



Regeneration of Polyol by Pentaerythritol-assisted Glycolysis of Flexible Polyurethane Foam Wastes

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ABSTRACT

The aim of this work is to introduce a novel chemical recycling approach for polyurethane foam wastes which can produce useful chemicals. Flexible polyurethane foam was dissolved in a mixture of diethylene glycol (DEG) and pentaerythritol (PE). PER is a useful choice in recycling processes because of its OH functional groups and its structural similarity to polyols. Meanwhile, there are several environmental advantages for chemical recycling of polyurethane foam wastes due to the capabilities of the above method. The trans-esterification process cleaves the urethane structure, producing OH containing chemicals. The product is separated in 2 phases which are both useful in the production of new PU foams. The split product was characterized by several analytical methods, e.g., chromatography and spectroscopy. Optimum reaction condition and the role of recycled polyol on foam properties were investigated, as well. The solvent system contained DEG/PER as 9/1 ratio. Sodium hydroxide was used as the catalyst and the optimum reaction time was 4 h. Separated phases of the product are reusable in polyurethane foam formulation: upper phase in flexible (40%) and lower phase in rigid (30%) foams to obtain desirable physical properties in the final product. This method can be introduced as a route for reduction of environmental hazardous materials, while regenerating valuable raw materials.

Key Words:

polyurethane;
waste;
recycling;
polyol;
pentaerythritol;
glycol.

INTRODUCTION

Nowadays, polyurethane products are an important part of everyday life. The variety of polyurethane products reaches from flexible and rigid foams over thermoplastic elastomers to adhesives, paints and varnishes. This variety of usage would result in a huge amount of consumption, causing some environmental problems [1]. Solid waste disposal has been a problem in today's world, and overall trends indicate that the overall amount of solid wastes that we generate

continues to increase. Because polyurethanes are used in so many diverse applications and industrial uses, they enter the municipal solid wastes stream, usually by ways of discarded consumers and industrial products. These products, e.g., upholstered furniture, mattresses and automobile parts, are frequently durable goods with a long lifespan.

By weight, approximately 1.3 million tons of waste polyurethanes are generated each year only in the

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US as part of the municipal solid wastes representing five percent of all current plastic wastes [2]. Public awareness of environmental issues has increased enormously, especially of the facts that the world has limited natural resources and a limited capacity to manage the volume of wastes which human activities generate. Recycling of polymeric products has been investigated to enhance the environmental protection and avoid landfilling. Among different polymers, recycling of polyurethane has always posed some challenges. Polyurethane disposal is generally via landfill, incineration or recycling. In 2003, 55.4% of all the municipal solid wastes generated ended up in landfills, while only 30.6% was recycled.

Traditional approaches to recycling such materials include mechanical regrinding and then using the reground materials as fillers [3]. Chemical recycling of polyurethanes by hydrolysis or aminolysis is for the most part considered economically uncompetitive compared to formulating with virgin raw materials. It should be mentioned that the high temperature and pressure required for hydrolytic decomposition of urethane structure [4] in addition with difficulties in reformulation of aminolysis products have prevented these methods to be useful and feasible in industrial scale [5,6].

Glycolysis, known as alcoholysis which was developed during 1980s, is the most widely used chemical recycling method for PU wastes. The main aim of this process is the recovery of polyols from waste PU for the production of new materials. A process for preparing a recyclate polyol obtained from glycolysis of PU comprises reacting scrap PU with a short-chain compound containing at least 2 OH groups in the presence of a catalyst with addition of a cyclic carbonate. The process results in a recyclate polyol having a low primary aromatic amine content which may be used to produce cellular or non-cellular polyurethanes, polyurea-polyurethanes, or polyisocyanurates. The reacting agent is usually diethylene glycol (DEG) and co-reagent would be diethanol amine (DEA). A reaction temperature below 180°C lowers catalyst activity severely and above 220°C produces undesired side reactions towards amines. For the catalyst it is important that the formation of aromatic amines must be avoided. Since the glycol acts as reactant and solvent at the

same time, the optimum glycol/PU ratio of 60/40 is usually used [7,8]. The interest in glycolysis method has brought it in the centre of recycling research activities in the last decade.

