, 488, 460, 552, and 553 μm , respectively. Figure 1 shows that when the graphene content is low, such as 0.1 or 0.2 phr, the average cell size is smaller and more uniform. When the amount of graphene is increased, the cell size in the PU foam/graphene composite is not uniform (Figure 1(f)). This may be because when the amount of graphene is large (0.4 phr), the filler tends to aggregate much more than when the amount of filler is small (0.2 phr). The aggregated fillers may not act as a nucleating agent properly, therefore, the cell size of the PU foam is not uniform and larger cells are observed. When the amount of graphene was more than 0.4 phr, the cell structure collapsed easily, therefore, cell formation of the PU foam was not able to obtain properly. From these results, it is suggested that graphene (0.2 phr) may contribute to reduce cell size of the PU foam.

Figure 2(a)–(f) shows cell morphology of the cryogenically fractured cross-sectional surfaces of the PU foam with TEMS content of 0.0, 0.1, 0.2, 0.5, 0.7, and 1.0 phr, respectively. Figure 2 shows that when the TEMS content was 0.5 phr, the cell size of the PU foam reached a minimum, which was similar to the behavior that Figure 1 shows. These results are likely due to the lower surface tensions of the polyol/TEMS mixture compared that of the polyol solution without TEMS,

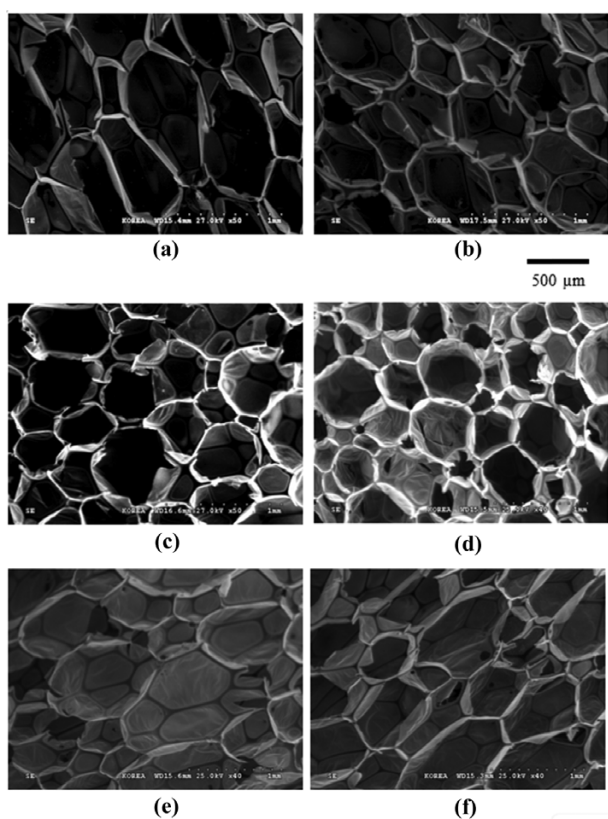


Figure 1. Scanning electron micrographs of polyurethane foam with graphene content (phr): (a) 0.0, (b) 0.05, (c) 0.1, (d) 0.2, (e) 0.3, and (f) 0.4.

