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A Fluoride Degradable Crosslinker for Debond-on-Demand Polyurethane Based Crosslinked Adhesives

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Abstract

The requirement to consider the whole lifecycle of products including disassembly and recycling has resulted in considerable interest in debond-on-demand adhesives. These smart materials undergo controlled loss of adhesive strength when subject to a specific stimulus. This paper reports the design of a crosslinked polyurethane (PU) adhesive which incorporates a fluoride responsive degradable group. The crosslinked PU (CLP) adhesive showed a 28 % increase in adhesive bonding strengths by lap shear testing (14.6 MPa) when compared to structurally analogous linear PU (LPU) adhesive (11.4 MPa). After 3 hours in contact with fluoride ions, the CLP exhibited a 55 % loss in adhesive bonding strength (from 14.6 MPa to 6.7 MPa) as a consequence of selective degradation of covalent bonds at the crosslinking sites. This work introduces a new route to dismantle components adhered with the widely used PU adhesives, facilitating recovery of valuable materials, and dramatically reducing waste.

1. Introduction

The desire for high strength adhesives that exhibit healable and debondable properties range of environmental conditions has driven interest in this field over several decades.[1] Many different types of adhesives have been developed including hot-melt adhesives,[2–6] pressure sensitive adhesives[7–9] as well as reactive [10,11] and crosslinked adhesives [9,12,13]. Formulations commonly used to produce in

crosslinked adhesives include those containing acrylates,[8,13–16] epoxy resins [17–21] and polyurethanes.[2,12,22] Whilst crosslinked materials generally offer higher mechanical strengths over a greater temperature range than structure analogous linear adhesives [23] they are by their nature, insoluble and cannot be melt processed. This intractability means they cannot be heated and extruded or solvent cast into position to enable bonding. Consequently, they are typically produced by mixing two tractable formulations that can react and harden *in situ*. In addition, selective removal of the adhesive to facilitate debonding at the end of life is problematic owing to the insolubility of the material. Consequently, several recent studies have sought to produce a new class of materials that offers the advantage of high strength and low creep of a crosslinked system, yet the debonding and solubility properties a typical linear hot melt adhesive.[24]

An approach to producing debondable crosslinked adhesives is to introduce functionalities at the crosslinking points that dissociate when exposed to a stimulus. For example, thermal stimulus can be used to induce Diels-Alder adducts to undergo reversible [4 + 2] cycloaddition reaction. [25–30] Heat has also been used to break the S-S bonds in disulfide bridges.[31] UV light has been used as a stimulus (to facilitate the reversible cycloaddition reactions in anthracene based adhesives.[32–34]

A new adhesive architecture has recently been produced by Phillips and co-workers where the crosslinking monomers are chemo-responsive and can be cleaved under specific conditions. [35] The material was bonded to glass substrates (0.51 ± 0.10 MPa) and the debonding time after addition of fluoride could be programmed by systematically varying the structure of the polymer. However, this elegant system contains features that may hinder universal use: bonding was achieved by evaporation of solvent from the THF swollen crosslinked gel, which releases undesirable VOCs,[36,37] the crosslinked material was synthesised using expensive ruthenium based catalyst (Grubbs' 2nd generation catalyst), and the degradable group required seven synthetic steps to synthesise in an overall yield of 10.2%.

We have recently reported the synthesis of fluoride degradable linear PU based adhesives. [38,39] The uncrosslinked, linear structure of these materials results in adhesives that can be thermally rebonded through multiple cycles without loss of adhesive qualities, but that also degrade on the addition of fluoride ions which results

in a permanent reduction in adhesive performance. The materials are comprised of diisocyanate linkers with hydrogenated polybutadienes or polyester soft segments. The common structural feature of these adhesives was the *tert*-butyldimethylsilyl (TBS) protected bifunctional degradable unit (BDU **1**) which can be synthesised in just 2 steps from commercially available materials. [38,40] The highest performing composition exhibited strong adhesive bond strengths (>11 MPa) achieved at an easily accessible bonding temperature (60 °C). In an effort to further improve the bonding strength of these materials we proposed to use a trifunctional fluoride degradable unit (TDU **2**). This TBS protected tri-benzyl alcohol, obtained in just two synthetic steps, was recently developed as the structural component in a chemical warfare agent detection system,[41] but has not been used in materials chemistry.

