

Microwave-assisted Glycolysis of Rigid Polyurethane Foam Waste Using 2-Methyl-1,3-propanediol for the Synthesis of Functional Polyester Polyols and Polyurethane Coatings

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Abstract Polyurethane (PU) foams are extensively utilized in various industries owing to their lightweight, excellent thermal insulation, and robust mechanical properties. However, their widespread use generates significant quantities of waste, which poses serious environmental and disposal challenges. In this study, rigid PU foam was effectively depolymerized via microwave-assisted glycolysis, using 2-methyl-1,3-propanediol (MP-diol) as the glycolysis agent and sodium hydroxide (NaOH) as the catalyst. The depolymerization process was systematically optimized by varying the reactant ratio, catalyst concentration, reaction temperature, and microwave power. The resulting oligomeric product (PUF-O) was characterized by physical, chemical, and spectroscopic analyses. Subsequently, a series of polyester polyols (PEs) was synthesized by partially and fully replacing neopentyl glycol with an oligomeric product in formulations containing adipic acid and phthalic anhydride. These PEs further reacted with commercial polyisocyanates to form polyurethane coatings. Characterisation was conducted to analyse the optical, mechanical, chemical, and anticorrosive properties of the cured films were characterized. The PUF-O-based polyols exhibited performance comparable to those synthesized using conventional raw materials, demonstrating the feasibility of incorporating recycled PU foam into high-performance coating systems. This work not only provides a sustainable strategy for recycling PU waste but also offers a viable route to value-added products.

Keywords Polyurethane rigid foam; Glycolysis; Microwave assisted; 2-Methyl-1,3-propanediol (MP-diol); Polyurethane coatings

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INTRODUCTION

Polyurethane (PU) is one of the most versatile and widely used classes of polymers and is popular in industry because of its superior mechanical properties, excellent thermal insulation, chemical resistance, and low density.^[1–3] It has applications in a variety of sectors, including automotive, construction, electronics, furniture, and packaging.^[4,5] Among the different forms of PU, rigid polyurethane foams are extensively employed as insulating materials in building and refrigeration systems. Owing to their durability and non-biodegradability, the extensive use of PU foams has created a growing problem with both post-consumer and industrial wastes, posing a notable environmental challenge.^[3,6]

The disposal of PU waste through incineration or landfilling is becoming increasingly undesirable because of environmental regulations, economic constraints, and the potential release of toxic gases during thermal degradation.^[7] The urgent need for sustainable waste management has led to the development of efficient recycling strategies for PU waste,

which is a critical research priority for both academia and industry. Among the available methods, chemical recycling has gained particular attention because it enables the breakdown of crosslinked PU structures into useful building blocks or oligomers that can be reused in new polymer formulations.^[8]

Glycolysis is one of the most widely explored and researched chemical recycling methods for PU. This involves the use of low-molecular-weight glycol as a cleavage agent. This method yields hydroxyl-terminated products that can be used for new polymer synthesis.^[9–11] In recent studies, various glycols such as ethylene glycol, diethylene glycol, and glycerol have been evaluated for their effectiveness in PU waste depolymerization.^[12–16] The selection of glycol significantly influenced the reaction efficiency, oligomeric structure, and performance of the recycled product. Despite these promising outcomes, conventional glycolysis methods often require long reaction times and high temperatures, which limits their scalability and energy efficiency.^[17,18]

To overcome these challenges, microwave-assisted glycolysis has emerged as an innovative approach, offering rapid and selective heating, shorter reaction time, and improved reaction.^[19,20] Notably, Nikje *et al.* have made significant contributions to the field of chemical recycling via microwave-

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assisted glycolysis. Systematic studies have explored the effects of various cleavage agents, catalyst systems (metal hydroxides and acetates), reaction times, and microwave power levels on depolymerization efficiency and polyol recovery from both flexible and rigid foams.^[21–25] In addition to Nikje's extensive contribution, a recent study by Donadini demonstrated the degradation of rigid polyurethane foam using diethylene glycol and potassium acetate under microwave irradiation, achieving higher reaction rates and polyol recovery compared to conventional techniques.^[26]

In this context, 2-methyl-1,3-propanediol (MP-diol) has been identified as a promising glycolytic agent, owing to its unique structural features. MP-diol possesses two primary hydroxyl groups along with a methyl substituent on the central carbon, which imparts flexibility and reduces the crystallinity of the resulting oligomeric products by increasing the free volume. Many high-performance coatings use polyester polyols made from MP diol as raw materials to improve their weathering resistance and mechanical properties.^[27–30] In our previous study on the glycolysis of PET, MP-diol was successfully utilized as a depolymerizing agent, and the subsequent oligomers obtained were utilized in UV-curable coatings.^[31]

In this study, rigid PU foam waste was depolymerized using MP diol under microwave irradiation in the presence of sodium hydroxide (NaOH) as a catalyst. Process parameters, including reactant ratios, catalyst concentration, temperature, and microwave power, were systematically optimized to maximize oligomer recovery. The resulting oligomeric products (PUF-O) were characterized using instrumental and end-group analyses. To demonstrate its potential in material circularity, the recovered PUF-O was employed in the synthesis of polyester polyols (PEs) through partial or complete replacement of conventional monomers. These polyols were subsequently used to formulate polyurethane (PU) coatings, which were evaluated for their mechanical, chemical, optical, and anticorrosive performance. These findings not only reveal the feasibility of microwave-assisted glycolysis for PU waste recycling but also illustrate a viable pathway for upcycling recovered materials into functional, high-performance coating systems.

EXPERIMENTAL

Materials

The PU rigid foam waste was obtained from Hamilton India Pvt.,

Ltd. The foam was obtained from a pre-consumer waste feedstock in the form of blocks, flakes, and pieces. The following chemicals were obtained from SD Fine Chemicals India Ltd.: analytical grade MP-diol (2-methyl-1,3-propanediol), sodium hydroxide (used as a catalyst), acetic anhydride, pyridine, neopentyl glycol, adipic acid, phthalic anhydride, xylene, methyl ethyl ketone (MEK), dimethylformamide (DMF) and potassium hydroxide. All chemicals were used as received without further purification. Fascat-4100 catalyst was obtained from Pidilite Industries, Mumbai. The polyisocyanate crosslinkers Desmodur N-75 (aliphatic-HDI-based) and Desmodur Z-4470 (cycloaliphatic-IPDI based) were obtained from Covestro, Mumbai.

Microwave-assisted Glycolysis of Polyurethane Waste Using MP-diol

Glycolysis was carried out using a MAS-II microwave reactor (Sineo Microwave Chemistry Technology), which was fitted with a reflux condenser and an integrated magnetic stirring system. Rigid PU foam was ground into fine foam powder or granules (<5 mm) and fed into three-necked microwave reactors. Depolymerization of the rigid PU foam was carried out using MP-diol in the presence of sodium hydroxide as a catalyst under a nitrogen atmosphere. The depolymerization ingredients PU foam and glycol were taken on a weight basis, whereas the catalyst was taken on a percentage basis of PU foam weight, as shown in Fig. 1. The reaction mixture was heated to the desired reaction temperature and maintained until the foam was completely dissolved. Furthermore, the process parameters were optimized to obtain a product with a lower hydroxyl value by varying the ratio of raw materials, reaction temperature, microwave power, and catalyst concentration, as tabulated in Table 1. The optimized parameters were further confirmed by repeatability and reproducibility of the batches.

