**North Dakota Soybean Council**

***Project Title:* Optimization of Novel Soybased Resin for Commercial Acceptance**

PI’s: Dilpreet S Bajwa and Dean C Webster

Project Date: July 1, 2016 - December 31, 2017

**Technical Summary**

The long term goal of the project is to develop a cost effective soybased resin/adhesive system with desirable properties for wood composite products. The primary goal of this project was to reduce the curing time of novel Epoxidized Sucrose Soyate (ESS) technology so it can be easily accepted by wood composite industries. The research team from North Dakota State University has shown that ESS can be a safe and effective alternative to commercial formaldehyde based adhesives that are under severe scrutiny due to formaldehyde emission and chemicals known to cause allergies or skin and eye irritation. EPA has described formaldehyde as a cancer causing carcinogenic compound and California has restricted its use in wood composites.

The main project tasks for project were 1) Evaluate the curing kinetics of ESS resin using most effective cross linker and catalysts blends and optimize/reduce the curing time so it matches with industry standards. 2) Manufacture particleboards with ESS resin blends under lowest curing time and test their physical and mechanical properties. 3) Develop a technical bulletin and product specification sheet for sharing with wood composite industries.

In the task 1, to reduce resin curing time a set of two cross-linkers (MTHPA, MHHPA) and three catalysts (AC8, BV-CAT7 and BV-CAT7FC) were selected for use in ESS resin. The catalyst were supplied by Broadview Company, ND. A design of experiment was created with two curing times (5, 10 min), four processing temperature (130oC, 150oC, 175oC, and 190oC) which resulted in 48 unique formulations. Initial characterization was conducted through evaluating curing kinetics via differential scanning calorimetry (DSC) and bond strength using lap shear testing. After initial characterization and screening six formulations were selected for detailed analysis as shown in Table 1.

Table 1: Preliminary ESS Resin-Crosslinker-Catalyst Formula Characterization

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Primary Resin | Catalyst | Crosslinker | Mixture Ratio | Name | DSC (P/F) |
| ESS | BV-CAT7FC | MHHPA | 25:20:01 | Formula 1 | Pass |
| 50:40:01 | Formula 2 | Pass |
| MTHPA | 25:20:01 | Formula 3 | Pass |
| 50:40:01 | Formula 4 | Fail |
| BV-CAT7 | MHHPA | 25:20:01 | Formula 5 | Pass |
| 50:40:01 | Formula 6 | Pass |
| MTHPA | 25:20:01 | Formula 7 | Pass |
| 50:40:01 | Formula 8 | Fail |
| AC-8 | MHHPA | 10:08:01 | Formula 9 | Fail |
| MTHPA | 10:08:01 | Formula 10 | Fail |

The analysis of shear stress values and curing kinetics further helped us to narrow down to one most promising formulation (Formula 2) which constituted ESS resin, MHHPA as crosslinker and BV-CAT7FC as catalyst as shown in figure 2. The curing time was 5 and 10 min and processing temperature of 175°C and 190°C. In task 2 particleboards were manufactured by blending formula 2 with commercial resin MDI at two different levels (50% and 75%).

Figure 2. Mean Shear Stress for ESS based Formulas and blends with MDI resin.

The particleboards were manufactured using resin, wax, and pine wood flour. The boards are made by spraying the wood flour with resin and wax while being agitated in a rotary mixer to evenly disperse the resin and wax throughout all the wood fibers. The freshly coated fibers are then placed into a mold that has been preheated to the curing temperature and then immediately placed in the press. Once in the press a pressure of 10 metric tons was applied within a minute and held there for the desired curing time, with the time starting once the 10 ton pressure is reached. After pressing the boards were removed from the mold and left to cool and equilibrate in standard lab conditions for at least 24 hours. The particleboards were tested for water absorption, flexural strength, and resin bond strength properties. Water absorption was evaluated by immersing low density boards in water bath for 2 hrs and calculating percentage water absorption. The strength properties (modulus of elasticity and modulus of rupture) were evaluated following ASTM D1037 method using universal testing machine Instron. Bond strength was measured by evaluating its resistance to tensile failure by following the ASTM D1037 procedure.

The result of physical and mechanical properties are shown in table 2. In terms of density there isn’t too much to say outside of the fact that all the specimen’s fall within the definition of medium density particleboard. This continues with the water absorption test for the first 2-hour mark. There are clear differences between the different formulas but no real trend to draw any conclusions off. However, when looking at water absorption at the 24-hour mark there are clear trends worthy of note. Starting with the most obvious and that being the % change in volume over the 24-hour period.

This study helped to identify two or more promising cross-linking chemistries which can reduce the cure time of ESS based particleboards without compromising the physico-mechanical properties. The use of anhydride crosslinkers MHHPA and MTHPA and fast acting catalyst in BV-CAT7 and BV-CAT7FC can reduce the curing time of particleboards from 30 min to 5 or 10 min when pressed under 175 or 190°C. Further ESS resin can be easily blended with commercial adhesives up to 50% without a significant loss in physico-mechanical properties of particleboards. Further ESS resin can be easily blended with commercial adhesives up to 50% without a significant loss in strength and durability properties. The biobased, and sustainable nature of ESS resin can be a safe alternative to formaldehyde adhesives and the low curing temperature of this ESS resin helps manufacturers to conserve energy.

Table 2: Physical and Mechanical Properties of ESS and MDI based Particleboards

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| Formula | Cure Time | Cure Temperature | Density (kg/m^3) | 2h % Volume Change | 2h % Mass Change | 24h % Volume Change | 24h %Mass Change |
| 100% MDI | 5 min | 175 °C | 556.37 | 10.97% | 11.09% | 21.77% | 24.77% |
| 190 °C | 548.70 | 18.06% | 29.63% | 25.91% | 42.33% |
| 10 min | 175 °C | 563.35 | 20.64% | 22.10% | 32.84% | 39.92% |
|   | 190 °C | 566.72 | 24.91% | 41.23% | 34.00% | 55.80% |
| 25% Formula 2 75% MDI | 5 min | 175 °C | 552.87 | 17.42% | 13.47% | 38.68% | 48.96% |
| 190 °C | 557.00 | 26.53% | 44.59% | 34.78% | 61.10% |
| 10 min | 175 °C | 561.26 | 21.65% | 24.42% | 36.16% | 40.46% |
| 190 °C | 564.00 | 26.81% | 45.32% | 37.29% | 60.81% |
| 50% Formula 2 50%MDI | 5 min | 175 °C | 552.84 | 10.26% | 7.11% | 40.38% | 53.62% |
| 190 °C | 550.24 | 40.01% | 68.90% | 47.85% | 91.20% |
| 10 min | 175 °C | 557.14 | 22.43% | 22.63% | 43.89% | 45.09% |
| 190 °C | 569.92 | 28.25% | 40.67% | 44.17% | 57.90% |

The research team is grateful to the soybean farmers and ND Soybean Council for providing funding for this research project. The outcomes/results of this research are expected to diversify soy based products with increased demand for soybean oil the principal component of the epoxidized sucrose soyate resin.