Soybean Oil-based Non-Isocyanate Polyurethanes for Commercial Applications

Final Report

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Graphical Summary

The graphical summary of the work performed using soybean oil for adhesive and coating application is provided in Figure 1.

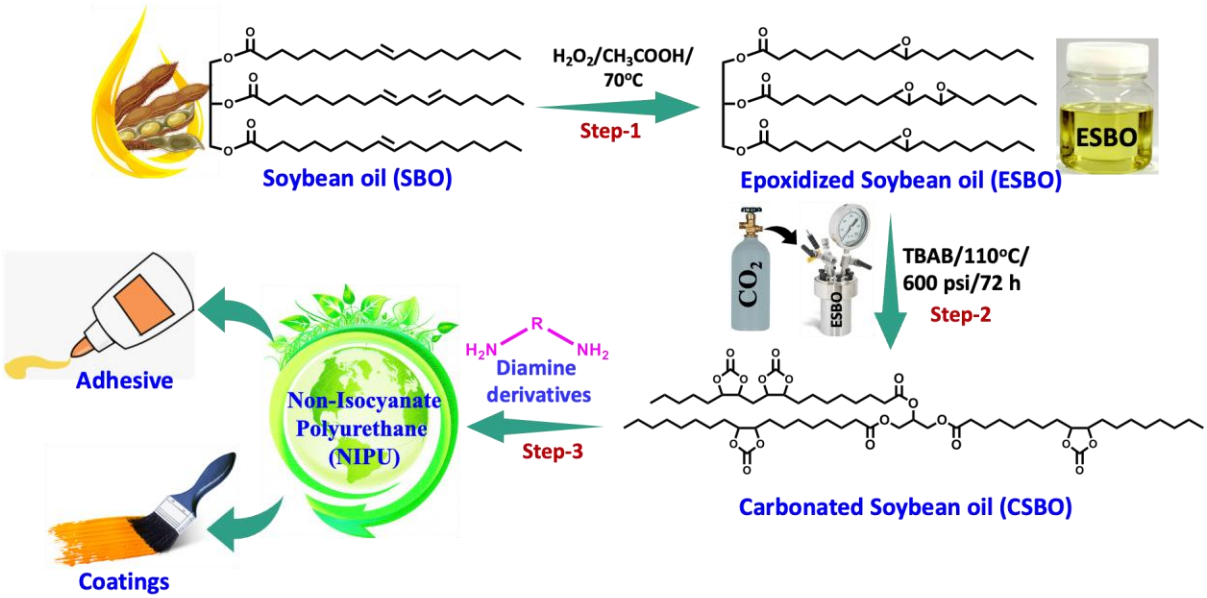

Figure 1. A schematic diagram for the synthesis of non-isocyanate polyurethane (NIPU) for adhesive and coating applications.

Detailed Report of the Work Performed

In continuation of our last submitted report, we have already discussed the thermal properties (DSC and TGA analysis), water affinity (contact angle), and shore D hardness of the NIPU materials (CSBO_CYS, CSBO_DETA, and CSBO_TETA). Herein, we elaborately discussed the flammability test, degree of cross-linking (gel content), chemical resistance performance, and also the ink repellence properties of the NIPU coating materials.

Flammability Test:

Furthermore, we have carried out the flammability tests of all the NIPU wood-coated specimens (CSBO_CYS, CSBO_DETA, and CSBO_TETA) and uncoated wood as well to gain insight into the coating's performance when exposed to fire. Here we investigated the ignition time, burning time, and also the weight loss of the specimens during the flammability test. As indicated in Figure 1A, the uncoated wood sample caught fire quickly by showing an ignition time of ~3 sec, whereas the ignition time increases in NIPU-coated wood samples. It is worth mentioning that, compared to CSBO_CYS and CSBO_TETA coated samples, the maximum ignition time of ~5 sec was observed for the CSBO_DETA coated wood sample. Moreover, from the burning time data (Figure 1B), it was indicated that the uncoated wood exhibits the longest burning time of ~90 sec with maximum weight loss of ~7%, whereas in all NIPU-coated samples, the burning duration as well as weight loss decreases (Figure 1B). As shown in Figure 1C, the CSBO_DETA coated sample shows the lowest burning time of ~38 sec with a weight loss of ~1.5%, whereas the CSBO_CYS and CSBO_TETA coated samples exhibited burning times of ~54 sec and ~40 sec with relative weight losses of ~4.5% and ~2% respectively. The images of the flammability test of the uncoated wood coupon and all the NIPU-coated wood specimens have been displayed in Figure 1. However, the initial flammability tests revealed that all the NIPU samples can act as a protective coating material on the wood surface from fire exposure.

Note: We will further investigate the effect of coating in the flame test by varying the loading of diamine cross-linker during the fabrication of NIPU coating materials.