The recovery efficiency of the oligomeric product (PUF-O) was calculated using Eq. (1):

$$\text{Recovery of PUF-O (\%)} = \frac{m_{\text{PUF-O}}}{m_{\text{initial}}} \times 100 \quad (1)$$

where $m_{\text{PUF-O}}$ is the mass of the recovered polyurethane foam oil (g), m_{initial} is the initial mass of the polyurethane foam batch used for glycolysis (g).

To lower the resulting hydroxyl value of the recovered oligomeric products, the filtered material was vacuum distilled, which effectively eliminated most of the excess MP-diol. In every experiment, a fixed amount of the product was vacuum-distilled. Continuous exercises and trials were con-

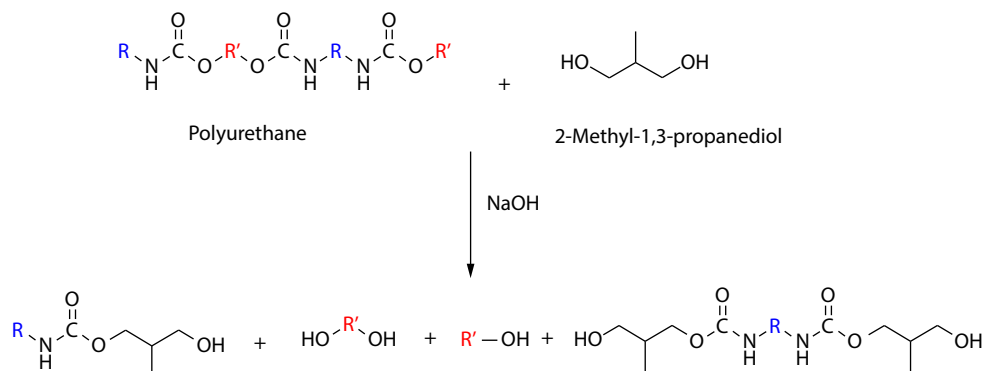


Fig. 1 Probable reaction products of PU waste with MP-diol.

Table 1 Depolymerization study of PU foam using microwave irradiation.

Varying ratio of raw materials						
No	Experiment	PU:MPD ratio	Catalyst	Temperature (°C)	Catalyst concentration (%)	Power (W)
1	MW-1	1:1	NaOH	190	3	500
2	MW-2	1:3	NaOH	190	3	500
3	MW-3	1:4	NaOH	190	3	500
4	MW-4	1:6	NaOH	190	3	500
5	MW-5	1:8	NaOH	190	3	500
6	MW-6	1:10	NaOH	190	3	500
Varying catalyst concentration						
No.	Experiment	PU:MPD ratio	Catalyst	Temperature (°C)	Catalyst concentration (%)	Power (W)
1	MW-7	1:3	NaOH	190	1	500
2	MW-8	1:3	NaOH	190	3	500
3	MW-9	1:3	NaOH	190	5	500
Varying reaction temperature						
No.	Experiment	PU:MPD ratio	Catalyst	Temperature (°C)	Catalyst concentration (%)	Power (W)
1	MW-10	1:3	NaOH	180	3	500
2	MW-11	1:3	NaOH	190	3	500
3	MW-12	1:3	NaOH	200	3	500
Varying microwave power						
No.	Experiment	PU:MPD ratio	Catalyst	Temperature (°C)	Catalyst concentration (%)	Power (W)
1	MW-13	1:3	NaOH	190	3	300
2	MW-14	1:3	NaOH	190	3	500
3	MW-15	1:3	NaOH	190	3	700
4	MW-16	1:3	NaOH	190	3	900
Repeatability and reproducibility						
No.	Experiment	PU:MPD ratio	Catalyst	Temperature (°C)	Catalyst concentration (%)	Power (W)
1	MW-R1	1:3	NaOH	190	3	700
2	MW-R2	1:3	NaOH	190	3	700
3	MW-R3	1:3	NaOH	190	3	700
4	MW-R4	1:3	NaOH	190	3	700

Table 2 Formulation of polyester polyol synthesis on equivalent basis.

Sample	PE-C	PE-20	PE-40	PE-60	PE-80	PE-100
PUF-O	—	0.24	0.48	0.72	0.96	1.2
Neopentyl glycol	1.2	0.96	0.72	0.48	0.24	—
Adipic acid	0.7	0.7	0.7	0.7	0.7	0.7
Phthalic anhydride	0.3	0.3	0.3	0.3	0.3	0.3
Fascat 4100(% on BW)	0.3	0.3	0.3	0.3	0.3	0.3

ducted to finalize the vacuum distillation process. Theoretically, the boiling point of MP-diol reduces to 147 °C at 100 mmHg, which was calculated using the temperature–pressure nomograph method. Various distillation batches were performed to determine the temperature range of distillation and extent to which MP-diol was recovered from the oligomeric product. Complete removal of excess MP-diol could make the final oligomeric product a viscous lump, which was undesirable. Hence, trials were conducted at various temperature ranges of vacuum distillation, keeping the weight of the product constant during each trial. The recovered product obtained using the optimized distillation conditions was effective in terms of the hydroxyl value and handling.

Synthesis of Polyester Polyol Using Glycolized Product

Six polyester polyols were prepared by reacting various equivalent proportions of the recovered PU foam oligomer (PUF-O) with 2-methyl-1,3-propanediol (MP-diol), neopentyl glycol (NPG), adipic acid (AA), and phthalic anhydride (PA), as detailed in Table 2. In these formulations, NPG was incrementally substi-

tuted with PUF-O at equivalent concentrations of 20%, 40%, 60%, 80%, and 100%, and the resulting polyols were labelled as PE-20, PE-40, PE-60, PE-80, and PE-100, respectively. For comparison, a control polyester polyol synthesized solely with NPG, AA, and PA, without any PUF-O, was designated as PE-C.

The calculated amounts of raw materials were introduced into a four-necked round-bottom flask equipped with a nitrogen inlet, Dean–Stark apparatus, and thermometer. The polyesterification reaction was conducted in the temperature range of 160–180 °C in the presence of 0.3% Fascat 4100 catalyst. This process was continued until the acid value of the mixture dropped below 15 mg_{KOH}/g. A representative schematic of the polyester polyol synthesis is shown in Fig. 2.

Preparation Procedure for Mild Steel Substrates

Mild steel panels measuring 15.00 cm × 10.00 cm × 0.05 cm were initially degreased using a cleaning solution for 10 min. Subsequently, they were rinsed with tap water and dried thoroughly. The surface preparation was completed by polishing the panels with emery paper.

Preparation of Polyurethane Coatings

The synthesized polyester polyols were crosslinked using two

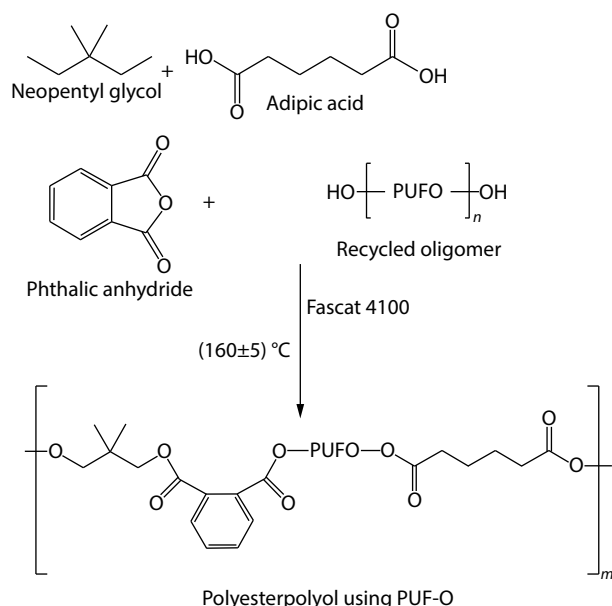


Fig. 2 Schematic representation of polyester polyol formation process.

types of polyisocyanates: an aliphatic type, Desmodur N-75 (HDI biuret), and a cycloaliphatic type, Desmodur Z4470 (IPDI trimer). The amount of crosslinker used was based on maintaining the NCO index of 1. A solvent mixture comprising xylene, methyl ethyl ketone (MEK), and dimethylformamide (DMF) in a 65:25:10 volume ratio was added in appropriate proportions to achieve the desired viscosity. Coating formulations were applied to the prepared substrates using a flow-coating method to ensure a uniform film. After a 15-min flash-off period, the coated panels were cured in a hot air oven at 120°C for 20 min to ensure complete film formation.

