

Received: 08 August, 2023
Accepted: 08 September, 2023
Published: 09 September, 2023

***Corresponding author:** Anna Chrobok, Department of Chemical Organic Technology and Petrochemistry, Silesian Technical University, Faculty of Chemistry, ul. Krzywoustego 4, 44-100 Gliwice, Poland, E-mail: anna.chrobok@polsl.pl

Keywords: Non-isocyanate polyurethanes; NIPU; Vitrimers; Cyclic carbonates; Covalent adaptive networks

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Research Article

Reprocessable Non-Isocyanate Polyurethane Vitrimers

Anna Chrobok^{1*}, Damian Kielkiewicz² and Agnieszka Siewniak²

¹Department of Chemical Organic Technology and Petrochemistry, Silesian Technical University, Faculty of Chemistry, ul. Krzywoustego 4, 44-100 Gliwice, Poland

²Lukasiewicz Research Network, Institute of Heavy Organic Synthesis "Blachownia", ul. Energetyków 9, 47-225 Kędzierzyn-Koźle, Poland

Abstract

Non-isocyanate polyurethanes (NIPUs, polyhydroxyurethanes, PHUs), have emerged as sustainable alternatives to conventional isocyanate-polyol polyurethanes. However, the permanent cross-links in traditional linear, crosslinked polyhydroxyurethane polymer networks hinder their recyclability for high-value applications. In this study, we provide a comprehensive overview of polyhydroxyurethane vitrimers – polymers with intrinsic recyclability – containing dynamic covalent adaptable bonds that allow them to be reprocessed or self-healed under external stimuli such as heat or solvents. These materials exhibit a unique combination of the attributes of thermosets, such as improved heat stability, solvent resistance or enhanced mechanical properties, and the reprocessability of thermoplastics. Various strategies have been explored to enable the reprocessability of PHUs. External catalysts, such as 4-(dimethylamino)pyridine (DMAP) have been used to facilitate exchange reactions and promote reprocessing. Additionally, the use of functionalized silica nanoparticles as reinforcing fillers has influenced the material's behavior during reprocessing. Another method involved the incorporation of dynamic disulfide bonds to expedite reprocessing times for PHU networks, while dissociative dynamic chemistry has enabled self-healing behavior in certain partially cross-linked NIPUs. These advancements demonstrate the potential for tailoring the reprocessability and mechanical attributes of NIPUs, paving the way for sustainable and versatile polymeric materials, and addressing the environmental concerns associated with traditional polyurethanes.

Abbreviations

NIPU, PHU: Non-Isocyanate Polyurethanes, Polyhydroxyurethanes; PU: Polyurethanes; DMAP-4: (dimethylamino)pyridine; TAEA: Tris(2-aminoethyl)amine; MDA: 1,8-Methane Diamine; CC: Cyclic Carbonates; CSBO: Carbonated Soybean Oil; DA: Diels-Adler addition; DMTA: Dynamic Mechanical Thermal Analysis; DSC: Differential Scanning Calorimetry; POM: Polarized Optical Microscopy

Introduction

Polyurethanes (PU) are a versatile class of polymers formed through the polyaddition reaction of isocyanates with polyols. They are produced in various forms, including rigid and flexible foams, as well as non-porous materials such as coatings, adhesives, sealants, thermoplastics, elastomers, or thermosets for various industries [1-3]. However, there are

some hazards associated with the application of polyurethane materials, particularly concerning isocyanate monomers. The most common commercial process for the synthesis of diisocyanates is realized through the phosgene route. Phosgene is a lethal, toxic gas derived from the reaction between carbon monoxide and chlorine [4]. Isocyanates are also known to pose potential health hazards due to their toxicity – exposure can lead to respiratory irritation, skin sensitization, asthma, and lung disorders [5].

