

# Polyurethane Rigid Foam Derived from Reduced Sweet Whey Permeate

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Low-pressure propoxylation of reduced whey permeate (RWP) with different water contents (12.7 and 33.4%) under alkaline conditions afforded polyether polyols (PEP) which have been characterized by their <sup>1</sup>H-NMR, HPLC, hydroxyl number, viscosity, unsaturation content, and alpha color. The results were compared with those from lactitol- and sucrose-based polyether polyols (LPEP and SPEP, respectively). Compared with LPEP and SPEP, polyether polyols from reduced whey permeate (RWP-PEP) showed relatively higher hydroxyl number, lower viscosity, and a similar degree of unsaturation. RWP-PEP was generally darker in color. In addition, polyurethane rigid foams (PURFs) from RWP-PEP, LPEP, and commercial sucrose-glycerin-based polyols (Voranol-PEP) were prepared and characterized. All PURFs made from RWP-PEP have low densities (from 28 to 29 kg/m<sup>3</sup>) and high closed cell contents (>73%). Compressive strength ranged from 13 to 17 kg × 1000/m<sup>2</sup> at 10% deformation. These foams have similar thermal stability as the commercial foams.

**Keywords:** *Reduced sweet whey permeate; propoxylation; polyether polyol (PEP); hydroxyl number; viscosity; unsaturation; alpha color; PAPI-27; polyurethane rigid foam (PURF); core density; closed cell content; compressive strength; thermal gravimetric analysis*

## INTRODUCTION

The polyurethane rigid foam (PURF) market in the United States was 1005.5 million pounds (Woods, 1987), and the demand is increasing rapidly because of wide applications as insulation materials, lightweight construction materials, floatation materials, packaging/padding materials, etc. Rigid urethane foam is synthesized from polyether polyols (PEP) and polyisocyanate (PAPI) in the presence of catalyst, surfactant, blowing agent, and other additives, while polyether polyols are usually made from propoxylation of low-cost materials such as sugars and their derivatives. The most widely used sugar for preparing PEP in industry is sucrose (Meath and Booth, 1977; Knodel, 1975) while less used sugars include maltitol (Takeda Chemical Industries, 1965), alkyl glycosides (Wilham et al., 1975; Otey et al., 1963, 1976, 1978), and modified cornstarch (Cunningham et al., 1992). Whey permeate generated from the cheese industry causes a serious economic and environmental problem (Zall, 1992). Since lactose, the major solid component in whey permeate, has a structure similar to that of sucrose, it would be interesting to use lactose or whey permeate to make PEP and hence PURF. Some early work using lactose and whey permeate directly for PURF was reported by Hustad et al. (1969) and Viswanathan et al. (1984, 1990). The PEPs from both lactose and whey permeate show extremely low viscosity and high carbonyl content relative to sucrose-based PEP (SPEP) because extensive degradation of lactose occurs during propoxylation

under the high temperature and basic condition. The major cause for degradation of lactose is due to the presence of aldehyde functionality which is not present in sucrose (Binkley, 1988; Donnelly, 1990). The resulting foams from lactose-based PEP hence show inferior mechanical properties and low closed cell content when compared to those from sucrose (a nonreducing sugar). In order to make a PURF with usable properties, modification of the aldehyde group in lactose is important. An obvious approach is to reduce lactose to lactitol and then use lactitol as the initiator for PEP and hence PURF. An early study done by other researchers (Stockburger et al., 1966) together with our recent work (Wilson et al., 1996) have proven lactitol to be a better initiator than sucrose for making PEP and PURF. For example, lactitol required shorter reaction times for propoxylation and produced lighter colored polyols than sucrose; PEP produced from lactitol (LPEP) displayed similar physical properties to sucrose-based PEP; LPEP-based PURF, under the same reaction conditions, has a density less than 30 kg/m<sup>3</sup>. Lactitol is more expensive than sucrose, so at present it will be a disadvantage to replace sucrose for industrial applications. This problem may be overcome by using reduced whey permeate (RWP) directly as a low-cost substitute in making PEP and PURF. In this article, we report the synthesis and characterization of RWP and RWP-based PURFs.

