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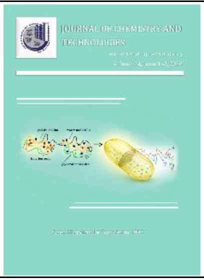

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# DEPOLYMERIZATION OF RIGID POLYURETHANE WASTE VIA CATALYTIC GLYCOLYSIS IN THE PRESENCE OF K, Na, AND Ca COMPOUNDS

Andrii R. Kovalchuk[1], Oleksandr S. Ivashchuk[1], Wolodymyr Ya. Suprun[2], Olha R. Burets[1], Volodymyr V. Reutskyy[3]

*[1]Department of Chemical Engineering, Lviv Polytechnic National University, 12 S. Bandera str., Lviv, 79013, Ukraine*
*[2]Martin-Luther University, Halle-Wittenberg, 11 Universitätsplatz, Halle, 06108 Germany*
*[3]Department of Physical, Analytical and General Chemistry, Lviv Polytechnic National University, 12 S. Bandera str., Lviv, 79013, Ukraine*

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## Abstract

**The process of chemical recycling of rigid polyurethane waste via catalytic glycolysis with diethylene glycol (DEG) was investigated. The purpose of this work was to study the influence of the nature of some alkaline earth compounds as homogeneous catalysts on the depolymerization of PU into a so-called recycled polyol with properties suitable for further application. The glycolysis process was carried out at a temperature of 220 °C for 40 min. with a mass ratio of polyurethane (PU) waste to DEG of 1 : 2.5. The K-, Na-, and Ca acetates and K carbonate, as homogeneous catalysts at constant mass concentrations (0.2 %, 0.4 %, and 0.6 %), were used. The effectiveness of depolymerization was evaluated by the physical and chemical properties of the obtained liquid products (i.e., recycled polyol), namely by hydroxyl-, acid-, and amine-number as well as by dynamic viscosity. It was found that the studied catalysts significantly intensify the depolymerization process compared to the non-catalytic reaction. Potassium carbonate at a concentration of 0.4 % allowed to obtain recycled polyol with a high hydroxyl number. The obtained results confirm the feasibility of K and Na acetates and carbonates for controlled depolymerization of solid PU waste.**

*Keywords*: polyurethane; depolymerization; catalysis; glycolysis; waste; recycling.

# ДЕПОЛІМЕРИЗАЦІЯ ВІДХОДІВ ТВЕРДОГО ПОЛІУРЕТАНУ МЕТОДОМ КАТАЛІТИЧНОГО ГЛІКОЛІЗУ В ПРИСУТНОСТІ СПОЛУК K, Na, Ca

Андрій Р. Ковальчук[1], Олександр С. Іващук[1], Володимир Я. Супрун[2], Ольга Р. Бурець[1], Володимир В. Реутський[3]

*[1]Кафедра хімічної інженерії, Національний університет "Львівська політехніка", вул. С. Бандери 12, Львів, 79013, Україна*
*[2]Мартін-Лютер Університет Галле-Віттенберг, Університетська площа 11, Галле, 06108 Німеччина*
*[3]Кафедра фізичної, аналітичної та загальної хімії, Національний університет "Львівська політехніка", вул. С. Бандери 12, Львів, 79013, Україна*

## Анотація

**Досліджений процес хімічної переробки відходів твердого поліуретану методом каталітичного гліколізу з використанням діетиленгліколю (DEG). Метою роботи було вивчення впливу природи каталізатора на отримання рециклькованих поліолів з властивостями, придатними для подальшого використання. Процес гліколізу проводили за температури 220 °C протягом 40 хв. при масовому співвідношенні відходів поліуретану (PU) до DEG 1 : 2.5. Проведено аналіз ефективності ацетатів K, Na, Ca та карбонату K, кожен з яких використовувався при масових концентраціях (0.2 %, 0.4 % та 0.6 %). Ефективність деполімеризації оцінювали за фізико-хімічними показниками отриманих рідких продуктів, а саме: гідроксильним, кислотним, амінним числом та динамічною в'язкістю. Встановлено, що досліджені каталізатори інтенсифікують процес деполімеризації порівняно з некаталітичною реакцією. Калій вуглекислий у концентрації 0.4 %, дозволив отримати рециклькований поліол з високим гідроксильним числом. Експериментальні дані підтверджують доцільність використання ацетатів та карбонатів лужних металів для керованої деполімеризації відходів твердого PU.**

*Ключові слова*: поліуретан; деполімеризація; каталіз; гліколіз; відходи; переробка.