New aspects of PU recycling are reported every year. Methanol-assisted recycling [9], adhesive production from waste PU [10], chemical recycling of mixed PUF wastes prior to separation [11] are some of the recent advances in this field. Meanwhile, researchers have also investigated the influence of glycolysis product on flexible PUF [12], split-phase recycling [13] and elastomer recycling [14].

Our research team has reported some studies on glycol-treated PU foams in the last few years. Integral skin PUF recycling [15,16], split-phase development [17], glycerin-assisted recycling [18] and application of microwave power source for chemical recycling of PUF [19-21] constitute the main research activities performed in our laboratory.

In the current work an attempt was made to use DEG-pentaerythritol as a new solvent-reactant mixture to investigate the recyclate specifications and also the effect of recyclate on formulated foams. According to our literature survey, there is no report indicating the application of this two-component solvent system in PUF recycling. Pentaerythritol contains 4 hydroxyl functional groups which would effectively accelerate the dissociation of urethane structure. Its multi-active sites for conducting a recycling process and its similarity with polyol structures are highly important. The upper phase of recyclate is useful in flexible PUF production. Also, regeneration of polyol by pentaerythritol-assisted recycling of polyurethane provides a high OH/molecular weight ratio in the lower phase which is useful for high quality rigid foam production.

EXPERIMENTAL

Materials and Apparatus

Diethylene glycol (DEG), pentaerythritol (PER) and sodium hydroxide were purchased from Merck, Germany. Polyether polyols (BAYNAT[®] PU-80IF04 for flexible foam and DaltoFoam TA[®] 14066 for rigid foam) containing all the required additives, isocyanate (MDI-Suprasec[®] 5005) and other

Table 1. Specifications of virgin polyols used in flexible and rigid PU foam formulations.

Property	BAYNAT® PU-80IF04	DaltoFoam TA® 14066
Appearance	Semi-clear liquid	Viscous yellow liquid
Viscosity	1740 cps (25°C)	5260 cps (25°C)
Specific gravity	1.03 g.cm ⁻³ (25°C)	1.06 g.cm ⁻³ (25°C)
Water content	1.2 %	2.3%
pH	10.1	9.5
OH number	345 ± 20	430 ± 20

chemicals from HUNTSMAN Co. were used in polyurethane flexible foam formulation. Technical data of virgin polyols and MDI-Suprasec® 5005 are listed in Tables 1 and 2, respectively. Spectroscopy grade tetrahydrofuran (Merck, Germany) and polyethylene glycol standards (Merck, Germany) for GPC analysis were used as received. Molecular weight distributions were determined by gel permeation chromatography (GPC). Chromatograms were obtained using a 6A Shimadzu instrument, Japan. Water content was measured by Metler-D18 Karl Fischer auto-titrator, Switzerland. Tensile strength of PUF samples was determined according to ASTM-D 638 by Instron 11 (USA) tensometer. Infrared spectra were obtained by Tensor 27 Bruker (Germany) FTIR spectrometer instrument in transmission mode.

Degradation of Flexible PU Foam in DEG-PER Mixture

Flexible polyurethane foam was decomposed by a mixture of diethylene glycol and pentaerythritol (in different mass ratios) in presence of NaOH as the

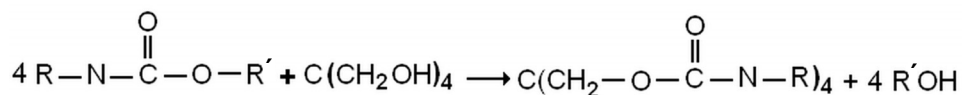
Table 2. Specifications of MDI used in flexible and rigid PU foam formulations.

SUPRASEC® 5005	
Appearance	Dark brown liquid
Viscosity	220 cps (25°C)
Specific gravity	1.23 g.cm ⁻³ (25°C)
NCO value	30.9 % by wt. NCO groups (group wt : 42)
Average functionality	2.7
Flash point	233°C
Fire point	245°C

catalyst. A reflux system containing a 1 L three-necked, round-bottom flask equipped with a condenser, thermometer, and agitator was used. The solvent mixture (200 g) was placed in the reaction flask and heated to 160°C. Then 1 g NaOH was dissolved in the mixture, temperature was increased to 200°C and 200 g of shredded PUF was added to the flask (15 g/min). Digestion occurred approximately as foam was in contact with solvent by an acceptable rate. Degradation process was continued for 5 h under atmospheric pressure and the product was separated into two phases of upper clear yellow solution and lower light brown upon cooling after 24 h. Both phases of the product were characterized using instrumental and classical analytical methods.