## MATERIALS AND METHODS

**General Procedures for Hydrogenation.** The procedures for reduction of whey permeate (product code 396, with a composition listed in Table 1, Foremost Ingredient Group, Baraboo, WI) was described earlier (Hu et al., 1996). After the reduction, the catalyst was removed by a Sorvall RC-5B Refrigerated Superspeed Centrifuge (DuPont, Wilmington, DE) at a temperature between 5 and 10 °C, and water was removed by a Flash-Evaporator (Buchler Instrument, Lenexa, KS) equipped with a cold tap under low pressure (1 torr). The

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**Table 1. Components of Dry Sweet Whey Permeate (SWP, Code 396, a Non-Hygroscopic Spray-Dried, Lactose-Rich Product from Sweet Dairy Whey)**

proximate analysis	typical (w %)
lactose	83.0
protein	3.5
fat	0.2
ash	8.5
water	4.8

**Table 2. Typical Foam Formulation**

component	part by weight
polyol <sup>a</sup>	100
Genetron 141b	35
DABCO R-8020	1.8
DABCO DC-193	1.5
PAPI 27	(105–110) <sup>b</sup>

<sup>a</sup> RWP-PEP, Voranol 360, LPEP, and SPEP. <sup>b</sup> Isocyanate to attain index of 105.

final water content of reduced whey permeate was measured by thermogravimetric analysis (TGA) (Shimadzu TGA-50, Columbia, MD) from room temperature to 105 °C at a rate of 50 °C/min, and held at 105 °C for 60 min under N<sub>2</sub>.

**Propoxylation of Reduced Whey Permeate.** The procedures for propoxylation of lactitol dihydrate (Purac America, Inc., Lincolnshire, IL) and RWP with KOH were described in our earlier study (Wilson et al., 1996), and the polyol workup was described as follows. The crude polyether polyols were dissolved in deionized water and the solutions were neutralized with diluted sulfuric acid. After concentration of water solution under reduced pressure followed by azeotrope of water with benzene, the PEP was dissolved in benzene again to precipitate the salts, followed by filtration, concentration, and drying at room temperature under high vacuum for 48 h.

**Polyol Characterization.** All <sup>1</sup>H-NMR analyses were run with a GE-300 instrument (General Electric, NMR-Instruments, Fremont, CA), with CDCl<sub>3</sub> as solvent, tetramethylsilane (TMS) as the internal standard, and trifluoroacetic anhydride as the shifting reagent. Hydroxyl number, unsaturation, and alpha color were determined according to ASTM methods D4274-88, D4671-87, and D4890-88, respectively. The end points for titration of hydroxyl number and unsaturation were determined by a microprocessor pH/millivolt meter 811 (Orion Research Inc., Boston, MA). Viscosity was determined using a controlled stress rheometer (Carri-Med, Ltd., Dorking, England) with a 2 cm disk having a 4° pitch and 110 μm truncation. For HPLC analysis (IBM LC/9533, IBM Instruments Inc., San Jose, CA), a 20 μL sample (0.2–0.3 mg/mL) was injected at a 0.5 mL/min flow rate with pure water as eluent. For separation, the samples were first passed through a Coregel-87C calcium form guard column and then a Coregel-87C calcium form analytical column (Interaction Chromatography, San Jose, CA) at 85 °C (Waters Temperature Control Module, Marlborough, MA). The separated samples were detected by a differential refractometer (Knauer, Rainin Instrument Co., Emeryville, CA) and analyzed by HP 3990 integrator. The deionized water was degassed and filtered through 0.2 μm HPLC filter paper.

**Foam Formation.** PURFs were prepared by adding PAPI 27 (polymeric diphenylmethane diisocyanate, equivalent weight 134; Dow Chemical, Midland, MI) to a premix of PEP (RWP-, lactitol-, or Voranol-based), DABCO R-8020 (amine catalyst; Air Products and Chemicals, Allentown, PA), surfactant DABCO DC-193 (silicone-glycol copolymer; Air Products and Chemicals), and blowing agent Genetron 141b (dichlorofluoroethane; Allied Signal, Morristown, NJ) in the ratios given by Table 2. The combined materials were mixed in a 16 oz paper cup for 10 or 20 s using a Barnant mixer (series 10, model 700-5400; Barnant Co., Barrington, IL) equipped with a turbine rotor (model 4544-10; 2 in. diam. 3/8 in. bore) at 2500–3000 rpm, then quickly poured into a 13 × 16 × 16 cm wooden mold which had been coated with wax. Foams were allowed to rise freely under ambient conditions then were

removed from the mold and cured 24 h at room temperature and ambient humidity.