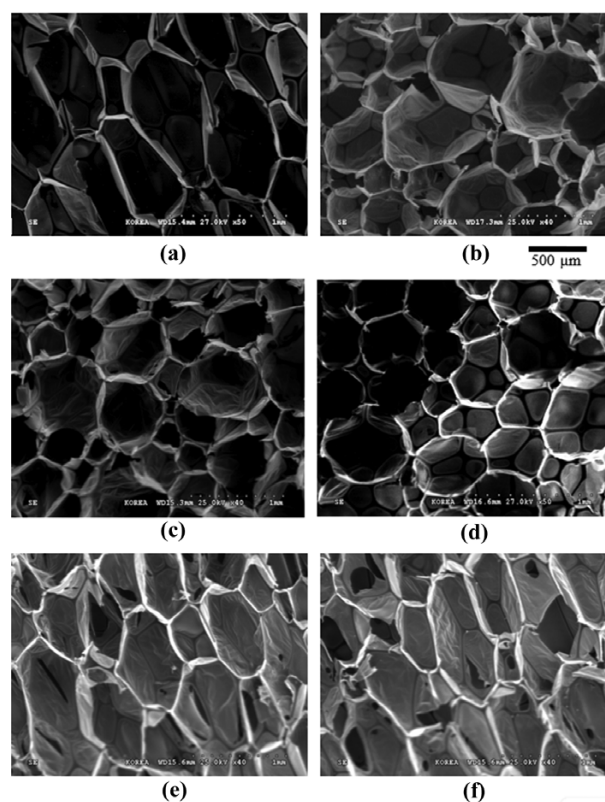


Figure 2. Scanning electron micrographs of polyurethane foam with tetramethylsilane (TEMS) content (phr): (a) 0.0, (b) 0.1, (c) 0.2, (d) 0.5, (e) 0.7, and (f) 1.0.

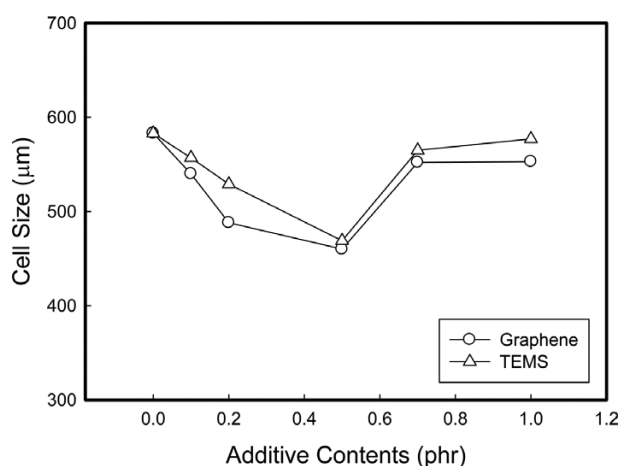
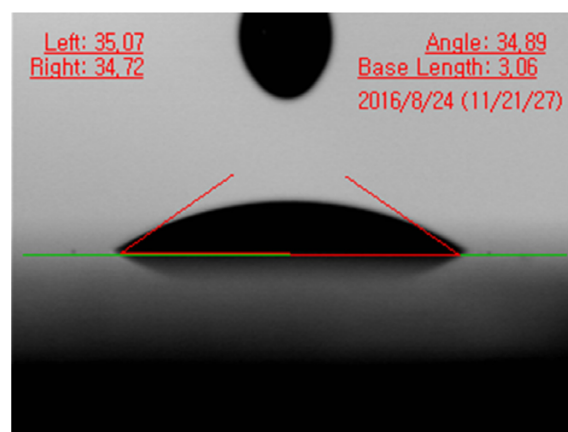


Figure 3. Average cell size of polyurethane foam with additive species and content: (○) graphene, (△) tetramethylsilane (TEMS). For the x-axis, the amount of graphene added was 0.0, 0.05, 0.1, 0.2, 0.3, and 0.4 phr.

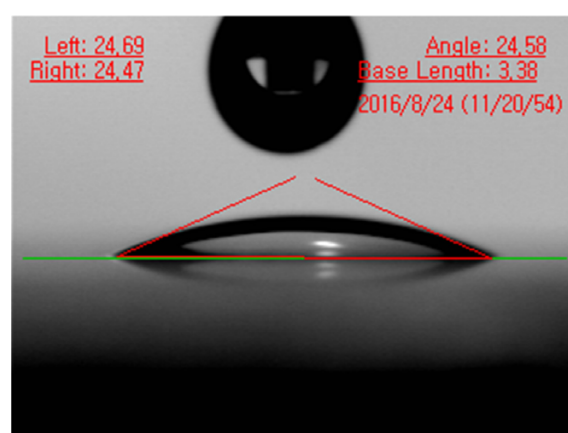
which allows it to be well-mixed in the polyol solutions and consequently a higher nucleation rate.⁵ It seems that the low-surface tension of the polyol/TEMS mixture prohibits the coalescence of bubbles and it promotes the formation of dense and small cells.¹⁵ Figure 3 shows average cell size of the PU foam with graphene and TEMS as a function of additive contents. For the x-axis in Figure 3, the amount of graphene added was 0.0, 0.05, 0.1, 0.2, 0.3, and 0.4 phr. The average cell size of PU foam/graphene composite is smaller than those of the PU foam with TEMS. From the results shown in Figure 3, it is suggested that graphene is more effective additive to reduce the cell size of the PU foam than the TEMS.