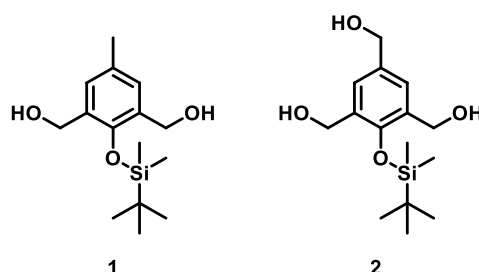
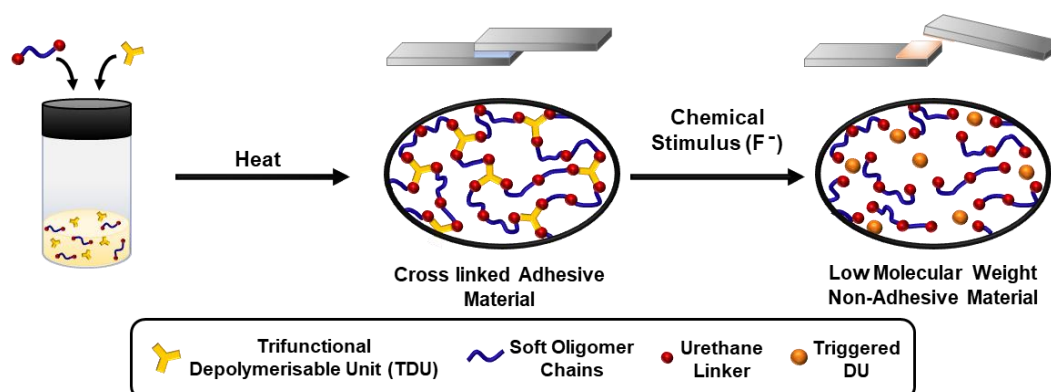


Figure 1 Structures of the bi- and tri-functional (**1** and **2**, respectively) degradable units.

Herein we report the fluoride responsive, crosslinked PU adhesive containing TDU **2** which can be synthesised without expensive catalysts and bonds surfaces without off-gassing e.g. araldite. Moreover, upon treatment of fluoride ions, the resulting polyurethane network degrades at the crosslinking points which dramatically reduces the adhesive strength (Scheme 1), allowing facile debonding of the substrates.



Scheme 1 Schematic showing the non-reversible nature of the fluoride responsive crosslinked adhesive.

2. Experimental

2.1. Chemicals

Stepanpol PC-205P-30 was kindly supplied by Alfa Chemicals. It was dried in a vacuum oven at 110 °C under 100 mbar vacuum for 1 hour prior to use. *Tert*-butyldimethylsilyl chloride (TBDMSCl) was purchased from Fluorochem and used as received. All other chemicals were purchased from Alfa Aesar and used as received.

2.2. Characterisation

¹H NMR (400 MHz) and ¹³C NMR (100 MHz) spectra were recorded on either a Bruker Nanobay 400 or a Bruker DPX 400. Samples for NMR spectroscopic analysis were prepared in DMSO-*d*₆. The data was processed using MestReNova Version 6.0.2-5475.

Infrared spectroscopic analysis was carried out on a PerkinElmer 100 FT-IR spectrometer equipped with a diamond attenuated total reflectance sampling attachment, and samples were analysed in neat form. The infrared spectroscopic data were processed using Microsoft Excel 365.

