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Research Article

Physical Properties of Soy-Phosphate Polyol-Based Rigid Polyurethane Foams

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Water-blown rigid polyurethane (PU) foams were made from 0–50% soy-phosphate polyol (SPP) and 2–4% water as the blowing agent. The mechanical and thermal properties of these SPP-based PU foams (SPP PU foams) were investigated. SPP PU foams with higher water content had greater volume, lower density, and compressive strength. SPP PU foams with 3% water content and 20% SPP had the lowest thermal conductivity. The thermal conductivity of SPP PU foams decreased and then increased with increasing SPP percentage, resulting from the combined effects of thermal properties of the gas and solid polymer phases. Higher isocyanate density led to higher compressive strength. At the same isocyanate index, the compressive strength of some 20% SPP foams was close or similar to the control foams made from VORANOL 490.

1. Introduction

Polyurethanes (PU) are important chemical products and over three quarters of the PU are consumed in the form of foams globally [1]. Isocyanates and polyols are two major components in polyurethane (PU) production, and they both rely on petroleum as feedstocks [2–4]. In recent years, various factors motivate foam researchers to investigate potential and renewable resources as feedstocks of polyols. United States is a major producer of soybean oils and the price of soybean oil is forecasted to be relatively stable, ranging near \$9.20–9.25 per bushel in the next decade [5]. Besides, refined soybean oils contain more than 99% triglycerides with active sites amenable to chemical reactions [6]. Therefore, soybean oil (SBO) is one of the most promising biobased resources as a feedstock of polyols for PU manufacture. Several synthetic approaches have been reported to introduce hydroxyl groups to the triglycerides: (1) hydroformylation followed by hydrogenation [7], (2) epoxidation followed by oxirane opening [8–10], (3) ozonolysis followed by hydrogenation [11, 12], and (4) microbial conversion [13]. It has been reported that flexible and rigid PU foams could be made with a mixture of petroleum polyol and soybean oil-based polyols [14–16].

Soy-phosphate polyol (SPP) is made from the SBO-derived epoxides with the presence of phosphate acid as catalyst by acidolysis reaction. Normally, the acidolysis reaction is carried out by mixing SBO-derived epoxides, o-phosphate acid ($\text{o-H}_3\text{PO}_4$), water, and polar solvents, and the oligomerization occurs instantly to produce clear, viscous, homogeneous SPP with a low acid value and high average functionality. In this reaction, SBO-derived epoxide is able to directly react with water to form diols because of their high reactivity through cleavage of the oxirane ring [17]. Phosphoric acid not only catalyzes ring-opening reaction but also is chemically involved to become part of the polyol [18, 19]. Guo et al. [19, 20] prepared SPP by using water and a significant amount of polar solvents to obtain high hydroxyl functionality while keeping the final acid value low.

The distilled water, acting as blowing agent, reacts with isocyanate to generate carbon dioxide, which blows the reactant mixture to form a cellular structure. It is an important parameter that influences the properties and performance of rigid PU foams. By varying the amount of distilled water, Thirumal et al. [21] studied the effect of foam density on the properties of rigid PU foams made from a petroleum polyol. They found that the strength properties increase with the increase in density of the foams and their relationship can

be roughly depicted by the Power law [22, 23]. Campanella et al. [24] analyzed the effect of foaming variables, such as water content, polyol reactivity, catalyst, surfactant, and isocyanate, on the structure and properties of soybean oil-based PU foams. Water was used as a reactive blowing agent to produce CO₂ with isocyanate. They found that an increase in the water content produced more CO₂ and resulted in a larger foam volume as well as lower foam density. Also, the foam morphology was influenced by the water content. An increase in the water content produced rigid PU foams with thinner foam cell walls and larger foam cells, changing the density and morphology of the foams.

Isocyanate, which is derived from petroleum, is another critical component in PU production. Its amount (or the isocyanate index) and property greatly affected the performance of PU foams [25, 26]. Guo et al. [27] investigated effect of isocyanate index (ranging from 110 to 130) on compressive strength of rigid PU foams made from soybean-oil-based polyol. They found that compressive strength of soy-polyol-based foams varied proportionally with isocyanate index when isocyanate index was over 100, indicating that the mechanical property of rigid foams could be modified by altering the amount of isocyanate used in the foaming formulation. They concluded that excess isocyanate used in the foam formulation gave rise to a more rigid structure in rigid PU foams because of the more complete conversion of OH groups in polyols and the reaction of isocyanate with moisture in the air. Tu et al. [28] studied the physical properties of water-blown rigid PU foams containing epoxidized soybean oil with an isocyanate index ranging from 50 to 110. They reported that the foam density decreased when the isocyanate index decreased to 60, but a sharp increase in density occurred at isocyanate index 50 due to foam shrinkage. Also, the compressive strength decreased with decreasing isocyanate index. Decreasing the isocyanate index would reduce isocyanate and meanwhile increase the bio-based content in PU formulations. In this study, the objective was to investigate the effect water (blowing agent) content and isocyanate index on the physical properties of SPP PU foams.