Figure 4 shows the contact angle measurements of the polyol solution without additive and the polyol/TEMS (0.5 phr) mixture which are 34.9 and 24.6 degrees, respectively. From the results of Figure 4, the surface tensions of polyol solution without additive and the polyol solution with TEMS (0.5 phr) are obtained to be 29.8 and 18.0 mN/m, respectively. When the TEMS was added, average cell size of the PU foam was getting more finer due to lower surface tension of the polyol/TEMS solution, thereby the nucleation rate and number of bubbles produced were increased and then the cell size becomes smaller. When the additives act as nucleating agents, the bubble size decreases, and the number of bubbles can increase.⁵ The results of Figures 2 and 3 suggest that the lower surface tension has affected the polyol/TEMS mixture to create enough nucleation sites and optimum additive content for the formation of PU foam with TEMS (0.5 phr).

Acoustic Damping Properties. Figure 5((a),(b)) shows the sound absorption coefficient (α) of the PU foam/graphene composite and PU foam/TEMS with the additive content and frequency, respectively. Figure 5 shows that the increase of sound absorption coefficient begins at a frequency of about 600 Hz, and continues up to about 3,100 Hz. This frequency



(a)

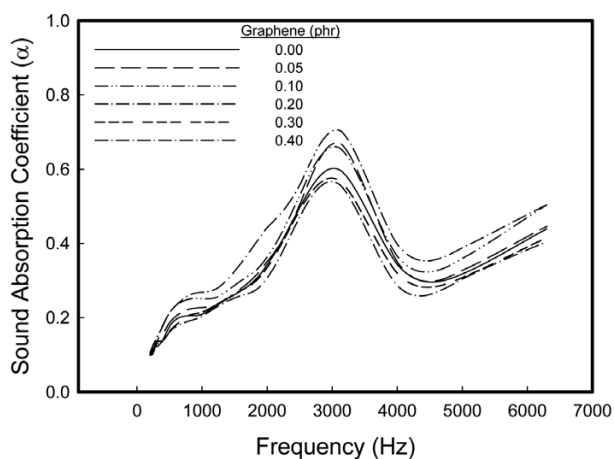


(b)

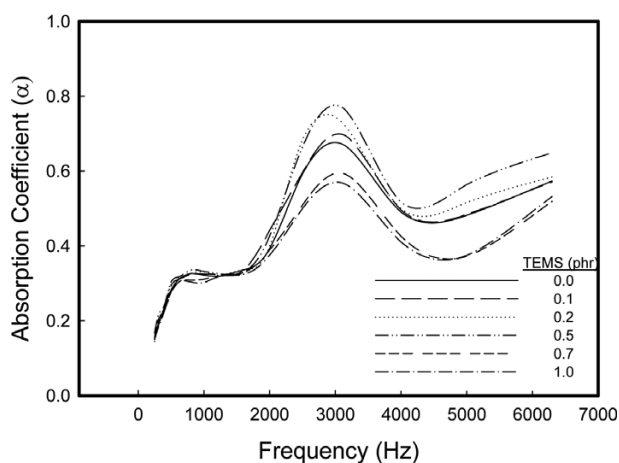
Figure 4. Measurements of contact angle of polyol and various additive mixtures: (a) polyol solution without additive, (b) polyol solution with tetramethylsilane 0.5 phr.

range includes a collection of sounds available in urban and industrial applications.¹⁰ Also, when the graphene content was 0.2 phr, a higher sound absorption coefficient was observed. For the PU foam/TEMS, a higher sound absorption coefficient was observed at the TEMS content of 0.5 phr.