Differential scanning calorimetric analysis used a TA Instruments DSC Q2000 with samples exposed from 200 °C to -10 °C to 200 °C at 5 °C/min; with an initial cycle from 20 °C to 110 °C to 20 °C at 10 °C/min. The typical sample mass was 5-6 mg, and the data was processed using TA Universal Analysis Version 4.7A and Microsoft Excel 365.

Lap shear adhesion samples were carried out in accordance with ASTM D1002 standards using an AML X5-500 single column universal tester, equipped with a 5 kN load cell and wedge grips. The aluminium coupons were cleaned for 20 minutes in methyl ethyl ketone and dried overnight in a desiccator. The coupons were then etched as previously reported. [39]

2.3. Synthesis

2.3.1. Synthesis of sodium 2,4,6-trimethylolphenate (4). [42]

Phenol (11.75 g, 0.13 mol) was dissolved in formaldehyde (33% in Methanol, 56 mL, 0.50 mol) at 0 °C. Water (10 mL) was added to the mixture, followed by NaOH (5.1 g, 0.13 mol) and stirred at 0 °C until all NaOH dissolved. The reaction mixture was then stirred at 25 °C for 24 h, before being poured slowly into vigorously stirred propan-2-ol (600 mL) at 0 °C. The precipitate was filtered under vacuum, washed with propan-2-ol (100 mL) before drying under high vacuum (0.1 mmbar) overnight to afford an off-

white powder (14.4 g, 70 %). The product was stored under vacuum when not in use. m.p. (DSC) 142 °C; ν_{max} (thin film, cm^{-1}) 3322, 2848, 2638, 1611, 1300, 1029, 754. δ_{H} (400 MHz, $\text{DMSO}-d_6$, ppm) 6.51 (2H, br, Ar-H), 4.44 (4H, br, Ar-CH₂-OH), 4.18 (2H, br, Ar-CH₂-OH). δ_{C} (100 MHz, DMSO, ppm) 126.5, 124.1, 64.8, 63.9. (m/z) found 229.0448 Da ($\text{C}_9\text{H}_{11}\text{O}_4\text{Na}_2$), calculated 229.0453 Da ($\text{C}_9\text{H}_{11}\text{O}_4\text{Na}_2$).

2.3.2. Synthesis of TDU 2 [40]

TBDMSCl (18.27 g, 0.12 mol) was added to a mixture of sodium 2,4,6-trimethylphenate (5.00 g, 24.3 mmol) and imidazole (8.26 g, 0.12 mol) in anhydrous DMF (300 mL) and stirred for 4 hours at 35 °C under nitrogen. The mixture was diluted in ethyl acetate (200 mL) and washed with water (2 × 200 mL). The organic mixture was collected, dried under MgSO_4 and concentrated to afford the crude oil, which was dissolved in methanol (100 mL) and diethyl ether (20 mL) and *p*-toluenesulfonic acid (0.15 g, 0.87 mmol) was added and stirred at ambient temperature for 1 hour. The mixture was diluted in ethyl acetate (100 mL), washed with saturated NaHCO_3 solution (100 mL), followed by brine solution (100 mL). The organic mixture was collected, dried over MgSO_4 and concentrated *in vacuo*. The crude was purified through a silica plug eluting with ethyl acetate to afford a white powder after evaporation of the solvent (3.56 g, 45 %). m.p. (DSC) 93 °C; ν_{max} (thin film, cm^{-1}) 3275, 2926, 2860, 1457, 1245, 886, 776. δ_{H} (400 MHz, $\text{DMSO}-d_6$, ppm) 7.26 (2H, s, Ar-H), 5.12 - 4.95 (3H, m, OH), 4.51 - 4.34 (6H, m, Ar-CH₂-OH), 0.98 (9H, s, Si-C-(CH₃)₃), 0.14 (6H, s, Si-(CH₃)₂). δ_{C} (100 MHz, DMSO, ppm) 156.1, 135.1, 132.1, 124.8, 63.0, 58.3, 26.0, 18.5, -3.6. (m/z) found 321.1489 Da ($\text{C}_{15}\text{H}_{26}\text{O}_4\text{NaSi}$), calculated 321.1498 Da ($\text{C}_{15}\text{H}_{26}\text{O}_4\text{NaSi}$).