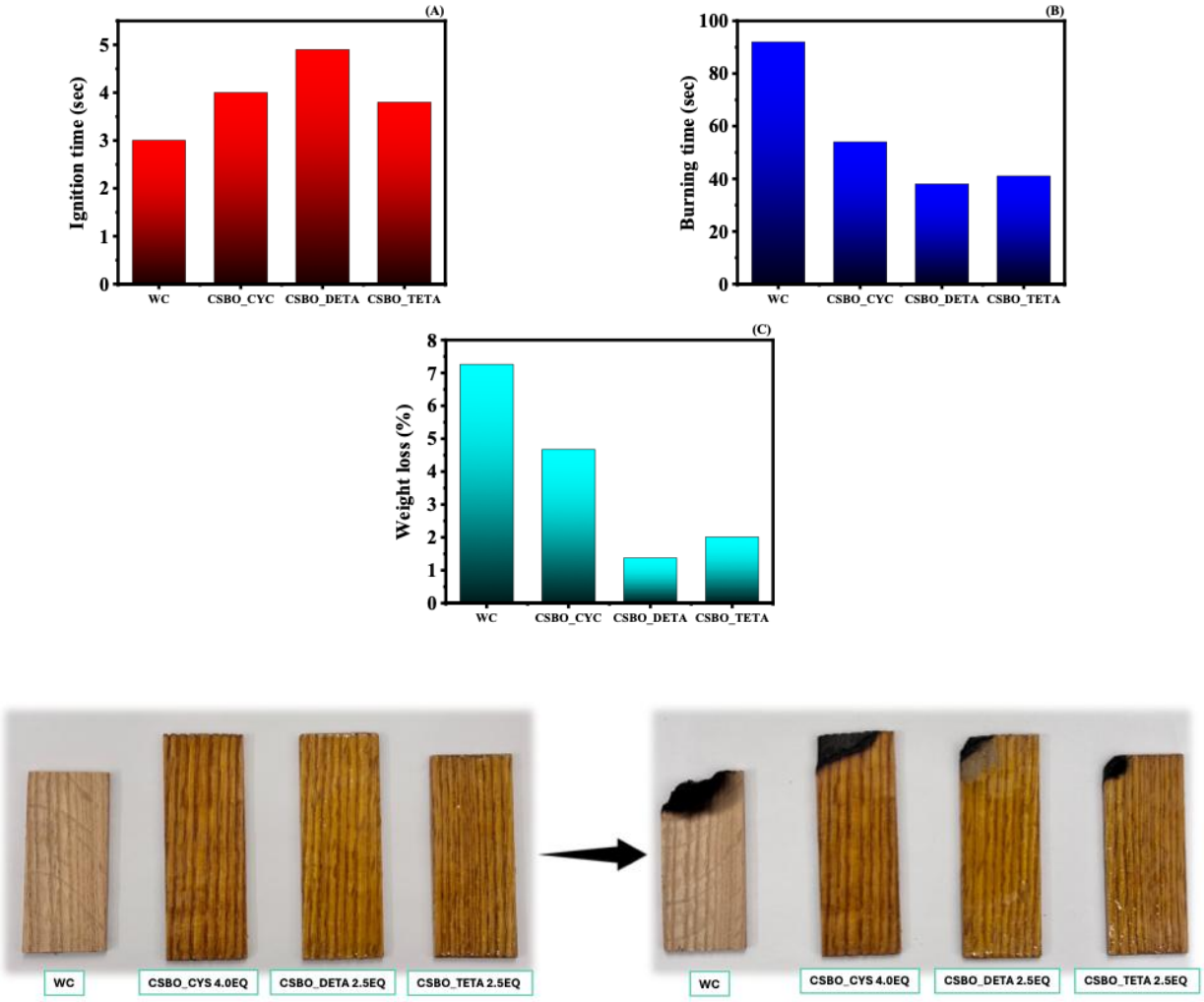

Figure 1. A comparative (A) ignition time, (B) burning time, and (C) %weight loss of uncoated wood with NIPU-coated wood specimens, and the image of the flammability test.

Gel content:

Furthermore, to support the cross-linked network of all the NIPU coating samples, the gel content was determined in two solvents, *e.g.*, water and anhydrous toluene. At first, the NIPU coating materials were immersed in the respective solvents and kept at room temperature for 24 h. After that, the solvents were removed and dried in an oven at 70°C under vacuum. As shown in Figure 2, all the synthesized NIPU coating materials exhibit >95% gel content in both toluene and water, which corroborates a high degree of cross-linking. As the conversion of cyclic carbonate into urethane using diamines results polar urethane linkage and hydroxyl groups, the synthesized NIPU materials

become hydrophilic and show relatively less gel content in polar solvent (water) compared to nonpolar solvent (toluene). The relative degree of hydrophilicity was also investigated in the water contact angle measurement of all the NIPU coating materials, where, depending upon the structural difference of the cross-linkers *e.g.* cystamine (CYS), diethylenetriamine (DETA), and triethylenetetramine (TETA) the hydrophilic/hydrophobic nature of the fabricated materials has been elucidated.