**Corresponding author: tel.: +380322582657; e-mail: oleksandr.s.ivashchuk@lpnu.ua*

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## Introduction

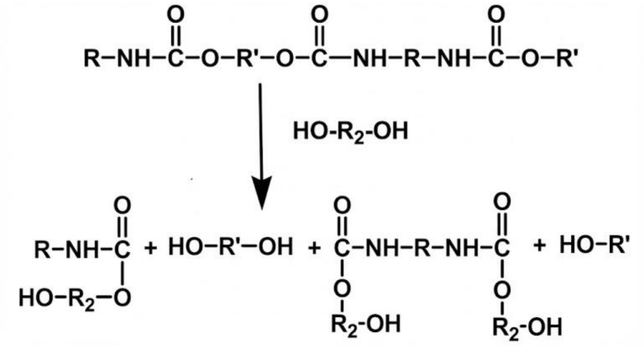
Polyurethanes (PU) are among the most versatile classes of polymers [1], with wide applications across industries, including construction, automotive [2], furniture production, coatings, adhesives, and biomedical materials [3]. The global PU market continues to grow rapidly [1], with its volume estimated to have reached 20 million tons [2] by 2025. However, the vast majority of polyurethane products, in particular rigid polyurethanes, are thermosetting polymers [4]. Their three-dimensional cross-linked structure precludes reprocessing by traditional thermoplastic extrusion [5], thereby creating environmental and economic problems.

The continuous growth of PU waste is a global environmental problem [6]. Traditional disposal methods, such as organized landfilling [5] and incineration, are ineffective and environmentally harmful. Landfilling results in long-term pollution and inefficient use of land resources, as PU waste undergoes prolonged anaerobic and biodegradation [6]. Incineration [7], although it allows for energy recovery, is accompanied by emissions of toxic gases, including amines, nitrogen oxides ($NO_x$), and hydrogen cyanide (HCN) [5]. In the context of the global transition to the principles of the circular economy [8], which entails maximizing the reuse of waste materials within the production cycle, the development of effective technologies for recycling PU waste is highly relevant and necessary [9].

Methods of PU waste recycling are divided into mechanical and chemical [10]. Mechanical recycling, which consists of grinding and subsequent use as a filler in new composite materials, is the simplest but also the least effective method [4]. This approach leads to so-called “downcycling”, i.e., obtaining a secondary product with lower performance characteristics and lower cost compared to the virgin material [10].

In contrast, chemical recycling [11] aims to depolymerize PU into a mixture of polyglycol oligomers [8], which can be used as a low-cost secondary raw material for the synthesis of new PU products. Methods of chemical recycling of PU include hydrolysis, aminolysis, alcoholysis, acidolysis [12], and glycolysis [13]. Among them, glycolysis is considered one of the most promising methods due to relatively mild process conditions [14], high-quality recovered polyols, and potential for industrial implementation [15].

Glycolysis of polyurethanes is a complex process of catalytic depolymerization [16], in which, under the action of an excess of diol (or low-molecular-weight glycol), the cleavage of the C–O bonds within the urethane (–NH–COO–) and ester (–C(=O) – O–) groups of the macromolecule occurs [3] (Scheme).



**Scheme. General scheme of the PU glycolysis reactions in the presence of excess glycol**

The stepwise reactions, such as the ester cleavage of the ester fragments, (-C(=O)-O-R′-), and the synchronous subsequent interaction with hydroxyl groups of glycol, (HO-$R_2$-OH), constitute a process of destructive transesterification, as a result of which PU macromolecules degrade, undergo transesterification, and form a mixture of various oligomeric polyols [10]. In addition to these target reactions, two side reactions are possible, leading to the formation of compounds with terminal amine and carboxyl groups [17].