A sustainable alternative to conventional PUs is Non-Isocyanate Polyurethanes (NIPUs), which can be obtained according to three general polymer synthetic methods: by step-growth polyaddition, polycondensation, and ring-opening polymerization [6,7]. The most promising and industrially significant is the synthesis of polyhydroxyurethanes in the reaction of multifunctional Cyclic Carbonates (CC) with primary diamines or polyamines obtained according to the

first-mentioned method. Apart from isocyanates elimination, another advantage is the possibility of carbon dioxide utilization in the cyclic carbonates synthesis in the reaction with epoxy compounds, also based on renewable raw materials like plant oils, terpenes, lignin or bio-glycols [8-11]. However, linear, crosslinked polyhydroxyurethane polymer networks cannot be recycled for high-value applications due to the presence of permanent cross-links, which prevent them from being melt-reprocessed [12]. Enabling the recycling of these polymers has become an important topic for sustainable development.

Polyhydroxyurethane vitrimers represent a new group of crosslinked polymeric materials that combine the excellent mechanical properties of thermosets with the reprocessability of thermoplastics [13,14]. Under normal conditions, they maintain chemical and mechanical stability, but through the introduction of dynamic covalent adaptable bonds are capable of network topology alternation. The networks constructed from such dynamic covalent bonded cross-links are referred to as Covalent Adaptive Networks (CANs), and exchange in these CANs occurs by either associative or dissociative pathways [15-17]. The exchange mechanism of a dissociative CAN is similar to the depolymerization of a polymer network, involving a process of breaking followed by re-forming [18]. With the temperature rising, the chemical bonds within the entire dissociative covalent adaptable network break faster than they can be joined, leading to a reverse decomposition reaction. This results in a reduction in the density of the crosslinking point and an increase in the mobility of the polymer, enabling the material to be processed and shaped [19]. When the temperature drops, the crosslink density of dissociative CANs increases, leading to the recovery of mechanical properties. In an associative pathway, the polymer maintains a constant crosslink density during the change, with bond breaking and reformation happening concurrently.

The introduction of CANs enables reprocessing or self-healing of the polymer under external stimuli, such as solvents, heat, pressure, pH, or UV light [20,21]. For reprocessing, physical and chemical recycling methods are used. Physical methods refer to a way of recycling by directly using materials by grinding into powders or pellets, with processing techniques including hot pressing, injection molding, welding, or extrusion molding [22-24]. Chemical recycling is a recovery method that uses chemical solvents or monomers to dissolve or degrade vitrimers into oligomers. The resulting materials are then separated from the solution for further processing [25]. Self-healing refers to a material's ability to recover from physical damage. Both physical and chemical approaches have been used to construct self-healing polymers. These include interchain diffusion and flow, phase-separated morphologies, shape-memory effects, heterogeneous self-healing systems, covalent-bond reformation and reshuffling, dynamics of supramolecular chemistry or combinations thereof [26].

While numerous studies have already been published on the general subject of vitrimers, only a few of them refer to polyhydroxyurethane ones. Below is presented an overview of research related to reprocessable and self-healable PHUs.

Methods of reprocessing

One approach to introducing the desired dynamic exchanges is to use external catalysts that can manipulate the exchange rate or mechanism [27]. The use of DMAP as a catalyst for network rearrangement in PHUs based on the poly(propylene glycol) dicyclocarbonate and tris(2-aminoethyl)amine (TAEA) or JEFFAMINE D-400 as the crosslinking agents were reported by Chen X, et al. [28]. These intrinsically reprocessable polymers were designed to achieve full property recovery via associative transcarbamylation (an exchange reaction) and reversible cyclic carbonate aminolysis (breaking and reconnecting the crosslinks). The same catalyst was used in the preparation of four PHU networks synthesized with two types of bio-based cyclic carbonates, Carbonated Soybean Oil (CSBO) and sorbitol ether carbonate, and two types of amines, Priamine 1074 and diethylene glycol bis(3-aminopropyl) ether [29]. In mild reprocessing conditions, CSBO-based PHU networks exhibit complete recovery of crosslink density and associated properties after multiple melt-state reprocessing steps. Besides the presence of reversible cyclic carbonate aminolysis and transcarbamylation exchange reactions, CSBO-based networks were shown, via a model reaction, to undergo a third dynamic chemistry based on a transesterification exchange reaction.

