# Canola-based Epoxy Resins for Bio-based Plastic Composites

Progress Report for 2009 National Canola Research Program

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## OVERVIEW AND PROJECT IMPACT:

The development of canola oil-based resins for commercial application to composite materials is the long-term objective of this project. Epoxies are already a well-established type of resin used in composites, and thus the current focus is on blends of canola oil-based epoxy and synthetic epoxy resins. The supporting objectives are stated below. By achieving these objectives, we are demonstrating that canola oil-based resins are suitable for high-value applications, thereby helping to create a new market for canola, fostering new business opportunities in the North Central U.S., and lessening our nation's dependence on imported petroleum.

## OBJECTIVES:

**Objective 1.** Scale up the conversion of canola oil to high-quality epoxy resin.

**Objective 2.** Produce competitive composite materials which are high in canola resin content.

**Objective 3.** Identify suitable applications and partners for the transfer of this technology.

## COOPERATING INVESTIGATORS:

Judith Espinoza-Perez, Grad. Res. Assist., Agricultural & Biosystems Engineering
Cole Gustafson, Co-PI, Agribusiness & Applied Economics
Darrin Haagenson, Research Specialist, Agricultural & Biosystems Engineering
Scott Pryor, Assistant Professor, Agricultural & Biosystems Engineering
Chad Ulven, Co-PI, Mechanical Engineering
Dennis Wiesenborn (PI), Agricultural & Biosystems Engineering
Undergraduate ABEN students: Mukesh Kumar, Ross Peterson, Tafseer Ansari, Aakanksha Rastogi, Vishal Verma

## SUMMARY:

Funding for this project commenced Fall 2006. Production of the canola oil-based epoxy resin (epoxidized canola oil, ECO) has progressed very well. For example, process conditions have been identified that achieve >98% conversion with 90% yield ECO, by adapting processes reported for other epoxy resins to ECO. Our process uses a heterogeneous catalyst that is readily recovered and reused, unlike the liquid acid catalysts traditionally used. We also showed that t-butyl alcohol can replace the toluene solvent, thereby eliminating a significant workplace and environmental hazard. An additional alternative was found, when using the heterogeneous catalyst, which is that the solvent can actually be omitted without loss of product quality; yield decreased 10%, but this loss was largely eliminated with catalyst reuse. Our ECO process has thus far been scaled up to 300 g batches without loss of product quality. An upscaled reactor

developed by senior-level undergraduates in the ABEN program produced a 1-kg batch of ECO with >80% conversion. This scaled-up reactor, when perfected, will enable us to better demonstrate the practical applications of canola epoxy resin.

Our ECO was blended with a synthetic epoxy system at 30, 35 and 40% of the total resin weight, combined with E-glass and cured (Figure 1). Although the resulting composite specimens had lower flexure strength and glass transition temperature than the zero-ECO control, the flexure modulus and toughness were similar to the control (Table 1). Thus, composites prepared using ECO-blends should perform well in applications requiring flexibility and toughness. Alternative curing agents will be explored to enhance flexure strength and glass transition temperature.

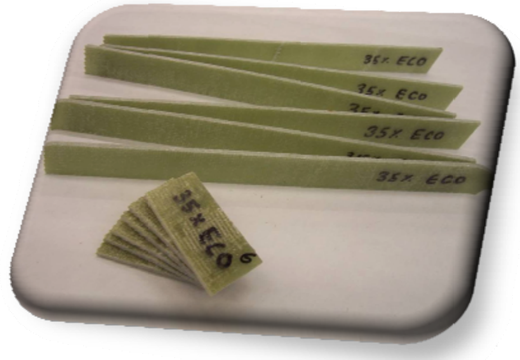

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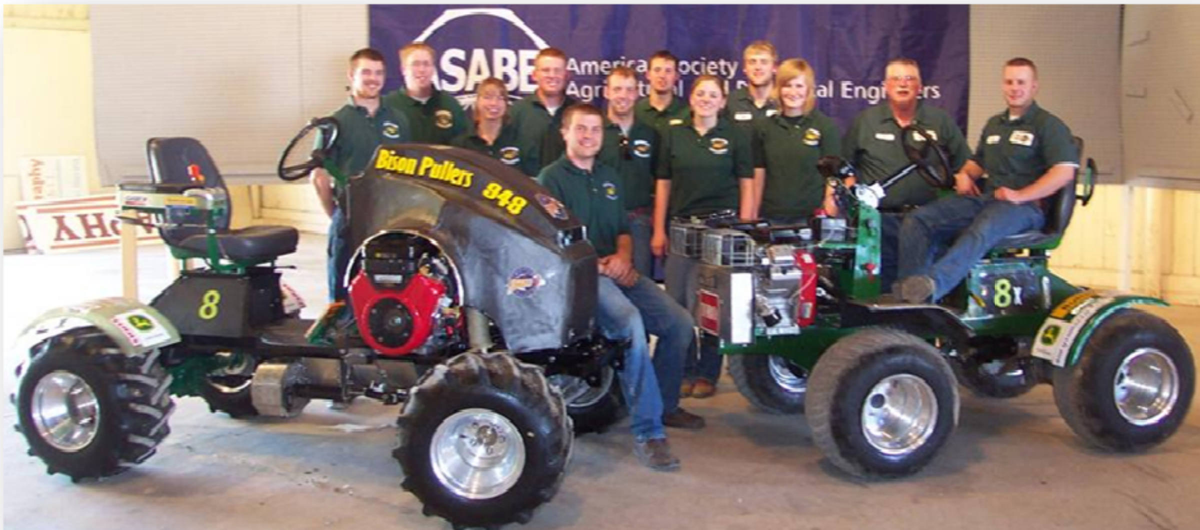
**Table 1. Properties of composite samples prepared using 0, 30, 35 and 40% canola oil-based epoxy resin (ECO) blended with Resinfusion™ 8603 cured with ECA 100KA.**