According to [17], the stronger the Lewis base, the more side amine compounds are formed during the base-catalyzed depolymerization-transesterification of PU macromolecules.

In general, transesterification can proceed under both acid- and base-catalytic conditions. Acid catalysis under conditions of PU depolymerization-transesterification has several disadvantages, as the use of strong acids leads to several side reactions with bases, including rapid catalyst deactivation and equipment corrosion. In this regard, mild base catalysis using alkali and alkaline earth metal compounds is recommended for PU depolymerization.

In this study, diethylene glycol (DEG) was used as a highly active and readily available glycolysing agent [15]. Furthermore, the presence of the ether fragment (-$CH_2$-O-$CH_2$-) in the DEG molecule can positively influence the physico-chemical properties of newly synthesized polyurethane products [1].

Glycolysis products are typically a homogeneous liquid mixture consisting of excess glycol [14] and various oligomers containing terminal hydroxyl groups. The presence of reactive HO-groups allows further application of the obtained recycled polyols for the synthesis of new polyurethane materials [18] via conventional reactions with di-isocyanates.

Non-catalytic glycolysis is a slow process [16], requiring long reaction times and high temperatures (up to 250 °C), making its implementation technologically challenging [14]. The use of catalysts allows the process to be intensified at temperatures below 250 °C. Various fundamental catalysts [19] for PU glycolysis are known, including amines (diethanolamine) [20], organometallic compounds (tin octoate, titanium butylate) [21], and alkali and alkaline earth metal compounds (hydroxides, carboxylates, and carbonates) [22]. Recently, more complex systems have been investigated, including ionic liquids [23] and magnetic heterogeneous catalysts [24], to improve the separation efficiency of the spent catalysts from reaction products.

Alkali and alkaline earth metal acetates and/or carbonates [20] attract special attention due to their availability, low cost, and high catalytic activity.

This work aimed to investigate the catalytic activity of sodium, potassium, and calcium acetates, as well as potassium carbonate, in the glycolysis of rigid polyurethane waste using diethylene glycol, and to determine their influence on the physicochemical properties of the resulting recycled polyol.

## Experimental

Foamed PU used in furniture production was employed as the raw material in the study. Before use, the PU was ground to a particle size of 2÷5 mm.

The glycolysis process was carried out in a 250 cm$^3$ glass three-necked flask (reactor) equipped with a mechanical stirrer and a thermocouple, which was thermostated in a glycerol bath (Fig. 1).

In a typical experiment, 75 g of DEG was charged to the flask and heated to 220 °C with constant stirring at 300 rpm. A weighed portion of the corresponding catalyst (mass fraction: 0.2 %, 0.4 %, or 0.6 %) was added to the preheated DEG and stirred until complete dissolution. After that, 30 g of ground PU was added to the mixture in equal small portions about 2.0±0.1 g, over a strictly controlled period of 10 min. The reaction time (40 min) was recorded starting immediately after the addition of the final portion of PU. Throughout the entire process, the temperature was maintained at 220 ± 5 °C. Upon completion, the heating was switched off, the stirrer was stopped, and the glycerol bath was lowered. The reaction mixture was allowed to cool in the reactor for 8 minutes to mitigate thermal shock, after which it was transferred to a separate vessel for final cooling to room temperature. A control experiment was conducted using a procedure similar to that employed in the catalyzed experiment, but without a catalyst.

A thermocouple was placed in the neck I to monitor the temperature of the solution inside the flask. PU loading was carried out through neck II.

The final samples of PU depolymerizate (so-called recycled polyol) were analyzed by titrimetric methods for the content of active hydroxyl groups, for the formation of amine and acid compounds, and for dynamic viscosity.

