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FABRICATION OF SOY OIL EPOXY RESIN BASED COMPOSITES

by

VINAY PRABHAKAR PAI

A THESIS

Presented to the Faculty of the Graduate School of the

MISSOURI UNIVERSITY OF SCIENCE AND TECHNOLOGY

In Partial Fulfillment of the Requirements for the Degree

MASTER OF SCIENCE IN MECHANICAL ENGINEERING

2008

Approved by

Dr. Virgil Flanigan, Advisor Dr. Shubhen Kapila Dr. K. Chandrashekhara

ABSTRACT

A renewable resource derived epoxidized ester - Epoxidized Allyl Soyate (EAS) yields a versatile epoxy resin system that can be used in pure form combination with bis - phenol epoxy systems to obtain high strength composite materials. EAS containing composites materials exhibit higher toughness and impact resistance than the traditional bis-phenol epoxy composites. These resin systems were found to be compatible with different fibers and screens such as glass fibers, glass mats, carbon fibers and metallic wire screens including those obtained from an automobile tire recycling process. The materials were tested for mechanical properties including tensile and flexure strengths. The highest tensile strength was observed with stainless steel screen incorporated composites, the ultimate tensile strength of such material was found to be ~ 39 MPa. Tensile strengths of material obtained by incorporating glass fiber, aluminum screen, stainless steel screen and steel wire mesh comprised of steel wires recovered from scrap tires were evaluated. Tensile strengths of materials with stainless screen and screen made from scrap tire steel were an order of magnitude higher than the materials with glass fiber or aluminum screens.

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TABLE OF CONTENTS

	Page
ABSTRACT	iii
ACKNOWLEDGMENTS	iv
LIST OF ILLUSTRATIONS	vi
LIST OF TABLES	vii
SECTION	
1. INTRODUCTION	1
2. EXPERIMENTAL	5
2.1. MATERIALS	5
2.2. TRANSESTERIFICATION OF EPOXIDISED SOYBEAN OIL (ESC)5
2.3. PREPARATION OF EPOXIDISED ALLYL SOYATE (EAS)	7
2.4. FREE RADICAL POLYMERIZATION OF EAS	
2.5. RECOVERY OF BRAIDED STEEL WIRES FROM SCRAP TIRES	9
2.6. PLASTIC AND COMPOSITE SAMPLE PREPARATION	10
2.7. TENSILE STRENGTH TEST	11
2.8. FLEXURAL STRENGTH TEST	
2.9. IMPACT DROP TEST	13
3. RESULTS AND DISCUSSION	14
3.1. TENSILE STRENGTH	14
3.2. FLEXURAL STRENGTH	
3.3. IMPACT DROP TEST	25
4. CONCLUSIONS	27
5. RECOMMENDATIONS	
BIBLIOGRAPHY	30
VITA	

LIST OF ILLUSTRATIONS

Figure	Page
2.1. Transesterification of ESO	6
2.2. Structure of Palmitic Acid (C16:0)	6
2.3. Structure of Stearic Acid (C18:0)	6
2.4. Structure of Oleic Acid (C18:1)	7
2.5. Structure of Linoleic Acid (C18:2)	7
2.6. Structure of Linolenic Acid (C18:3)	7
2.7. Free Radicals from Benzoyl Peroxide	9
2.8. Block Diagram of Low Temperature Pyrolizer	10
2.9. Tensile Testing on Instron 4489	11
2.10. Flexural Testing on Instron 4469	
2.11. Drop Test Apparatus with Plumb Bob	13
3.1. Molecular structure of LS-682K, Lindride 56Vand BTDA	14
3.2. Tensile Test Curve - Lindride 56V vs. LS682K vs. BTDA	15
3.3. Tensile Test Curve-EAS Composites	16
3.4. Tensile Test Curve - SF and SPI	
3.5. Tensile Test Curve – EPON + EAS	19
3.6. Tensile Test Curve – EPON: EAS Composite	
3.7. Tensile Test Curve - 70:30 EPON: EAS + Soy Flour	
4.1. Tensile and Flexural Strength Comparison	

LIST OF TABLES

Table	Page
3.1. Tensile Strength Data of the 3 Hardening Agents	15
3.2. Tensile Strength Data-EAS Composites	17
3.3. Tensile Strength Data - SF and SPI	
3.4. Tensile Strength Data – EPON + EAS	
3.5. Tensile Strength Data - EPON: EAS Composite	21
3.6. Tensile Test Data - 70:30 EPON: EAS + Soy Flour	
3.7. Flexural Strength Data for the Hardening Agents	
3.8. Flexural Strength Data for the EAS Composites	
3.9. Flexural Strength Data – EPON + EAS	
3.10. Flexural Strength Data – EPON: EAS Composite	
3.11. Flexural Strength Data – 70:30 EPON, EAS + Soy Flour	
3.12. Impact Drop Test Results	