2.3.3. Preparation of Crosslinked PU adhesive and lap shear samples

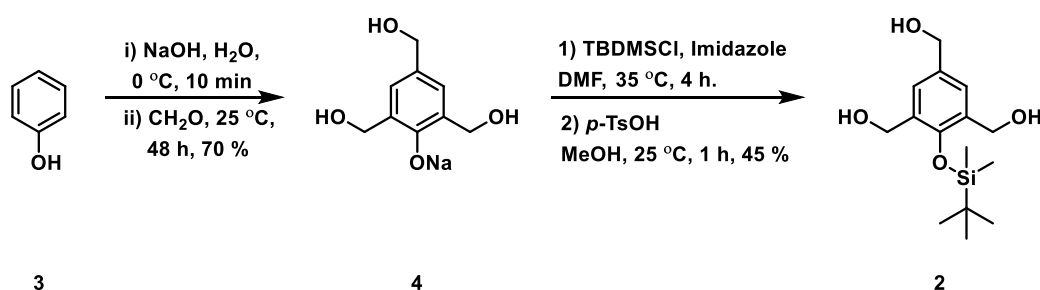
4,4'-methylene diphenyl diisocyanate (0.66 g, 2.67 mmol) was added to Stepanpol PC-205-P 30 (5.00 g, 1.33 mmol) and stirred for 3 hours at 120 °C under an inert atmosphere. The prepolymer (500 mg) was added to a glass vial containing the TDU 2 (26 mg, 88 μmol) and mixed at 110 °C with a spatula and immediately spread onto a lap shear coupon over a 12 × 26 mm area, before being clamped with another lap shear coupon and cured at 120 °C for 1 hour.

2.3.4. Preparation of Linear PU adhesive and lap shear samples.

The linear PU was synthesised and adhered to aluminium coupons as previously reported. [39]

3. Results and Discussion

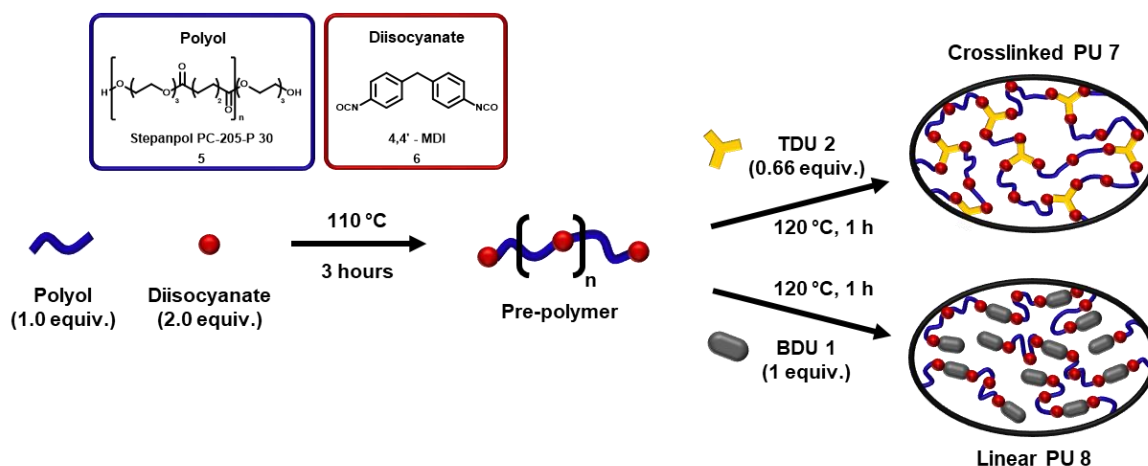
The TDU **2** was prepared as recently reported [41] with minor modifications (Scheme 2). Phenol **3** is reacted with formaldehyde under basic conditions to give the sodium phenolate **4**, which was reacted with *tert*-butyldimethylsilyl chloride in DMF at 35 °C to afford a tetra protected intermediate which was not isolated but immediately subject to selective deprotection with *p*-toluenesulfonic acid in methanol to give the targeted TBS protected TDU **2** at 45% yield, slightly higher than previously recorded yields (38%) [41].