2. Materials and Methods

2.1. Materials. The petroleum-based polyol and isocyanate used in this study were VORANOL 490 and PAPI 27, respectively. Both were obtained from Dow Chemical Co. (Midland, MI). Their specifications are shown in Tables 1 and 2, respectively. Soy-phosphate polyol (SPP) was prepared at the University of Missouri and its specification is shown in Table 3. It was made by an acidolysis reaction as shown in Figure 1 [18]. Full-epoxidized soybean oil (7.0% oxirane, Vikoflex 7170, Arkema, King of Prussia, PA) was reacted with 1.5% o-H₃PO₄ (85% aqueous from Fisher Scientific), added drop wise in a beaker under vigorous mechanical stirring at room temperature for 5 min [18]. Dimethylcyclohexylamine and pentamethyldiethylenetriamine were used as catalysts. They were from Sigma-Aldrich (St. Louis, MO). Dabco DC 5357 (Air Products and Chemicals, Allentown, PA) was used as a surfactant.

TABLE 1: Specifications of polymeric isocyanate PAPI 27 [29].

Properties	Values
Density, g/mL at 25°C	1.23
Average molecular weight	340
Functionality	2.7
NCO content by weight, %	31.4
Viscosity, cps at 25°C	150–220
Acidity, %	0.017
Vapor pressure, mm Hg at 25°C	<10 ⁻⁵
Decomposition point °C	230

TABLE 2: Specifications of VORANOL 490 [30].

Properties	Values
Density, g/cm ³	1.11
Hydroxyl number, mg KOH/g	484.8
Functionality	4.3
Average molecular weight, g	460
Flash point, 0°C	31.4
Water content, %	0.007
Viscosity, mPa.s (at 25°C)	6180

TABLE 3: Specifications of soy-phosphate polyol [18].

Properties	Values
Hydroxyl number, mg KOH/g	240
Viscosity, mPa.s (at 25°C)	3946.8
Acid number	0.5

2.2. Experimental Design and Foaming Formulations. This experiment consisted of two stages. In the first stage, the effect of water content (2, 3, and 4%) with different soy-phosphate polyol (SPP) percentages (0, 10, 20, 30, 40, and 50%) on the properties of rigid PU foams were studied. Table 4 shows the foaming formulations for rigid SPP-based polyurethane foams (SPP PU foams). Based on the results obtained from the first stage, the water content was fixed at 3% at the second stage to study the effect of isocyanate index and SPP percentage on the properties of SPP PU foams. The formulations are shown in Table 5.

2.3. Foam Preparation. The water-blown rigid polyurethane foams were made through one-shot and free-rising method. A total of 100 g of VORANOL 490 and soy-polyol mixture were weighed in a plastic container. Other B-side components such as water, catalysts, and surfactant were added successively and all B-side materials were mixed with an electric mixer at 3450 rpm for 2 min. The mixture was allowed to degas for 2 min. Thereafter, preweighed isocyanate (A-side material) was added rapidly and mixed at the same speed for 10 s. Then, the reacting mixture was poured quickly into a wooden mold (11.4 × 11.4 × 21.6 cm) with aluminum lining and the foam was allowed to free-rise and set at ambient

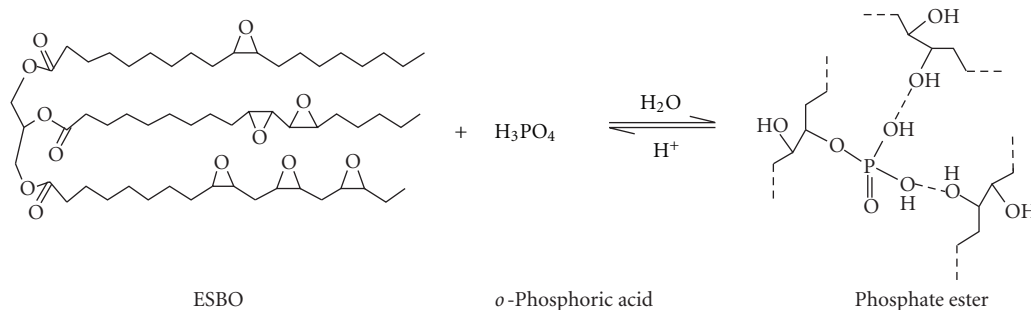


FIGURE 1: Schematic representation of soy-phosphate formation.

