Chapter

Isocyanate-Free, Room-Temperature Curing Polyurethanes for Structural Applications

Bruno Traber, Heiko Jung and Raphael Schaller

Abstract

This study presents the development and characterization of novel hybrid non-isocyanate polyurethanes (NIPUs) for structural adhesive applications. A one-pot synthesis method at room temperature was developed, combining polyfunctional cyclic carbonates, diethylenetriamine, and epoxy. Kinetic investigations revealed significant differences between bi- and tricyclic carbonates, with tricyclic variants demonstrating superior curing kinetics. The hybrid NIPUs exhibited remarkable lap-shear strengths of 14–16 MPa on untreated aluminum and planed beech wood after 12 hours of room-temperature curing. The absence of isocyanates offers health and safety advantages in various applications. The versatility of hybrid NIPUs was demonstrated through their broad adhesion spectrum and adaptable mechanical properties, positioning them as promising candidates for diverse industrial applications, including battery component encapsulation and multi-material laminates. While challenges remain, particularly in the availability of higher-functional cyclic carbonates, this research opens new avenues for high-performance adhesive systems.

Keywords: polyurethanes, non-isocyanate polyurethanes, room-temperature curing, structural, metal adhesion

1. Introduction

In 1937, Otto Bayer's pioneering discovery of polyurethanes laid the foundation for a polymer class now recognized for its exceptional versatility and applicability, as characterized by the urethane bond in its structure [1]. The diversity of polyurethanes is rooted in their modular synthesis, which allows the incorporation of a wide range of different repeating units. This adaptability enables fine-tuning of the material's physical and chemical properties, ranging from thermoplastics to highly reactive compounds. As a result, polyurethanes serve an expansive array of applications, including but not limited to foams, coatings, textile fibers, and adhesives [2–4].

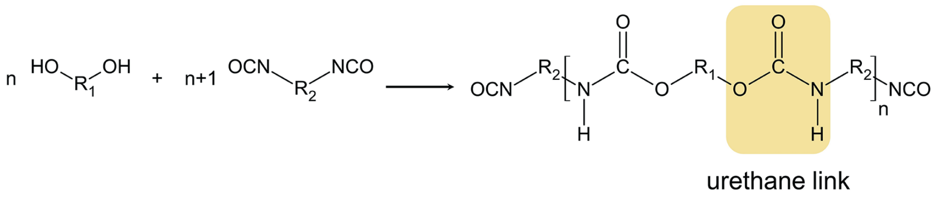

Figure 1.
Reaction equation of the polyaddition between polyol and isocyanate to form polyurethane.

The traditional synthesis of polyurethanes relies on a polyaddition reaction between polyols and isocyanates, typically employing at least bi-functional precursors (**Figure 1**). However, isocyanates pose health concerns, necessitating cautious use and obligatory training. Recent regulatory developments in the European Union [5] have catalyzed a quest for alternative synthetic pathways for polyurethanes. While conventional polyurethanes have dominated the literature with over a million publications in the past two decades, the emerging field of non-isocyanate polyurethanes (NIPUs) represents a niche, with approximately 4500 publications and 100 patents during the same period (**Figure 2**).

Within the research landscape, four primary approaches for synthesizing NIPUs have emerged [6, 7]:

1. Polyaddition of polyfunctional cyclic carbonates and amines: The advantage of this synthetic route is that with each urethane group, an additional functional group, a primary or secondary hydroxy group, is formed (**Figure 3**). Furthermore, no condensation products are formed during the polyaddition, which can be carried out at moderate temperatures (<120°C). An additional benefit of this synthesis is the biodegradability, potential bioavailability, and low toxicity of the cyclic carbonates.