Characterization

Physical and chemical characterization

The recovered oligomeric products and the subsequently synthesized polyester polyols (PE-C, PE-20, PE-40, PE-60, PE-80, and PE-100) were evaluated for their physical and chemical properties. The analyses included the determination of percent non-volatile matter (%NVM), visual appearance, viscosity, acid value, and hydroxyl value, following relevant ASTM standard test methods.

Instrumentation

Fourier transform infrared (FTIR) spectroscopy was performed using a Perkin-Elmer Spectrum 100 instrument equipped with an Eco-ATR (Bruker, Germany). Spectral data were acquired from KBr pellets at 2 cm^{-1} resolution over 40 scans. The infrared spectral analysis focused on specific vibrational zones: broad O—H stretching vibrations (in the ranges of $4000\text{--}3000$ and $1000\text{--}400\text{ cm}^{-1}$), symmetric C=O stretching bands ($1800\text{--}1600\text{ cm}^{-1}$) and absorption regions linked to alcohol functional groups and C—O stretching ($1400\text{--}1000\text{ cm}^{-1}$), which provided insights into the molecular structure of the synthesized materials.

The molecular weights of both the recovered oligomers and the synthesized polyester polyols were determined via gel permeation chromatography (GPC) using an Agilent 1100

series system. HPLC-grade tetrahydrofuran (THF) was used as the eluent, and calibration was performed using monodisperse polystyrene standards.

Differential scanning calorimetry (DSC) analysis was conducted using a DSC Q-100 system (TA Instruments, USA) under nitrogen atmosphere. Calibration was performed using standard materials such as indium and *n*-octane. The samples were heating the samples from 25°C to 120°C at a controlled rate of $10^\circ\text{C}/\text{min}$ under a continuous nitrogen purge at $50\text{ mL}/\text{min}$ to maintain an inert environment.

Thermal stability was assessed using thermogravimetric analysis (TGA) on the same DSC Q-100 instrument. The samples were heated from 100°C to 650°C at $10^\circ\text{C}/\text{min}$ under a nitrogen flow of $50\text{ mL}/\text{min}$. For this analysis, the coating material was removed from the substrate before testing.

The anticorrosive behavior of the coated steel substrates was evaluated *via* electrochemical measurements using a VersaSTAT-3 workstation (AMETEK, Princeton Applied Research, Oak Ridge, TN, USA). Experiments were conducted at 30°C in 3.5% NaCl solution using a conventional three-electrode setup consisting of a saturated calomel reference electrode, a platinum counter electrode, and a coated panel as the working electrode. In all tests, the exposed surface area of the coating was maintained at 21 cm^2 . Each measurement was performed in triplicates to ensure reproducibility and data reliability.

Coating properties

Gloss measurements were conducted using a Rhopoint-Novogloss glossmeter, following the guidelines of ASTM D523-67 to evaluate the light reflectance properties of the coated surfaces. The coating adhesion was assessed using both the cross-cut test ASTM D-3359-02 and pull-off adhesion as per ASTM 454. The hardness was evaluated using the pencil hardness method in accordance with ASTM D-3363-00. The flexibility and mechanical durability of the coatings were tested using conical mandrels ASTM D 522 and ASTM D 2794 respectively. The solvent resistance was determined by conducting double rub tests using methyl ethyl ketone (MEK) and xylene, following ASTM D-4752-0. The gel content of the cured coatings was quantified *via* solvent extraction using a ternary solvent system (xylene:MEK:DMF = 65:25:10, V:V:V) over 24 h. The remaining insoluble fraction was dried at 80°C in a hot-air oven for another 24 h and then weighed to assess the degree of curing.^[32] The water uptake of the crosslinked coatings was evaluated in accordance with ASTM D570-98. Dried coating films of known weights were immersed in water at ambient temperature for 24 h. After Post-immersion, the samples were gently dried using a paper towel and their weights were recorded. The water absorption (%) was calculated using the following Eq. (2)

$$\text{Water absorption (\%)} = (w_b - w_a) / w_a \times 100 \quad (2)$$

where w_a is the weight of coating film after immersion (g), w_b is the weight of coating film before immersion (g).

RESULTS AND DISCUSSION

Glycolysis of PU Waste Using MP-diol

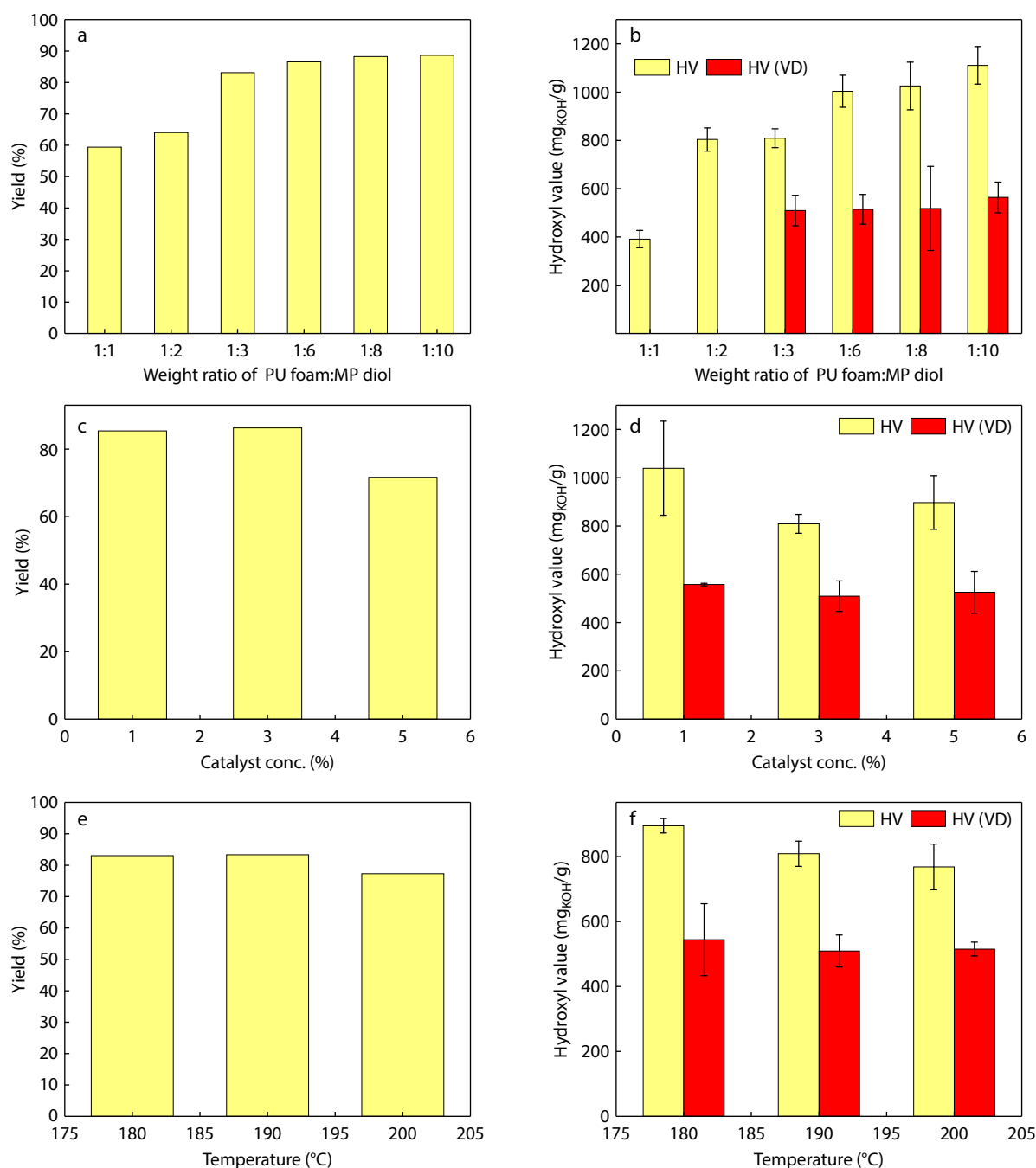
Influence of reaction conditions on hydroxyl number and recovery percentage of the oligomeric product

