



Effects of PEG 3350 and PEG 8000 Molar Ratio in Polyurethane for Underwater Acoustic Attenuation Performance

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Abstract: In this study, we investigated the changes in the structural and mechanical properties of polyurethane (PU) induced by blending it with poly (ethylene glycol) (PEG) of two different molecular weights, viz., PEG 8000 and PEG 3350, at different molar ratios as soft segments. The properties of the resulting PU/PEG specimens were characterized using dynamic mechanical analysis, differential scanning calorimetry, etc. As the content of PEG 8000 increased, the Young's modulus of the blend increased by 124.8%, and its degree of crystallinity increased by 12.37%, whereas the degree of phase separation decreased by 31%. Furthermore, the PU/PEG specimens with a mixture of PEG 8000 and PEG 3350 exhibited broader and higher $\tan \delta$ peaks compared with those of the specimens with PEG of a single molecular weight. These results indicate that the use of PEG mixtures in PU leads to superior sound absorption.

Keywords: Polyurethane, PEG, crystallinity, phase separation, loss factor

Introduction

Submarines play an increasingly important role in modern naval warfare. Owing to their ability to navigate underwater, submarines are less detectable than surface vessels.¹ To minimize acoustic detectability by sound navigation and ranging (SONAR) systems, various noise-reduction design and analysis technologies have been developed and applied to submarine structures.²

Acoustic attenuation in materials can be achieved through various mechanisms, including wave redirection, scattering by inhomogeneities, mode conversion at boundaries, and intrinsic absorption via conversion to heat in viscoelastic materials.³ To develop effective sound-absorbing materials, a comprehensive consideration of all these attenuation methods is necessary. Polymer materials are widely used for underwater sound absorption due to their viscoelastic properties that convert acoustic energy into heat. The acoustic performance is determined by the impedance mismatch between the material and the surrounding medium, which is water. To reduce acoustic reflection, it's crucial to minimize this impedance difference.¹ Polymeric materials

such as PU and epoxy exhibit acoustic impedances similar to water, making them advantageous for underwater sound absorption.⁴⁻⁶ The acoustic attenuation of a material can be determined by its loss tangent ($\tan \delta$). Higher values of loss tangent at a given temperature and frequency correspond to greater acoustic attenuation.³

Polyurethane (PU) is a block copolymer with a structure of alternating soft segments (SS) and hard segments (HS). PUs exhibit microphase separation between these segments, which is influenced by factors such as monomer polarity, segment length, and the SS/HS ratio.⁷ This phase separation influences the local mobility of the soft segments and scattering at the interfaces, thereby correlating with the sound absorption performance. Polyurethane offers significant design flexibility, as its properties can be tailored by controlling a combination of factors, including the type of polyol and isocyanate, the molecular weight of the soft segment, and the choice of chain extenders or crosslinking agents.⁸ Furthermore, its excellent compatibility with various additives makes it suitable for use in composite materials, which is advantageous for achieving acoustic attenuation over a wide frequency range.⁹

Previous research on PUs as a sound-absorbing material has analyzed the effect of varying polyol molecular weights,

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but the study comparing the performance of blends of two different polyol molecular weights is difficult to find.¹⁰ Even in studies that blended two types of polyols, the influence of molecular weight alone could not be accurately determined because the polyol types were different.¹¹ Therefore, this study aimed to understand the effect of PEG molecular weight variation on sound absorption performance by blending PEGs with different molecular weights. PEG-3350 ($M_n=3350$ g/mol) and PEG-8000 ($M_n=8000$ g/mol) were used to analyze the influence of the average molecular weight and molecular weight distribution of PEG on the acoustic performance of polyurethane.

The soft segment component was categorized into two groups: polyurethane synthesized with PEG of a single molecular weight and polyurethane prepared using a mixture of PEGs with different molecular weights. The effects of varying PEG composition on the mechanical strength, viscoelastic behavior, and temperature dependent properties of the polyurethane were systematically investigated. The influence of soft-segment composition on phase separation behavior and crystallinity, and consequently on the overall physical properties, was elucidated. Based on these results, this study provides fundamental insights for the design of polyurethane-based acoustic damping materials applicable to submarine sound absorbing systems.