2. Polycondensation between polycarbonates and polyamines or polycarbamates and polyols: The approach appears comparable to the previously described poly-

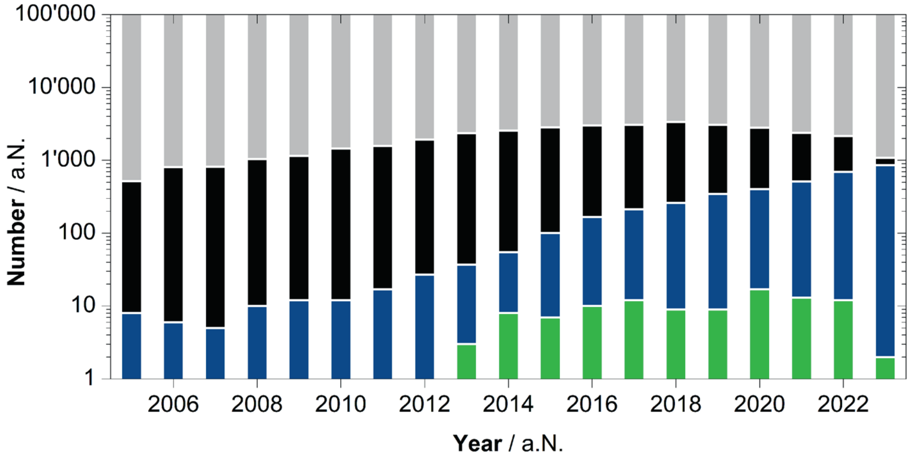

Figure 2.
Activities in filing patents and publishing articles on polyurethanes in the period from 2005 to 2023. Patents: 🟩 *isocyanate-free polyurethanes and* ⬛ *polyurethanes. Articles:* 🟦 *isocyanate-free polyurethanes and* ⬜ *polyurethanes.*

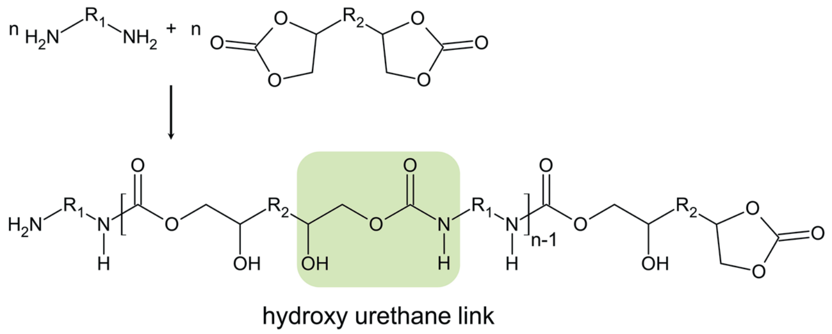

Figure 3.
Reaction equation of the polyaddition between amine and cyclic carbonate for the isocyanate-free formation of poly (hydroxyurethane).

addition. These routes use polycarbonates and polyamines or polycarbamates and polyols with functionalities of at least two. **Figure 4** shows this is a polycondensation reaction where alcohol is produced as an undesired condensate, which is either distilled off or remains in the product.

3. Ring-opening polymerization of six- or seven-membered cyclic carbamates: This method initially appears to be elegant and free of by-products. However, it often requires very high temperatures, around 200°C, and catalysts such as sodium hydride to shift the equilibrium in the desired direction (**Figure 5**). Additionally, phosgene is typically used in the production of cyclic carbamates.

4. In situ formation of isocyanate groups, e.g., via Lossen, Hofmann, or Curtius rearrangement: In these complex synthetic pathways, the specified starting compounds undergo a rearrangement to form in situ isocyanates, which react with alcohol to produce urethanes (**Figure 6**). A disadvantage of this rearrangement is that the process, strictly speaking, is not isocyanate-free, as isocyanates are formed (albeit transiently) during the reaction.

