

Rigid Polyurethane Foams Made from High Viscosity Soy-Polyols

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ABSTRACT: This study investigated the physical properties of water-blown rigid polyurethane (PU) foams made from VORANOL®490 (petroleum-based polyether polyol) mixed with 0–50% high viscosity (13,000–31,000 cP at 22°C) soy-polyols. The density of these foams decreased as the soy-polyol percentage increased. The compressive strength decreased, decreased and then increased, or remained unchanged and then increased with increasing soy-polyol percentage depending on the viscosity of the soy-polyol. Foams made from high viscosity (21,000–31,000 cP) soy-polyols exhibited similar or superior density-compressive strength properties to the control foam made from 100% VORANOL® 490. The thermal conductivity of foams containing soy-polyols was slightly higher than the control foam. The maximal foaming temperatures of foams slightly decreased with increasing soy-polyol percentage. Micrographs of foams showed that they had many cells in the shape of sphere or polyhedra. With increasing soy-polyol percentage, the cell size decreased, and the cell number increased. Based on the analysis of isocyanate content and compressive strength of foams, it was concluded that rigid PU foams could be made by replacing 50% petroleum-based polyol with a high viscosity soy-polyol resulting in a 30% reduction in the isocyanate content. © 2012 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* 127: 1623–1629, 2013

KEYWORDS: rigid polyurethane foams; soybean oil-based polyol; high viscosity; compressive strength; foaming temperature; thermal conductivity; SEM

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INTRODUCTION

Rigid polyurethane (PU) foams have excellent attributes such as low thermal conductivity, low density, high strength-to-weight ratio, and low moisture permeability. They play an important role in industries and are widely used in transportation, refrigeration, packaging, automotive industry, and building construction. Therefore, demand of rigid PU foams has been growing at a rapid rate throughout the world.^{1–3} Polyol and isocyanate are two main raw materials of rigid polyurethane foams, both of which are derived from petroleum.^{1,2} In recent years, increasing costs of petroleum and growing environmental concerns encourage researchers to explore renewable and bio-based resources as feedstocks in manufacture of PU foams.³

Because of the great yield of soybeans and stable price of soybean oils in the US, soybean oil-based polyols (SBOPs) have attracted considerable attention to replace petroleum-based polyols in production of PU foams.⁴ PU foams made from SBOPs have been reported in the literature. John et al. studied the reaction kinetics of PU foams made from modified SBOPs. They concluded that PU foams could be successfully made from SBOPs and their properties could be changed by controlling

variables like water content, isocyanate index, and catalysts.⁵ Narine et al. compared the mechanical and thermal properties of PU foams made from castor oil, canola oil-based polyol, and SBOP. Their results indicated that the differences in foam performance were related to the differences in the number and position of OH-groups, dangling chains in the starting materials, and their cellular structure.⁶ Campanella et al. made flexible PU foams from three SBOPs with different functionalities and positions of OH-groups (primary or secondary OH-groups). They found that SBOP containing primary OH-groups was the most reactive and produced foams with smaller and more uniform cells.⁷ When only the secondary OH-groups were present, SBOP with higher functionality was more reactive and produced smaller and more uniform cells. Veronese et al. studied the properties of rigid PU foams made from soybean oil-based polyol and castor oil-based polyol. These bio-based polyols had an OH number between 393 and 477, and a viscosity ranging from 51 to 2187 cP at 25°C, lower than 7050 cP of the petroleum-based polyol Voranol 466N (Bayer SA, Brazil). They reported that foams made from SBOP with a higher OH number had higher T_g because of higher crosslinking density.⁸ In addition, bio-based PU foams showed slightly inferior mechanical

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properties than the control. Banik and Sain investigated the effects of various factors like catalyst, blowing agent, functionality and viscosity of soy-polyols on the structures and rigidity of rigid PU foams. Their results showed that higher dosage of catalyst or higher functionality soy-polyols gave rise to a more rigid foam structure. Also, when soy-polyols with the same OH number, foams made from soy-polyol with a higher viscosity of 4500 cP had higher compressive strength and were more rigid than those made from a lower viscosity (1250 cP) soy-polyol.⁹ While the effect of hydroxyl number, the position of hydroxyl groups (primary or secondary) and functionality of polyols on the properties of rigid polyurethane foams is well known, the effect of viscosity of polyols is not. The objective of this study was to investigate the effect of high viscosity SBOPs with a viscosity up to 31,351 cP on various physical properties of rigid polyurethane foams such as density, compressive strength, thermal conductivity, foaming temperature, and microstructure.