Experimental

1. Materials

The following chemicals were purchased from Sigma-Aldrich and used in the study: polyethylene glycol (PEG-3350, $M_n=3350$ g/mol), polyethylene glycol (PEG-8000, $M_n=8000$ g/mol), dimethylolpropionic acid (DMPA), trimethylolpropane (TMP), dibutyltin dilaurate (DBTDL), and 4,4'-methylenebis(cyclohexyl isocyanate) (H12MDI). H12MDI was dried with 4-Å molecular sieves prior to use.

2. Fabrication of PU Membranes

The PU membranes were synthesized following a procedure adapted from the previously reported methods.¹² Each chemical was prepared based on a molar ratio of 1 : 0.08 : 0.08 : 2.2 for PEG, DMPA, TMP, and H12MDI, respectively. The sample names corresponding to different PEG composition ratios are listed in Table 1. Two types

Table 1. Nomenclature Based on PEG Composition and Hard Segment Weight Percent of PUs

Name of sample	PEG3350 (mol%)	PEG8000 (mol%)	HS wt% (=X _{HS})
PU-100	100%	0%	14.69
PU-80	80%	20%	11.87
PU-40	40%	60%	8.58
PU-0	0%	100%	6.72

of PEGs with different molecular weights were dissolved and pre-mixed using a hotplate stirrer at 250 RPM for 12h. Subsequently, PEG, DMPA, and TMP were placed into a four-neck round-bottom flask equipped with an external exhibit thermometer, vacuum line, mechanical stirrer, and heating jacket. The mixture was stirred under vacuum at 85°C for 1h. A solution of H12MDI and DBTDL was added dropwise into the flask, and the reaction was continued for 3.5h at 85°C. The synthesized PU was dried in an oven at 45°C for 12h to remove trapped air bubbles. The PU was further processed into films using a mini hot press (QM900S, QMESYS, Uiwang, South Korea) under a pressure of 25 MPa, yielding specimens with a thickness of approximately 0.1 mm. The films were then cut into appropriate sizes according to the subsequent characterization methods.

3. Characterization of PUs

To analyze the transmission spectra of the PU membranes, Fourier-transform infrared (FT-IR) spectroscopy (Frontier, PerkinElmer, Waltham, MA, USA) equipped with an attenuated total reflection (ATR) accessory was employed. Spectra were recorded in the range of 500–4000 cm^{-1} . The FT-IR spectra were collected every 30 minutes during polymerization to monitor the reaction progress. To deconvolute the peaks at 1737 cm^{-1} and 1699 cm^{-1} , the Gaussian–Lorentzian Cross fitting function was used and the degree of phase separation was calculated based on the fitted peak areas. The fitting quality was evaluated using the coefficient of determination (R^2), which was maintained above 0.997 for all four resolved peaks.

The thermal behavior of the PU membranes was analyzed using a differential scanning calorimeter (DSC, Q2000, TA Instruments, New Castle, DE, USA). Samples were prepared as square specimens (1cm $\times$ 1cm) with a mass of approximately 5.5 mg. To eliminate the previous thermal history, each sample was subjected to two heating–cooling

cycles over a temperature range of -90°C to 100°C at a heating/cooling rate of $5^{\circ}\text{C}/\text{min}$. The DSC measurements were used to determine the amorphous content, glass transition temperature (T_g), melting temperature (T_m), and crystallization temperature (T_c) of soft segment. T_g was defined as the midpoint of the intersection between the extrapolated baselines and the inflection point on the DSC curve.

The mechanical strength of the PU membranes was evaluated using a universal testing machine (Instron, Norwood, MA, USA) equipped with rubber-coated grips and a 100-kgf load cell, following the ASTM D882 standard and procedures reported in the previous studies. Test specimens were prepared with a width of 10 mm, a gauge length of 30 mm, and a thickness ranging from 100 to 300 μm . Tensile tests were conducted at a crosshead speed of 5mm/min, and the yield strength and Young's modulus were determined from the stress-strain curves to analyze the elastic deformation behavior.

The viscoelastic properties of the PU membranes were characterized using a dynamic mechanical analyzer (DMA 850, TA Instruments, New Castle, DE, USA). Rectangular specimens (5 mm $\times$ 30 mm, thickness 100–200 μm) were prepared for testing. Temperature-ramp experiments were conducted in tension mode at a frequency of 100 Hz over a temperature range from -70 to 30°C , with a heating rate of $5^{\circ}\text{C}/\text{min}$. To ensure linear viscoelastic behavior, the oscillation amplitude was maintained at 10 μm , and a preload force of 0.01N was applied. The storage modulus (E'), loss modulus (E''), and loss factor ($\tan\delta$) were obtained to evaluate the damping behavior and thermal transitions of the PU membranes.