1. INTRODUCTION

Transesterification of soybean oil with allyl alcohol and subsequent epoxidation of the allyl ester or transesterification of epoxidized soybean oil with allyl achohol yield a versatile epoxy resin that can be used for fabrication of composite material with good mechanical properties (1-4). The resin is compatible with bis-phenol based epoxy resins and the mixed resins have been shown to yield materials with superior toughness. Epoxidized allyl esters of soybean oil represent a new class of viable and sustainable resource based resins for the fabrication of plastics. In addition, such resins offer added advantage of yielding materials that are more readily biodegradable and environmentally benign. However, in order to successfully compete with plastics derived from the traditional resource (petroleum), properties of renewable resource based plastics must be comparable to those of the traditional plastics. In addition, materials should be amenable to commonly used processing techniques such as compression molding, extrusion molding and injection molding (5, 6). Finally, the materials must be cost competitive.

Soybeans are a good source of oil, carbohydrates and proteins. As a result, this crop has attracted considerable attention as a source for industrial materials including plastics. Soybean oil has received the most attention as plasticizers or a source of polymer resin (3). Soy protein isolate (SPI) has also been used for fabrication of biocompatible plastics (7). Properties of such plastics are dependent on the characteristics of soy proteins such as the tertiary structure of the soyproteins; however, such structures can be modified through physical, chemical and enzymatic methods. In attempts to develop renewable resource based plastics, SPI has been blended with glycerol, a by-product of biodiesel production. However, these blends generally yield materials with poor mechanical properties and stability (8). To enhance the mechanical properties and reduce the hydrolytic susceptibility of SPI plastics, SPI has been blended with other biodegradable polymers (9, 10). These materials have also been treated with denaturing agents, such as urea, to increase glass transition temperature (11). Various plasticizers and cross linking agents such as ethylene glycol, stearic acid and propylene glycol have also been added to improve mechanical properties of soy protein isolate based plastics (12, 13). Effect of processing parameters such as temperature and pressure, on the properties of the soy protein plastics has been examined (14). However, despite the optimization of various parameters, SPI plastics-especially those formulated with glycerol–generally possess poor mechanical and thermal properties. The problem is in part related to lack of interaction between soy protein molecules and glycerol, which acts only as a plasticizer.

To overcome this problem, only soy oil derived epoxy resins and soy proteins were used for fabrication of plastics. In these materials the two components EAS and SPI interact chemically e.g. amine functionalities of basic amino acids and hydroxyl of serine in SPI react with oxirane functionality of the resin to yield cross linked polymers. Cross linking is enhanced with anhydride or amine cross linking – curing agents. As a result dense cross-linked networks with good strength and modulus have been obtained (15, 16). Plastics with greater strength can also be obtained by adding EAS to a commercial epoxy resin; providing viable low-cost, high-performance thermoset plastics (17, 18). The current study was undertaken to assess feasibility of obtaining plastic materials through interactions between EAS in combination with different fibers and screens such as glass fibers, glass mats, carbon fibers, metallic wire screens and metal screens obtained from recycled automobile tires.

It has been estimated that several hundred million scrap tires are stockpiled in the U.S. In addition, several million scrap tires are generated annually (19). Tires in landfills are a significant nuisance and source of perpetual problems (20). Reutilization of tire chemicals has been a subject of research for many years. The major impediment to design of a more environmentally acceptable disposal and reclamation process is the high cost. Pyrolytic processes hold considerable potential for reclamation of tire materials. Scrap tires have been used as an energy source in power plants, cement kilns, athletic and recreational applications and railroad ties, or recycled in highway barriers. Pyrolysis is thermal degradation of organic molecules in an oxygen-free environment. The application of pyrolysis as a recycling process was reported as early as the 1920s. Pyrolysis studies on scrap tires have shown to produce light aromatics such as benzene, toluene, xylenes and styrene (21). Investigations of a low-cost self fueled, self-inerting pyrolysis process on a bench-scale and at a pilot scale for recovery of value added materials have been done. This has shown that the pyrolysis temperature is the most influential parameter which determined the type and concentration of organic products, e.g. at temperatures between 300 - 450oC. Braided steel wires make up approximately 10% of a tire by weight. Essentially, all of this material was recovered through the low temperature process. The recovered braided steel accounted for approximately 10% of the total product weight. The steel was utilized in fabrication of composite material with soybean oil derived epoxidized allyl soyate (EAS) resin developed in our laboratories. This resin forms dense cross-linked polymers which possess higher glass transition, strength and modulus. EAS holds high potential for the fabrication of renewable resource based composites (22).