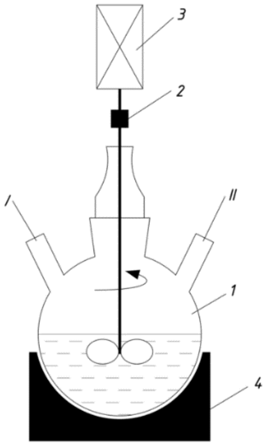


**Fig. 1. Scheme of the experimental setup for PU glycolysis: 1- flask, 2 – silicone sleeve, 3 – electric stirrer drive, 4 – thermostated glycerol bath, I – thermocouple, II – polyurethane loading**

The hydroxyl number (HON) was determined in accordance with ISO 2554:2014. The acid number (AcN) was determined in accordance with ISO 2114:2000 by titrating the sample with KOH solution. The amine number (AmN) of the samples was determined by acid-base titration in accordance with ISO 25761:2014.

The dynamic viscosity was measured at 20 °C using a Lamy Rheology First Plus rotational viscometer equipped with an SVTR 9 measuring system and a rotating spindle.

## Results and Discussion

In all experiments, complete dissolution of rigid polyurethane was observed within 40 minutes. This indicates the successful depolymerization of the cross-linked polymer network and its conversion into a liquid product.

Visually, all obtained recycled polyols were brown, viscous liquids.

*Table*

**Dynamic viscosity (ν) and hydroxyl value (HON) of PU depolymerizates (recycled polyol) in the presence of K, Na, Ca compounds (T = 220°C, τ = 40min)**

| Catalyst | Catalyst content, % | ν, mPa·s 20° | $HON_{RM}$, mg KOH·g$^{-1}$ | Δ HON | $RC_{HON}$, % |
|---|---|---|---|---|---|
| Initial mixture | - | 36 | 778* | – | – |
| Without catalyst | - | 726 | 712 | –66 | –8.5 |
| Ca-acetate | 0.2 | 734 | 730 | –48 | –6.2 |
| | 0.4 | 790 | 756 | –22 | –2.8 |
| | 0.6 | 397 | 753 | –25 | –3.2 |
| K-acetate | 0.2 | 686 | 786 | +8 | +1.0 |
| | 0.4 | 786 | 732 | –46 | –5.9 |
| | 0.6 | 629 | 735 | –43 | –5.5 |
| K-carbonate | 0.2 | 776 | 749 | –29 | –3.7 |
| | 0.4 | 268 | 798 | +20 | +2.6 |
| | 0.6 | 257 | 786 | +8 | +1.0 |
| Na-acetate | 0.2 | 581 | 547 | –231 | –29.7 |
| | 0.4 | 619 | 557 | –221 | –28.4 |
| | 0.6 | 544 | 590 | –188 | –24.2 |

*Calculated HON value relative to the initial composition of the reaction mixture with a DEG content of 75 %

The absolute change of the hydroxyl value (ΔHON) to DEG at their concentration of 75 % and the relative change ($RC_{HON}$) were calculated using the following equations, respectively:

$$\Delta HON = HON_{RM} - HON_{DEG75} \qquad (1)$$

$$RC_{HON} = \frac{\Delta HON}{HON_{DEG75}} \cdot 100\% \qquad (2)$$

where $HON_{DEG75}$ is the hydroxyl value relative to the initial composition of the reaction mixture (i.e., 75 %);

$HON_{RM}$ is the final hydroxyl value of the PU depolymerization reaction mixture.

It is advisable to evaluate the efficiency of the depolymerization-transesterification (glycolysis) process by four parameters: hydroxyl (HON), amine (AmN), and acid (AcN) values, as well as the viscosity of the reaction mixture depolymerizate (recycled polyol). Deeper depolymerization leads to the formation of lower-molecular-weight oligomers, thereby decreasing the viscosity of the final reaction mixture. Simultaneously, the cleavage of a larger number of urethane bonds in the PU macromolecule leads to a change in HON due to the formation of various hydroxyl-containing polyols (Scheme 1).

It should be noted that, for the synthesis of PU products, depending on the final product requirements, various diols (polyols) with hydroxyl values ranging from 50 to 1000 are used. Furthermore, the viscosity of diols should not be high (less than 1000 mPa·s) to ensure optimal processing during mixing with diisocyanates.