The objective was to optimize the process parameters to obtain

a product with a lower hydroxyl value so that the product can be used in surface coating applications. From the obtained results of the preliminary trials and from the literature searches of the relevant works, the process parameters mentioned below were considered influential over the resultant product. The depolymerization study was carried out by varying the ratio of raw materials (PU foam: MP-diol), setting the reaction temperature -190°C , reaction power of 500 W using NaOH catalyst 3% PU foam weight, and its effect on the recovery efficiency (%) of the recycled oligomeric product and hydroxyl value with and without vacuum distillation was studied, as shown in Figs. 3(a) and 3(b), respectively. Batches with various ratios of raw materials

(1:1, 1:2, 1:3, 1:6, 1:8, and 1:10) were designated as MW-1, MW-2, MW-3, MW-4, MW-5, and MW-6, respectively. It was observed that batches with raw material ratios of 1:3, 1:6, 1:8, and 1:10 resulted in a percent recovery of recycled oligomeric products in the range of 80%–90%. The hydroxyl values were determined before and after vacuum distillation for all batches, except for the raw material ratios of 1:1 and 1:2. These batches resulted in high-viscosity products with low recovery of recycled oligomeric products. The MW-3 batch with a raw material ratio of 1:3 resulted in an oligomeric product with excellent percent recovery and less hydroxyl value, with and without vacuum distillation.

The next set of PU foam depolymerization reactions were



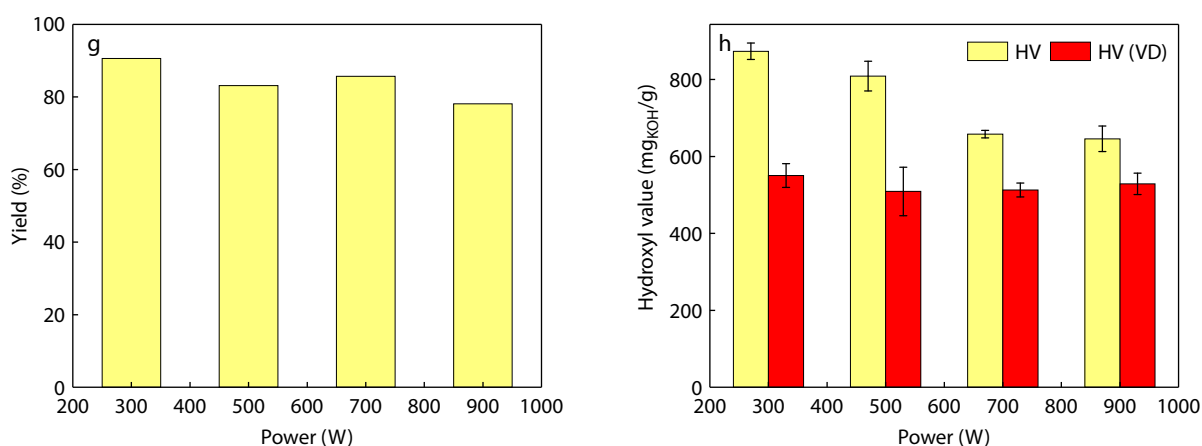


Fig. 3 Impact of process variables on hydroxyl content and recovery efficiency of the oligomeric product: (a) varying ratios of raw materials on recovery efficiency (%), (b) varying ratios of raw materials on OH value, (c) varying catalyst concentrations on recovery efficiency (%), (d) varying catalyst concentrations on OH value, (e) varying reaction temperatures on recovery efficiency (%), (f) varying reaction temperatures on OH value, (g) varying reaction power.

carried out by varying the catalyst concentration and ratio of raw materials PU foam:MP-diol = 1:3, set reaction temperature as 190 °C, and reaction power as 500 W. The depolymerization reactions were carried out using NaOH catalysts with various concentrations of 1%, 3%, and 5% of the total weight of PU foam, and the batches were designated as MW-7, MW-8, and MW-9, respectively. Furthermore, its effect on the percentage recovery of the recycled oligomeric product and hydroxyl value with and without vacuum distillation was studied, as shown in Figs. 3(c) and 3(d), respectively. The extent of depolymerization was observed to be optimum for MW-8 in terms of recycled oligomeric product with excellent percent recovery and lower hydroxyl value compared to MW-7 and MW-9. In the next step of the PU foam depolymerization study, the reaction temperature was varied by keeping all other reaction parameters constant, such as the ratio of raw materials PU foam:MP-diol = 1:3, 3% NaOH catalyst, and power of 500 W. The depolymerization reactions were performed at 180, 190, and 200 °C and designated as MW-10, MW-11, and MW-12, respectively. Furthermore, its effect on the percentage recovery of the recycled oligomeric product and hydroxyl value with and without vacuum distillation was studied, as shown in Figs. 3(e) and 3(f), respectively. Batch MW-11 with a reaction temperature of 190 °C resulted in better recovery of the recovered oligomeric product as compared to MW-10 and MW-12. No significant effect was observed on the hydroxyl values of the in-process or the final end products for the given range of reaction temperatures.

Further, the PU depolymerization study was carried out by varying the reaction power while keeping other reaction parameters constant, such as the ratio of raw materials PU foam:MP-diol = 1:3, reaction temperature 190 °C and 3% NaOH catalyst. The reactions were carried out by varying the microwave power to 300, 500, 700 and 900 W, designated as MW-13, MW-14, MW-16, and MW-17, respectively. Furthermore, its effect on the percentage recovery of the recycled oligomeric product, foam dissolution time, and hydroxyl value with and without vacuum distillation was studied, as shown in Fig. 3(g). The results revealed that foam dissolution time decreased as the reaction power increased. The hydrox-

yl values without vacuum distillation showed an increasing trend with increasing reaction power, indicating that the rate of PU foam depolymerization increased with increasing reaction power. In view of these findings, it was observed that MW-16 would be the best optimized process parameter for PU foam depolymerization using microwave irradiation. Reproducibility and repeatability studies were also carried out for the optimized process parameters, as given in Table 3, and designated as MW-R1, MW-R2, and MW-R3.