The current study was undertaken to assess the feasibility of obtaining plastic materials through interactions between EAS in combination with different fibers and screens such as glass fibers, metallic wire screens and screens obtained from recycled automobile tires.

2. EXPERIMENTAL

2.1 MATERIALS

The Epoxidized Soybean Oil (ESO) was obtained from Crompton Corporation, Middlebury, CT. The food grade soybean oil was obtained from the local supermarket. Allyl alcohol was obtained from Fisher Scientific, Saint Louis, MO. The hardeners Lindride 56V and LS-682K were obtained from Lindau Chemicals, Columbia, SC. 3, 3' 4, 4' Benzophenone Tetra Carboxylic Dianhydride (BTDA) was obtained from Sigma Aldrich, Saint Louis, MO. Soy Protein Isolate (SPI) (PRO-FAM 974) was obtained from Archer Daniel Midland, Decatur, IL. Soy Flour (SF) was obtained from local Missouri Farmer Association (MFA) store. EPON resin SU2.5 was obtained from Hexion Specialty Chemicals, Pueblo, CO. The stainless steel and aluminum screens were obtained from a local hardware store. The braided steel wires from scrap tires were obtained by subjecting the scrap tire to pyrolysis. The 0°-90° glass fiber mat was obtained from US Composite, West Palm Beach, FL.

2.2 TRANSESTERIFICATION OF EPOXIDISED SOYBEAN OIL (ESO)

ESO reacts with allyl alcohol in the presence of sodium producing a mixture of Fatty acid alkyl allyl esters and glycerol. In a 3 step reversible reaction di and monoglycerides are formed. The exothermic reaction is as shown in Figure 2.1.

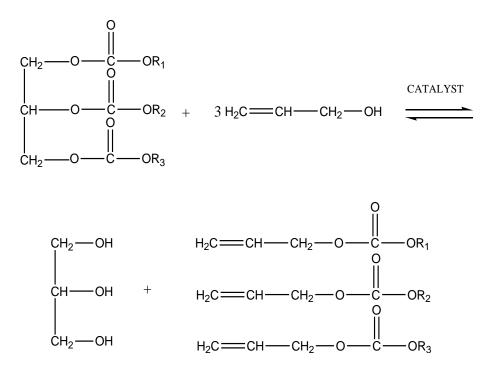


Figure 2.1: Transesterification of ESO

The fatty acid composition of soybean oil is Palmitic Acid (10%), Stearic Acid (5%) Oleic Acid (26%), Linoleic Acid (52%) and Linolenic Acid (7%). The chemical structure of these fatty acids is as shown in Figures 2.2 to 2.6

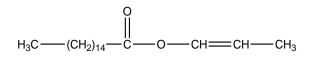


Figure 2.2: Structure of Palmitic Acid (C16:0)

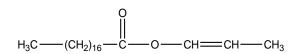
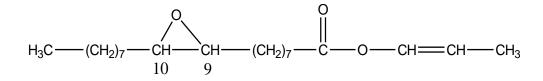
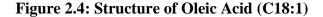


Figure 2.3: Structure of Stearic Acid (C18:0)





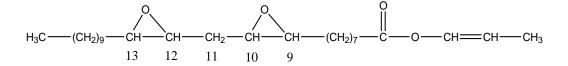


Figure 2.5: Structure of Linoleic Acid (C18:2)

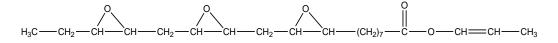


Figure 2.6: Structure of Linolenic Acid (C18:3)

2.3. PREPARATION OF EPOXIDISED ALLYL SOYATE (EAS)

1000gm of Epoxidized Soybean Oil (ESO) was weighed and transferred to a two neck round bottom flask with a 5 liter capacity. Allyl alcohol was weighed and placed in the round bottom flask into which 3.5gm sodium metal was weighed and added. The sodium taken was 1% by weight of Allyl alcohol. Epoxidized Soybean Oil and Allyl Alcohol are taken in the ratio 1:0.35, respectively. The flask was placed in an electrical heating mantel and attached to a water cooled condenser in a well functioning fume hood. A thermometer was inserted into the flask through the second opening. The contents of the flask were heated to 95°C and allowed to reflux at this temperature for four hours. After four hours heat was turned off and the reaction mixture was allowed to cool down to the room temperature. The flask was then detached from the condenser and its contents transferred to a single neck flask. The single neck flask was connected to a rotary evaporator and unreacted allyl alcohol was removed from the reaction product under vacuum. Once all of the unreacted allyl alcohol had been removed from the ester, the contents of the flask were transferred to centrifuge tubes and centrifuged at 4,500 rpm for 40 minutes. The EAS resin layer which forms the top layer was separated from the glycerol which forms the bottom layer.