Results and Discussion

1. FT-IR Analysis of PUs

Figure 1(a) shows the FT-IR absorption spectrum of the synthesized PU. The characteristic absorption peaks of polyurethane confirm that the polymerization was successfully carried out. The absorption band at 1100 cm^{-1} corresponds to the C–O–C stretching vibration of PEG. The bands at 1240 cm^{-1} and 1540 cm^{-1} correspond to amide II and amide III modes, associated with the coupled vibrations of N–H bending and C–N stretching in the urethane group. The absorption peak at 2261 cm^{-1} , corresponding to the

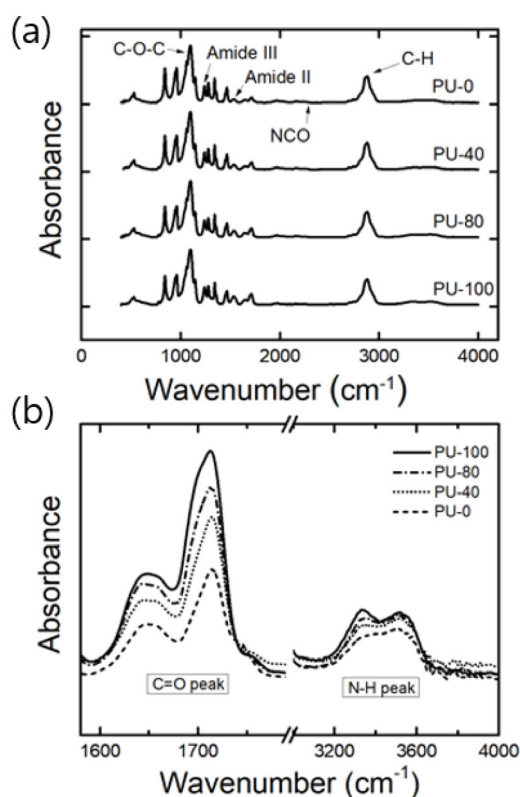


Figure 1. (a) FT-IR spectra of PUs synthesized with different PEG3350/PEG8000 ratios and (b) enlarged C=O and N-H stretching regions.

stretching vibration of the NCO group of isocyanate, was used to monitor the reaction progress. Its intensity gradually decreased during polymerization and became constant when the reaction reached completion, as residual NCO remained due to the NCO/OH molar ratio of 1.83. The absorption band at 2880 cm^{-1} corresponds to the C–H stretching vibration of the polyurethane backbone.¹³

Figure 1(b) shows the absorption spectra of the carbonyl (C=O) and N–H stretching regions. PU exhibits two major vibration regions: the N–H stretching band ($3200\text{--}3500\text{ cm}^{-1}$) and the C=O stretching band ($1600\text{--}1750\text{ cm}^{-1}$). As the average molecular weight of PEG increased, the intensities of both the C=O and N–H peaks tended to decrease. This trend can be attributed to the reduced density of urethane linkages caused by the higher fraction of soft segments in the PU membranes. Hydrogen bonding between the carbonyl and N–H groups may occur due to the polar nature of the urethane linkage, leading to observable peak shifts depending on the extent of hydrogen bonding.¹⁴

The degree of phase separation between the soft and hard segments in the polyurethane was calculated using the

following equation.¹⁵

$$DPS = \frac{R}{R + 1}$$

$$DPM = 1 - DPS$$

$$R = \frac{A_b}{A_f}$$

DPS : degrees of phase separation

DPM : degrees of phase mixing

R : carbonyl hydrogen bonding index

A_b : absorption intensity of hydrogen bonded carbonyl (1699 cm^{-1} peak)

A_f : absorption intensity of free carbonyl (1737 cm^{-1} peak)

The absorption bands observed in the range of 1737–1699 cm^{-1} correspond to the C=O stretching vibrations of urethane linkages. This region can be deconvoluted into three distinct components: the 1737 cm^{-1} band is assigned to free C=O groups that are not involved in hydrogen bonding, the 1717 cm^{-1} band represents *disordered hydrogen-bonded* C=O groups in amorphous domains, and the 1699 cm^{-1} band