**Foam Characterization.** The foams were cut into test samples with an auto band saw and all specimens conditioned at 21 °C and 65% relative humidity for 48 h prior to characterization. Apparent core densities were determined by caliper and analytical balance. Closed cell content of the foam (size of 20 × 20 × 20 mm) was determined by an air pycnometer (Model 930, Beckman Instruments, Inc., Fullerton, CA) according to ASTM D 2856-94, procedure C. The compressive properties (foam size 40 × 40 × 50 mm) were measured in accordance with ASTM D1621-73 using an Instron 1122 (Instron Corp., Canton, MA) with a full scale load of 50 kg and a cross-head speed of 5 mm/min. Thermal stability of polyurethane rigid foams was also determined by TGA from room temperature to 550 °C at a rate of 20 °C/min in air.

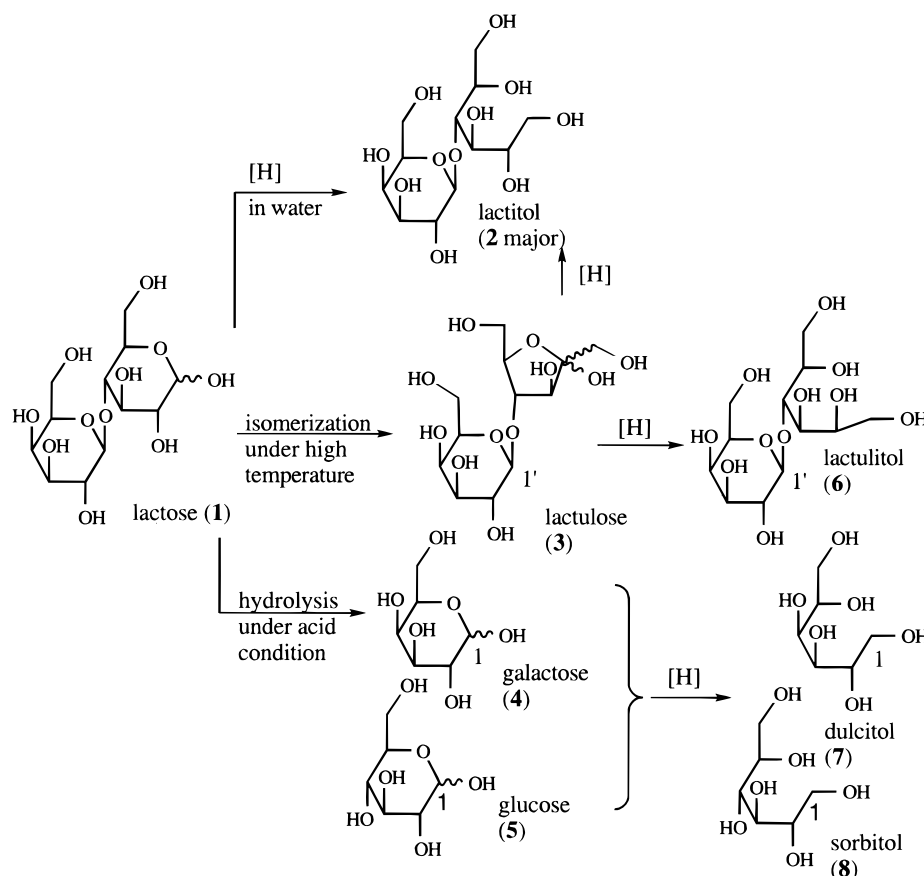
## RESULTS AND DISCUSSION

**Reduction of Sweet Whey Permeate.** Our early study (Hu et al., 1996) has shown that many side products can be formed during the reduction of sweet whey permeate (SWP, see Scheme 1). The percentage of each component is highly dependent upon the reaction conditions (e.g., SWP concentration, reaction time, temperature, hydrogen pressure, and amount of Raney Ni catalyst). Among the side products, lactulose, galactose, and glucose, together with lactose (starting material) are reducing sugars (lactulose interconverts with lactose under high temperature), and lactitol, lactulitol, ducitol, and sorbitol are the nonreducing sugars. The presence of these reducing sugars facilitates browning products when they are propoxylated. The nonreducing sugars have increased alkaline and heat stability (Donnelly et al., 1990), and hence degradation is negligible (Wilson et al., 1996). Therefore, it is desirable to generate nonreducing sugars while minimizing reducing sugars. The optimal conditions we chose throughout this research for reduction of SWP were as follows: 150 g of dry SWP was dissolved in 190 g deionized water with a reaction temperature of 120 °C, initial hydrogen pressure of 1500 psi, 40.5 g Raney Ni, and 5 h reaction time. Under these conditions, whey permeate lactose (**1**) can be completely converted into its nonreducing forms with lactitol (**2**) as the major product, and lactulitol (**6**), ducitol (**7**), and sorbitol (**8**) as minor products (Hu et al., 1996).

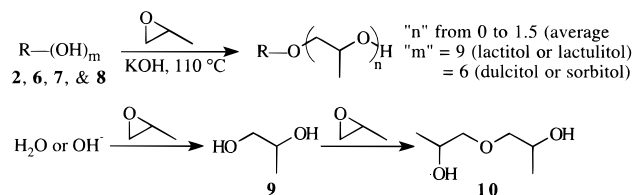