Scheme 2 Synthesis of the trifunctional degradable group (TDU) **2**.

In our previous work, [38,39] the hydrogenated polybutadiene (HPB) based PU showed butt-tensile adhesion (ca. 7 MPa) at 140 °C. Through structure property optimisation studies [39] we found that structurally analogous PUs containing the commercially available polyester, Stepanol, (**5**) increased the butt tensile adhesion (ca. 10-12 MPa) while reducing bonding temperatures to 60 °C. This was a result of introducing crystalline regions within the soft midblock of the supramolecular polymer. Therefore, we selected the same Stepanol **5** polyol to prepare a bifunctional isocyanate prepolymer with 4,4'-methylene diphenyl diisocyanate (MDI (**6**)) which in turn would be reacted with the TDU **2** to create a crosslinked adhesive material.

Addition of Stepanol **5** to MDI **6** at 110 °C for 3 hours (OH : NCO = 1 : 2) resulted in the formation of an isocyanate terminated prepolymer. Subsequent addition of neat TDU **2** at 120 °C resulted in the mixture solidifying within 1 hour suggesting the successful formation of the crosslinked PU **7** (Scheme 3). For comparison, the previously reported linear PU **8** is also shown.



Scheme 3 Synthesis of the crosslinked PU adhesive **7** from an isocyanate terminated prepolymer (where $n = 1-2$). As a comparison, the previously reported linear PU **8** is shown

As expected from a crosslinked material, CPU **7** did not dissolve in any selected solvent, although did swell markedly in solvents such THF and acetone (Figure 2). For comparison, the linear PU **8** is readily soluble in polar solvents including tetrahydrofuran and chloroform. [38,39]