MATERIALS AND METHODS

Materials

The isocyanate used in this study was PAPI®27 with functionality 2.7. The petroleum-based polyether polyol was VORANOL® 490. Both PAPI® 27 and VORANOL® 490 were bought from Dow Chemical Company (Midland, MI). The high viscosity soy-polyols, A124, A125, and A73, were made from fully epoxidized soybean oil (ESBO) by alcoholysis reaction in Dr. Galen Suppes's lab in the Department of Chemical Engineering at the University of Missouri. In the alcoholysis reaction, ESBO was combined with ethylene glycol using *p*-toluenesulfonic acid as catalyst to promote the oxirane ring opening and form a hydroxyl group molecule.¹⁰ Detailed specifications of these polyols are shown in Tables I and II. Dimethylcyclohexylamine and pentamethyldiethylenetriamine, obtained from Sigma–Aldrich (St. Louis, MO), were used as catalysts. Dabco® DC 5357 was used as surfactant and was bought from Air Products & Chemicals (Allentown, PA). Distilled water was used as the blowing agent.

Foaming Formulation

The foaming formulation of rigid polyurethane foams made with different high viscosity soy-polyol percentages is shown in Table III.

Foam Preparation

The rigid polyurethane foams were made through one-shot and free-rising method. A total of 100 g of VORANOL® 490 and

Table I. Properties of Petroleum-Based Polyol and High Viscosity Soybean Oil-Based Polyols

Polyol	Viscosity (cP) at 22°C	ASTM hydroxyl number	Acid number
A124 (soy-polyol)	12,682	265	3.25
A125 (soy-polyol)	21,514	255	3.15
A73 (soy-polyol)	31,351	243	2.80
VORANOL® 490 (commercial petroleum-based polyether polyol)	9000	490	0

Table II. Viscosity (cP) of Soybean Oil-Based Polyols at Different Temperatures

	35°C	45°C	55°C	65°C
A124	4759.5	2321.3	1293.5	752.8
A125	6848.3	3477.1	1980.0	1076.1
A73	7817.7	4534.0	2416.8	1425.9

soy-polyol mixture were weighed into a plastic container and preheated in the oven (65°C) until the center temperature of the polyols reached 40–45°C for A124 and A125, and 55–65°C for A73. The polyols were then taken out of the oven and mixed with an electric stirrer at 3450 rpm for 15 s. Other B-side components such as water, catalysts and surfactant were added and mixed for 10–15 s. The mixture was allowed to degas for 2 min. Thereafter, preweighed isocyanate (A-side material) was added and mixed at the same speed for 10 s. The reacting mixture was then poured quickly into a wooden mold ($11.4 \times 11.4 \times 21.6 \text{ cm}^3$) with aluminum lining and the foam was allowed to rise and set at ambient conditions (23°C) for curing. The thermal conductivity testing is conducted in 24–48 h and other physical properties are tested after 7 days.

Foam Property Measurements

The apparent density was measured in accordance with American Society of Testing Materials (ASTM) Designation: D1622-08 (2008).¹¹ The compressive strength was measured in accordance with ASTM Designation: C1621-10 (2010) by a TA.HDi Texture Analyzer with XTRA Dimension software (Texture Technologies Corp., Scarsdale NY).¹² The dimension of the specimens was $6.35 \times 6.35 \times 3.81 \text{ cm}^3$ in both density and compressive strength.

Table III. Foaming Formulation of Rigid Polyurethane Foams with High Viscosity Soybean Oil-Based Polyol

Ingredients	Concentration, php ^a	
	Control PU foams	Soybean oil-based PU foams (SBO PU foams)
B-side materials		
VORANOL® 490 (Petroleum-based Polyol)	100	90, 80, 70, 60, 50
High Viscosity Soy-Polyol	0	10, 20, 30, 40, 50
Dimethylcyclohexylamine (gelling catalyst)	0.84	0.84
Pentamethyldiethylenetriamine (blowing catalyst)	1.26	1.26
Dabco® DC 5357 (surfactant)	2.5	2.5
Distilled water (blowing agent)	3.0	3.0
A-side material		
Isocyanate index ^b of PAPI® 27	110	110

^aThe concentration of all ingredients are expressed in parts per hundred parts of polyol, which conventionally dictates that the sum of all polyols adds up to 100 parts. ^bThe amount of isocyanate is based on the isocyanate index. The isocyanate index is the amount of isocyanate used relative to the theoretical equivalent amount.