Among these methods, the polyaddition of polyfunctional cyclic carbonates and amines proves to be particularly promising. It leads to the formation of

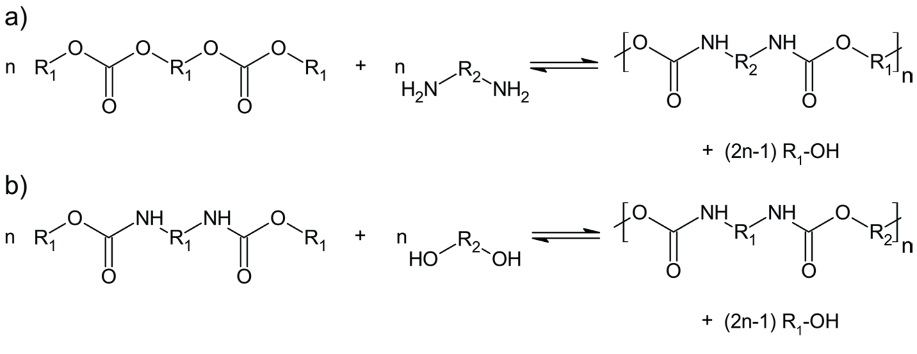

Figure 4.
These polycondensation routes employ (a) polycarbonates with polyamines or (b) polycarbamates with polyols to synthesize NIPUs.

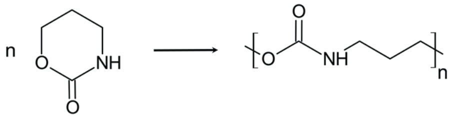

Figure 5.
Ring-opening polymerization of cyclic carbamates to NIPU.

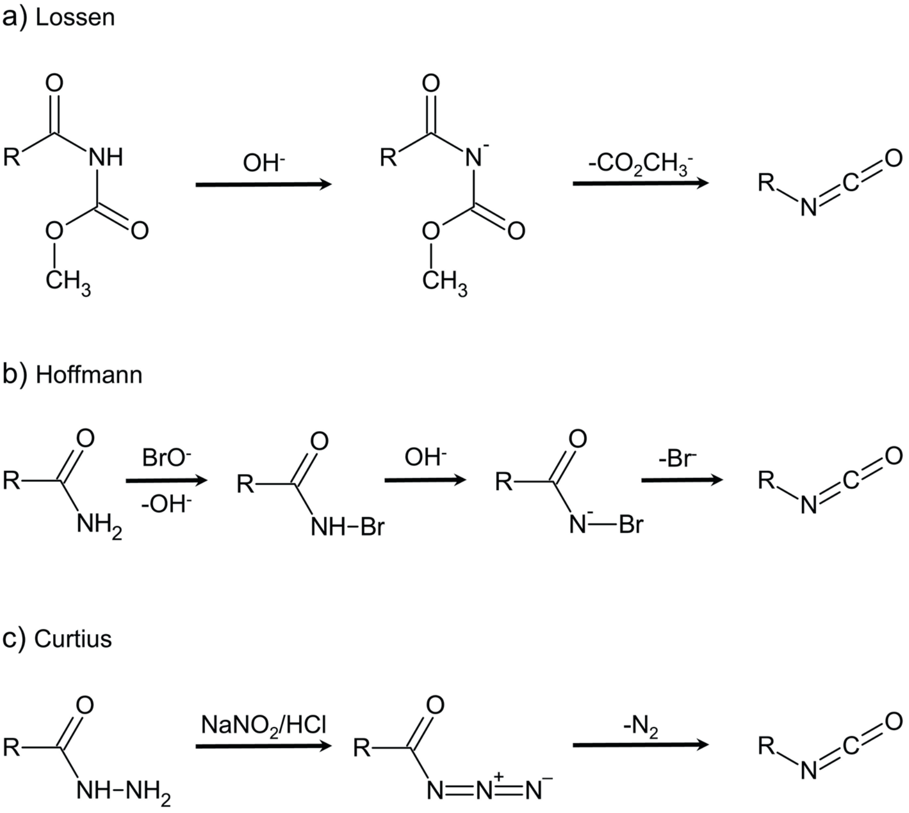

Figure 6.
(a) Lossen, (b) Hoffmann, or (c) Curtius rearrangement: In these complex synthetic routes, the specified starting compounds react through a rearrangement to form in situ generated isocyanates.

poly(hydroxyurethane) (PHU), which reveals urethane bonds and primary or secondary hydroxy groups (**Figure 3**). The motivation for this work lies not solely in the research and development of isocyanate-free polyurethanes but also in optimizing their properties for specific adhesive applications. In the course of this development, a promising combination of epoxides, polyfunctional cyclic carbonates, and amines was identified. These hybrid NIPUs can be tailored regarding their rigidity, flexibility, and adhesion spectrum, making them pre-destined for applications in adhesives, potting compounds, or impregnating resins. The scientific literature frequently describes multi-step processes for producing these hybrid NIPUs. These methods typically involve the initial synthesis of a prepolymer, followed by its further processing in a second step. Such processes are often complex and time-consuming, requiring multiple isolation and purification steps [8].