TABLE 4: Foaming formulation of rigid polyurethane foams with different soy-phosphate polyol percentages.

Ingredients	Parts by weight ^a	
	Control PU foams	SPP PU foams
<i>B-side materials</i>		
VORANOL 490 (petroleum-based polyol)	100	90, 80, 70, 60, 50
Soy-phosphate polyol (soybean oil-based 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)	2.0, 3.0, 4.0	2.0, 3.0, 4.0
<i>A-side material</i>		
Isocyanate index ^b of PAPI 27	110	110

^aAll recipes and calculations are based on 100 total parts by weight 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.

TABLE 5: Foaming formulation of rigid polyurethane foams with different isocyanate indices.

Ingredients	Parts by weight ^a	
	Control PU foams	SPP PU foams
<i>B-side materials</i>		
VORANOL 490 (petroleum-based polyol)	100	80, 50
Soy-phosphate polyol (soybean oil-based polyol)	0	20, 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
<i>A-side material</i>		
Isocyanate index ^b of PAPI 27	70, 80, 90, 100, 110	70, 80, 90, 100, 110

^aAll recipes and calculations are based on 100 total parts by weight 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.

conditions (23°C) for 7 days before measurement except thermal conductivity which was conducted within 24–48 h.

2.4. Foam Property Measurements. The apparent density of foam samples was measured according to the American Society of Testing Materials (ASTM) designation: D1622-08

(2008) [31]. The apparent thermal conductivity of rigid foams (20 × 20 × 2.5 cm) was determined according to ASTM designation: C518-10 [32] and was tested by a Fox 200 heat flow meter instrument (LaserComp, Wakefield, MA), after foam curing at room temperature (25°C) for 24–48 h. The compressive strength was determined according to ASTM Designation: D1621-10 [33] by a TA.HDi Texture

Analyzer (Texture Technologies Corp., Scarsdale, NY). The compressive strength was also tested after foam was stored seven days at room temperature. The dimension of the specimen in both density and compressive strength was $6.35 \times 6.35 \times 3.81$ cm. Five specimens per sample were measured and the average was reported.

3. Results and Discussion

3.1. Effect of Water Content

3.1.1. Density. Figure 2 shows the effect of soy-phosphate polyol (SPP) percentage on the density of rigid PU foams with different water contents. At 2% water content, the density of PU foams decreased slightly with increasing SPP percentage. Because SPP has a lower hydroxyl number (OH number of 240) than the petroleum-based polyol, VORANOL 490 (OH number of 490), a higher percentage of SPP would require less isocyanate in the foam formulation. Furthermore, when the water content was fixed at 2%, the same amount of carbon dioxide was generated in the PU foams. Thus, all foam volumes remained essentially the same and the density of foams decreased slightly with an increase in SPP percentage. However, at 3% water content, the density of foams did not change significantly with increasing SPP percentage. In principle, higher water content in the foaming formulation process generated more volume or amount of carbon dioxide gas [34]. More carbon dioxide forced the cell walls of the foams to become thinner, causing a weaker three-dimensional network, which led to decreased closed cell percentage (Figure 3) [35–37]. Even though less isocyanate was present to take part in foaming with increasing SPP percentage, the foam volume still decreased due to increase in the open cell percentage. The effects of both decreasing amount of isocyanate and foam volume caused the density to remain more or less the same. At 4% water content, the density of foams did not change significantly except the foam with 50% SPP replacement. This was caused by the same reason as 3% water content, which was discussed above. When SPP replacement reached 50%, the closed cell content was only 60% (Figure 3) resulting in a much lower volume compared to other replacements. Therefore, the foam with 50% SPP replacement displayed a slightly higher density.