**Propoxylation of RWP with KOH.** Since RWP contains several nonreducing sugars, they all will act as initiators for propoxylation (Scheme 2). Another important substance that needs to be taken into consideration is water. Water is present in RWP because all of the nonreducing sugars (**2**, **6**, **7**, and **8**) are very hydroscopic. Water is usually used as a co-initiator in preparing PEP (Meath et al., 1977) and has the advantages of increased propoxylation speed, decreased overall PEP functionality and viscosity, improved PEP compatibility with PAPI, and unchanged raw material cost. Thus, the presence of water in RWP will add an advantage to the preparation of PEP and PURF. The water content in RWP could be measured by TGA analysis and RWPs with two levels of water content (i.e., 12.7 and 33.4%) were used for propoxylation. The PEPs prepared from RWP with a 12.7% water content were designated as RWP-PEP 1, 2, and 3 (series 1); with 33.4% of water in RWP, the PEPs were designated as RWP-PEP 1', 2', 3', and 4' (series 2, Table 3).

In our previous study of propoxylation of lactitol- and sucrose-dihydrate, various basic catalysts were probed.

**Scheme 1. Reduction of Sweet Whey Permeate: Major Product (Lactitol, 2), Minor Products (Lactulose, 3; Lactulitol, 6; Galactose, 4; Glucose, 5; Dulcitol, 7; and Sorbitol, 8)**



**Scheme 2. Reaction Products from Propoxylation of RWP (Including Lactitol, Lactulitol, Dulcitol, Sorbitol, and Water)**



Among them, KOH gave a relatively shorter reaction time and lower unsaturation compared to triethyl amine and potassium *tert*-butoxide. Hence, 1% KOH (w/w relative to the solid weight of RWP and assumed to contain 15% water) was used as the catalyst in the propoxylation of RWP. The amount of propylene oxide (PO) was based upon the PO/OH ratio (0.5–1.5, see Table 3). The number of OH groups was the sum of all sugar OH groups plus two OHs per water.

When PO/OH = 0.5, water initiated propoxylation gave 1,2-propanediol (9) which is clearly indicated in the <sup>1</sup>H-NMR spectrum of exhaustively trifluoroacetylated PEP (TEA-PEP from trifluoroacetic anhydride in Figure 1a). HPLC analysis of RWP-PEP 1 (lowest PO/OH), showed sugar PEP formed without lactitol (retention time or *t<sub>R</sub>* = 12.95 min), lactulitol (*t<sub>R</sub>* = 11.33 min), dulcitol (*t<sub>R</sub>* = 23.28 min), and sorbitol (*t<sub>R</sub>* = 24.34 min) left (Figure 1a, b, c, and d). This is true for both propoxylation series of RWPs. As the amount of PO increased (or PO/OH increased), the percentage of 9 significantly decreased because of the further propoxylation of the primary OH of 9. At PO/OH = 1.5, there was virtually no 1,2-propanediol left, but compound 10 was formed. From the above observations, we can