Preparation of PUF Samples by Product Phases

According to the characterization of recycling product, top and bottom phases were used in the formulations of flexible and rigid PU foams, respectively. Phases were separated and one shot method was applied for foam preparation. Polyol blends and additives were premixed in a 300 mL carton beaker by 2 min mixing followed by MDI addition. The pre-polymer was mixed for 10 s (beyond creaming time) and then allowed to rise freely, remaining at room temperature for post-cure step in the next 24 h. The starting formulation stoichiometry for flexible PUF was 100 parts by weight (pbw) of BAYNAT® PU 80IF04 and 65 pbw of SUPRASEC® 5005. The virgin rigid PUF was produced by 100 pbw of DALTOFOAM® TA 14066 and 133 pbw of SUPRASEC® 5005. Virgin polyol was substituted by recycling product of different ratios.



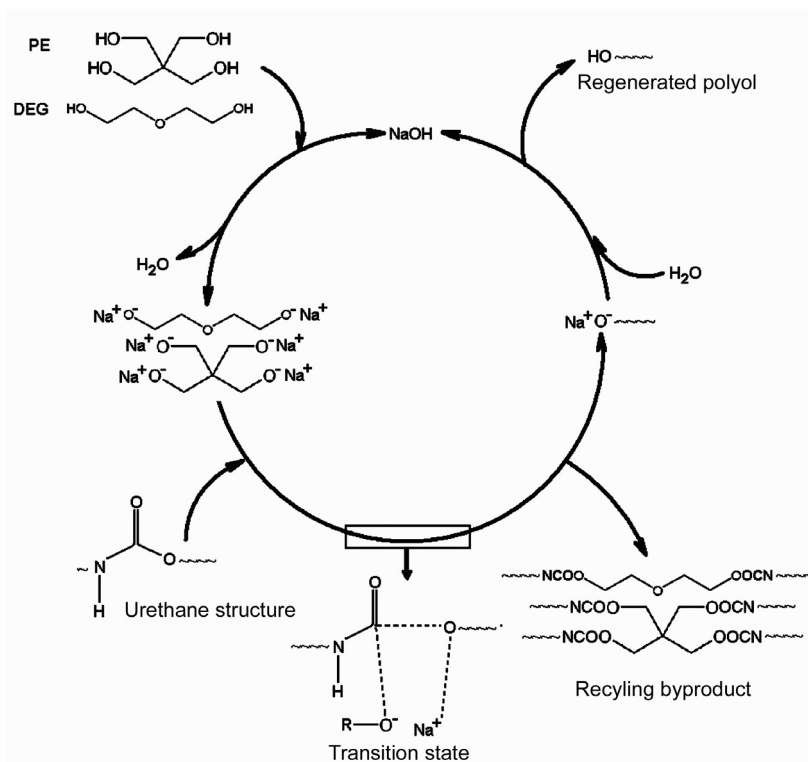
Scheme I. Reaction between polyurethane structure and pentaerythritol.

RESULTS AND DISCUSSION

Reaction Set

Previous research works confirmed that urethane bond cleavage would occur in presence of alkaline metal hydroxide as a catalyst [13-19]. A set of reactions was performed by different ratios of DEG/PER. Pentaerythritol contains 4 hydroxyl

groups and thus demonstrates high functionality trans-esterification reaction (Scheme I). The amount of reactants and product phases are given in detail in Table 3. Unreacted PER was vacuum filtered after phase separation. In all reactions, 100 g of PUF was reacted. Schematic mechanism of the reaction is depicted in Scheme II. DEG and PER would react with $-\overset{\text{O}}{\parallel}{C}-O-$ functional groups. A high amount of



Scheme II. Schematic mechanism of recycling process via trans-esterification reaction in presence of DEG and PER.

Table 3. Reaction set for PER assisted recycling of flexible PU foam (all data are in gram).