3.1.2. Closed Cell Percentage and Thermal Conductivity. The effect of SPP percentage on the closed cell percentage and thermal conductivity of rigid PU foams with different water contents was shown in Figures 3 and 4, respectively. In the foaming process, water reacted with isocyanate to produce carbon dioxide, which expanded the gelling polymer creating many small cells in the foam. At the same water content, the closed cell percentage of PU foams decreased with increasing SPP percentage. The functional hydroxyl groups of SPP were secondary, while in VORANOL 490 they were primary [38, 39]. It is known that the primary hydroxyl groups react three times faster than secondary groups with isocyanate [37, 40]. The lower reactivity between the SPP and isocyanate might have diminished the foam cell strength and meanwhile given

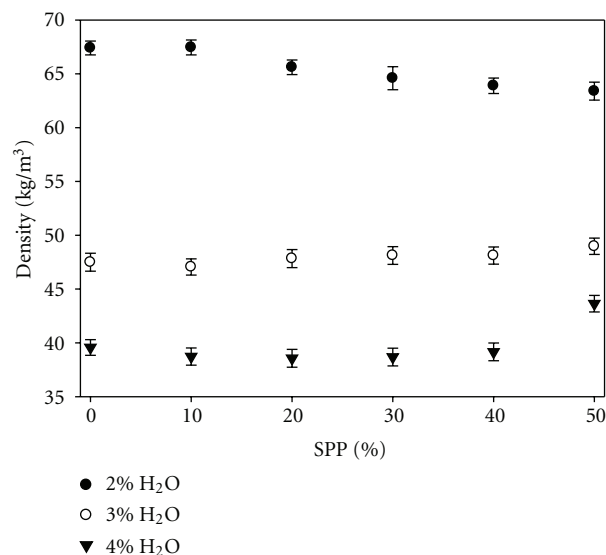


FIGURE 2: Effect of SPP percentage on density of rigid PU foams with different water contents.

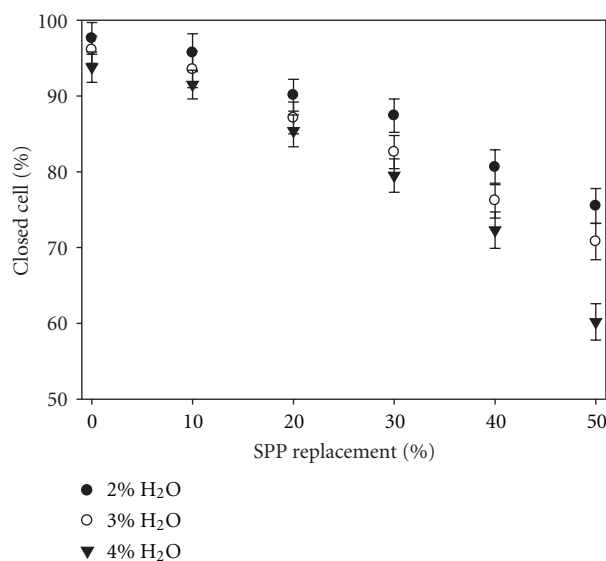


FIGURE 3: Effect of SPP percentage on closed cell percentage of rigid PU foams with different water contents.

rise to a weaker three-dimensional polymer network of cell walls to hold the pressure of carbon dioxide trapped in the cells. Therefore, with an increase of SPP percentage, the closed cell percentage in PU foams decreased.

The closed cell percentage decreased with increase in the water content. This was because at a higher water content, more carbon dioxide was generated leading to a larger foam volume and thinner cell walls. Thinner cell walls and higher gas volume might have weakened foam cell walls resulting in a lower closed cell percentage.

In Figure 4, at 2 to 4% water content, the thermal conductivity decreased from 0 to 20 or 30% SPP percentage and then increased. The thermal conductivity was mainly affected

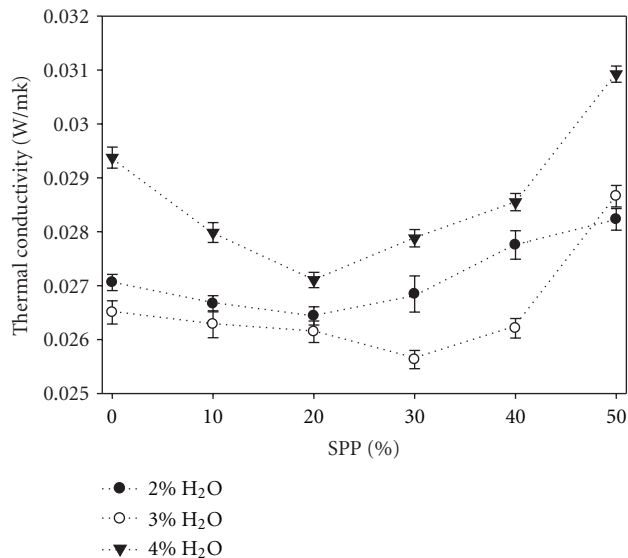


FIGURE 4: Effect of SPP percentage on thermal conductivity of rigid PU foams with different water contents.