In an alternative approach to circumvent these multi-step procedures, Ke et al. developed a one-pot synthesis method [9]. In this method, all substances are combined simultaneously. The procedure involves mixing an epoxy-containing compound with a cyclic carbonate and an organocatalyst—in this case, 1,4-diazabicyclo[2.2.2]octane (DABCO)—followed by a reaction with an amine-containing compound. Although this method avoids multiple process steps, it requires heating the reaction mixture, which may limit its applicability in commercial settings such as carpentry workshops or construction sites.

In the following essay, a new generation of hybrid NIPUs are presented that can be produced and processed via a one-pot synthesis at room temperature within 1 hour. These NIPU adhesives are compared and evaluated in terms of viscosity range and lap-shear strength with other structural adhesive classes.

2. Investigations on reaction kinetics

In our efforts to optimize NIPU formulations, we conducted extensive kinetic studies to explore the interactions between formulation ingredients. We quantified the time-dependent viscosity evolution of various formulations using plate-plate rheometry from Anton Paar at 20°C, with a shear rate of 100 s^{-1} over a one-hour period. This approach allowed us to uncover the optimal synergy between polyfunctional cyclic carbonates and diethylenetriamine concerning their reaction kinetics.

Figure 7 provides a compelling visualization of the viscosity profiles for the NIPU formulations under investigation, contrasting the effects of different catalysts on bi- and tricyclic carbonates. Our analysis revealed significant differences in reaction kinetics: formulations with bicyclic carbonates exhibited a moderate viscosity increase from an initial range of 10–50 Pa·s to approximately 450 Pa·s within 1 hour.

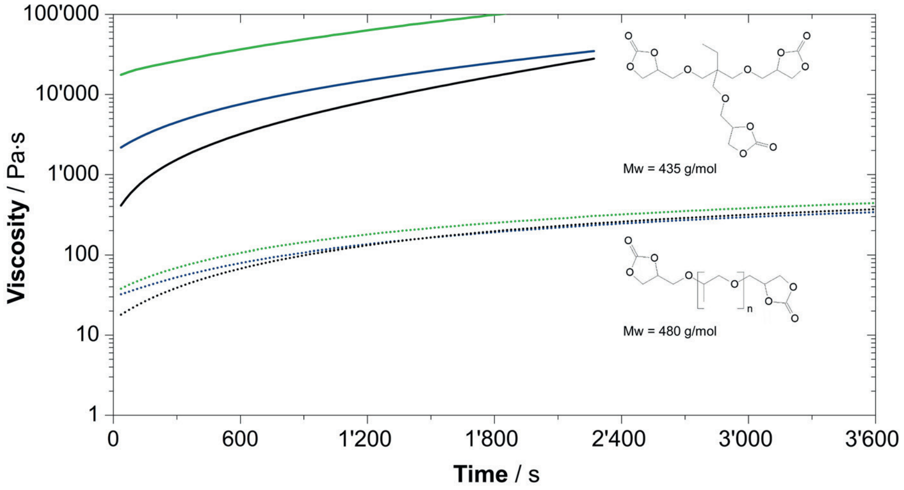

Figure 7.
Viscosity increase of NIPU formulations with polyfunctional cyclic carbonates and diethylenetriamine. A plate-plate rheometer was used at 20°C and a shear rate of 100 s^{-1}. Formulations with a bicyclic carbonate (poly(propylene glycol)α,ω-bis(cyclocarbonate), Mw = 480 g/mol): ••• uncatalyzed, ••• Catalyst 1, DBU, and ••• Catalyst 2, TBD. Formulations with a tricyclic carbonate (trimethylpropane-tri(cyclocarbonate), Mw = 435 g/mol): ▬ uncatalyzed, ▬ Catalyst 1, DBU, and ▬ Catalyst 2, TBD.