Table 2. Degree of Phase Separation of PUs

Name of Sample	R(=A _b /A _f)	DPS	DPM
PU-100	4.94	0.83	0.17
PU-80	2.96	0.74	0.26
PU-40	2.94	0.74	0.26
PU-0	1.10	0.52	0.48

corresponds to *ordered hydrogen-bonded* C=O groups in crystalline domains. Because the 1717 cm^{-1} component includes overlapping signals that cannot be clearly attributed to specific hydrogen-bonding states, it was excluded from the phase-separation analysis.^{16, 17} Additionally, a minor peak observed at 1651 cm^{-1} is attributed to the C=O stretching vibration of urea linkages, formed by a side reaction between residual isocyanate groups and atmospheric moisture.¹⁴

The carbonyl peaks at 1737 cm^{-1} and 1699 cm^{-1} were deconvoluted, as shown in Figure 2, and the degree of phase separation was calculated from the relative integrated areas of the two peaks. The calculated values are summarized in Table 2. The phase separation ratio was the highest (i.e. 83%) for PU-100, which contains the lowest average molecular weight PEG, and it decreased progressively with increasing PEG molecular weight, reaching 52% for PU-0. This indicates that the change in phase separation behavior is primarily driven by the increased hard-segment fraction associated with the HS wt%, rather than by the soft-segment molecular weight itself.^{18,19}

2. Thermal Analysis of PUs

Figure 3(a) shows the thermal curves obtained from the DSC analysis, and the corresponding soft-segment transition temperatures and crystallinities are summarized in Table 3. All thermal transition temperatures (T_m , T_c , and T_g) increased with increasing average molecular weight of PEG. Similarly, the crystallinity of the soft segment (X_c) also exhibited a positive correlation with PEG molecular weight, indicating that higher-molecular-weight PEGs tend to form more ordered crystalline domains. This trend is consistent with the previous reports that PEG8000 exhibits the highest degree of crystallinity among PEGs with different molecular weights.²⁰ The crystallinity of the polyurethane soft segment was calculated using the following equation.²¹

$$X_c = \frac{\Delta H_m}{(1 - X_{HS}) \times \Delta H_{100\%PEO}} \times 100\%$$

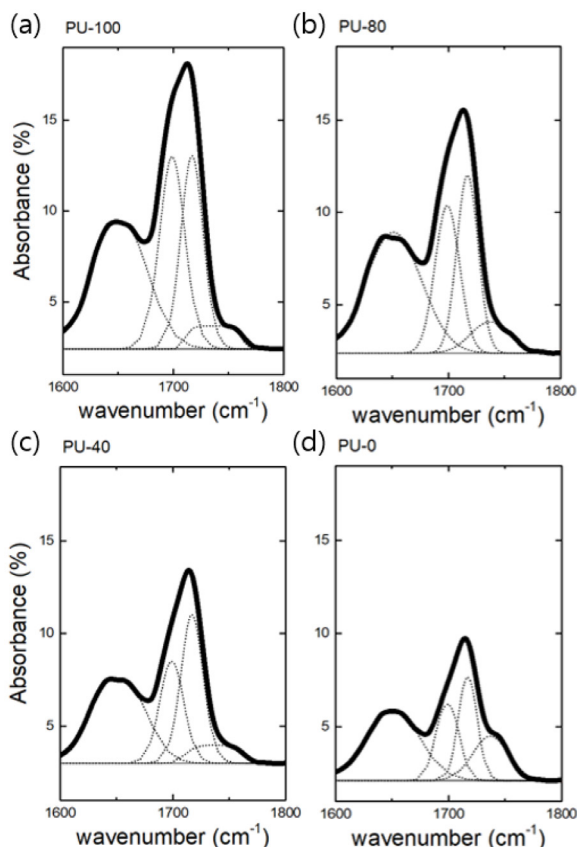


Figure 2. Deconvolution C=O peak (1600 cm^{-1} ~1800 cm^{-1}).