2.4 FREE RADICAL POLYMERIZATION OF EAS

Out of all the fatty acids present in soybean oil, Palmitic acid and Steraic acid require polymerization. Using EAS without polymerization will result in low quality, low performance resins. Hence, EAS is polymerized by adding Benzoyl Peroxide. Benzoyl Peroxide is added 1% by weight of EAS. This mixture is stirred using magnetic stirrer and heated to 120°C for 2 days. Benzoyl Peroxide decomposes to form free radicals which initiate the long chain polymerization. The free radicals thus, produced contain a free electron which requires another free electron for stability. This required free electron is obtained from the carbon double bond in allyl alcohol. The reaction is shown in Figure 2.7.

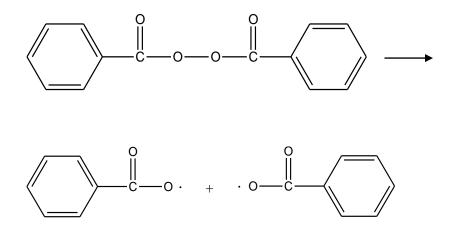


Figure 2.7: Free radicals from Benzoyl Peroxide

2.5 RECOVERY OF BRAIDED STEEL WIRE FROM SCRAP TIRES

Steel belts (braided wires) were recovered from scrap tires through pyrolysis in the low temperature thermal unit. The thermal treatment unit was designed to treat whole scrap tires without pre-treatment and generate recyclable products. The major recycled products of the process are char (carbon black substitute), steel, oil and low BTU gas composition. The process is based on an oxygen-free batch pyrolyzing system. This process was carried out at low temperatures ranging between 450 – 550°C. The overall system consists of three subassemblies: a scrap tire introduction device, a pyrolyzer unit, and a product separator. The process can be configured into a pseudo-continuous system with multiple processors (23, 24). Figure 2.8 shows the block diagram of the low temperature pyrolizer.

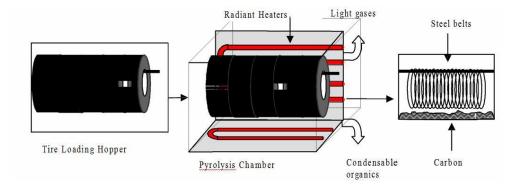


Figure 2.8: Block Diagram of Low Temperature Pyrolizer

2.6 PLASTIC AND COMPOSITE SAMPLE PREPARATION

A 5'X7' aluminum mould was fabricated at the CEST machine shop to prepare the samples. The mould was designed to prepare both neat resin and composite samples with a simple change in top strip. A provision was provided in the mould to hold the steel wire, screen and glass fiber. The mould was coated with 4 layers of Chem Trend Chemlease 41-90 Semi permanent release agent. This was later placed in a Precision Economy Laboratory oven set at 100°C for 1 hour. Various combinations of soy epoxy resins with EPON, BTDA, SPI or SF and liquid hardening agents were mixed using a pneumatic mixer (Model 1302, Grovhac Inc, Milwaukee, WS) for 45 minutes. This mixture was later poured into the pre heated mould and was allowed to cure for 1 hour at 80°C and later at 120°C for the next 6 hours. This curing time decreased to 2 hours whenever EPON resin was used.

2.7 TENSILE STRENGTH TEST

Uniaxial tensile testing was done on an Instron Universal Testing machine (Model.No.4484) at the Interdisciplinary Engineering's Testing Laboratory on campus. Instron Blue hill software was used for data acquisition. The testing for neat resins was done as per ASTM D638-92: Standard Test Method for Tensile Properties of Plastics. The testing for the composite samples was done as per ASTM D3039: Standard Test Method for Tensile Properties of Polymer Matrix Composite Materials. The results reported are averages of five samples. Each sample was tested at 0.01in/min crosshead speed. The specimen ends were clamped into the hydraulic jaws of the testing machine as shown in Figure 2.9.