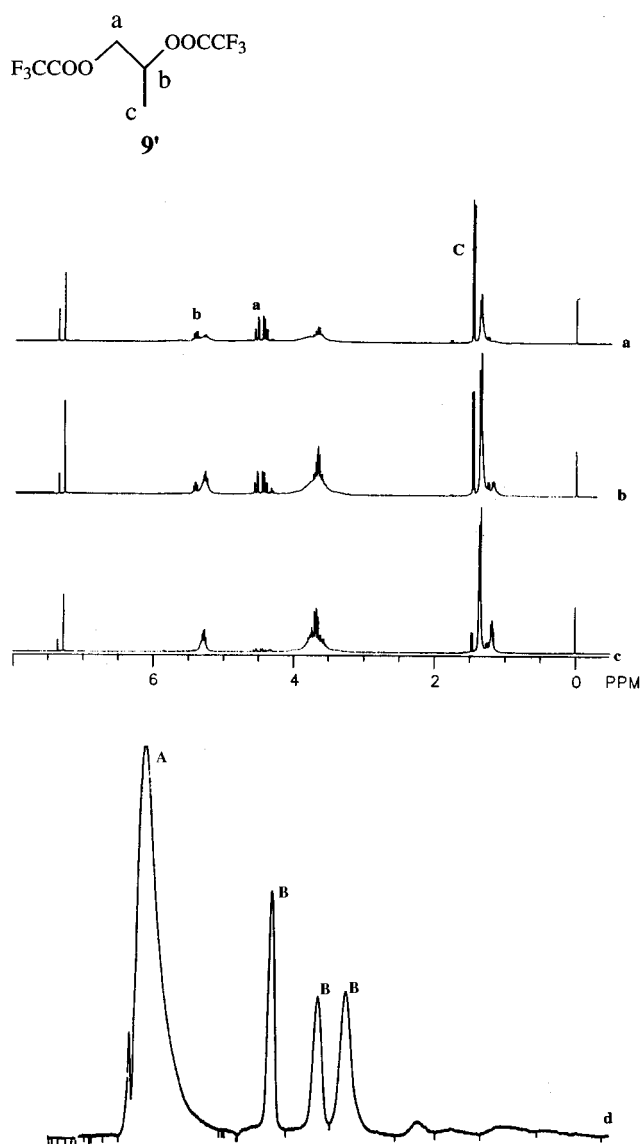
predict the acidity of different types of OH groups: nonreducing sugar O-H (both 1 and 2°) >HO-H>PEP O-H (both 1 and 2°). This also explains the narrow distribution of PEP formed during propoxylation (Figure 1d and Wilson et al., 1996).

**Effect of Water Content in RWP on Hydroxyl Number.** The amount of water in RWP will also considerably affect the hydroxyl number. In Figure 2, the hydroxyl numbers of various PEPs from two different series of RWP were plotted against PO/OH. For RWP with 33.4% water, an increased amount of propylene oxide decreased the overall OH number of PEP. This is also true for RWP with 12.7% water. Also from Figure 2, we can conclude that more water present in RWP actually gives a higher OH number under the same PO/OH ratio. For example, at PO/OH = 1.5, PEP from RWP with 33.4% water, 12.7% water, and the dihydrate (with 9.4% water) have OH numbers of 562.9, 501.0, and 429.3 meq KOH/g, respectively. For PO/OH between 0.5 and 1.5, all RWP-based PEP have OH numbers between 500 and 700 meq KOH/g, and hence they are suitable for spray and thin section pour-in-place types of foam applications.