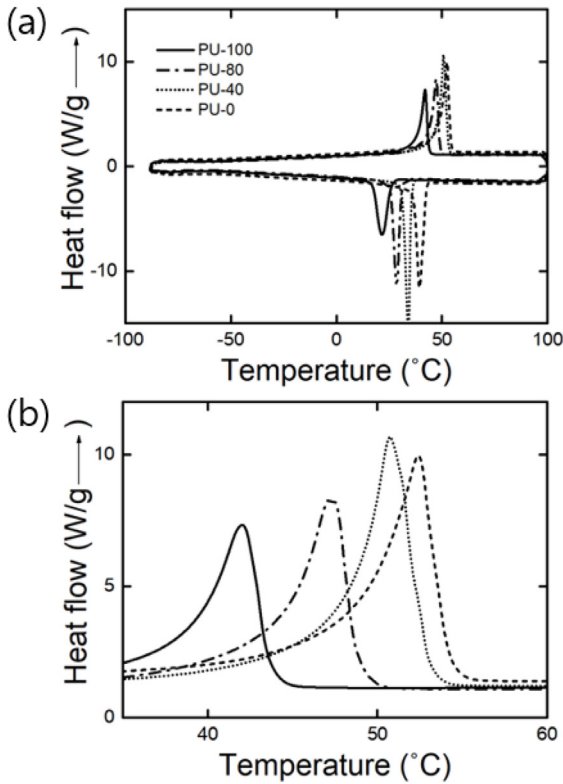


Figure 3. (a) DSC heat flow curves and (b) T_m peaks of PUs synthesized with different PEG33350/PEG8000 ratios.

The endothermic peak areas were integrated to quantify the melting enthalpy (ΔH_m) required for crystal melting, as shown in Figure 3(b). The theoretical melting enthalpy of 100% crystalline PEG ($\Delta H_{100\%PEO}$) was referenced from the literature and taken as 196.6 J/g.²⁰ The crystallinity of the hard segment (X_{HS}) was determined using the weight fraction of the hard segment (HS wt%).

The increase in T_g was also closely related to the enhanced crystallinity of the soft segment, which is consistent with previous studies describing the dependence of T_g on the degree of crystallinity.²² From a thermodynamic perspective, crystalline domains act as physical crosslinking points that restrict the mobility of adjacent amorphous chains. This restriction reduces the configurational entropy of the amorphous

phase and consequently requires higher thermal energy for segmental relaxation associated with the glass transition. Therefore, the rise in crystallinity leads to an increase in T_g .

3. Tensile tests of PUs

Figure 4 presents the stress–strain curves and a comparison of Young’s modulus for each sample. The tensile test results revealed that both Young’s modulus and ultimate tensile strength were strongly influenced by the average molecular weight of PEG, exhibiting an increasing trend with higher PEG molecular weight. The PU-0 sample exhibited a Young’s modulus of 288.2 MPa, which was 124.8% higher than that of PU-100.

A comparison between the crystallinity of the soft segment and the measured Young’s modulus revealed a consistent correlation between the two parameters. Both PU-0 and PU-40 exhibited relatively high crystallinity and modulus values, whereas samples with lower PEG molecular weight showed a progressive decrease in both crystallinity and modulus. These results clearly indicate that the crystalline domains within the soft segment contribute significantly to the mechanical rigidity of the polyurethane membranes.

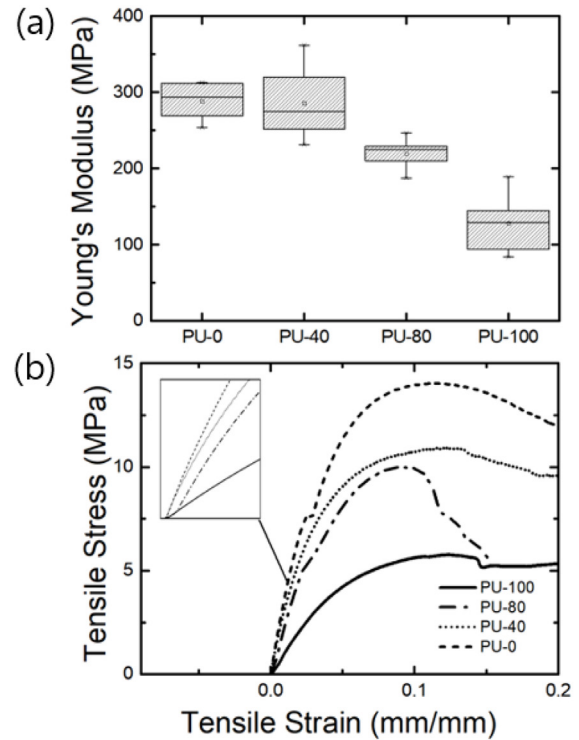


Figure 4. (a) Young’s Modulus and (b) Stress-Strain curve and Initial slope of PUs synthesized with different PEG33350/PEG8000 ratios.