Figure 2.9: Tensile Testing on Instron 4489

2.8 FLEXURAL STRENGTH TEST

Flexural testing was done on a Instron Universal Testing machine (Model.No.4469) at the Interdisciplinary Engineering's Testing Laboratory on campus. Instron Blue hill software was used for data acquisition. The testing was done as per ASTM D790: Standard Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials. The results reported are average of five samples. Each sample was tested at 0.01in/min crosshead speed. The testing was done using a 3 point flexure jig as shown in Figure 2.10.

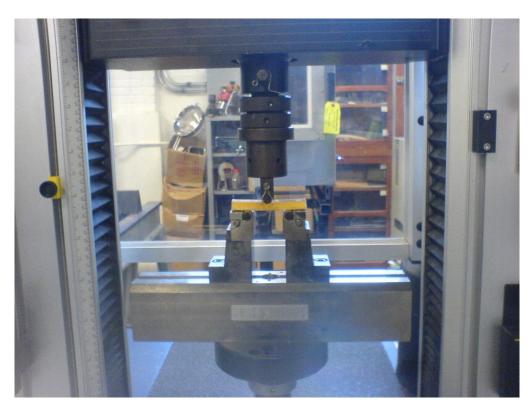


Figure 2.10: Flexural Testing on Instron 4469

2.9 IMPACT DROP TEST

A drop test apparatus was set up at the machine shop at CEST. The apparatus consisted of a camera tripod from which the plum bob was suspended using a pin. The plum bob weighs 439.4gm and its tip is machined at an angle of 45°. The height of fall is varied from 8 to 15 inches for different samples. When the pin is pulled the plum bob free falls from the measured height onto the sample which is supported on a circular end support. The sample is rigidly held between a aluminum end frame as shown in Figure 2.11.

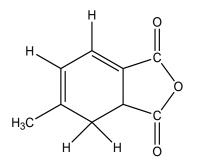


Figure 2.11: Drop Test Apparatus with Plumb Bob

3. RESULTS AND DISCUSSIONS

3.1 TENSILE STRENGTH

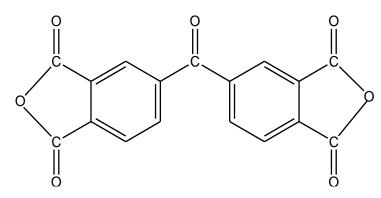
The EAS resin was tested with 3 different hardener combinations. Lindride 56 V, a 3-Methyl Hexohydro Phthalic Anhydride, LS-682K, a 3-Methyl Tetrahydro Phthalic Anhydride and 3, 3' 4, 4' Benzophenone Tetra Carboxylic Dianhydride (BTDA), a solid di-anhydride are the three hardening agents used. Figure 3.1 shows the molecular structure of the three hardening agents.



H H C C H H C C C H H H O O

3-Methyl tetrahydro phthalic anhydride

3-Methyl hexahydro phthalic anhydride



3, 3' 4, 4' Benzophenone Tetra Carboxylic Dianhydride

Figure 3.1: Molecular Structure of LS-682K, Lindride 56V and BTDA

Figure 3.2 shows the tensile strength curves for the plastic samples made using the three hardening agents.

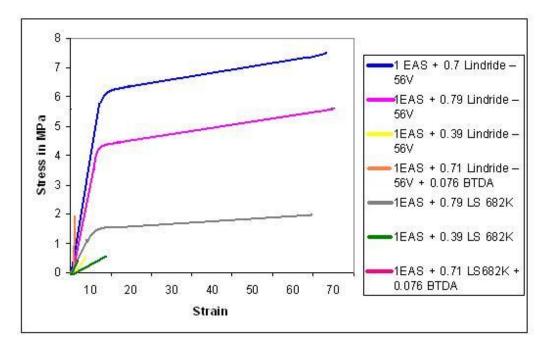


Figure 3.2: Tensile Test Curve-Lindride 56V vs. LS682K vs. BTDA

Table 3.1 shows the tensile strength data obtained from the plastic samples made by using the three hardening agents.

Composition	Tensile Strength (MPa)
1 EAS + 0.7 Lindride - 56V	7.3
1EAS + 0.79 Lindride - 56V	5.75
1EAS + 0.39 Lindride - 56V	0.35
1EAS + 0.71 Lindride - 56V + 0.076 BTDA	1.8
1EAS + 0.79 LS 682K	2.83
1EAS + 0.39 LS 682K	0.095
1EAS + 0.71 LS682K + 0.076 BTDA	0.61