Five specimens per sample were measured and the averaged result was reported. The surface foaming temperature was detected and recorded by an Omega Engineering OS 552A-MA-4 infrared thermometer equipped with a wireless transmitter and receiver (Omega Engineering Inc., Stamford, CT). The infrared thermometer focused on the center of the wooden mold (a circle of 2.54 cm in diameter) and was fixed at 40.64 cm above the mold. The interval of recording time was 1 s.¹³ The apparent thermal conductivity was measured in accordance with ASTM Designation: C518-10 (2010).¹⁴ After curing at ambient temperature for 24–48 h, the sample ($20 \times 20 \times 2.5 \text{ cm}^3$) was tested by a Fox 200 heat flow meter instrument (LaserComp, Wakefield, MA). The foam morphology was observed by a Hitachi S-4700 field emission scanning electron microscope (FESEM, Tokyo, Japan). A 3-mm cube foam sample was adhered to a substrate with silver glue and coated with gold powder by a plasma sputter. The sample was observed with an accelerating voltage of 5000 V and emission current of 9700 nA in FESEM.¹⁵

RESULTS AND DISCUSSION

Density

Figure 1 shows the density of rigid PU foams made from high viscosity soy-polyols mixed with VORANOL® 490 in different percentages. As shown, the density of soybean oil-based PU foams (SBO PU foams) decreased when increasing soy-polyol percentage from 0 to 50%. Soy-polyols have a hydroxyl number in the range from 243 to 265, lower than the hydroxyl number of 490 in VORANOL® 490. Thus, foams made from soy-polyols required less isocyanate than foams made from 100% VORANOL® 490. So were foams made from soy-polyols with a lower hydroxyl number than foams from soy-polyols with a higher hydroxyl number. In addition, the water content (blowing agent) in the foam formulation was fixed at 3% which generated the same amount of gas (CO_2) in the final foams. Therefore, the density decreased with increasing soy-polyol percentages in soybean oil-based rigid PU foams.^{16,17} Also, at the same soy-polyol percentage, SBO PU foams made from soy-polyol with a lower hydroxyl number had a slightly lower density.

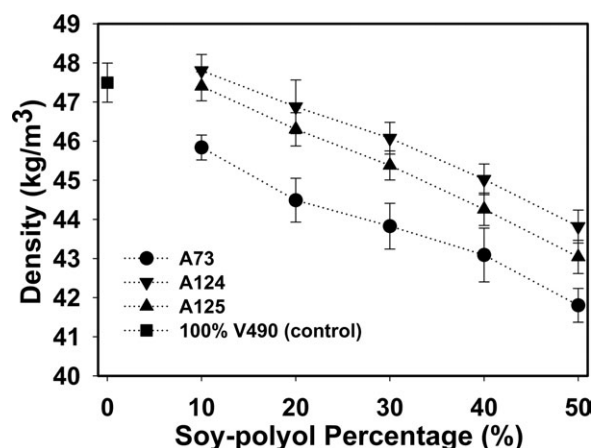


Figure 1. Density of rigid PU foams made from high viscosity soy-polyols mixed with VORANOL® 490.

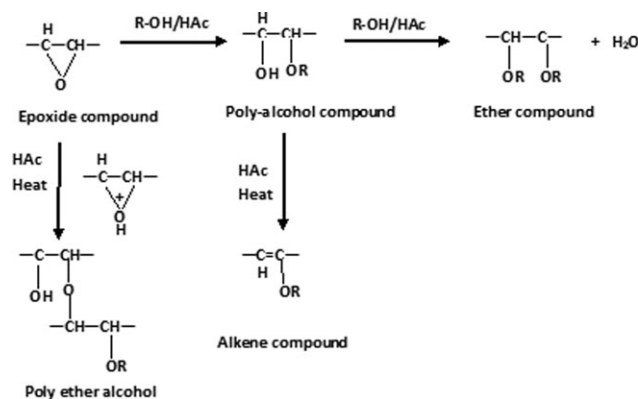


Figure 2. General mechanism for the alkoxy hydroxyl reaction (R-OH was an alcohol and HAc was an acid).¹⁸