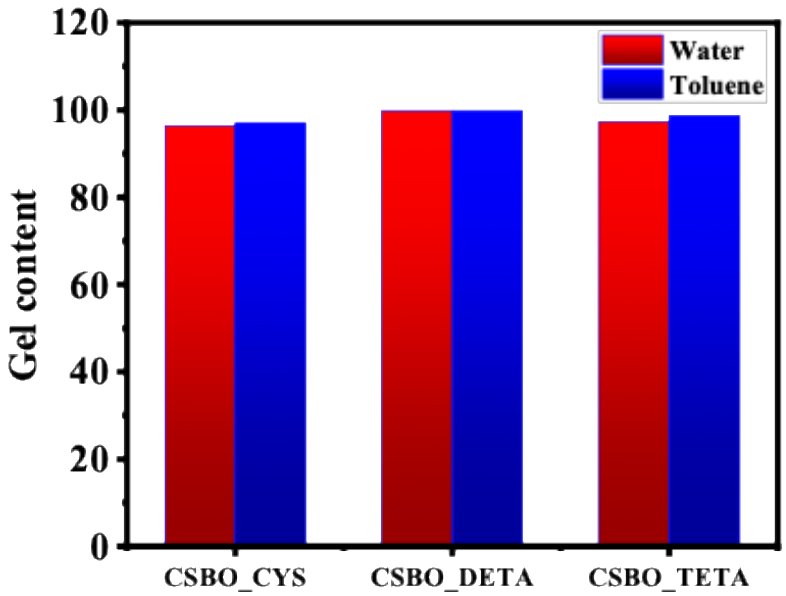

Figure 2. Gel content (GC%) of the three coating materials in water and toluene, respectively.

Chemical Resistance test:

The solvent resistance of all the NIPU coating materials has been investigated by partially coating each material in a stainless-steel metal coupon, followed by putting a droplet of saturated NaCl solution, 1(N) H_2SO_4, and 1(N) NaOH solution, both in the uncoated and coated part of the stainless-steel coupon. The solvent resistance of the coating was assessed after 1h and 24 h by wiping the surface with soft wet tissue, as shown in Figure 3. After 1 hour of exposure, no visual corrosion was observed for both uncoated and coated surfaces in NaCl and 1(N) NaOH solution, whereas a stain appeared in 1 (N) H_2SO_4 only for the uncoated surface of the stainless-steel coupon. However, after 24 h, all the NIPU-coated surfaces remained undamaged in saturated NaCl solutions, but in 1(N)

H_2SO_4 solutions, both the uncoated and coated surfaces showed corrosion. Moreover, 1(N) NaOH solution showed staining on the uncoated stainless-steel surface, but the CSBO_CYS and CSBO_TETA coated surfaces remained undamaged after 24 h, while for the CSBO_DETA coated surface, corrosion already started after 24 h of exposure.

Note: Currently, the chemical resistance test is ongoing with 10% aq. HCL solution instead of 1(N) H_2SO_4 solution.

Figure 3. Chemical resistance test of uncoated and NIPU-coated stainless-steel coupons in NaCl (sat. solution), 1N H_2SO_4, and 1N NaOH solution.

Ink Repellence Behavior:

To investigate the ink repellence behavior of all three NIPU coating materials, using a typical oil-based red marker, draw a line both on the uncoated wood surface and the NIPU-coated surfaces (Figure 4). After that, it was observed that the red line on the uncoated wood surfaces could not be

removed even after wiping with tissue and leaving a permanent mark on that, whereas the red line in all the coated surfaces became easily removed by wiping with tissue paper, leaving no permanent ink marks (Figure 4). The above ink repellent behavior of all the coated materials demonstrated that these NIPU-based coatings can be used for the prevention of permanent ink, which can inhibit the ink deposition on the surface of the coated substrates.

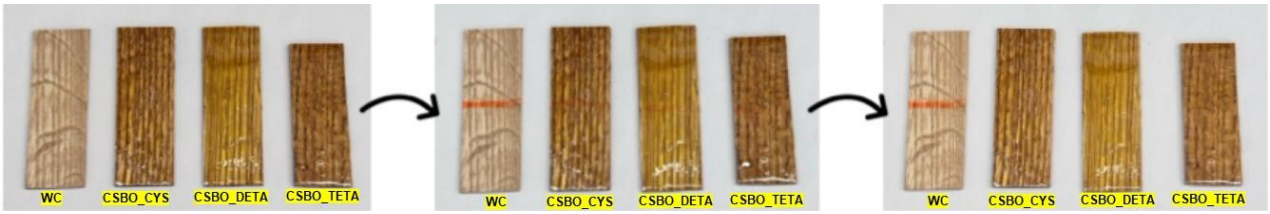

Figure 4. Ink repellence test of uncoated and NIPU-coated wood coupons.