Reaction	DEG	PER	Total product	Upper phase	Lower phase	Remaining PER
1	65	35	150.9	52.4	98.5	28.9
2	70	30	174.3	69.9	104.4	19.2
3	75	25	176.8	72.2	104.6	17.5
4	80	20	182.4	76.8	105.6	11.2
5	85	15	188.2	77.1	111.1	6.3
6	90	10	196.6	83.3	113.3	0.9

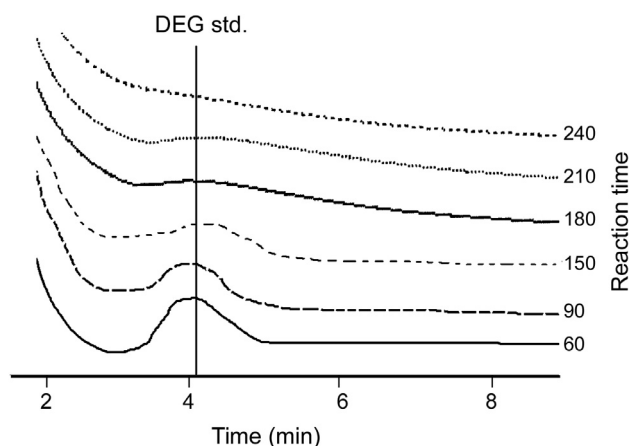


Figure 1. HPLC chromatograms of upper phase in recycling product for determination of DEG content.

PER reduces the reactivity of OH groups in the solvent systems. Interestingly, the product would be increased by increasing the DEG/PER ratio, while the evaporated mixture is lowered. According to the data obtained in experimental set, DEG/PER ratio was set as 9/1. It was under this condition that other experimental parameters were investigated.

In order to optimize the reaction, 2 parameters were investigated: the molecular weights of product phases by GPC and the remaining DEG in recycling product by HPLC analysis. In order to determine the molecular weights of the products, PEG with different MWs (400, 800, 1000, 1600, 2000, 4000 and 10000) were used as internal standards in order to perform the calibration model. As shown in Figures 1 and 2, DEG

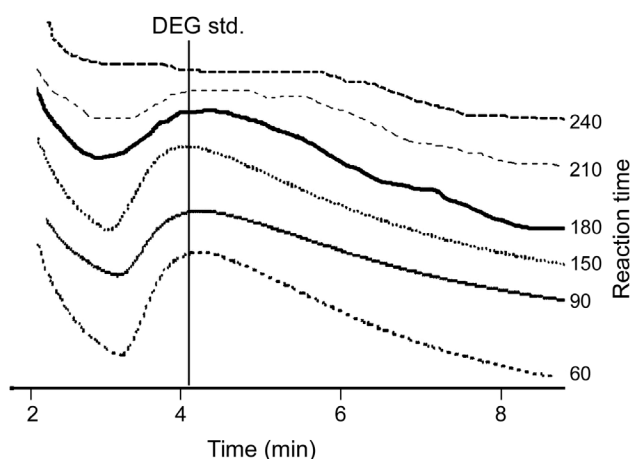


Figure 2. HPLC chromatograms of lower phase in recycling product for determination of DEG content.

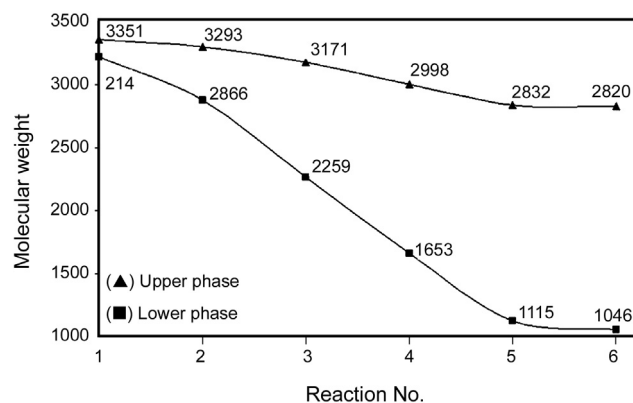


Figure 3. Molecular weights of recycling products phases obtained by GPC.

content of both phases would be near 0.0% and this is important to obtain the best reaction time. Where, the MWs of both phases would be at minimum in reaction number 6 (Figure 3). Product of the reaction 6 at 240 min was cooled overnight, being separated as 2 phases.