Characterization of the product via chemical and spectroscopic techniques

The obtained oligomeric product was a dark brown viscous liquid, which was further analyzed for viscosity and molecular weight using gel permeation chromatography. The optimization study of PU foam depolymerization revealed that the oligomeric product recovered using vacuum distillation resulted in hydroxyl values and viscosities in the range of 450–550 mg_{KOH}/g of the sample and 700–800 cps respectively. The number-average molecular weights (M_n) of PUF-O in the optimized batches are tabulated in Table 3, and the overlay of the GPC chromatogram is shown in Fig. 4.

Table 3 Number average molecular weights (M_n) of PUF-O.

Sample	M_n (g/mol)	PDI
MW-R1	404	2.53
MW-R2	508	2.18
MW-R3	541	2.05
MW-R4	553	2.1

The oligomer recovered after vacuum distillation was characterized using FTIR spectroscopy (Fig. 5). The transmission band at 3313 cm⁻¹ attributed to the presence of hydroxyl groups. There were no band between 2270 and 2100 cm⁻¹, which is the characteristic absorption band of N=C=O, indicating that isocyanate groups were absent. The band at 1704 cm⁻¹ showed the presence of a C=O linkage in urethane. This could be due to the presence of unreacted foam or incomplete depolymerization of polyurethane, which can be inferred from the band at 1725 cm⁻¹. The presence of bands at

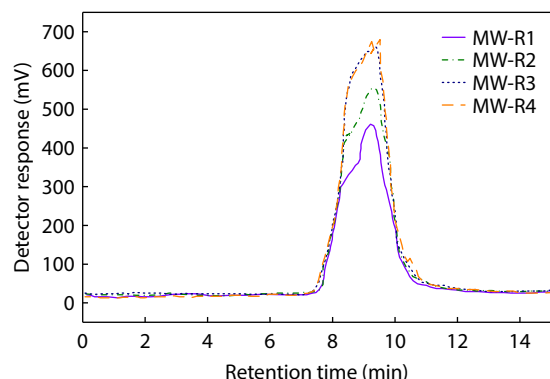


Fig. 4 GPC chromatograms of PUF-O.

1607 and 1528 cm^{-1} could be due to the formation of amine compounds during the depolymerization reaction, which were further concentrated in the reaction mixture with time.^[33] The bands at 2925 and 2879 cm^{-1} represent the stretching vibrations of the C—H bonds in aliphatic carbons.

Synthesis of Polyester Polyol Using Glycolized Product

Characterization of polyester polyols: physical, chemical and spectroscopic analysis

The synthesized polyester polyols (PE-C, PE-20, PE-40, PE-60, PE-80, and PE-100) were also analyzed for NVM (%), yield (%), acid value (AV) and hydroxyl value (HV) as given in Table 4.

Table 4 Characterization of polyester polyols (PEs).

Sample	NVM (%)	AV	HV (Theoretical)	HV (Expt.)	Yield (%)
PE-C	98.78	12.74	93.08	92.92	97.87
PE-20	99.35	14.22	84.66	115.98	98.06
PE-40	98.95	11.87	77.63	95.69	97.98
PE-60	98.54	13.54	71.69	88.94	97.43
PE-80	99.15	13.41	66.59	92.14	98.05
PE-100	99.65	15.98	62.17	84.94	98.34

All polyester polyol systems exhibited high non-volatile matter (NVM) and product yields, ranging between 98%–99% and 97%–98%, respectively. The hydroxyl values across the synthesized batches were consistently in the range of 80–115 $\text{mg}_{\text{KOH}}/\text{g}$, indicating suitable reactivity for polyurethane applications.

Fourier transform infrared spectroscopy (FTIR) was used to characterize both the polyester polyol synthesized from the recovered oligomeric product (PE-PUF-O) and the reference conventional polyester polyol (PE-C), as illustrated in Fig. 6. A broad absorption band in the region of 3400–3500 cm^{-1} confirmed the presence of OH functional groups in both samples. Distinct peaks at approximately 2955 cm^{-1} were attributed to aliphatic and aromatic C—H stretching vibrations. The presence of ester linkages was confirmed by a prominent absorption band around 1700 cm^{-1} . PE-PUF-O exhibited an additional sharp band at 1602 cm^{-1} , which corresponds to the carbonyl stretching of the urea linkage. This distinctive peak provides clear evidence of the successful incorporation of the recycled PU foam-derived oligomer in the polyester polyol structure.

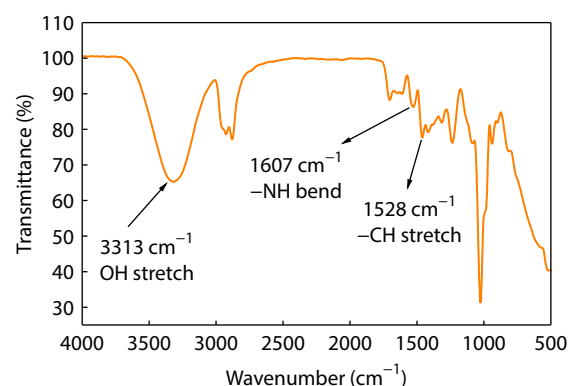


Fig. 5 FTIR spectrum of recycled polyol obtained.

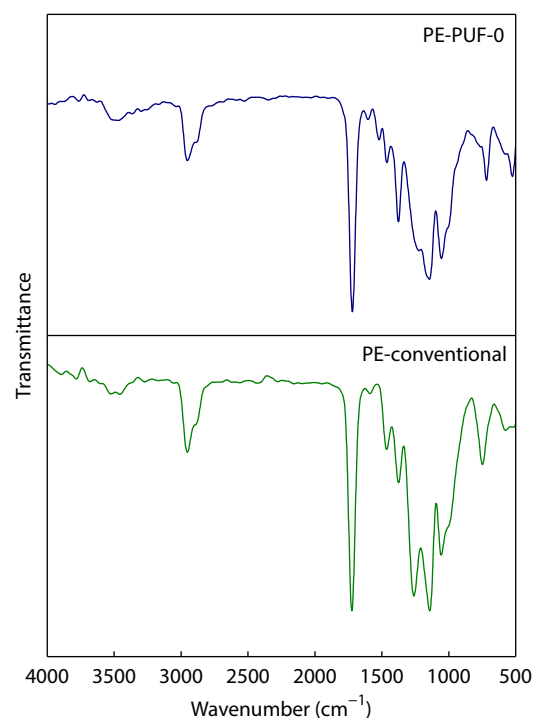


Fig. 6 FTIR spectra of PE-C and PE-PUF-O.

Coating properties

The physical, mechanical, chemical, thermal, and anticorrosive properties of polyester polyols (PE-C, PE-20, PE-40, and PE-60) cured with aliphatic and cycloaliphatic crosslinkers were evaluated. The coatings exhibited complete curing, transparency, and no visible defects such as pinholes, blisters, or phase separation. The average film thickness for all coatings was in the range of 25–45 μm . When measured with a digital gloss meter at 60°, the gloss of the polyester-polyol-based PU coatings was found to vary in the range of 80–100 GU.

Mechanical properties

A comprehensive evaluation of the coatings was conducted to examine various mechanical attributes, such as adhesion performance, surface hardness (pencil hardness test), resistance to abrasion (scratch resistance), flexibility, impact durability, and pull-off strength, as summarized in Table 5. Adhesion to metal substrates was assessed using the cross-hatch technique, where all formulations showed a 5B rating, irrespective of the type of

Table 5 Mechanical and chemical properties of coatings.