In contrast, those containing tricyclic carbonates demonstrated a dramatic rise to over 10,000 Pa·s in just 30 minutes. This pronounced increase in viscosity for tricyclic carbonates indicates enhanced curing kinetics at ambient temperature, highlighting their potential for rapid-curing NIPU systems.

The kinetic studies were conducted meticulously under controlled conditions, utilizing two distinct catalysts—1,5,7-triazabicyclo[4.4.0]dec-5-ene (TBD) and 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU)—each at a 1% w/w concentration, while maintaining a stoichiometric ratio of 1:1 between carbonate and amine fractions.

This systematic approach not only facilitated a comprehensive evaluation of reaction kinetics but also laid the groundwork for developing optimized NIPU formulations with enhanced processing and performance characteristics. Future research may focus on further refining catalyst systems and fine-tuning the carbonate-amine stoichiometry to improve curing kinetics and achieve tailored properties for specific applications. Such advancements could potentially revolutionize the field of non-isocyanate polyurethanes, opening new avenues for their use in diverse industrial sectors.

3. Initial hybrid formulations

For preliminary experiments with hybrid NIPU formulations (**Table 1**), polyfunctional cyclic carbonates, diethylenetriamine, and epoxy were utilized.

The conversion of the carbonate with the amine to form C-N bonds was facilitated through targeted catalysis using organocatalysts. This choice of catalyst eased the

Formulation	**A**	**B**	**Remark**
Ingredients and composition	% w/w	% w/w	—
Epoxy	53.3	29.7	—
Cyclic carbonate	32.7	48.8	A: bi-, B: tricyclic carbonate
Catalyst	1.0	1.0	TBD catalyst
Amine	13.0	20.5	—
Properties	**A**	**B**	**Method**
Initial viscosity/Pa•s	6.0	324.0	DIN EN ISO 10364
Pot life at 20°C/min	5.0	6.0	DIN EN ISO 10364
Lap-shear strength/MPa A: planed beech wood B: untreated aluminum	6.0	9.8	A: DIN EN 12765 B: DIN EN 1465
Storage modulus at 20°C/MPa	1200.0	1200.0	ASTM D5279
Tensile strength/MPa	36.4	46.6	DIN EN ISO 527-1:2019
Strain at break/%	6.2	9.0	DIN EN ISO 527-1:2019
Glass transition temperature T_G/°C	51.6	49.6	ASTM D5279

Table 1.
Initial hybrid NIPU adhesive formulations A and B with their compositions and specific properties such as start viscosity, pot life at 20°C, lap-shear strength, storage modulus, glass transition temperature, and tensile properties.

desired crosslinking at room temperature, avoiding the need for curing at elevated temperatures (typically up to 120°C for several hours), which often leads to unwanted side reactions. Organocatalysts, such as tertiary amines and hydrogen-bond donors, exemplified by TBD or thiourea, effectively activate the carbonyl groups of cyclic carbonates at room temperatures [10]. The one-pot synthesis of the initial formulations followed a six-step protocol:

1. Homogenization of the epoxy and the polyfunctional cyclic carbonate using a speedmixer (2000 min^{-1} for 1 minute).

2. Integration of the catalyst followed by further homogenization (2500 min^{-1} for 3 minutes).

3. Thermal treatment to dissolve the catalyst in the epoxy-carbonate mixture (80°C for 40 minutes).

4. Cooling to room temperature and re-homogenization (2500 min^{-1} for 3 minutes).

5. Incorporation of diethylenetriamine at room temperature (2000 min^{-1} for 1 minute).

6. Immediate processing of the mass by casting or application for adhesive bonding at room temperature.

Detailed information regarding the synthesis methodology is disclosed in the patent application "Verfahren zur Herstellung eines hybriden nicht-isocyanathaltigen Polyurethans" by Lutz and Odermatt [11]. For industrial scaling, these hybrid NIPUs are designed as two-component adhesive systems, where Component A comprises the mixture of epoxy, cyclic carbonate, and catalyst. At the same time, Component B represents the amine component.