FTIR Analysis of Regenerated Polyols Structures

Fourier transform infrared spectroscopy was used to study the chemical structure of recycling product phases and comparing them with virgin polyols. Some of the main similarities, observed in molecular structure of recycling product and virgin polyols are explained below:

BAYNAT® PU 80IF04 and upper phase:

- Intensive band of the aliphatic ether group associated to hydroxyl end groups at 1110 cm^{-1} ;
- Bending vibrations of methylene groups in 1372 and 1456 cm^{-1} ;
- Stretching vibrations of carbon-hydrogen bonds in aliphatic carbons at $2870\text{-}2971\text{ cm}^{-1}$ spectral region;
- Stretching vibration of OH group at 3486 cm^{-1} .

DALTOFOAM® TA 14066 and lower phase:

- Stretching vibrations of OH groups and C-O ether at 1105 cm^{-1} ;
- Stretching vibrations of methylene group at $2871\text{-}2970\text{ cm}^{-1}$ region;
- Bending vibrations of methylene groups at 1375 , 1465 , 1520 and 1625 cm^{-1} and also assigned to remaining additives [19].

Comparing Regenerated and Virgin Polyols

Specific gravity of the separated phases was similar to virgin polyols (flexible polyol: 1.03 g/cm³, top phase: 1.09 g/cm³, rigid polyol: 1.06 g/cm³, and bottom phase: 1.13 g/cm³). One of the most important parameters that affect the injection process in PU machines is the viscosity of polyol. Higher values of viscosity would cause several problems, e.g., heterogeneity in prepolymer, over-loaded pressure of polyol stream which destroys the injection moulding machine, etc. Viscosity of BAYNAT® PU 80IF04 is 1740 cps, while the upper phase of recycled polyol has a viscosity of 1592 cps. Regenerated polyol in lower phase has a viscosity about 5660 cps while virgin rigid polyol's viscosity is 5260 cps (all at 25°C).

Water as a blowing agent has an important function in the synthesis of PU foam. Water content of both virgin polyols and both regenerated polyols were determined by automated Karl-Fischer titrator. The water contents of both product phases were less than virgin polyols (flexible polyol: 1.2 w/w%, upper phase: 0.8 w/w%, rigid polyol: 2.3 w/w%, and lower phase: 1.3 w/w%).

The pH of all 4 polyols was determined by digital pH meter. The pH was similar for flexible polyol and upper phase (10.1 and 9.8, respectively) but with higher amounts of alkaline components in the lower phase of the recycling product, the pH would be higher in comparison with virgin rigid polyol (9.9 and 9.5, respectively). Certainly, the most important parameter would be MW which determines application potential of the products phases. Higher molecular weight of OH containing product provides higher cross-linking density and also more flexibility of the formulated foam. The upper and lower phases were applied in cold cure flexible and rigid foam formulations, respectively.

Application of Product Phases in New Foam Formulation

The main aim of this study was to regenerate raw materials and according to comparative tests on the product, upper phase was reused in the synthesis of flexible foam and the lower phase was used in rigid foam production. The starting formulation of typical cold cure flexible and rigid polyurethane foams is

given in detail in Experimental section. It is evident that higher than 30% of the recycled polyol can be tolerated by increasing the MDI usage. In fact, the narrow molecular weight distribution of recycled polyol enhances the functionality that parallels OH-value of the polyol. In other words, while increasing the percentage of recycled polyol, it is necessary to add more MDI.

One of the other ways to compare recycled and virgin polyols is to compare their reactivities by recording the time factor in foaming procedure. Regenerated phases were used in polyol blends up to 50%. All ingredients and their quantities in foam synthetic process in addition to reactivity parameters (time control records) are detailed in Tables 4 and 5 for flexible and rigid foams, respectively.

In the physical properties test measurements two regenerated foams were compared with those of the virgin based foams. S8 from Table 4 (flexible sample) and S₆ from Table 5 (rigid sample) are the typical foam samples. As shown in Table 6, physical properties of the foam samples containing regenerated polyols demonstrate reliable results which ensure the application of recycling products in foam synthesis. In other words, improvement in physical properties of rigid foam is in accordance with incremental increase in cross-link density of the synthesized foam in the presence of PER based oligomers. These oligomers are hyperbranched and highly functional. It is also noticeable that some characteristics of flexible foam would be decreased in relation with open cells which are more prominent in recycled polyol containing samples because of high functionality in low molecular weight oligomers.