Table 3. Transition Temperatures and Crystallinity of PUs

Name of Sample	T_g	T_c	T_m	ΔH_m	X_c
PU-100	-42.31	21.60	42.06	66.49	39.64
PU-80	-40.26	28.55	47.30	77.76	44.87
PU-40	-38.28	34.05	50.71	94.39	52.51
PU-0	-35.34	39.39	52.38	95.39	52.01

4. Dynamic Mechanical Analysis of PUs

Figure 5 shows the temperature dependent loss factor ($\tan\delta$) curves of the PU membranes. All samples exhibited a distinct $\tan\delta$ peak around -5°C , corresponding to the glass transition of the soft segment. The PU membranes containing mixed PEGs (PU-40 and PU-80) exhibited 13.3% higher $\tan\delta$ values on average, across the entire temperature range compared to those prepared with a single PEG (PU-0 and PU-100). Notably, the mixed systems exhibited relatively larger $\tan\delta$ values in the low temperature region (below -40°C), while the peak positions remained nearly unchanged. The single-PEG samples displayed a sharp $\tan\delta$ peak corresponding to the glass transition of their respective soft segments. In contrast, the PU membranes synthesized with mixed-PEG showed broader $\tan\delta$ peaks over a wider temperature range, attributed to the combined effect of two distinct T_g values.

Although PU-0 and PU-40 exhibited similar crystallinity and Young's modulus values, their loss factors differed significantly. PU-0 showed a lower loss factor compared to PU-40, which can be attributed to its relatively lower degree of phase separation and reduced interfacial energy dissipation. Polyurethanes containing a single PEG soft segment tend to form well ordered domains with regular chain packing, as the uniform soft segment length promotes structural periodicity. In contrast, when two PEGs of different molecular weights are incorporated, mixed soft-segment domains are formed, exhibiting a broader distribution of domain sizes and morphologies.²³ In copolymer systems, variations in the molecular weight distribution of the soft

segments can disrupt the long-range order of phase-separated structures and induce microphase imbalance, thereby increasing enhancing the interfacial area density between domains.^{24, 25} This expansion of interfacial area provides additional pathways for stress-induced energy dissipation, leading to an enhanced loss factor. Consequently, the higher and broader $\tan\delta$ peaks observed in mixed-PEG PUs suggest that the loss factor is strongly influenced by phase separation behavior, domain arrangement heterogeneity, and the presence of multiple soft-segment T_g relaxations. Therefore, the improved underwater acoustic attenuation is attributed to the optimized heterogeneous microphases and interfacial dissipation, not simply to the decrease in crystallinity.

Conclusion

In this study, the effects of the average molecular weight and molecular weight distribution of PEG on the mechanical and viscoelastic properties of PU membranes were systematically investigated. The PU samples synthesized with mixed molecular weight PEGs (PU-40 and PU-80) exhibited higher loss factor ($\tan\delta$) values in the low-temperature region below the glass transition, compared with those synthesized using single molecular weight PEGs (PU-0 and PU-100). Both tensile strength and crystallinity showed a clear increasing trend with higher average PEG molecular weight, ranging from PU-100 to PU-0. Notably, PU-40 demonstrated a tensile strength comparable to that of PU-0, while exhibiting a broader and higher $\tan\delta$ peak. These results indicate that an optimized ratio of PEGs with different molecular weights can effectively enhance damping performance without compromising mechanical strength. This finding provides valuable insight for the design of polyurethane-based underwater acoustic absorption materials with tunable viscoelastic properties.

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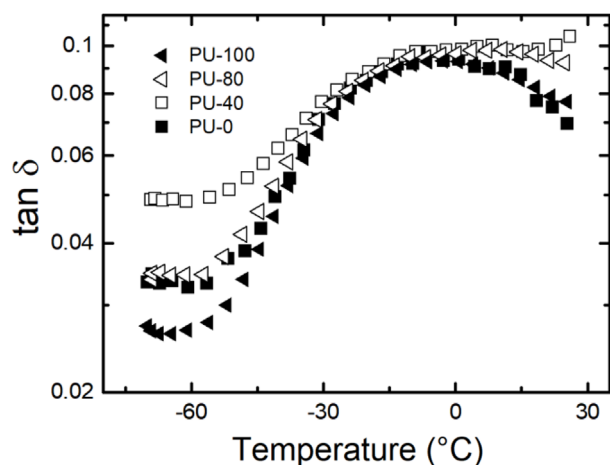


Figure 5. Loss factor of PUs synthesized with different PEG3350/PEG8000 ratios.

Conflict of Interest: The authors declare that there is no conflict of interest.

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