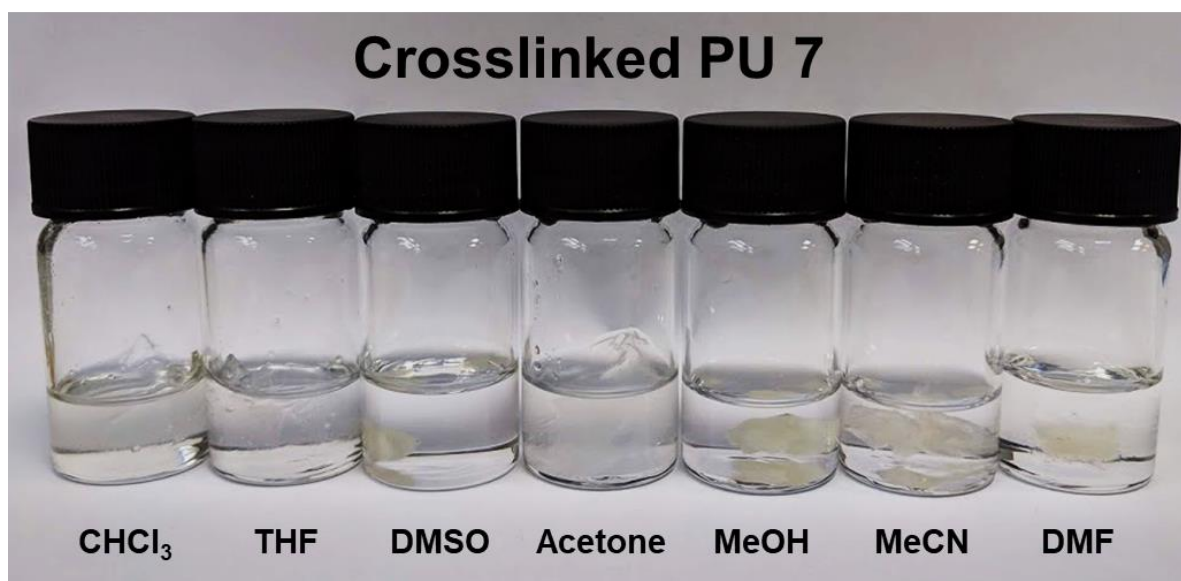


Figure 2 Samples of the crosslinked PU **7** soaked in various solvents for 24 hours at 35 °C. Swelling was most pronounced in CHCl₃, THF and acetone.

The thermal properties of CPU **7** were investigated by differential scanning calorimetry (DSC) (Figure 3). A melting transition (T_m) at 48 °C and a crystallisation transition (T_c) at 16 °C were detected, which are close to those of the of the pristine Stepanpol **5** ($T_m \approx 54^\circ\text{C}$, $T_c \approx 36^\circ\text{C}$). No transitions were observed between 80 – 100 °C in the DSC

thermogram, which would indicate the presence of any unreacted TDU **2** (T_m 93 °C); therefore, suggesting complete consumption of TDU **2**.

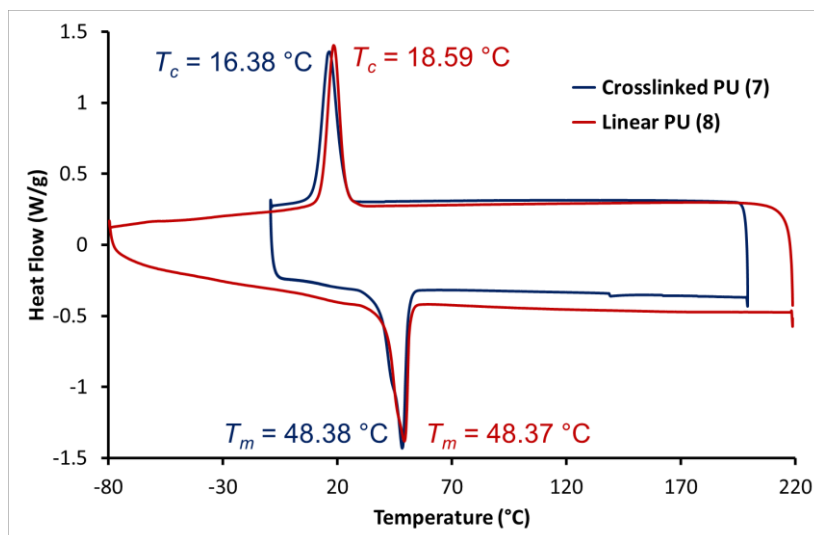
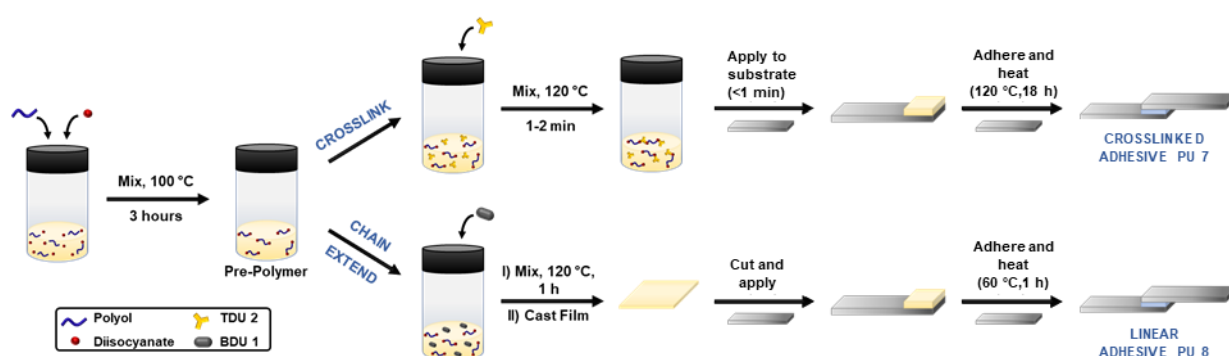