Figure 6 shows the average value of sound absorption coefficient of the PU foam/graphene composite and PU foam/TEMS with additive content, which is obtained from the results of Figure 5. For the x-axis in Figure 6, the amount of graphene added was 0.0, 0.05, 0.1, 0.2, 0.3, and 0.4 phr. The values of the sound absorption coefficient shown in Figure 6 were averaged in the frequency ranges between 1,600 and 2,500 Hz, which covers the sensitive frequency region of the human ear.¹⁰ Table II summarizes the values of the sound absorption coefficient of the PU foam/graphene and PU foam/TEMS shown in Figure 6. For the PU foam/graphene composite at the content of 0.05, 0.1, and 0.2 phr, the sound absorption of the PU foam showed higher than that of the PU foam without additives (Table II). In particular, at the graphene content of 0.2 phr, the sound absorption coefficient showed their highest value compared



(a)



(b)

Figure 5. Sound absorption coefficient (α) of polyurethane foam with additive species and frequency: (a) graphene, (b) tetramethylsilane (TEMS).

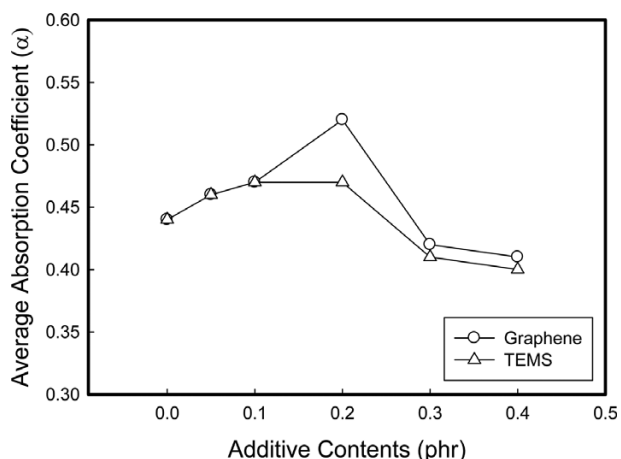


Figure 6. Average sound absorption coefficient (α) of polyurethane foam with additive species and content: (○) graphene, (△) tetramethylsilane (TEMS). For the x-axis, the amount of graphene added was 0.0, 0.05, 0.1, 0.2, 0.3, and 0.4 phr.

Table II. Average Sound Absorption Coefficient (α) of the Polyurethane (PU) Foam with Graphene and Tetramethylsilane (TEMS)

Additive Content (phr)	PU Foam/Graphene ^a	Additive Content (phr)	PU Foam/TEMS ^a
0.00	0.44	0.00	0.44
0.05	0.46	0.10	0.46
0.10	0.47	0.20	0.47
0.20	0.52	0.50	0.47
0.30	0.42	0.70	0.41
0.40	0.41	1.00	0.40

^aThe values of sound absorption coefficient were averaged in the frequency ranges between 1,600 and 2,500 Hz.

the other additive species and compositions, which were 0.52. This behavior can be explained from the morphological results of Figure 1 that the smaller and more uniform cells (at 0.2 phr) increase the sound absorption coefficient of the foams. From Table II, the sound absorption coefficient of the PU foam/graphene (0.5 phr) composite increased by 18.2% compared with that of the PU foam without graphene.

Another factor to the sound absorption that might be considered is that the sound damping effects of the PU foam/graphene composite can be attributed to the large surface of the PU foam-filler (graphene) interface, where energy can be dissipated by interfacial sliding and stick-slip behavior.^{8,23,24} In such cases, the acoustic energy directly dissipates to thermal energy. It is believed that the presence of small voids between the matrix and fillers is favorable for improving the sound absorption, due to the increase in tortuous paths of the foams.¹⁰ The results of the morphological and acoustical properties suggest that the graphene that can act as a nucleating agent in the formation of the foams decreases the cell size and increases the tortuous paths of the foams, and this small cell size consequently increases the acoustic damping properties of the PU foam/graphene composite.

For the PU foam with TEMS, the values of the sound absorption coefficients showed slightly lower than that of the PU foam/graphene composite (Table II). This may be because the cell size of the PU foam with TEMS was somewhat larger than that of the PU foam with graphene. From Table II, the sound absorption coefficient of the PU foam with TEMS (at 0.5 phr) increased by 7.0% compared with that of the PU foam without TEMS. From the results of the sound absorption coefficient of the PU foam, it is suggested that graphene is more effective additive than the TEMS.