 Table 3.1: Tensile Strength Data of the 3 Hardening Agents

It was observed that the samples with combination of EAS and Lindride 56V produced the best results. This was followed by combinations of EAS with LS-682K and BTDA, respectively. Lindride 56 V has a saturated structure and when mixed with the liner EAS resin, it produced a more homogenous mixture when compared with the others. When BTDA was used, it produced an inhomogeneous mixture thereby making the sample very brittle. The results obtained with EAS and LS-682K combination where higher than those obtained with BTDA, but lower than those obtained from Lindride 56V. It was also observed that EAS and Lindride 56V combination worked out best when they were taken in ratio 1:0.7 by weight, respectively. Hence, it was decided to pursue further research with this combination. Figure 3.3 shows the tensile test curves obtained from the composite samples made using 1 part of EAS and 0.7 parts of Lindride 56V

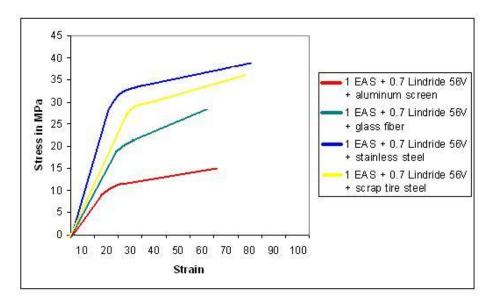


Figure 3.3: Tensile Test Curve-EAS Composites

EAS and Lindride 56 V in the ratio 1:0.7 by weight were used as the resin. Different composite materials were prepared using $0^{\circ}-90^{\circ}$ glass fiber, aluminum screen, stainless steel screen and recovered steel wires from tires. Table 3.2 shows the tensile test data obtained from the composite samples made using 1 part of EAS and 0.7 parts of Lindride 56V

Composition	Tensile Strength (MPa)
1 EAS + 0.7 Lindride 56V + aluminum screen	15.0
1 EAS + 0.7 Lindride 56V + glass fiber	27.4
1 EAS + 0.7 Lindride 56V + stainless steel	39.7
1 EAS + 0.7 Lindride 56V + scrap tire steel	35.8

Table 3.2: Tensile Strength Data-EAS Composites

It was observed that the tensile strength increased from a modest 7.3MPa for a neat resin to as high as 39.7MPa when stainless steel screen was incorporated. It was observed that the results for the recovered steel wires from tires were close to the results obtained from the stainless steel screen sample. During testing it was observed that in the case of recovered steel wires, there was de-bonding between the resin and the steel wires. During testing, the composite failed, as the resin failed while the steel wires did not. These recovered steel wires had a coat of carbon from the tires over them. This was suspected to be the cause of failure. Before preparing the next sample, these wires were sand blasted at the Machine Shop at the Mechanical Engineering Department to remove the carbon deposits. This carbon free wires were used to prepare another sample, which when tested gave the same results as the one with carbon deposits.

To the combination of EAS and Lindride 56V in the ratio 1:0.7 by weight, 0.2 parts by weight of Soy Flour and Soy Protein Isolate were added to see any improvement in its properties. Figure 3.4 shows the tensile test curves of the plastic samples with Soy Flour and Soy Protein isolate.

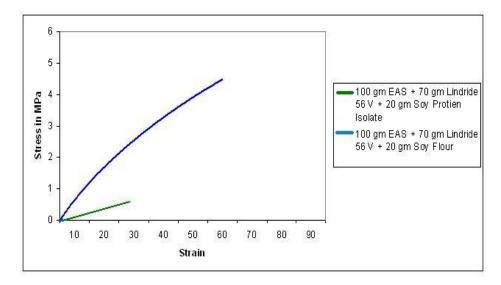


Figure 3.4: Tensile Test Curve - SF and SPI

Table 3.3 shows the tensile test data for the plastic samples with Soy Flour and Soy Protein isolate.

Composition	Tensile Strength (MPa)
100 gm EAS + 70 gm Lindride 56 V + 20 gm Soy	
Protien Isolate	0.4137
100 gm EAS + 70 gm Lindride 56 V + 20 gm Soy	
Flour	5.171

Table 3.3: Tensile Strength Data - SF and SPI

It was observed that with the addition of Soy Flour and Soy Protein Isolate to the neat resin, the strength decreased. The sample with Soy Protein Isolate displayed Hypo-Elastic properties. Hence, it was decided not to prepare composite samples with this formulation.

In order to further enhance the mechanical properties of the samples, the resin EPON was added in different quantities. The quantity of EPON and EAS was varied in the ratio 100:0, 90:10, 80:20, 70:30, 60:40, 50:50, respectively, by weight. The quantity of the hardener Lindride 56V was kept constant at 40% in all the cases. Figure 3.5 shows the tensile test curves for the plastic samples with EPON and EAS.