Compressive Strength

The soy-polyols, A73, A124, and A125, were made from opening oxirane rings of fully epoxidized soybean oil (ESBO) by alcoholysis reaction. In the alcoholysis and addition polymerization reaction, ESBO was combined with ethylene glycol using *p*-toluenesulfonic acid as catalyst to promote the opening of epoxy rings and form hydroxyl groups in the molecule (Figure 2).¹⁸ High viscosity soy-polyols contain a large amount of ether chains in the molecules due to the formation of oligomeric ethers. During the polymerization between the hydroxyl and isocyanate groups, these soy-polyol ether bonds are also embedded in the cross-linking structure of PU polymers (Figure 3).

The compressive strength of rigid PU foams made from high viscosity soy-polyols mixed with VORANOL® 490 in different percentages is shown in Figure 4. Polyols with a higher hydroxyl number need more isocyanate for foaming and therefore produce PU foams with a higher cross-linking density. In general, higher density and/or higher cross-linking density result in foams with a higher compressive strength.^{15,19–21} As soy-polyol percentage increased, both the density and cross-linking density decreased. But the three blends exhibited three different compressive strength curves. Except slight difference in hydroxyl numbers, these soy-polyols had quite different viscosity. As shown in Figure 1, a higher viscosity soy-polyol would have a

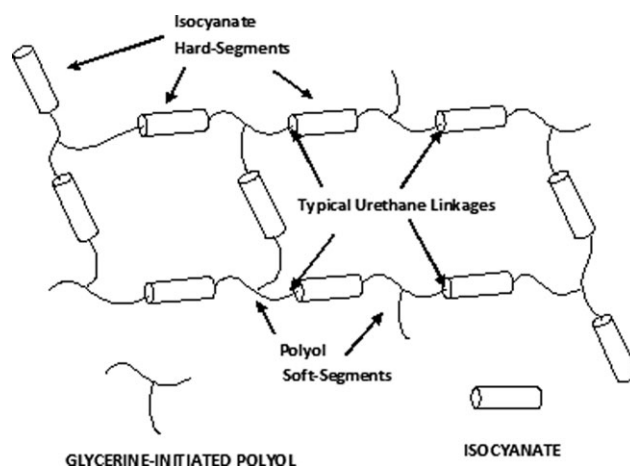


Figure 3. Cross-linking structure in polyurethane.¹⁹

higher molecular weight and contain more ether compounds and polyether alcohol compound in the molecules, and foams made from a higher viscosity soy-polyol would have more ether chains in the polymer network.¹⁰ Although A73 (31,351 cP viscosity) had a lower hydroxyl number than VORANOL® 490 and the density of foams made from A73 decreased with increasing soy-polyol percentages, the compressive strength remained roughly the same level from 0 to 30% and then increased gradually with further increase in soy-polyol percentages. In fact, the compressive strength of foams made from 50% A73 was similar to the control foam made from 100% VORANOL® 490. Also in Figure 4, the compressive strength of foams made from A125 (21,514 cP viscosity) decreased to 30% and then increased up to 50%, similar to foams made from A73 (31,351 cP viscosity). On the other hand, the compressive strength always decreased with increasing soy-polyol percentages from 0 to 50% for foams made from A124 (12,682 cP viscosity). Decreases in the compressive strength with increasing soy-polyol percentages were expected due to decreases in foam density (Figure 3) and cross-linking density, caused by using less isocyanate and hence less urethane linkage formation. Increases of compressive strength when soy-polyol was increased from 30 to 50% for foams from two soy-polyols (A73 and A125) with viscosity higher than 20,000 cP were not expected, however. These compressive strength results of foams made from A73 and A125 seem to suggest that the soy-polyol ether bonds, that were more abundant in foams from higher viscosity soy-polyols,¹⁰ might have functioned like urethane linkages to support the structure to a certain extent when foams were under loading. Further studies are needed to confirm the relationship of the molecular structure and compressive strength of foams.