| Samples | Flexure Properties | | Toughness (J) | Glass transition |
|---|---|---|---|---|
| | Strength (MPa) | Modulus (GPa) | | $T_g$ (°C) |
| CONTROL | 372 ± 2 | 16.9 ± 0.6 | 2.7 ± 0.2 | 105.8 ± 0.2 |
| ECO 30 | 266 ± 9 | 14.6 ± 1.1 | 2.3 ± 0.1 | 75.9 ± 0.5 |
| ECO 35 | 181 ± 6 | 12.5 ± 0.6 | 2.8 ± 0.2 | 64.5 ± 0.6 |
| ECO 40 | 120 ± 4 | 9.6 ± 0.7 | 2.8 ± 0.4 | 69.2 ± 0.3 |

Composites such as those shown in Figure 1 may be used to create strong, light-weight exterior shields for machinery. A demonstration of the practical application of composites containing ECO was undertaken in 2008-09 by a group of senior-level students in the capstone design course for Agricultural & Biosystems Engineering at NDSU. A number of students in this department voluntarily participate in the national ASABE quarter-scale tractor competition, and the NDSU team placed 8th in the nation in the 2008 event (Figure 2). The students recognized that, because of competition weight restrictions, the low weight of ECO-composite shields relative to steel shields will give the team a competitive advantage. An entirely new tractor must be created for each competition, and the hood for the 2009 competition (Figure 2) was designed with the intention of incorporating canola epoxy resin.

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Practical demonstration of the use of ECO will be invaluable as we explore the transfer of ECO to industrial applications, much as demonstrations of biodiesel in bus fleets and public vehicles aided public recognition and acceptance of that new, bio-based product.

ECO may be further modified for a wide variety of other applications.  For example, ECO can be converted to a polyol for conversion to a canola-based polyurethane, or it may be converted to acrylics.  We produced test samples of acrylated canola oil which was cured using UV light by Dr. Chen in the Center for Nanoscale Science & Engineering of NDSU.  UV-curing will be attractive for some industrial applications, and we will pursue funding to further this line of research.

Engineering data obtained under objectives 1 and 2 are the foundation of cost estimates now underway, as these relationships will delineate conversion rates and quantities of inputs required. An industry analysis by Standard Industry Classification (SIC) code will reveal commercial demand within the Northern Plains region as well as the rest of the country.  A competitive analysis will compare net delivery costs of new composites developed with existing products, including transportation, by supplier.  The above analyses will be used to identify potential industry partners and specific applications, and ultimately to measure performance and predict product costs in the most promising applications.

**CONCLUSION:**

Techniques for producing the resin and incorporating the resin into composites are now advanced, and much data has been collected. Composites containing up to 40% canola-based resin in the matrix had very good toughness and flexibility, but lower strength compared to synthetic resins. Prototype shielding for small tractors incorporated up to 10% canola resin and had excellent mechanical properties. The process for making the resin must be scaled up in order to make possible large-scale applications with high canola resin content. Key areas for future work are the investigation of alternative curing agents and improved adhesion between the matrix and fibers.

**Peer-reviewed Journal Article:**

Espinoza-Perez, J.D., D.P. Wiesenborn, D. Haagenson, C.A. Ulven, S. Pryor. 2009. Production and characterization of epoxidized canola oil. *Trans ASABE* 52(4):1289-1297.

**Article in Review:**

Espinoza-Perez, J.D., C.A. Ulven, D.P. Wiesenborn. Epoxidized high-oleic vegetable oils applied to composites. Submitted to *Trans ASABE*.

**Presentations at Scientific Society Meetings:**

Ansari, M., D. Wiesenborn, D. Haagenson, J. Espinoza-Perez. 2009. E.C.O Shield (Senior design project). North Central ASABE/CSBE Conference, Brookings, SD, Sept. 17-19.

Espinoza-Perez, J.D., D. Haagenson, R. Brudvik, C.A. Ulven. D.P. Wiesenborn. 2009. Epoxy resin from high-oleic oils applied to composites. ASABE Annual International Meeting, Reno, NV, Jun 21-24. (published abstract)

**Outreach/Extension Audiences**

This research is showcased during tours of the NDSU Pilot Plant, which in 2008-09 included tours for Undersecretary of Agriculture Gail Buchanan, the NATURE program, ND Department of Commerce, and NDSU Harvest Bowl honorees. Other visitors to the Pilot Plant in recent years included Governor Hoeven, Senator Conrad, North Dakota Empower, and various government staff and news media personnel who accompanied these visits. This research was also presented via posters or talks in 2008-09 to ND Research & Technology Conference, Fargodome; BioOpportunities Conference, Ramada Plaza Suites, Fargo; and Northern Canola Growers Association, NDSU Alumni Center. This research was also cited in the November 2008 issue of Biomass Magazine (J. Kram, “Plastics from the Prairies”).

The level of outreach activity is anticipated to increase for 2009-2010, as the tangible outcomes have increased in number and significance.