Sample		Mechanical properties							
		Dry film thickness (μm)	Gloss (60°)	Cross cut adhesion	Pencil hardness	Scratch hardness (gm)	Impact resistance-intrusion (kg-cm)	Impact resistance-extrusion (kg-cm)	Pull off adhesion (MPa)
PE-C	N-75	25–35	95±3	5B	6H	1200	81.6	81.6	1.67
	IPDI	35–45	98±3	5B	6H	1700	81.6	81.6	1.87
PE20	N-75	30–45	90±3	5B	6H	1200	81.6	81.6	1.57
	IPDI	30–40	96±3	5B	6H	1800	81.6	74.8	2.32
PE40	N-75	25–30	92±3	5B	6H	1300	81.6	81.6	1.96
	IPDI	25–35	95±3	5B	6H	2000	74.8	47.6	1.95
PE60	N-75	30–40	88±3	5B	6H	1350	81.6	81.6	2.13
	IPDI	35–45	92±3	5B	6H	2050	54.4	54.0	2.23
PE80	N-75	25–35	87±3	5B	6H	1400	81.6	81.6	1.97
	IPDI	35–45	89±3	5B	6H	2100	47.6	47.6	2.25
PE100	N-75	35–45	83±3	5B	6H	1400	81.6	81.6	1.93
	IPDI	30–40	85±3	5B	6H	2100	40.8	40.8	1.86
Sample		Chemical properties							
		Acid resistance (24 h)	Alkali resistance (24 h)	Water resistance (24 h)	Hydrolytic stability (4 h boiling water)	Solvent resistance (Xylene)	Solvent resistance (MEK)	Gel content (%)	Water absorption (%)
PE-C	N-75	A	C	A	B	>200	<125	93.89	3.89
	IPDI	A	C	A	B	>200	<125	95.43	2.45
PE20	N-75	A	C	A	B	>200	<125	94.56	3.58
	IPDI	A	C	A	B	>200	<125	96.23	2.05
PE40	N-75	A	C	A	B	>200	<125	94.78	3.35
	IPDI	A	C	A	B	>200	<125	94.89	1.95
PE60	N-75	A	C	A	B	>200	<125	92.97	2.92
	IPDI	A	C	A	B	>200	<125	95.67	1.69
PE80	N-75	A	C	A	B	>200	<125	91.55	2.97
	IPDI	A	C	A	B	>200	<125	93.78	2.15
PE100	N-75	A	C	A	B	>200	<125	91.65	3.43
	IPDI	A	C	A	B	>200	<125	93.05	2.19

A: no change observed; B: gloss reduction; C: blistering and softening of the film; D: film removal.

curing agent used. This strong coating-substrate interaction is likely due to the presence of urethane linkages, which enhance the interfacial bonding with metal surfaces in the polymer matrix.

The superior bond strength across all coating systems was further confirmed by pull-off adhesion tests when benchmarked against the standard formulations. The pencil hardness of all cured samples indicated excellent surface hardness for both types of crosslinker. An improvement in scratch resistance was observed with an increase in the recycled oligomer content, especially with cycloaliphatic crosslinkers. This enhancement is likely due to the more tightly packed polymer network generated by the synergistic effect of the oligomer and rigid crosslinker structure.

The conical-bend mandrel method was employed to assess the flexibility of the coatings. Coatings cured with aliphatic crosslinkers maintained high flexibility across all formulations, and a noticeable reduction in flexibility was observed in films cured with a cycloaliphatic crosslinker that contained 40%–100% recycled oligomers. This reduction was probably a result of the increased structural rigidity introduced by the coating formulations. Impact resistance testing, which measures the coating's response to a sudden mechanical force from a free-falling weight, showed that aliphatic cured coatings have better resilience. However, the formulations containing higher amounts of recycled oligomer and

cured with a cycloaliphatic hardener displayed reduced impact strength, possibly because of the increased brittleness from the rigid chemical structure of the hardener and oligomer combination.

Chemical properties

The chemical resistance of the coated samples was evaluated in acidic, alkaline, and aqueous environments, as shown in Table 5. The specimens were submerged in 10% hydrochloric acid, 10% sodium hydroxide, and distilled water for 24 h. After post-immersion, the samples were inspected for visible signs of deterioration. All the coating formulations demonstrated strong resistance to acidic and aqueous exposure. However, alkaline conditions caused slight softening in some films, which was likely due to the partial degradation of ester linkages *via* saponification. To assess hydrolytic stability, the PU coatings were subjected to boiling water immersion in accordance with ASTM B-1308. After 4 h of exposure, the panels were examined for surface integrity. Most systems retained their structural integrity, although a minor reduction in the gloss was observed. Solvent resistance was analyzed using a rubbing test with methyl ethyl ketone (MEK) and xylene, targeting the interaction of the coating with polar and non-polar solvents. Across all crosslinker types, the coatings showed high resistance to xylene, enduring 200 double rubs without a substantial loss of gloss. Conversely, their performance against MEK was comparatively lower, with the

coating generally failing before reaching 125 double rubs. This behavior can be attributed to the polar nature of the urethane linkages, which may be more susceptible to MEK-induced softening or dissolution.

Water absorption and gel content

Water uptake tests were conducted on all coatings, and the results are summarized in Table 5. Each coating sample with a known initial mass was immersed in water at room temperature for a 24-h period. After immersion, the samples were weighed again to determine the extent of the water absorption. Systems incorporating a higher proportion of recycled oligomers in the polyester polyol formulation showed a noticeable decrease in water absorption compared to the standard formulation. Additionally, the coatings cured with cycloaliphatic isocyanate absorbed less water than those cured with aliphatic isocyanates. This behavior is attributed to the denser and more rigid network of the IPDI, which likely shields hydroxyl functionalities more effectively than the longer chain structure of HDI, thereby limiting moisture penetration. The extent of curing and cross-linking was also assessed after 24 h of curing. The cured films were immersed in a solvent blend comprising xylene, MEK, and DMF at a 65:25:10 ratio for another 24 h. The mass loss from each film was recorded after post-immersion, and the gel content was calculated accordingly. The results indicated that coatings cured with aliphatic crosslinkers have comparatively lower gel content than those cured with cyclic aliphatic systems, supporting the superior structural integrity imparted by IPDI.

Thermal properties

The thermal behavior of the coatings was analyzed by differential scanning calorimetry (DSC), the glass transition temperatures (T_g) of coatings cured with aliphatic (HDI) and cycloaliphatic (IPDI) hardeners are presented in Fig. 7. For the coatings cured with N75 crosslinkers, the T_g values for systems PE-C-N75, PE-20-N75, PE-40-N75, PE-60-N75, PE-80-N75, and PE-100-N75 were measured at 31.07, 32.57, 33.84, 48.15, 51.86 and 52.38 °C, respectively. The coatings cured with IPDI exhibited higher T_g values for the systems PE-C-IPDI, PE-20-IPDI, PE-40-IPDI, PE-60-IPDI, PE-80-IPDI, PE-100-IPDI were 47.60, 50.23, 52.61, 53.63, 55.96 and 57.45 °C, respectively. The inference drawn from the thermograms suggests that increasing the amount of recycled oligomeric product increases the glass transition tem-

perature. Additionally, the coatings cured with IPDI consistently had higher T_g values than those cured with HDI. This difference can be attributed to the cycloaliphatic structure of the IPDI, which restricts chain mobility and creates a denser, more tightly crosslinked polymer network compared to HDI.