Thus, an effective catalyst should yield a PU depolymerizate (recycled polyol) with low viscosity, moderate HON values (300–800 mg KOH·g$^{-1}$), and low content of free amine and acid compounds.

Analyzing the results of Table regarding the hydroxyl value of the final reaction mixture, the following series of catalytic activity of the investigated catalysts can be established:

*K-carbonate > K-acetate > Ca-acetate > Without catalyst > Na-acetate*

*798 > 786 > 756 > 712 > 590 (mg KOH·g$^{-1}$)*

First of all, it should be noted that the HON for pure (99.6 %) DEG is 1055 mg KOH·g$^{-1}$, and for the initial reaction mixture with 75 % DEG content, it is 778 mg KOH·g$^{-1}$. The use of Na-acetate significantly reduces the total HON of the depolymerizate, indicating greater consumption of DEG at deeper stages of depolymerization and transesterification. It should be taken into account that the final product is a mixture of initial unreacted DEG and newly formed oligo- or polyglycols with terminal HO-groups, which possess different reactivity towards acetic anhydride [25]. A detailed analysis of the reaction mixture is only possible by means of liquid chromatography and/or $^{1}$H-, $^{13}$C-NMR spectroscopy.

Dynamic viscosity is presented as a catalytic activity series arranged in ascending order, from the minimum achieved value to the highest:

*K-carbonate < Ca-acetate < Na-acetate < Without catalyst < K-acetate*

*257 < 397 < 544 < 629 < 629 (mPa·s)*

The amine value indicates the non-selective formation of side amine compounds in the final reaction mixture. The catalytic activity series is presented below:

*Without catalyst < K-acetate < Ca-acetate < K-carbonate < Na-acetate*
*1.6 < (5.1–6.3) < (5.3–6.6) < (5.3–9.2) < (7.8–14.0) (mg KOH·g$^{-1}$)*

Accordingly, Na-acetate promotes the formation of side amine compounds during PU depolymerization.

The acid value indicates the formation of products with free acid (carboxyl) fragments. The catalytic activity series, arranged in ascending order of the yield of acidic side products, is as follows:

*Na-acetate < K-acetate < K-carbonate < Without catalyst < Ca-acetate*
*(0.45–1) < (0.9–4.5) < (1.5–4.6) < (1.74–1.75) < (1.9–4.6) (mg KOH·g$^{-1}$)*