Brief Cost Analysis

As the polyurethane foam wastes are easily accessible, the main effective parameters that define the economic feasibility of the proposed recycling approach are the prices of the chemicals and energy costs. While the approximate price of pentaerythritol (1.9 USD per kg), DEG (0.6 USD per kg), NaOH (0.3 USD per kg) and assuming 0.2 USD for achieving 1 kg of waste PUF, the chemicals cost for the recycling process with 90% yield would be 0.52 USD per kg of product. In addition, energy cost for this process is approximately 0.19 USD per kg of

Table 4. Cold cure flexible PU foam formulation, properties and reactivity (ingredients: pbw; time: s).

Sample code	BAYNAT® PU 80IF04	MDI Suprasec® 5005	Recyclate upper phase	Water	Cream	Gel	String	End of rise	Tack free	Appearance
S ₁	100	100	0	0.0	5	6	30	44	60	Excellent
S ₂	90	100	10	0.0	5	6	28	40	58	Excellent
S ₃	80	100	20	0.2	6	7	28	40	54	Good
S ₄	70	100	30	0.3	6	7	26	37	49	Good
S ₅	70	105	30	0.5	6	6	28	40	55	Excellent
S ₆	60	100	40	0.5	5	7	28	37	50	Good
S ₇	60	110	40	0.7	6	8	29	40	49	Good
S ₈	60	115	40	0.8	5	7	26	45	52	Excellent
S ₉	50	120	50	0.8	5	5	27	39	44	Good
S ₁₀	50	125	50	1.0	5	6	25	40	49	Good

Table 5. Rigid PU foam formulation, properties and reactivity (ingredients: pbw; time: s).

Sample code	Polyol Dalto Foam TA® 14066	MDI Suprasec® 5005	Recyclate lower phase	Water	Cream	Gel	String	End of rise	Tack free	Appearance
S ₁	100	100	0	0.0	5	6	30	44	60	Excellent
S ₂	90	100	10	0.0	5	6	28	40	58	Excellent
S ₃	80	100	20	0.3	6	7	26	42	52	Good
S ₄	70	100	30	0.3	6	7	25	35	47	Good
S ₅	70	110	30	0.6	5	6	25	41	52	Good
S ₆	60	100	40	0.6	5	6	26	38	50	Good
S ₇	60	110	40	0.8	6	7	26	39	48	Medium
S ₈	60	115	40	0.8	4	6	24	40	51	Good
S ₉	50	120	50	1.0	4	5	24	33	36	Medium
S ₁₀	50	130	50	1.2	4	6	23	36	40	Medium

Table 6. Comparative quality control of foams formulated by virgin and regenerated polyols.

Physical characteristic	Rigid	S ₆	Flexible	S ₈
Core density (kg m ⁻³)	35	39	60	58
Tensile strength vertically (kPa)	111	230	187	165
Elongation-at-break (%) vertically	10	18	45	38
E-modulus (kPa) vertically	1558	5205	2025	3216
Tensile strength horizontally (kPa)	108	216	205	219
Elongation-at-break (%) horizontally	9	15	47	50
E-modulus (kPa) horizontally	1388	4256	1388	4256
Compression set (%)	14	12	0.2	3.5
Dimensional stability (-30°C / 24 h) (%)	OK	OK	OK	OK
Dimensional stability (70°C / 24 h) (%)	OK	OK	OK	OK
Compression set (75%, 22 h) (%)	12.0	13.0	8.5	9.0

recyclate. Thus, the total recycling cost would be 0.71 USD per kg.

It is noticeable that the costs for human resources and capital investments for such an industrial plant must be also considered as both also depend on the manufacturing scale. The average price of virgin polyol is 2.7 USD per kg and thus application of 40% of recycling product (estimated overall price of 1 USD) in foam formulation would provide a rate of 0.68 USD per kg of mixed polyol. If the recycling product is utilized in the production of only 50000 tones per year (with approximate consumption of about 300000 tones annually and polyol/isocyanates ratio is 1/1) then 17 million USD would be saved. The environmental friendly aspects of proposed recycling approach and its capabilities for job opportunity would be of high importance, as well.

CONCLUSION

The regenerated polyol provided by pentaerythritol-assisted glycolysis of flexible PUF wastes is capable of high quality foam formulation production. By optimizing the recycling approach, it is possible to replace about 40% of the virgin polyol by glycolysis product. Although the economic aspects of the proposed method are of high interest, and the environmental pollution reduction is the main advantage of the studied polyol regeneration approach.

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