Thermogravimetric analysis (TGA) was performed to assess the thermal stability of the coating formulations. Fig. 8 shows the thermograms of the percentage weight loss as a function of temperature for both aliphatic and cycloaliphatic crosslinked systems. Across all formulations, the onset of degradation was observed within the range of 200–300 °C. Coatings incorporating cycloaliphatic crosslinkers exhibit improved thermal resistance compared to their aliphatic counterparts, highlighting the influence of the crosslinker structure on thermal performance. This difference in stability can be attributed to the nature of the crosslinkers used in each polyester polyol system.

Anticorrosive properties

Corrosion protection is a coating system that is governed by several critical parameters. These include the chemical nature of polyester polyol, structural characteristics of the crosslinker, resultant polymer matrix, and level of moisture permeability through the film.

To investigate corrosion resistance, accelerated environmental testing and electrochemical analyses were conducted. Fig. 9 depicts the visual condition of the coated substrates before and after exposure to a corrosive environment. The test involved subjecting the coated panels to a 3.5% sodium chloride solution under controlled conditions (35 °C) for a continuous period of 500 h. The ability of a coating to prevent corrosion is largely influenced by its impermeability and the extent of network formation within the cured structure. The denser the structure, the lower the free volume present in the polymer matrix, and hence, the lower the chances of permeation of corrosive species. When comparing the two types of hardeners, the cycloaliphatic hardener displayed enhanced resistance to surface-level electrochemical degradation, indicating a more robust corrosion-inhibiting characteristic. This improvement is attributed to the denser crosslinked matrix created by the IPDI-based system and the inherent rigidity of the oligomeric segments recovered from the PU foam waste. Despite these advantages, the corrosion performances of

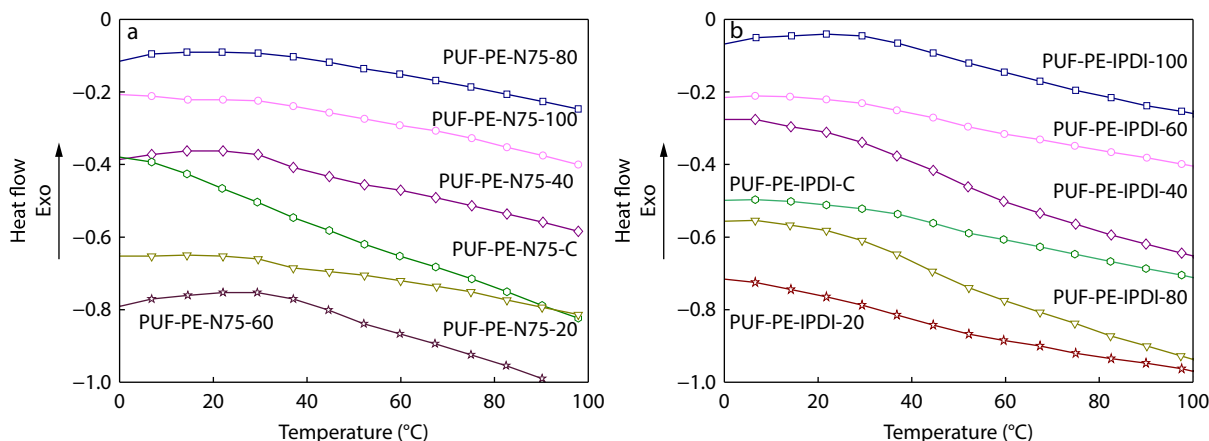


Fig. 7 Thermal transition profiles of coatings cured with (a) aliphatic crosslinker (b) cycloaliphatic crosslinker.

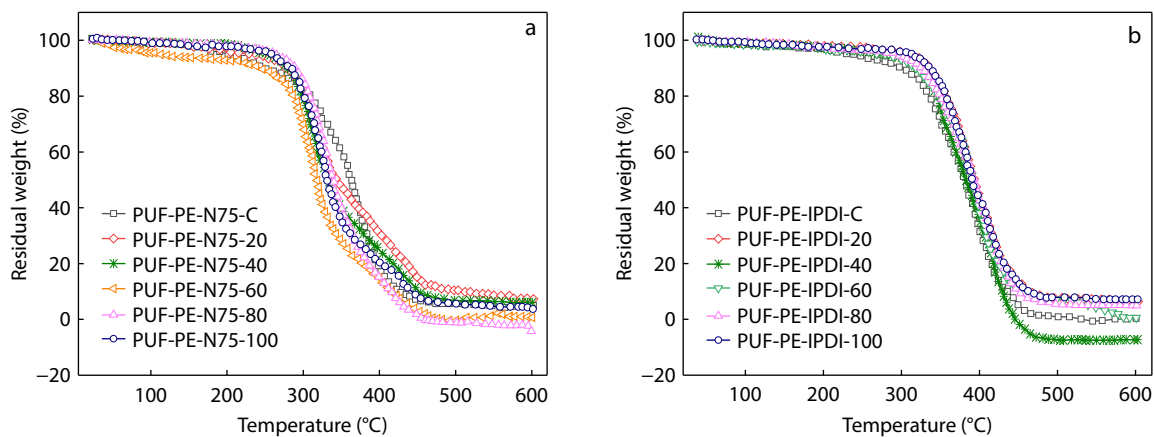


Fig. 8 TGA thermograms of coatings cured with (a) aliphatic crosslinker (b) cycloaliphatic crosslinker.



Fig. 9 Salt spray test panels of (a) PE-N75 (0, 500 h), (b) PE-IPDI system (0, 500 h).

both crosslinking chemistries are comparable to those of coatings formulated with conventional polyols.

Electrochemical impedance analysis (EIS) is a standard approach to quantitatively evaluate the corrosion protection offered by coatings. By interpreting the Bode plots, parameters such as the electrical resistance and capacitance of the coat-

ing can be extracted using equivalent circuit modelling. These metrics offer insights into the barrier effectiveness of the coating, as further illustrated by the Nyquist plots presented in Fig. 10 for both aliphatic and cycloaliphatic crosslinkers. The circuit model employed for data fitting, shown in Fig. 10 includes elements such as R_s (electrolyte re-

distance), C_c (capacitance of the coating layer), R_{po} (resistance due to pores or coatings), C_{dl} (interfacial double layer capacitance) and R_c (resistance related to charge transfer at the interface).^[34–36] Fig. 11 does not appear in the maintext. Please supply it in the proper place.

The resistance of a coating is often referred to as the pore resistance, which reflects its capacity to restrict the electrical current flow. Systems crosslinked with IPDI demonstrated greater resistance values than those crosslinked with HDI (Table 6). This is likely due to the inherent differences in the molecular structures of isocyanates. Specifically, the cycloaliphatic configuration of IPDI tends to yield a more rigid and tightly packed polymer network than the more flexible structure provided by aliphatic N-75. Furthermore, the corro-

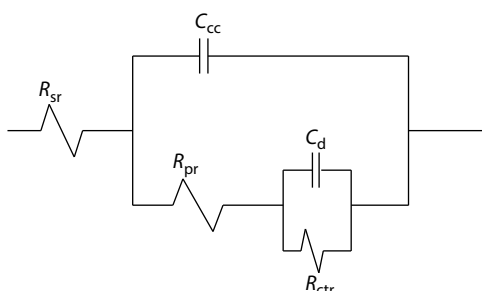


Fig. 10 Equivalent circuit model for coating.