by two factors: the conduction of the polymer phase and convection of the gas trapped in the cells [37, 41]. With increasing SPP percentage in the foam formulation process, less isocyanate was present to participate in the polymerization leading to less polymer phase. Therefore, the thermal conductivity was reduced when increasing the SPP percentage from 0 to 20 or 30%. On the other hand, the amount of carbon dioxide trapped in the foams decreased with increasing SPP percentage due to lower closed cell percentage (Figure 3). Carbon dioxide has a lower thermal conductivity (~ 0.0146 W/mK) than air (~ 0.024 W/mK) at room temperature [42]. The thermal conductivity of SPP PU foams was contributed by both polymer phase and gas phase. When the SPP was higher than 20 or 30%, the effect of decreasing closed cells (less carbon dioxide in the foam) might have exceeded the effect of decreasing polymer phase. This led to a higher thermal conductivity.

It is interesting to note that the thermal conductivity of foams at 3% water content was lower than those at 2% and followed by those at 4% at the same SPP percentage. As mentioned above, thinner cell walls (also less polymer phase) or higher closed cell percentage contributes to a lower thermal conductivity. Higher water content produced more gas, larger foam volume, and, therefore, thinner cell walls in the foam. At 4% water content, foams had the lowest closed cell percentage but more gas volume. At 2% water content, foams had the highest closed cell percentage but the least amount of carbon dioxide trapped in the cells as well as the thickest cell wall. It appears foams at 3% water content had the lowest thermal conductivity was due to the balanced effects from the closed cell percentage and amount of gas trapped in the cells.

3.1.3. Compressive Strength. Figure 5 shows the effect of SPP percentage on the compressive strength of rigid PU foams with different water contents. At 2% water content, the compressive strength decreased with increasing SPP percentage.

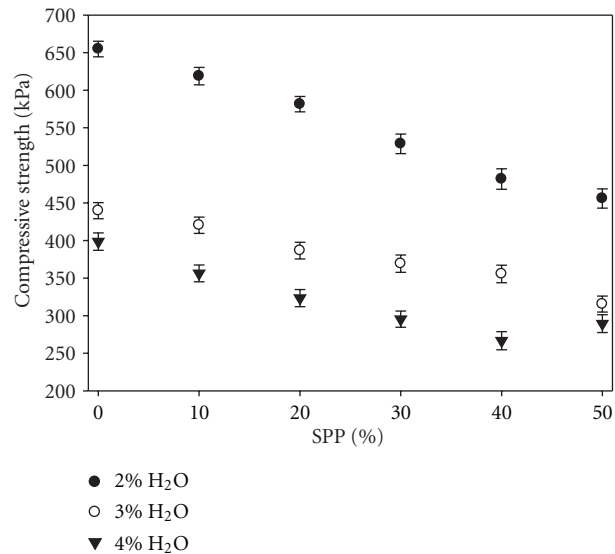


FIGURE 5: Effect of SPP percentage on compressive strength of rigid PU foams with different water contents.

Density and the number of cross-links of polymer network are two main factors that affect property of compressive strength of PU foams. Higher density and/or more cross-links give rise to a higher compressive strength [43, 44]. As the SPP percentage increased, less isocyanate was present in foaming. Therefore, fewer cross-links were generated by the reaction of hydroxyl and isocyanate functional groups [15, 45]. In addition, the foam density decreased slightly as the SPP percentage increased (Figure 2). Therefore, with increasing SPP percentage, the compressive strength decreased, due to both decreases of cross-links and density in PU foams.

Increasing the water content from 2 to 4% reduced the compressive strength (Figure 5). This was mainly due to the effect of water content on the foam density—higher density would result in higher compressive strength. As shown in Figure 2, the density of foams at 2% water content was higher than at 3% and followed by at 4%. Therefore, the compressive strength of foams followed the same order.