Density-Compressive Strength and Isocyanate Density-Compressive Strength

Figure 5 presents the density-compressive strength property in rigid PU foams made from high viscosity soy-polyols mixed with VORANOL® 490 in different percentages using 3% water content as blowing agent. The square symbols are rigid PU foams made from 100% VORANOL® 490 using water content from 2 to 4% as blowing agent. As shown, there was a linear

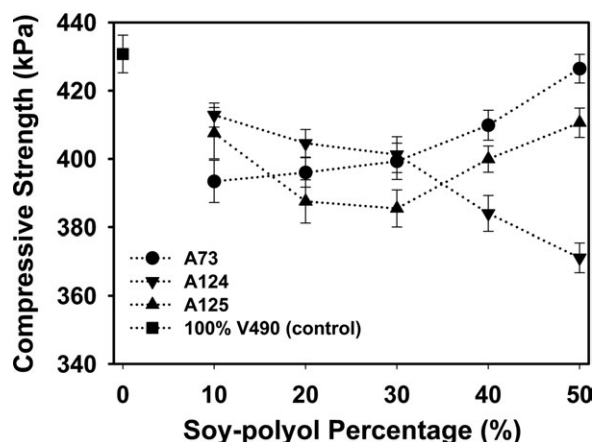


Figure 4. Compressive strength of rigid PU foams made from high viscosity soy-polyols mixed with VORANOL® 490.

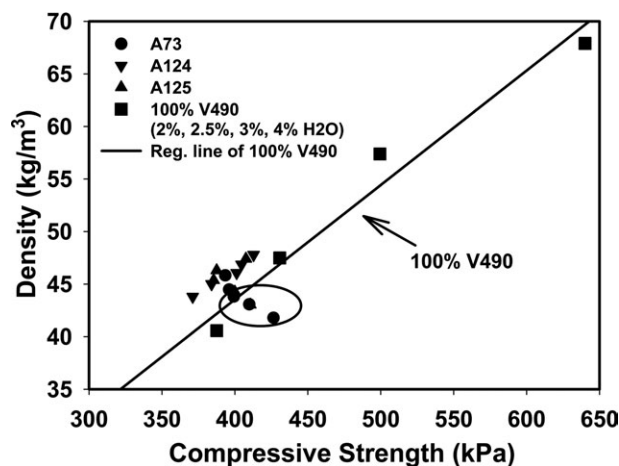


Figure 5. Density-compressive strength of rigid PU foams made from high viscosity soy-polyols mixed with VORANOL® 490.

relationship between the density and compressive strength in rigid PU foams made from 100% VORANOL® 490. This phenomenon was commonly observed in rigid PU foams.^{22,23} Foams located on the left-hand side of the regression line indicated that their density-compressive strength property was inferior to the control foam. Conversely, foams on the right-hand side of the line were superior to the control. From Figure 5, it was encouraging that foams made from mixing 40–50% A125 or 30–50% A73 with VORANOL® 490 were similar or superior to the control foam in terms of density-compressive strength property. A review of polyol properties (Table I) indicates that this comparable performance of soy-based foams might depend on soy-polyols having a minimum viscosity of about 22,000 cP. Density-strength property became even better when the soy-polyol viscosity increased to 31,000 cP.

The property of isocyanate density-compressive strength of rigid PU foams made from high viscosity soy-polyols mixed with VORANOL® 490 in different percentages is shown in Figure 6. Similar to Figure 5, there was a linear relationship between the isocyanate density and the compressive strength in rigid PU foams made from 100% VORANOL® 490. Foams on the right-

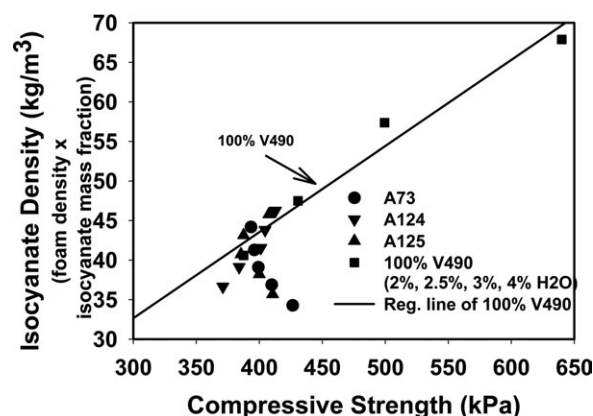


Figure 6. Isocyanate density-compressive strength of rigid PU foams made from high viscosity soy-polyols mixed with VORANOL® 490.

hand side of the regression line were superior to the control foams in the isocyanate density-compressive strength property (i.e., having higher compressive strength at the same isocyanate density or lower isocyanate density at the same compressive strength). It was interesting that most SBO PU foams displayed comparable or superior isocyanate density-compressive strength property to the control.