Figure 3 DSC thermogram of the Stepanol CLP **7**. (Ramp rate 5 °C/min)

As a consequent of the crosslinked architecture of CPU **7** it is not possible to melt process the material as possible with typical linear hot melt adhesives. Therefore, bonding was carried out using a simple procedure (Scheme 5). Firstly, the isocyanate terminated prepolymer was made by mixing MDI **6** and Stepanol **5** (NCO:OH = 2:1) at 100 °C. After 3 h, TDU **2** was added to the prepolymer and rapidly hand mixed before being applied to a lap shear coupon (12 × 26 mm) within a minute. After application of the coupon, the adhesive was cured for 18 hours at 120 °C. In comparison, the linear PU adhesive **8** was first solvent cast into a hot-melt film before being applied to the substrates (Scheme 5) thereby increasing assembly time from start to finish.



Scheme 5 Preparation of the crosslinked and linear PU adhesives and application to substrate.

Photos of the bonded aluminium substrates with the crosslinked PU adhesive 7 are shown in Figure 4 (A-C). The bonded area was 12 × 26 mm (conforming to ASTM D1002 standards) and the bonded samples underwent strength testing in the lap shear geometry at a strain rate of 1 mm/min. The samples fractured by cohesive failure as shown by the residual adhesive on both surfaces of the joint (Figure 4D). This indicates loss of strength from the bulk adhesive, and not loss of strength from the adhesion between the PU and aluminium surface.

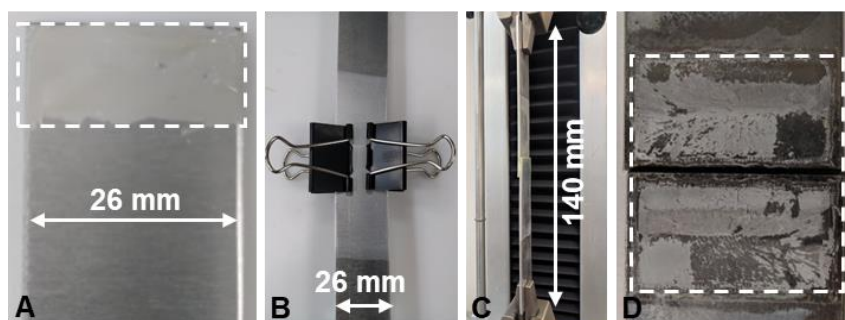


Figure 4 Photos of the lap shear samples: (A) the paste spread over 12 × 27 mm area on a pre-treated aluminium coupon, (B) two aluminium coupons clamped together sandwiching the adhesive paste, (C) the aluminium lap shear sample in the tensile apparatus, and (D) the adhesive bond after breaking showing cohesive failure (adhesive remaining on both side of substrate).

Figure 5 shows the lap shear testing data for the crosslinked PU **7** and linear PU **8** in their pristine state (blue hashed bars). Introducing crosslinking into the system resulted in a significant (27%) increase in lap shear modulus compared to the linear system. As the main advantage of these materials is the on-demand debonding property, the samples were immersed in a 0.025 M TBAF/acetonitrile solution for 3 hours and then dried at 40 °C for 1 hour prior to adhesive testing. The crosslinked material underwent a much greater reduction in lap shear modulus (55%) after degradation than the linear material (36%).