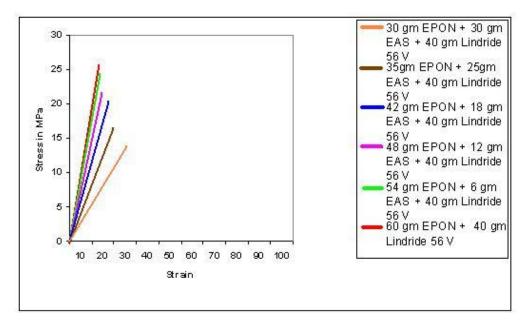


Figure 3.5: Tensile Test Curve – EPON + EAS

Table 3.4 shows the tensile test data for the plastic samples with EPON and EAS.

Composition	Tensile Strength (MPa)
30 gm EPON + 30 gm EAS + 40 gm Lindride 56 V	14.48
35gm EPON + 25gm EAS + 40 gm Lindride 56 V	16.13
42 gm EPON + 18 gm EAS + 40 gm Lindride 56 V	20.68
48 gm EPON + 12 gm EAS + 40 gm Lindride 56 V	21.18
54 gm EPON+ 6 gm EAS + 40 gm Lindride 56 V	23.72
60 gm EPON + 40 gm Lindride 56 V	24.13

 Table 3.4: Tensile Strength Data – EPON + EAS

It was observed that as the quantity of EPON went up and that of EAS came down, the sample became more rigid and brittle. The sample with combination 100:0 EPON and EAS, respectively, gave the highest tensile strength. It was observed that the combination 70:30 EPON and EAS, respectively, gave optimum tensile and flexural strength. It was seen that addition of EPON increased the transparency of the samples. EPON and EAS in the ratio 100:0, 70:30, 60:40 and 50:50 by weight with 40% Lindride 56V by weight were used to prepare composite samples with stainless steel screen. Figure 3.6 shows the tensile test curves.

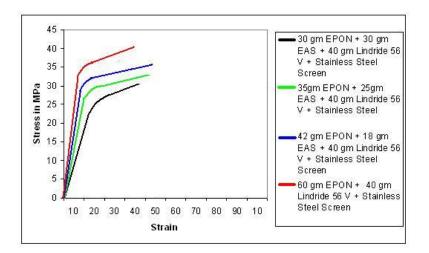


Figure 3.6: Tensile Test Curve – EPON: EAS Composite

Composition	Tensile Strength (MPa)
30 gm EPON + 30 gm EAS + 40 gm Lindride 56 V +	
Stainless Steel Screen	31.05
35gm EPON + 25gm EAS + 40 gm Lindride 56 V +	
Stainless Steel	33.82
42 gm EPON + 18 gm EAS + 40 gm Lindride 56 V +	
Stainless Steel Screen	35.42
60 gm EPON + 40 gm Lindride 56 V + Stainless Steel	
Screen	40.51

Table 3.5 shows the tensile test data for the composite samples.

 Table 3.5: Tensile Strength Data - EPON: EAS Composite

These composite samples were found to be very rigid. The results thus, obtained were found to be better than the samples with only EAS and Lindride 56V. Hence, it can be concluded that addition of EPON enhances the performance of these composites. To the 70:30 EPON, EAS with 40% Lindride 56 V sample, 10gm, 20gm and 30 gm of Soy Flour were added. Figure 3.7 shows the tensile curves for the sample with Soy Flour.

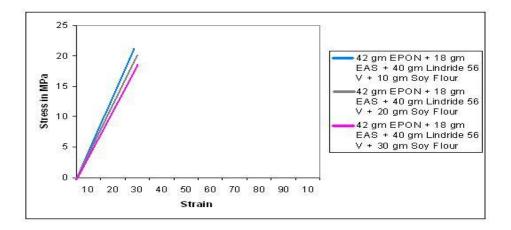


Figure 3.7: Tensile Test Curve - 70:30 EPON: EAS + Soy Flour

Table 3.6 shows tensile test data for the sample with Soy Flour.

Composition	Tensile Strength (MPa)
42 gm EPON + 18 gm EAS + 40 gm Lindride 56 V + 10 gm	
Soy Flour	21
42 gm EPON + 18 gm EAS + 40 gm Lindride 56 V + 20 gm	
Soy Flour	20.68
42 gm EPON + 18 gm EAS + 40 gm Lindride 56 V + 30 gm	
Soy Flour	18.96

Table 3.6: Tensile Test Data - 70:30 EPON: EAS + Soy Flour

It was seen that addition of Soy Flour up to 20gms did not have any considerable effect on the performance of the plastics. When this quantity was increased to 30gms, a decrease in strength was observed. Hence, it can be concluded that for optimum results the quantity of Soy Flour must be limited to 20gms.