Reprocessable PHU network nanocomposites were prepared by Chen X, et al. using silica nanoparticles with different surface functionalities as reinforcing fillers and DMAP as a vitrimerization catalyst [30]. Different behavior was observed depending on the nanoparticles' surface functional groups. Hydroxyl and amine functional groups can participate in transcarbamylation exchange reactions and reversible cyclic carbonate aminolysis reactions, which constitute the dynamic chemistry associated with reprocessable PHU networks, providing routes for the loss of cross-link density after reprocessing, and leading to losses in mechanical properties. On the other hand, the use of not functionalized silica significantly enhanced Young's modulus and tensile strength, and based on values of the rubbery plateau modulus allowed it to fully recover its cross-link density after a reprocessing step. Reactive Polyhedral Oligomeric Silsesquioxanes (POSS) were also used by Hu S, et al. for reinforcing reprocessable PHU network composites [31]. With functionalized POSS serving as a fraction of the cross-linkers, the PHU-POSS network nanocomposites exhibited significantly enhanced storage modulus at the rubbery plateau region relative to the neat PHU network. With up to 10% POSS loading, these network composites can be melt-state reprocessed at 140 °C with 100% property recovery showing excellent creep resistance at elevated temperatures.

A five-member cyclic carbonate based on novolac epoxide was used by Ge W, et al. as the precursor of polyhydroxyurethane thermosets, while a trifunctional polyetheramine served as the curing agent, and DMAP as a transcarbamylation catalyst [32]. To improve properties, nanocrystalline cellulose modified via surface-initiated RAFT/MADIX polymerization of N-vinylpyrrolidone was used as the nanoreinforcement agent.



In comparison with plain PHU, the nanocomposites displayed enhanced thermomechanical properties, as measured by glass transition temperatures and tensile mechanical properties. Remolding of cut material at elevated temperatures showed its good shape memory and reprocessing properties.

Wu H, et al. presented degradable and self-healing vitrimers based on non-isocyanate polyurethane elastomer. These vitrimers were fabricated by copolymerizing di(trimethylolpropane) and chloroformic acid-propyl ester-based bis(six-membered cyclic carbonate) with amino-terminated liquid nitrile rubber. Reprocessing and self-healing behavior were achieved through the alteration of the network topologies by transcarbonation exchange reactions between hydroxyl and carbonate groups at elevated temperatures. Additionally, 75% of the pure di(trimethylolpropane) monomer was recovered through the hydrolysis and decarbonization of the elastomer containing the dynamic networks in a strong acid solution (HCl) [33].

PHU networks derived from six-membered bis-CC and TAEA, displaying vitrimer properties without the use of an external catalyst, were proposed by D. J. Fortman, et al. [34]. After reprocessing grounded PHU at 160°C for 8h, under elevated pressure, samples exhibited similar glass transition temperature, and retained about 75% of the tensile properties of the virgin material. The vitrimer behavior was explained by a hydroxyl-mediated associative transcarbamylation reaction activated by mechanical stress. In another paper, the authors also evaluated the influence of PHU structure on polymer reprocessability and concluded that five-membered CC, compared to six-membered CC decomposes easier at elevated temperatures due to the reversion of PHU linkage and subsequent side reactions [35].

1,8-Methane Diamine (MDA), derived from turpentine was used as a curing agent for cyclic-carbonated soybean oil (CSBO) for preparing fully bio-based PHUs without solvents and catalysts. The amino groups from MDA facilitate bond exchange under autocatalysis in PHUs, and the rigid structure improves the thermal and mechanical properties of PHUs [36].