Figure 7 shows the relation between the sound absorption coefficient and cell size of the PU foam/graphene composite and PU foam/TEMS. Figure 7 shows that the smaller cell size increased the sound absorption coefficient of the foams. This is probably because the smaller cells increased the tortuosity of the foams, thereby increasing the sound absorption

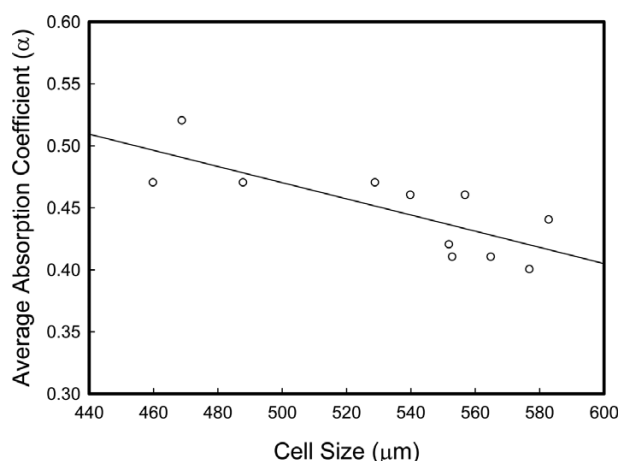


Figure 7. Relationship between average sound absorption coefficient (α) and cell size of polyurethane foams prepared at different additive content and species (graphene and tetramethylsilane).

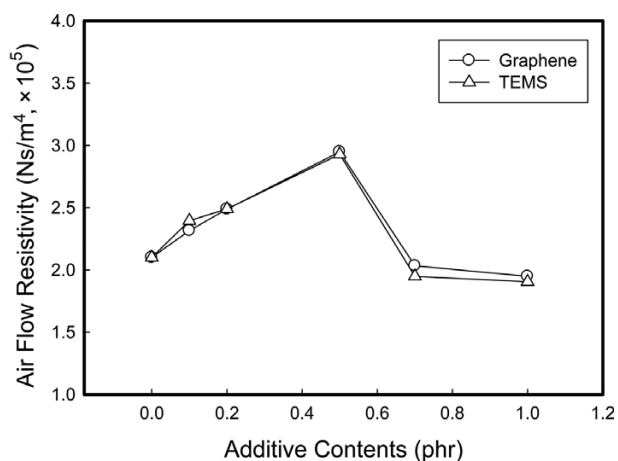


Figure 8. Airflow resistivity of polyurethane foams with additive species and content: (○) graphene, (△) tetramethylsilane (TEMS). For the x-axis, the amount of graphene added was 0.0, 0.05, 0.1, 0.2, 0.3, and 0.4 phr.

coefficient of the foam.^{10,14} This result is somewhat consistent with the result by Ghaffari Mosanenzadeh *et al.*¹⁵ that average cell sizes between 250 and 500 μm of the poly(lactic acid) open-cell foams showed the best sound absorption performance. The sound absorption coefficient and airflow resistivity are also closely related, and the flow resistivity plays a major role in the sound absorption of a foam.^{10,15,25} More detailed relation between the sound absorption coefficient and airflow resistivity is discussed in the next section.

Airflow Resistivity. Figure 8 shows the airflow resistivity of the PU foam/graphene composite and PU foam/TEMS, while Table III summarizes the airflow resistivity of the PU foam with the additive species and additive content. For the x-axis in Figure 8, the amount of graphene added was 0.0, 0.05, 0.1, 0.2, 0.3 and 0.4 phr. Flow resistivity is one of the most effective factors of the sound absorption of cell structured porous

Table III. Air Flow Resistivity (Ns/m^4) of the Polyurethane (PU) Foam with Graphene and Tetramethylsilane (TEMS)

Additive Content (phr)	PU Foam/ Graphene	Additive Content (phr)	PU Foam/ TEMS
0.00	210,000	0.00	210,000
0.05	231,500	0.10	239,500
0.10	248,800	0.20	249,000
0.20	294,800	0.50	292,900
0.30	203,400	0.70	194,800
0.40	194,900	1.00	190,500