In summary, the water content had a significant effect on the physical properties of SPP PU foams. Higher water content generated more carbon dioxide. Therefore, foams had larger volume, lower density, as well as lower compressive strength. Based on the previous results, SPP rigid PU foams with 3% water content had the best thermal conductivity, medium density, and compressive strength. Therefore, the effect of isocyanate index was investigated at 3% water content.

3.2. Effect of Isocyanate Index

3.2.1. Density. Figure 6 shows the effect of isocyanate index on the density of rigid PU foams at 20 and 50% SPP. As shown, with the exception of 50% SPP PU foam at index 70, foam density increased when increasing the isocyanate index. Increasing isocyanate index raised the actual amount of isocyanate in the polymerizing reaction, thereby increasing

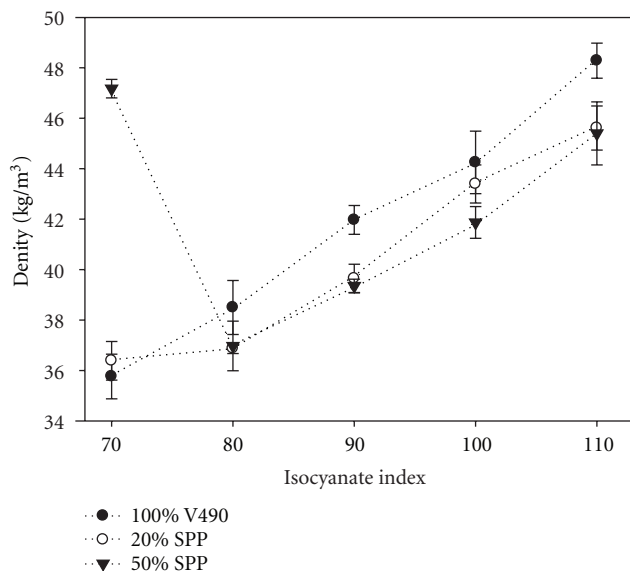


FIGURE 6: Effect of different isocyanate index on density of rigid PU foams with different SPP percentages.

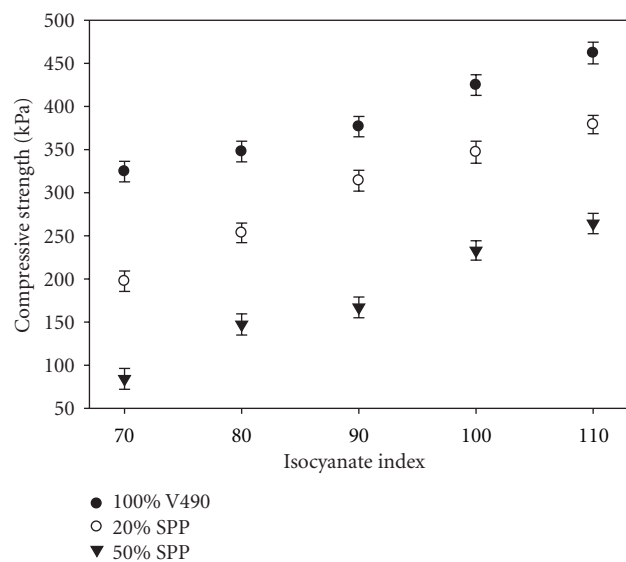


FIGURE 7: Effect of different isocyanate index on compressive strength of rigid PU foams with different SPP percentages.

the total amount of polymer network [46]. On the other hand, the foam volume was dictated by the amount of carbon dioxide generated by the reaction of water and isocyanate. This reaction was independent of the isocyanate index since isocyanate was not limiting when reacting with water. Therefore, the foam volume did not change with the SPP percentage which was also observed in the experiment. With more isocyanate in the foam formulation (increasing isocyanate index), foam density increased.

3.2.2. Compressive Strength. Figure 7 shows the effect of different isocyanate indices on compressive strength of rigid PU