**Effect of Water Content in RWP on Viscosity and Other PEP Properties.** Usually, high functionality (*f<sub>n</sub>*) initiators such as sucrose (*f<sub>n</sub>* = 8) and lactitol (*f<sub>n</sub>* = 9), after propoxylation when PO/OH is between 1 and 2, result in highly viscous PEPs. These PEPs are usually difficult to handle when making PURF. Different kinds of co-initiator can be added to dissolve sucrose or lactitol, making propoxylation faster and reducing PEP viscosity. Among the co-initiators (water, glycerol, ethylene diamine, propylene glycol, etc.), water is most widely used. Figure 3 shows the relationship

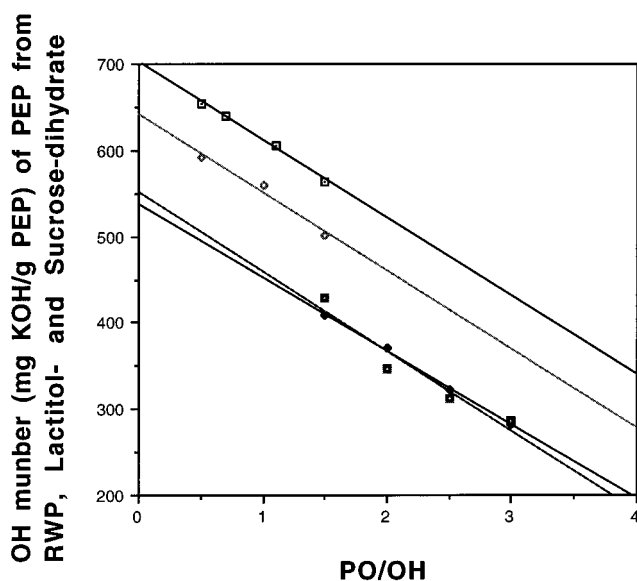
**Table 3. Properties of RWP-PEP, LPEP, Voranol-PEP, and Their Derived PURF**

	PEP Series								lactitol•2H <sub>2</sub> O 9.4% water LPEP	commercial / Voranol-360
	RWP									
	12.7% water			33.4% water						
	1	2	3	1'	2'	3'	4'			
PO/OH	0.5	1	1.5	0.5	0.7	1.1	1.5	1.5	/	
unsaturation (meq/g)	≈0	0.0036	0.0037	≈0	≈0	0.0012	0.0046	0.003	/	
alpha-color	>500	>500	>500	>500	>500	>500	>500	300	/	
	PURF Series								LPEP- PURF	Voranol- PURF
	1	2	3	1'	2'	3'	4'			
mixing time (s)	/	10	10	/	20	20	20	10	10	
cream time (s)	/	50	56	/	68	60	55	64	83	
rise time (s)	/	156	195	/	180	140	145	214	190	
cell size (mm)	/	<0.3 × 0.3	≈0.9 × 1.3	/	≈0.9 × 1.7	≈1.4 × 1.8	≈0.9 × 1.1	<0.5 × 0.5	<0.3 × 0.3	
closed cell content	/	93.3	88.2	/	82.3	73	80.5	85.2	86.9	
foam density (kg/m <sup>3</sup> )	/	29.02	30.37	/	28.84	29.61	29.04	28.9	31.99	
compressive strength (kg × 1000/m <sup>2</sup> )	/	14.8	13	/	15.6	13.5	16.3	12.37	13.72	
onset degradation (°C)	/	269	286	/	259	283	270	298	305	
offset degradation (°C)	/	356	375	/	357	360	343	365	380	



**Figure 1.** <sup>1</sup>H-NMR exhaustive trifluoroacetylation of (a) RWP-PEP 1 with PO/OH = 0.5, (b) RWP-PEP 2 with PO/OH = 1, (c) RWP-PEP 3 with PO/OH = 1.5, and (d) HPLC analysis of RWP-PEP 1: A (LPEP), B (other PEPs).

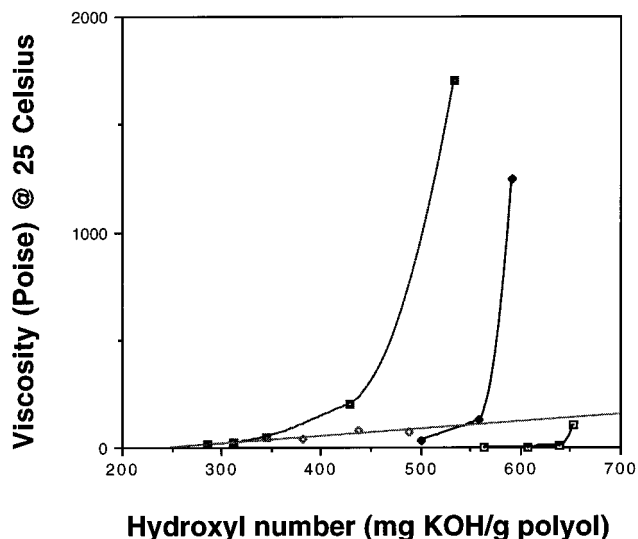
between OH number and viscosity for the same two series of RWP-PEPs listed in Table 3. The viscosities