Zhao W, et al. proposed polyhydroxyurethanes prepared from bis(six-membered cyclic carbonate), bio-based dimer fatty diamine, and lignin, used in amounts up to 50 % of the mass of CC and diamine [37]. The resulting polymers were reprocessed by cutting and then reshaping via hot-pressing, and after four times of reprocessing still retain 83.1% of their initial tensile strength. This was a result of the high content of hydroxyl groups in PHU and lignin. These groups can easily undergo transcarbamylation reactions with carbamate groups at high temperatures and render fast reconstruction of the covalent crosslinking networks.

PHU networks are reprocessable via transcarbamylation reactions even in the absence of external catalysts, but this process takes several hours at temperatures above 150 °C. The reprocessing times of PHU networks were dramatically shortened by incorporating dynamic disulfide bonds. Fortman, et al. prepared PHU using cystamine, derived from the amino

acid cysteine as a comonomer with a six-membered bis(cyclic carbonate) and tris(2-aminoethyl)amine as a crosslinking agent [38]. Despite good mechanical properties, the obtained materials showed rapid stress relaxation, enabling reprocessing with quantitative recovery of cross-link density as measured by DMTA (dynamic mechanical thermal analysis) after only 30 min of elevated-temperature compression molding. In these polymers, the cross-links may reorganize through at least two pathways: disulfide exchange or transcarbamylation of the urethane linkages. To assess the contributions of each of these processes, the authors compared the cystamine-containing polymers with identical networks in which the disulfide linkages were omitted by replacing cystamine with hexamethylenediamine. In polymers containing disulfides, stress relaxation occurred multiple orders of magnitude more rapidly at similar temperatures and with significantly lower energy of activation, suggesting that the disulfide exchange is the dominant pathway.

Another mechanism used for network rearrangement was dissociative dynamic chemistry, which allowed for the self-healing behavior of partially cross-linked NIPUs synthesized via a Diels-Adler (DA) addition reaction between polyurethane prepolymers with furan terminal groups (F-NIPUs) [39]. These F-NIPUs were synthesized through a ring-opening reaction of diglycerol bis(cyclic carbonate) with isophoronediamine and furfurylamine, and an unsaturated polyester prepared from melt polycondensation of maleic anhydride, adipic acid, and butanediol. The electron-withdrawing carbonyl groups in the unsaturated polyester make the C = C bonds reactive and easy to conduct a DA reaction with furan groups, resulting in thermal reversibility of the NIPUs. The cross-linking of the polymer was verified by swelling analysis, and self-healing properties were confirmed by DSC (differential scanning calorimetry) and POM (polarized optical microscopy) methods. Cracks on the NIPU surface disappeared within 8 minutes at 120 °C.

Conclusion

With the increasing awareness of environmental issues, the possibility of extending the service of thermosetting polymers through self-healing and recycling provides a more sustainable and resource-efficient approach in the field of polymeric materials. In this paper, we presented a wide range of associative and dissociative mechanisms, including transcarbamylation, reversible cyclic carbonate aminolysis, transesterification, and disulfide exchange, used to develop reprocessable non-isocyanate polyurethane vitrimers. However, there are still many opportunities for improvements, such as shortening the synthesis of cyclic carbonates, increasing the reactivity of polyhydroxyurethanes, and improving their water-resistant and mechanical properties. Future work will focus on overcoming these drawbacks and replacing fossil-based raw materials with renewable resources like plant oils, fatty acids, glycerol, cardanol, or lignin. Other areas of interest include new composite and nanocomposite materials, also made with bio-based reinforcements, as well as applications such as 3D printing filaments. The increased demand for recyclable and healable composite materials obtained through dynamic



bond exchange provides prospects for polyhydroxyurethane vitrimers to revolutionize the development of sustainable polymers.

Funding/Acknowledgments

We would like to thank the Ministry of Education and Science of Poland under project No DWD/4/21/2020 for the financial support.

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