4. Isocyanate-free polyurethane hybrids for structural bonding

Based on preliminary findings, all subsequent formulations for hybrid NIPU adhesives with structural properties were engineered using the tricyclic carbonate trimethylpropane-tri(cyclocarbonate) with a molecular weight of 435 g/mol, selecting TBD as the catalyst due to its superior catalytic efficiency and an epoxy. **Figures 8** and **9** illustrate the application domains of various structural adhesives, presenting the relationship between lap-shear strength and initial viscosity. These two properties were measured as follows:

1. Lap-shear strength tests were performed on planed beech wood referred to DIN EN 12765 at durability class C1 (**Figure 8**) and untreated aluminum EN AW-5754 alloy according to DIN EN 1465 (**Figure 9**). All adhesives were prepared and mixed under controlled conditions of 20°C and 65% relative humidity prior to application. The substrates were used as received without pretreatment, with the following dimensions (length × width × thickness):

a. Beechwood: 80.0 mm × 20.0 mm × 5.0 mm, with a 10 mm overlap

b. Aluminum: 80.0 mm × 25.0 mm × 1.6 mm, with a 12.5 mm overlap

The lap-shear specimens were subjected to a pressure of 0.8 N/mm^2 for 12 hours at 20°C. Subsequently, the specimens underwent a 7-day conditioning period in a climate-controlled environment maintained at 20°C and 65% relative humidity. Lap-shear tests were performed using a ZwickRoell universal testing machine equipped with a 50 kN load cell and tested at a 10 mm/min crosshead speed.

2. Initial viscosity was measured using plate-plate rheometry from Anton Paar at 20°C with a shear rate of 100 s^{-1} according to DIN EN ISO 10364.

On beech wood, 2 K epoxy adhesives, atypical for wood joints, exhibit good lap-shear strengths of up to 13 MPa in the low viscosity range (200–4000 mPa•s).

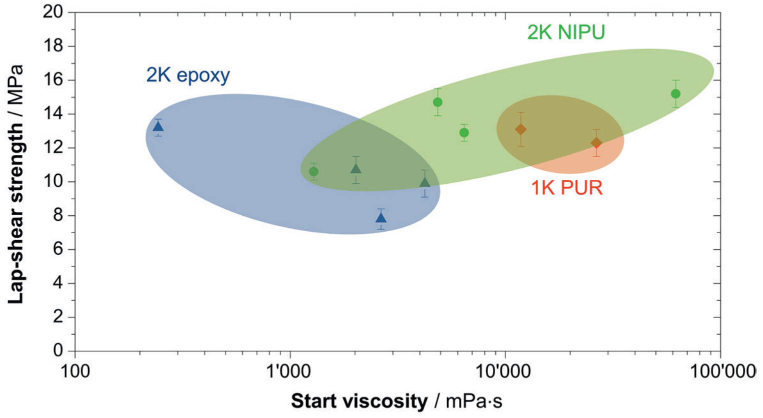

Figure 8.
Lap-shear strength on planed beech wood versus initial viscosity (measured after mixing) of structural adhesives: ▲ *2 K epoxy,* ◆ *1 K PUR, and* ● *2 K NIPU.*