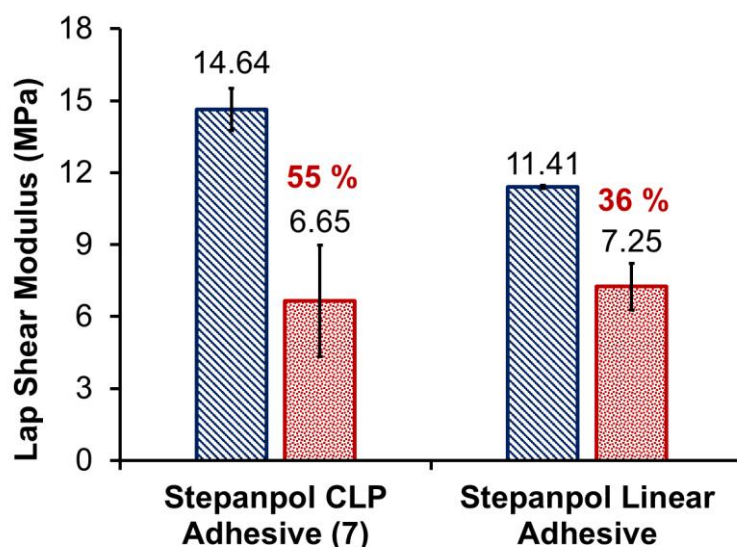
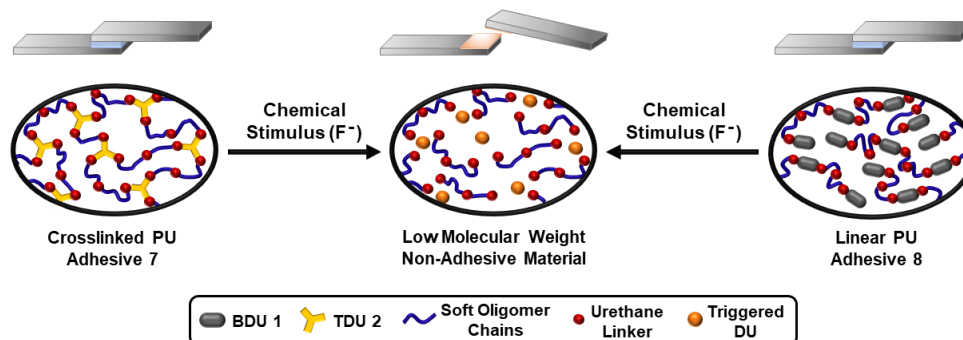


Figure 5 Lap shear tests results for: (blue hatched bars) pristine CLP and Linear PU and (red dot bars) after treatment with 0.025 M TBAF in acetonitrile for 3 hours. The % in red show the change in lap shear modulus between the pristine and degraded samples. Errors were calculated from the standard deviations ($n = 3$).

The crosslinked and linear materials are both derived from the same pre-polymer (see scheme 5) which is either chain extended or crosslinked by the degradable groups (BDU 1 or TDU 2). Thus, chemical degradation of either CLP **7** or LPU **8** results in the same polymeric residue, with a molecular weight that is related to common pre-polymer. This is shown schematically in scheme 6 and accounts for the similar lap shear modulus for the degraded products from CLP **7** and LPU **8** (ca. 7 MPa).



Scheme 6 Schematic showing the degradation of the linear and crosslinked adhesives.

4. Conclusion

In this paper, we report the synthesis of a novel crosslinked fluoride responsive PU. The CLP adhesive was constructed from an isocyanate terminated polyester and a trifunctional degradable crosslinker. The polyester based material did not dissolve in a variety of solvents. However, as the polymeric network incorporated crystallite regions, the adhesive showed a melting transition at ca. 48 °C. Adhesion was obtained

through a reactive *in situ* method on the aluminium lap shear specimens. The crosslinked adhesive showed a 28 % increase in adhesive bonding strength when compared to previously reported linear polymeric adhesives. Finally, the debonding on demand nature of the adhesive was tested, resulting in a 55 % loss in adhesive bonding strength when treated with a TBAF solution for 3 hours.

5. Conflicts of interest

The authors declare no competing financial interest.

6. Acknowledgements

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