Because of a hydroxyl number of about 240, soy-polyols produced foams having reduced isocyanate contents (VORANOL®490 has a hydroxyl number of 490). Figure 6 summarizes the impact of isocyanate content in the foams on compressive strength. At lower isocyanate densities, most soy-polyols provided the advantage of reducing isocyanate content. The greatest reductions were obtained by A73 soy-polyol with a viscosity of 31,000 cP, and about 30% reduction in isocyanate content was achievable. These results indicated that rigid PU foams could be made by replacing 50% petroleum-based polyol with a high viscosity soy-polyol resulting in a 30% reduction in the isocyanate content.

Thermal Conductivity

The effect of high viscosity soy-polyols mixed with VORANOL® 490 on the thermal conductivity and closed cell percentage of rigid PU foams is shown in Figures 7 and 8, respectively. In general, the thermal conductivity of SBO PU foams increased as soy-polyol percentages increased. Also, all foams made from soy-polyols had a slightly higher thermal conductivity than the control foams made from 100% VORANOL® 490. VORANOL® 490 was a petroleum-based polyether polyol with a linear chemical structure and had primary hydroxyl groups; while soy-polyols were derived from vegetable oil and had secondary hydroxyl groups located in the middle of the triglyceride alkyl chains.^{24,25} During polymerization between the secondary hydroxyl groups and isocyanate, steric hindrance to crosslinking would occur because of the bulky aromatic isocyanate.^{26,27} Thus, soy-polyols had a lower reaction rate with isocyanate than VORANOL® 490. In the control foams, the polymer-forming reaction rate and blowing reaction rate was balanced. In SBO PUF, the polymer-forming

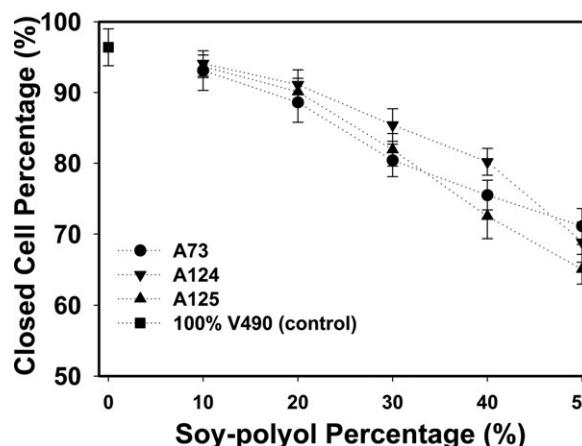


Figure 8. Closed cell percentage of rigid PU foams made from high viscosity soy-polyols mixed with VORANOL® 490.

reaction rate was actually slower than the blowing rate, so the cell walls were not sufficiently strong as in the control foams, and became easier to burst during the expansion of bubbles.¹⁵ Figure 8 showed that the closed cell percentage of SBO PUF gradually decreased with increasing soy-polyol percentage, which in turn increased the foam thermal conductivity.

Foaming Temperature

The foaming temperature was taken on the center of the foam surface during foaming using an infrared temperature sensor. Even though A73 soy-polyol was preheated to 55–65°C while A124 and A125 soy-polyols were preheated to 40–45°C, their foaming temperature curves were similar. Typical foaming temperature results of rigid PU foams made from high viscosity soy-polyols are shown in Figure 9. As shown, the maximal foaming temperatures of foams slightly decreased with increasing soy-polyol percentages. These results were not unexpected. Both the polyol-isocyanate and water-isocyanate reactions are exothermic. In SBO PUF, the secondary hydroxyl groups and a lower hydroxyl number in soy-polyols resulted in a lower reactivity as well as less exothermic when they reacted with