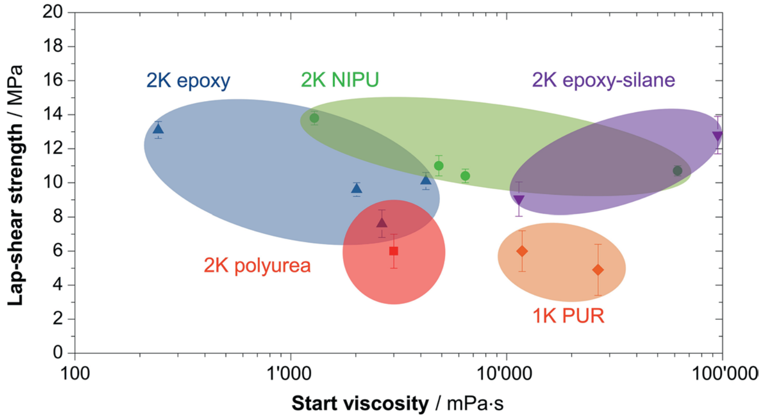

Figure 9.
Lap-shear strength on untreated aluminum EN AW-5754 versus initial viscosity of structural adhesives: ▲ *2 K epoxy,* ■ *2 K polyurea,* ◆ *1 K PUR,* ▼ *2K epoxy-silane, and* ● *2 K NIPU.*

Reactive one-part polyurethane (1 K PUR) systems achieve lap-shear strengths of 12–13 MPa at typically higher initial viscosities >10,000 mPa•s. Hybrid NIPUs demonstrate the highest lap-shear strength of over 15 MPa across the broad viscosity range, with wood failure occurring in more than 60% of cases, indicating their versatility and effectiveness.

On untreated aluminum EN AW-5754, 1 K PUR and 2 K polyurea adhesives exhibit predictably poor adhesion due to the lack of surface pretreatment. In contrast, 2 K epoxy and 2 K epoxy-silane adhesives demonstrate superior performance, achieving lap-shear strengths ranging from 10 to 14 MPa.

Notably, hybrid NIPUs attain comparable results to 2 K epoxy and 2 K silane-modified adhesives on untreated aluminum, primarily exhibiting cohesive failure while maintaining their characteristic polyurethane tensile properties, as illustrated in **Figure 10**. This performance suggests that hybrid NIPUs may offer a promising alternative for bonding untreated aluminum surfaces without compromising the beneficial properties of polyurethane-based adhesives.

Stress-strain curves were generated in accordance with DIN EN ISO 527-1:2019. Epoxy and hybrid NIPU samples were prepared and mixed under controlled conditions of 20°C and 65% relative humidity before being injected into 5A tensile specimen molds. The specimens were then subjected to a 7-day conditioning period in a climate-controlled environment maintained at 20°C and 65% relative humidity. Tensile tests were conducted using an Instron universal testing machine equipped with a 30 kN load cell and tested at a 10 mm/min crosshead speed.

The stress-strain diagram (**Figure 10**) shows that by increasing the NIPU content in the epoxy, a transition from brittle to elastic behavior with strain-hardening behavior can be achieved. **Table 2** presents the corresponding lap-shear strengths. At an epoxy: NIPU ratio of 78:22, the highest shear strengths on the planed beech wood are achieved, with approximately 80% wood failure. On the aluminum alloy EN AW-5754, the maximum shear strength of 15.9 ± 1.0 MPa was obtained at an epoxy: NIPU ratio of 55:45, exhibiting cohesive failure in the adhesive. This result is remarkable for a polyurethane-based adhesive formulation on untreated aluminum.

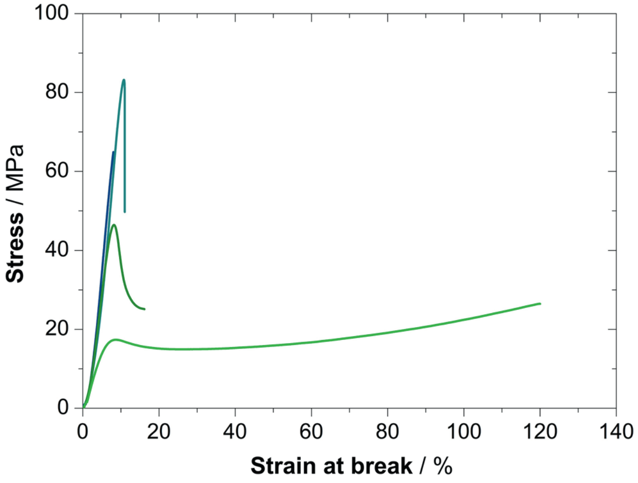

Figure 10.
The stress-strain diagram shows a transition from epoxy to a brittle and tough hybrid NIPU adhesive: Epoxy with ▬ *0% w/w,* ▬ *22% w/w,* ▬ *33% w/w, and* ▬ *45% w/w NIPU content.*