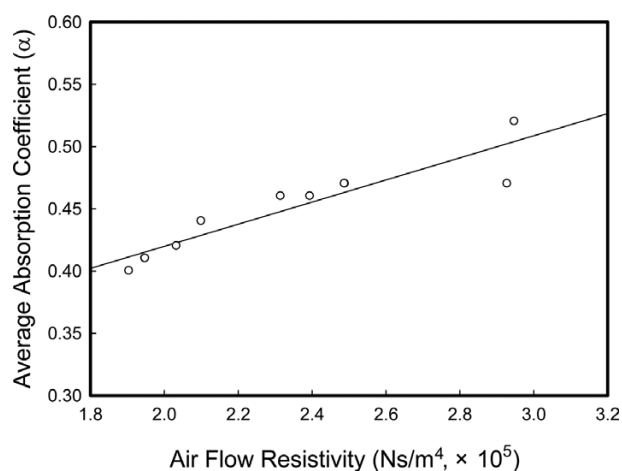


Figure 9. Relationship between average sound absorption coefficient (α) and airflow resistivity of polyurethane foams prepared at different additive content and species (graphene and tetramethylsilane).

materials. It is reported that the increase of flow resistivity improves the sound absorption property of porous materials.^{10,22} Figure 8 shows that for both the PU foam/graphene and PU foam/TEMS, the additive content of 0.2 and 0.5 phr increased the flow resistivity which were 294,800 and 292,900 Ns/m^4 , respectively (Table III). The flow resistivity of the PU foam/graphene and PU foam/TEMS was increased by 40.3 and 39.5% compared with that of the PU foam without additive. The increase in flow resistivity of the PU foam is probably because of the increase in tortuous paths by the small cells of the foams.

Figure 9 shows the relation between the sound absorption coefficient and airflow resistivity of the PU foam/graphene composite and PU foam/TEMS. Figure 9 suggests that the sound absorption coefficient and airflow resistivity are closely related. The results of morphologies, acoustic properties and airflow resistivity shown in Figures 6-9 suggest that a major contribution to increase the sound absorption coefficient is the decreased cell size of the foams, which is probably caused by the nucleating effect of the additives during the formation of the foams, which results in increasing the tortuous paths of the foams.

Conclusions

In this study, the sound absorption coefficient and airflow resistivity of the PU foam/graphene composite and PU foam/TEMS were investigated with the additive content. For the PU foam/graphene composite (0.2 phr), the values of sound absorption coefficient and airflow resistivity were 0.52 (frequency range of 1,600 to 2,500 Hz) and 294,800 Ns/m⁴, respectively, which showed the highest values investigated in this study. The sound absorption coefficient of the PU foam/graphene (at 0.2 phr) increased by 18.2% compared with that of the PU foam without graphene. This may be due to the PU foam/graphene (0.2 phr) showing the smallest cell size among the graphene compositions investigated in this study. The increased sound absorption coefficient that might also be considered is that the sound damping effects of the PU foam/graphene composite can be attributed to the large surface of the PU foam-filler (graphene) interface, where the acoustic energy directly dissipates to thermal energy by interfacial sliding and stick-slip behavior. For the PU foam/TEMS (0.5 phr), the values of sound absorption coefficient and airflow resistivity were 0.47 (frequency range of 1,600 to 2,500 Hz) and 292,900 Ns/m⁴, respectively, which showed that the sound absorption coefficient increased by 7.0% compared with that of the PU foam without TEMS.

The results of cell size, sound absorption coefficient and airflow resistivity of the PU foam with solid-type additive (graphene) and liquid-type additive (TEMS) suggest that reduction in cell size of the PU foam increases the sound absorption coefficient and airflow resistivity of the PU foam. The results of morphological, acoustic properties and airflow resistivity suggest that the graphene can act as a nucleating agent effectively in the formation of the foams, decreasing the cell size, and increasing the tortuous paths of the foams; and this small cell size consequently increases the acoustic damping properties of the PUF.

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