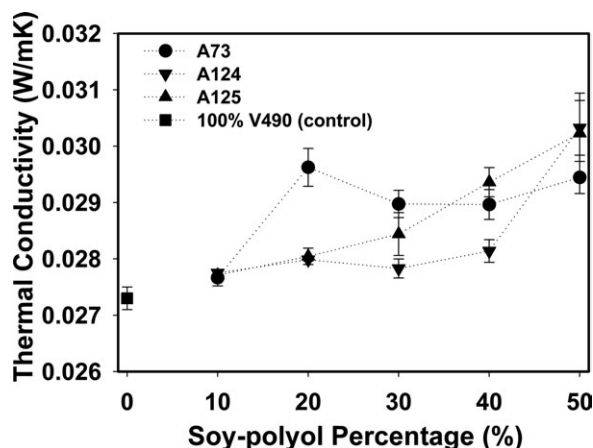


Figure 7. Thermal conductivity of rigid PU foams made from high viscosity soy-polyols mixed with VORANOL® 490.

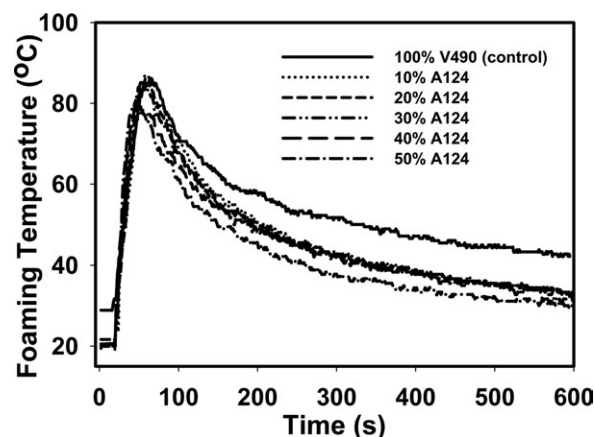


Figure 9. Foaming temperatures of rigid PU foams made from high viscosity soy-polyol A124 mixed with VORANOL® 490.