**Figure 2.** Hydroxyl number of various RWP-PEP (□) series 2, (◇) series 1, (■) LPEP, and (◆) SPEP.

of RWP- and lactitol-based PEP decrease as the extent of propoxylation increases, but for most RWP-PEPs, the viscosities are very close to that of commercial PEP (straight line in Figure 3). In contrast to the low viscosity of lactose-based PEP reported by Viswanathan (1984 and 1990), the low viscosities of RWP-PEPs are due to the extremely low viscosity of 1,2-propanediol **9** and **10** from propoxylation of water. This is confirmed by the light color (having an  $\alpha$ -color slightly higher than 500) and unsaturation determination (far less than 0.04 meq/g for industry applications) of all the RWP-PEPs (see Table 3).

**PURF Properties.** PURFs from RWPs with lower viscosities were synthesized and their properties were compared to those from LPEP and Voranol-PEP foams (used as standards, see Table 3). A simple method for measuring the different reactivities of these PEPs toward PAPI is to measure the macroscopic phenomena of the various time parameters such as cream time, rise time, and tack-free time. Since the foam properties are highly dependent upon the mixing time, the type and amount of catalyst, blowing agent, and isocyanate index, we needed to fix these parameters in each case. In our previous study (Wilson et al., 1996), 10 s of mixing time



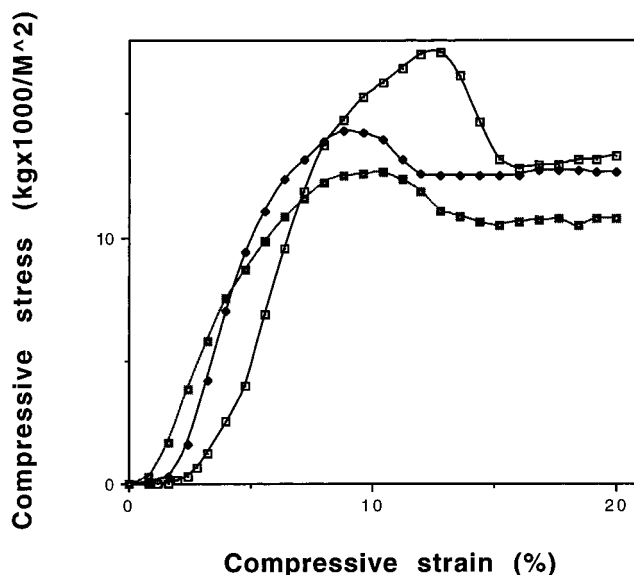
**Figure 3.** Viscosity of PEP as a function of PO/OH ratio: (■) lactitol PEP, (◆) RWP-PEP series 1, (□) RWP-PEP series 2, and (◇) commercial PEP (Dow Voranol for PURF).

resulted in good foaming for both lactitol- and Voranol-based PEP because of their higher reactivities. For PEPs derived from RWP containing 33.4% water, there is not enough foaming when all other parameters are kept constant due to the low reactivity of these PEPs. Hence, the resulting foams have high densities and toughness. In order to obtain low-density PURF, we applied 20 s mixing time for the 33.4% water series of PEP in foam formation. On the other hand, 10 s mixing time is sufficient for PEPs from RWP containing 12.7% water to react with PAPI.