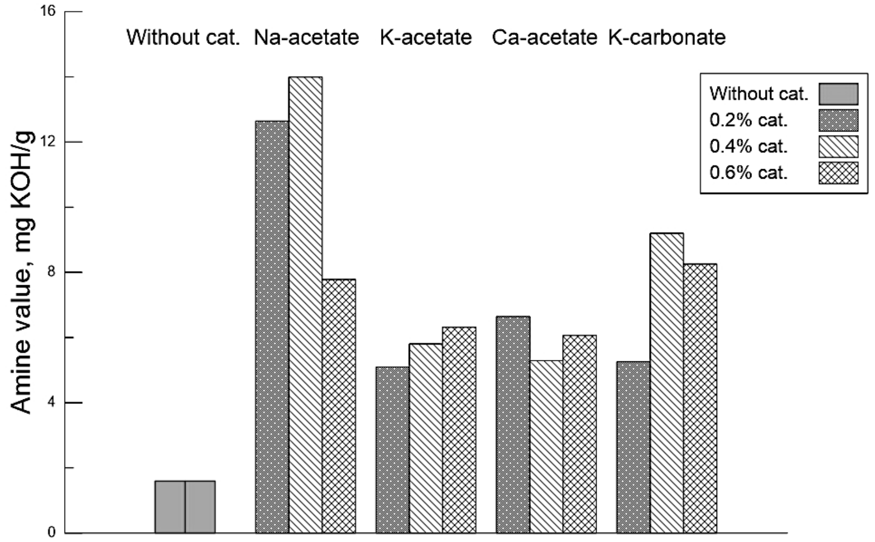


**Fig. 2. Influence of catalyst concentration on the amine value of the depolymerizate**

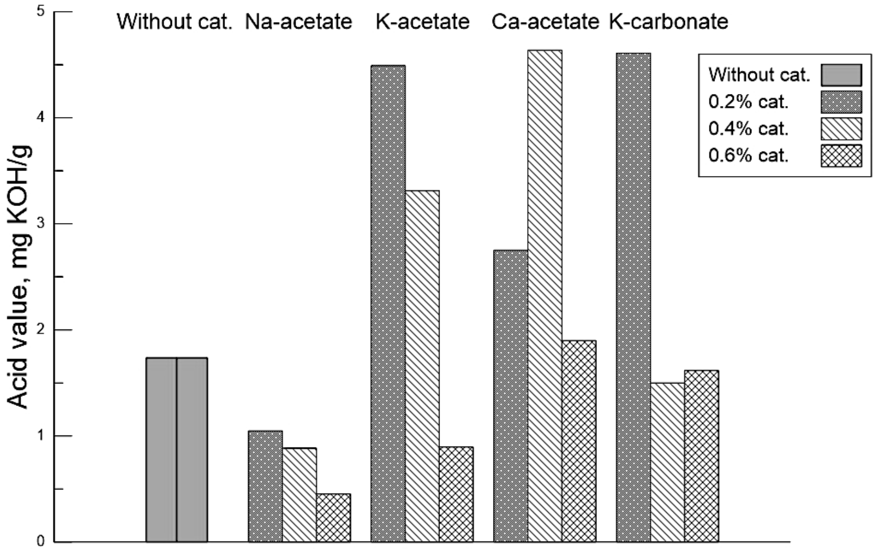


**Fig. 3. Influence of catalyst concentration on the acid value of the depolymerizate**

Furthermore, the final values of acid (AcN) and amine (AmN) numbers of the reaction mixture are also indicators and extremely important parameters for the entire selective PU depolymerization transesterification process and the properties of the recycled polyol.

The highest amine value is observed with sodium acetate at 0.2 % and 0.4 %, where AmN is 12.6–14 mg KOH g$^{-1}$ (Fig. 2). This indicates extensive degradation of the polymer, with the formation of a large amount of side amines. Potassium carbonate, despite its high activity, also shows an increased level of amines (8.3–9.2 mg KOH·g$^{-1}$) compared to potassium and calcium acetates (AmN in the range of 5–7 mg KOH·g$^{-1}$). Higher basicity accelerates the target depolymerization reaction and, to a certain extent, promotes side-reaction pathways. AcN values are generally low, indicating minimal free acid formation. The lowest acidity values (AcN) of PU recycled polyol were obtained when the sodium acetate (0.9 at 0.4 %) and the potassium acetate (0.9 at 0.6 %) were used (Fig. 3).

## Conclusions

The present investigation on the catalytic glycolysis of rigid PU waste revealed that the depolymerization efficiency is influenced not only by the catalyst's basicity but also by the nature (type) of the cation (K, Na, Ca). While all alkaline earth compounds intensified the process compared to the non-catalytic reaction, potassium carbonate was identified as the most effective and selective catalyst.

Specifically, the investigation highlights a critical divergence in selectivity between potassium and sodium compounds. The significant decrease in hydroxyl number (HON) observed with sodium acetate indicates that this catalyst promotes side reactions that consume hydroxyl groups on DEG, leading to the formation of products with lower functionality and a higher amine number (up to 14 mg KOH $g^{-1}$). This confirms that high catalytic activity does not always equate to high selectivity for polyol recovery.

In contrast, potassium carbonate at an optimal concentration of 0.4 % achieved a balance: it produced the deepest depolymerization (dynamic viscosity up to 268 mPa·s) while preserving a high hydroxyl number (up to 798 mg KOH $g^{-1}$). The presence of extremums in viscosity and acid values at specific concentrations suggests that 0.4 % represents the-optimum reaction conditions for the K-carbonate-catalyzed glycolysis and transesterification processes, beyond which competitive side reactions or saturation effects may hinder the quality of the final reaction products. Therefore, for applications requiring low-viscosity polyols with preserved functionality, potassium carbonate is the recommended or preferred catalyst.

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