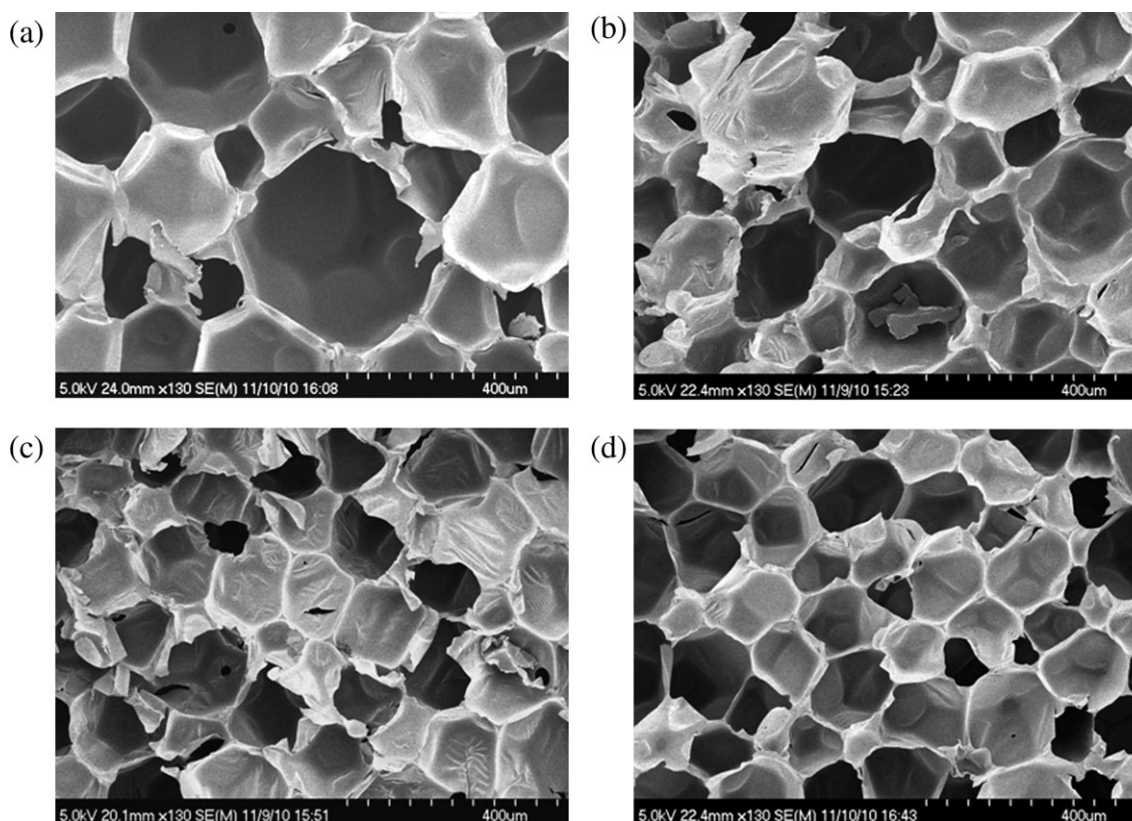


Figure 10. Scanning electron micrographs of rigid PU foams made from high viscosity soy-polyol A73 mixed with VORANOL® 490. (a) 100% VORANOL® 490 (control); (b) 10% A73; (c) 30% A73; (d) 50% A73.

isocyanate than VORANOL® 490, which contained primary hydroxyl groups and a higher hydroxyl number.

Microstructure

Figure 10 displays the scanning electron micrographs (SEM) of rigid PU foams made from high viscosity soy-polyols, A73, mixing with VORANOL® 490 in different percentages. The micrograph of the control foam made from 100%VORANOL® 490 is shown in Figure 10(a). It was observed that the rigid PU foams were composed of many cells in the shape of sphere or polyhedra. When the soy-polyol percentages increased from 0 to 50%, the foam cell size gradually decreased and meanwhile the foam cell number increased. Similar results were found for foams made from two other soy-polyols, A124 and A125. This change in the foam microstructure might be partially due to the difference of the chemical structure between the soy-polyols and petroleum-based polyether polyol. Soy-polyols were triglyceride with three pendant chains while petroleum-based polyether polyol was linear in chemical structure. During polymerization, the branched and pendant chains of triglycerides in soy-polyols were able to offer more nucleation sites for bubble formation and growth than VORANOL® 490. In addition, because soy-polyols had a significantly higher viscosity than VORANOL® 490, the viscosity of the reacting polyol-isocyanate liquid mixture would increase with increasing soy-polyol percentage. The viscosity increase of the reacting liquid system would reduce the coalescence among bubbles, therefore decreasing foam cell size and increasing foam cell number.²⁸

CONCLUSION

Rigid PU foams were made with high viscosity soy-polyols mixed with VORANOL® 490 in different percentages. Because soy-polyols had a lower hydroxyl number than VORANOL® 490 and the water content (blowing agent) was fixed, the density of SBO PU foams decreased with increasing soy-polyol percentages. The compressive strength of SBO PU foams made from A125 (21,514, cP viscosity) decreased to 30% and then gradually increased up to 50%. For foams made from A73 (31,351 cP viscosity), the compressive strength remained roughly at the same level from 0 to 30% and then gradually increased up to 50%. SBO PU foams made from 50% A73 and 50% VORANOL® 490 displayed comparable property to the control foam in compressive strength. In terms of density-compressive strength, SBO PU foams made from A125 and A73 showed comparable or superior property to the control foams when A125 percentage was at 50%, and A73 percentage was at 30–50%. Additionally, most SBO PU foams displayed comparable or superior isocyanate density-compressive strength property to the control foams. The results revealed that rigid PU foams can be made by replacing 50% petroleum-based polyol with a high viscosity soy-polyol leading to a 30% reduction in the isocyanate content. In thermal conductivity, SBO PU foams had slightly higher values than the control foams. This was caused by a lower closed cell content of PU foams made from soy-polyols than that in the control foams. The maximal foaming temperatures of foams slightly decreased with increasing soy-polyol percentage.

This was due to the lower reactivity of secondary hydroxyl groups in soy polyols with isocyanate. The micrographs presented that with increasing soy-polyol percentages the foam cell size decreased and cell number increased. This change was attributed to the difference in chemical structure between soy-polyols and VORANOL® 490. In addition, with increasing soy-polyol percentages, the viscosity increase of the reacting liquid system reduced the coalescence among bubbles, consequently decreasing cell size and increasing cell numbers. Based on the analysis of isocyanate content and compressive strength of foams, it was concluded that rigid PU foams could be made by replacing 50% petroleum-based polyol with a high viscosity soy-polyol resulting in a 30% reduction in the isocyanate content.

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