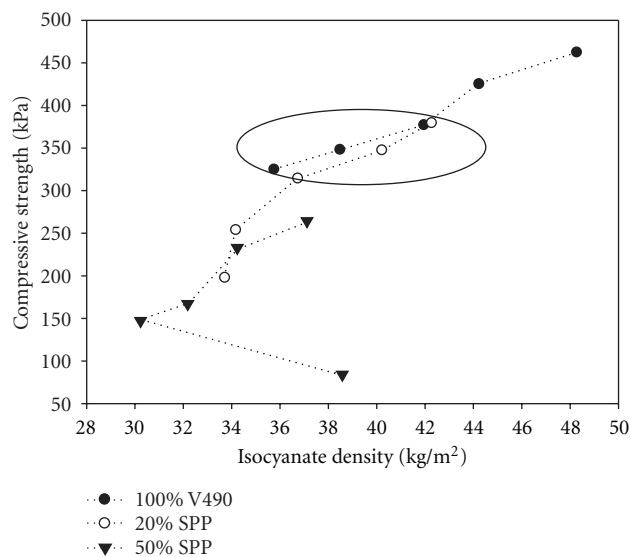


FIGURE 8: Results of compressive strength versus isocyanate density.

foams at 20 and 50% SPP. Raising the isocyanate index increased the compressive strength of foams. This was because the more the isocyanate used in the foam formulation, more cross-links were formed from the reaction of isocyanate and the hydroxyl groups in the polyols. In addition, foam density increased with the isocyanate index (Figure 6). Both increases in cross-links and density contributed to higher compressive strength.

The compressive strength of 50% SPP foam was less than the 20% SPP foam and both SPP foams were less than the control (Figure 7). This might be due to the difference in the reactivity of isocyanate with the hydroxyl groups that were in the petroleum polyol in the control (primary) and those in the SPP (secondary). This might have resulted in less cross-links when increasing the SPP percentage from 0 to 50%. In addition, the density of control foam was also greater than the SPP foam leading to higher compressive strength.

3.2.3. Compressive Strength versus Isocyanate Density. Figure 8 displays the results of compressive strength versus isocyanate density of rigid PU foams. The isocyanate density was calculated by percent of isocyanate multiplying by the density of PU foams (Table 6). As shown, at the same SPP percentage, higher isocyanate density foams were harder or had higher compressive strength. While the compressive strengths of 50% SPP PU foams were always inferior to those of 100% VORANOL 490 PU foams, it is interesting to note that some 20% SPP PU foams were close or similar to control in compressive strength at the same isocyanate density.

4. Conclusion

In summary, water-blown rigid polyurethane foams were produced by combining 0 to 50% soy-phosphate polyol (SPP) with petroleum-based-polyol VORANOL 490 using

TABLE 6: Calculation of isocyanate density.

SPP	Isocyanate index	Foam density (kg/m ³)	Theoretical isocyanate amount (g)	Actual isocyanate amount (g)	Isocyanate mass fraction	Isocyanate density (kg/m ³) = Foam density × isocyanate mass fraction	Compressive strength (kPa)
0	110	48.29	176.62	176.62	1	48.29	461.99
	100	44.25	160.57	160.57	1	44.25	424.79
	90	41.97	144.50	144.50	1	41.97	376.59
	80	38.5	128.45	128.45	1	38.50	347.71
	70	35.77	112.40	112.40	1	35.77	324.48
20%	110	45.62	176.62	163.73	0.93	42.29	379.04
	100	43.40	160.57	148.85	0.93	40.23	346.97
	90	39.65	144.50	133.97	0.93	36.76	313.95
	80	36.88	128.45	119.08	0.93	34.19	253.47
	70	36.39	112.40	104.20	0.93	33.73	197.40
50%	110	45.40	176.62	144.40	0.82	37.12	264.28
	100	41.87	160.57	131.28	0.82	34.23	232.98
	90	39.36	144.50	118.15	0.82	32.18	167.07
	80	36.98	128.45	105.02	0.82	30.23	147.29
	70	47.18	112.40	91.88	0.82	38.56	84.15

2–4% water as the blowing agent. The results showed that the water content significantly affected the physical properties of the final SPP PU foams, such as density, compressive strength, and thermal conductivity—lowest water content produced foams with the least volume but the highest density and compressive strength. The lowest thermal conductivity was observed in SPP foams at 3% water content. Because the hydroxyl number of SPP was lower than VORANOL 490, compressive strength of SPP foams decreased when increasing the SPP percentage in the foaming formulation. Increasing the isocyanate index or isocyanate density raised the compressive strength of SPP foams. At the same isocyanate index, the compressive strength of some 20% SPP foams was close or similar to the control foams made from VORANOL 490.


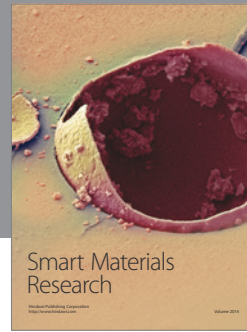
Acknowledgments

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