NIPU content in epoxy % w/w	Lap-shear strength planed beech wood MPa	Lap-shear strength untreated aluminum MPa
—00	11.3 ± 0.2	10.1 ± 0.4
—22	13.3 ± 0.4	10.8 ± 0.9
—33	12.9 ± 0.6	14.1 ± 0.5
—45	10.2 ± 0.2	15.9 ± 1.0

Table 2.
Lap-shear strength on planed beech wood and untreated aluminum EN AW-5754 of hybrid NIPU adhesives at various epoxy:NIPU proportions.

The adhesive demonstrates high toughness, with the epoxy contributing to adhesion on metals, supported by the additional hydroxy groups of the NIPU. This combination of properties opens new possibilities for developing high-performance, versatile adhesive systems for diverse structural applications.

5. Challenges and opportunities

The development and application of hybrid NIPU adhesives face several significant technical and economic challenges.

First, a fundamental issue lies in the generally low reactivity of cyclic carbonates, which impairs the efficiency of polyaddition reactions and leads to extended reaction times. This kinetic limitation can be problematic in industrial applications, negatively affecting processing speed and, consequently, process economics. Developing suitable catalyst systems to accelerate the reaction remains a central development goal.

Second, the current catalyst quantities are too high at 1% w/w. Therefore, optimizing catalyst systems to accelerate the polyaddition reaction is already a central research topic [12–14]. Advancements in this area could significantly increase processing speed and thus improve industrial applicability.

Third, the long-term stability of hybrid NIPU adhesives under application conditions has yet to be sufficiently researched. Comprehensive aging studies, including alternating climate change and weathering tests, are necessary to generate reliable data on the durability of bonded components.

Last, another challenge is the limited availability of higher-functional cyclic carbonates. While monofunctional cyclic carbonates are commercially available in large quantities, including from Huntsman or UBE Corporation, higher-functional variants are produced only on a laboratory scale, such as by Specific Polymers [15]. This limitation has significant implications for the scalability and economic feasibility of NIPU technology. Nevertheless, various approaches to producing cyclic carbonates from alternative sources are being explored in academia. Promising strategies include the use of polyether polyols from the fermentation of polysaccharides or the utilization of lignin or glycerine with subsequent cycloaddition of CO_2 as an abundant and cost-effective renewable raw material source to produce cyclic carbonates [6, 16–18]. Further activities also exist in areas where NIPU has carbonated soybean oil and a diamine comprising 100% renewable carbon content [19].

6. Conclusions

Hybrid NIPUs demonstrate their potential in structural bonding. In this context, initial formulations achieved remarkable lap-shear strengths of 14–16 MPa on untreated aluminum and planed beech wood, underscoring their suitability for demanding joints. The absence of isocyanates in these adhesive systems offers advantages in terms of health protection, particularly in carpentry workshops and construction sites, where exposure to potentially harmful substances can be minimized.

Of particular note are the surprising properties of hybrid NIPUs regarding their adhesion spectrum on untreated metal surfaces and their structural performance. Initial formulations achieved lap-shear strengths of nearly 16 MPa on untreated aluminum with a pressing time of 12 hours under room temperature conditions.

Further investigations indicated that new hybrid NIPU formulations with even higher lap-shear strengths in combination with increased toughness and reduced pressing times are achievable. This discovery opens new application fields for polyurethane adhesives in areas where pronounced metal adhesion is critical. A promising application area is the encapsulation of battery components in electromobility. Further, the expanded adhesion spectrum of hybrid NIPUs predestines them as laminating resins in multi-material laminates, such as those used in ski construction. This versatility in substrate compatibility and advantageous mechanical properties position hybrid NIPUs as a potential novel solution for complex bonding tasks across various industrial sectors.

Ultimately, the most significant limitation to the economic feasibility of this technology is the availability of higher-functional cyclic carbonates.

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