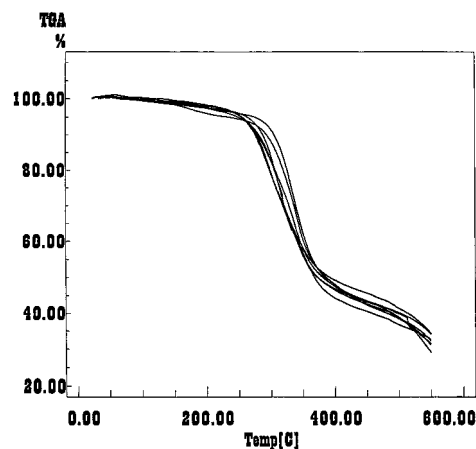
From Table 3, we can see that the PEP from RWP containing 33.4% water shows relatively lower reactivity than LPEP or Voranol-PEP, while the 12.7% water PEPs show similar reactivities to the standards. On the other hand, PEP 2', 3', or 4' gave foams with larger cell size (since the foams are freely blown, the cells are anisotropic and the cell size is indicated by both parallel and perpendicular directions of foam rise in diameters estimated by a caliper) due to the extreme heat released during the foam formation process. This may be a result of the longer mixing time. RWP-PEP 2- and PEP 3-based foams resulted in cell sizes (<0.3 mm of diameter for RWP-PEP 2) similar to that of LPEP and Voranol PEP foams. Both series of RWP-PURFs show good uniformity.

An important PURF application is based upon its insulation properties. Higher closed cell content corresponds to lower thermal conductivity. RWP-based PURF has a closed cell content from 73 to 93% according to our measurement. Although at least 90% closed cell content is required for insulation applications, our results are based upon ASTM D2856-94 procedure C which does not correct for cells opened during sample preparation. Larger error in closed cell content is expected with foams having larger cells, such as RWP-PEP 3'-based foam with cell size being about  $1.4 \times 1.8$  mm. With the same ASTM method procedure A, we predict the closed cell content is at least 90% for foams from PEP 3, 2', 3', and 4' (similar to those from LPEP and Voranol-PEP foam).

In addition to high closed cell contents, all the RWP-PEP-PURFs have densities at about 29–30 kg/m<sup>3</sup> (compared to 28.9 kg/m<sup>3</sup> of LPEP foam and 31.99 kg/m<sup>3</sup> of Voranol-446 foam). This low density may result



**Figure 4.** Compressive strength curve (in direction of foam rise) of PURF prepared according to formulations given in Table 2 using RWP-PEP 4' (□), LPEP (■), and Voranol-360 (◆).



**Figure 5.** TGA of PURF from RWP-PEP, LPEP, and Voranol-446-PEP.

from longer mixing time (20 s) or higher functionality ( $f_n = 9$ ) of LPEP (Wilson et al., 1996). An extremely important advantage of the low-density foam from RWP-PEP is that less blowing agent can be used to achieve higher density and improve other foam properties (e.g., increase foam compressive strength by increase in foam density).

The compressive strength of RWP-PEP-PURF was also characterized. Figure 4 is a typical stress/strain curve of PEP 4'-based foam. As we can see, foam from RWP has a similar curve to those from LPEP- and Voranol-based foams while showing higher resilience (maximum stress reached after 10% strain). All other RWP-PEP-PURFs have a similar curve with a maximum stress appearing after 10% strain (Table 3), but PURF derived from RWP with more water content (here 33.4%) showed higher toughness than the foam from RWP with less water (12.7%). Decreasing the amount of blowing agent (freon) can result in even stronger material from RWP-PEP.

Because most PURF is used in building construction, it is important to know its thermal stability. An indirect measurement of this property is by TGA analysis of the foam. Figure 5 depicts the TGA curves for all the PURFs from RWP-PEP, LPEP, and Voranol-446-PEP.

PURFs from RWP showed very similar thermal behavior to those of LPEP and Voranol, and the curves are well overlapped. The onset degradation temperatures (from 259 to 286 °C) of all the RWP-based foams are slightly lower than those of LPEP and Voranol and the offset degradation temperature is very close for all foam (from 343 to 375 °C). The slightly lower onset degradation temperature may be due to the larger cell size or low density. Since most PURF can be used in the temperature range of -200 to +150 °C, foams based upon RWP are suitable substitutes.

**Conclusions.** Low-cost reduced whey permeates with various water contents are suitable substitutes for sucrose in preparing low viscosity, lightly colored polyether polyols with low functionality and high hydroxyl number. Polyurethane rigid foams derived from the these polyols were shown to have low densities, high closed cell contents, strong mechanical properties, and high thermal stability.

#### ACKNOWLEDGMENT

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