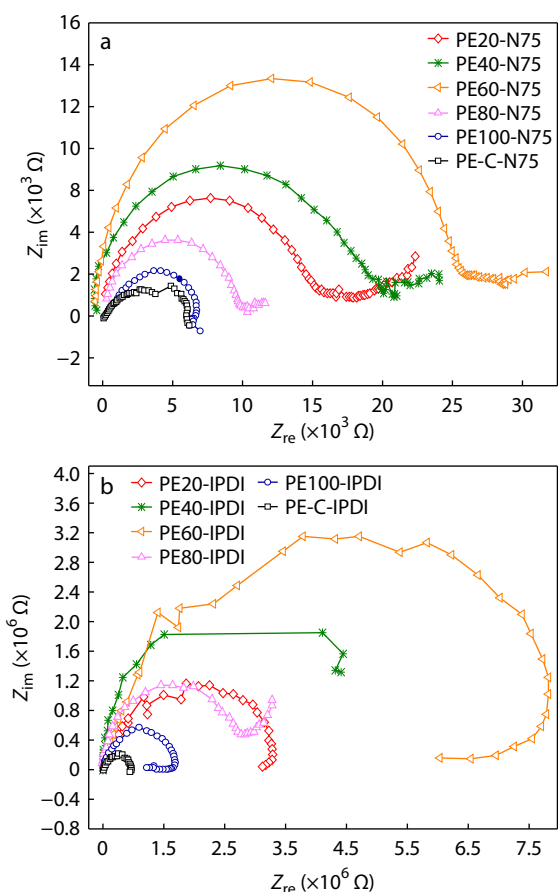


Fig. 11 Nyquist plots of (a) PE-N75 system, (b) PE-IPDI systems.

Table 6 Fitted resistance and capacitance parameters for coating systems.

Coatings	Resistance (Ω)	Capacitance (F)
Aliphatic crosslinker (N-75)		
PE-C-N75	4.42×10^2	0.50×10^{-6}
PE 20-N75	1.66×10^3	0.18×10^{-6}
PE 40-N75	4.52×10^5	1.16×10^{-9}
PE 60-N75	1.10×10^6	1.13×10^{-9}
PE 80-N75	1.45×10^4	2.06×10^{-9}
PE100-N75	8.06×10^2	5.49×10^{-9}
Cycloaliphatic crosslinker (IPDI)		
PE-C-IPDI	9.29×10^2	0.52×10^{-9}
PE 20-IPDI	6.10×10^4	1.90×10^{-9}
PE 40-IPDI	1.78×10^6	1.03×10^{-9}
PE 60-IPDI	4.31×10^9	0.83×10^{-9}
PE 80-IPDI	2.72×10^4	1.60×10^{-9}
PE100-IPDI	1.80×10^4	2.00×10^{-9}

sion resistance improved in both systems with up to 60% substitution of polyester polyol using recycled oligomers. Among the tested formulations, the PE-60 composition cured with IPDI exhibited superior resistance to corrosion. The capacitance indicates the ability of the coating to prevent permeation of water or electrolytes into the film. The lower the capacitance values, the better water or electrolyte permeation is prevented. The IPDI-based coatings consistently show lower capacitance than those based on HDI. The denser and more structured network formed by cycloaliphatic isocyanate may govern these trends. These findings are consistent with the observed water absorption trends. An increase in parameters such as crystallinity, hardness, and rigidity led to a corresponding decrease in capacitance. Consequently, the PE-IPDI system exhibited excellent barrier characteristics, as evidenced by its minimal water and ion diffusion.

CONCLUSIONS

The present study successfully demonstrated the microwave-assisted depolymerization of rigid PU waste using excess 2-methyl-1,3-propanediol and NaOH as a catalyst. PU:MP diol ratio of 1:3, reaction temperature 190 °C, and 3 wt% catalyst loading led to a yield of over 80% of the oligomeric product with a hydroxyl value in the range of 500–500 mg_{KOH}/g. The recovered oligomers were further utilized in the synthesis of polyester polyols, which were subsequently used to formulate polyurethane coatings. Among the series of coatings developed, the formulation containing 60% replacement of conventional polyol with recovered PUF-O and crosslinked with a cycloaliphatic hardener exhibited superior coating properties, including enhanced hardness, chemical resistance, and corrosion protection. Overall, this study confirms that MP-diol is a highly efficient glycolysis agent for rigid PU waste, and the integration of microwave irradiation offers a sustainable, energy-efficient pathway to convert polymer waste into functional value-added materials.

Conflict of Interests

The authors declare no interest conflict.

Data Availability Statement

The related data of this paper is data that should not be shared and can be obtained from the author for reasonable reasons. The author's contact information as.sabnis@ictmumbai.edu.in

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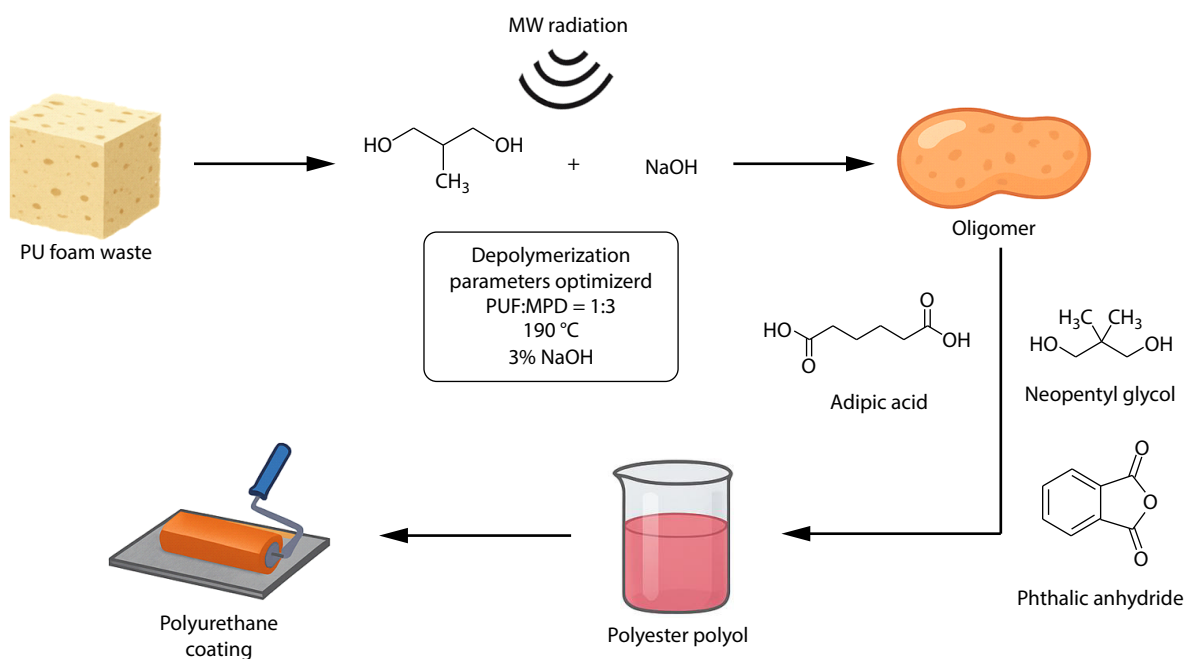
Graphical Abstract

Microwave-assisted Glycolysis of Rigid Polyurethane Foam Waste Using 2-Methyl-1,3-propanediol for the Synthesis of Functional Polyester Polyols and Polyurethane Coatings

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Polyurethane (PU) foam waste was depolymerized using microwave-assisted glycolysis with a polyol (MPD) and NaOH catalyst under optimized conditions (PUF:MPD = 1:3, 190 °C, 3% NaOH) to produce oligomers. These oligomers were then reacted with adipic acid, neopentyl glycol, and phthalic anhydride to synthesize a recycled polyester polyol, which was subsequently used to prepare the polyurethane coatings.



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