3.2. FLEXURAL STRENGTH

The flexural strength of the materials when used with the three hardening agents is mentioned in table. It can be observed that as in the case of tensile test, the optimum combination is obtained by using Lindride 56V as the hardening agent with EAS in the ratio 1EAS:0.7Lindride 56V by weight. Table 3.7 shows the flexural strength data for the plastic samples made with the three hardening agents.

Composition	Flexure Strength (MPa)
1 EAS + 0.7 Lindride - 56V	0.41
1EAS + 0.79 Lindride - 56V	0.37
1EAS + 0.39 Lindride - 56V	0.045
1EAS + 0.71 Lindride - 56V + 0.076 BTDA	0.1
1EAS + 0.79 LS 682K	0.13
1EAS + 0.39 LS 682K	0.013

 Table 3.7: Flexural Strength Data for the Hardening Agents

This combination is further used to prepare different composite materials using glass fiber, aluminum screen, stainless steel and recovered steel wires. Table 3.8 shows the flexural strength data for the composite samples made with 1 part of EAS and 0.7 parts of Lindride 56V.

Composition	Flexure strength (MPa)
1 EAS + 0.7 Lindride 56V	0.41
1 EAS + 0.7 Lindride 56V + aluminum	0.43
screen	
1 EAS + 0.7 Lindride 56V + glass fiber	0.44
1 EAS + 0.7 Lindride 56 V+ stainless steel	0.92
1 EAS + 0.7 Lindride 56V + scrap tire steel	0.84

Table 3.8: Flexural Strength Data for the EAS Composites

There is an increase in strength with the use of composite materials. As seen from the tensile test, the optimum results are obtained for the sample with stainless steel screen. The sample with recovered steel wires from tires came very close to the one with stainless steel screen, just as observed in tensile test. Table 3.9 shows the flexural strength data for the plastic samples with EPON.

Composition	Flexural Strength (MPa)
30 gm EPON + 30 gm EAS + 40 gm Lindride 56 V	5.516
35 gm EPON + 25 gm EAS + 40 gm Lindride 56 V	6.724
42 gm EPON + 18 gm EAS + 40 gm Lindride 56 V	7.584
48 gm EPON + 12 gm EAS + 40 gm Lindride 56 V	6.851
54 gm EPON + 6 gm EAS + 40 gm Lindride 56 V	6.153
60 gm EPON + 40 gm Lindride 56 V	4.826

 Table 3.9: Flexural Strength Data – EPON + EAS

In order to further enhance the mechanical properties of the samples, the resin EPON was added in different quantities. The quantity of EPON and EAS was varied in the ratio 100:0, 90:10, 80:20, 70:30, 60:40, 50:50, respectively, by weight. The quantity of the hardener Lindride 56V was kept constant at 40% in all the cases. It was observed that addition on EPON increased the flexural properties by a large percentage. Addition of EPON makes the sample tough and rigid. It can be seen that the combination with 70:30 EPON and EAS gives the optimum results. Table 3.10 shows the flexural strength data for the composite samples with EPON.

	Flexural
Composition	Strength (MPa)
30 gm EPON + 30 gm EAS + 40 gm Lindride 56 V + Stainless	
Steel Screen	5.516
35gm EPON + 25gm EAS + 40 gm Lindride 56 V + Stainless	
Steel Screen	6.724
42 gm EPON + 18 gm EAS + 40 gm Lindride 56 V + Stainless	
Steel Screen	7.584
60 gm EPON + 40 gm Lindride 56 V + Stainless Steel Screen	6.851

 Table 3.10: Flexural Strength Data – EPON: EAS Composite

A very high increase in flexural strength was seen when EPON was added to the composite sample. As in the previous case, the sample with 70 parts of EPON and 30 parts of EAS gives the optimum results. To the 70:30 EPON, EAS with 40% Lindride 56 V sample, 10gm, 20gm and 30 gm of Soy Flour were added. Table 3.11 shows the flexural strength data for the plastic sample with Soy Flour.

CompositionFlexural Strength
(MPa)42 gm EPON + 18 gm EAS + 40 gm Lindride 56 V + 10 gm
Soy Flour7.86142 gm EPON + 18 gm EAS + 40 gm Lindride 56 V + 20 gm
Soy Flour7.20542 gm EPON + 18 gm EAS + 40 gm Lindride 56 V + 30 gm
Soy Flour5.861

 Table 3.11: Flexural Strength Data – 70:30 EPON, EAS + Soy Flour

It was seen that there was a marginal increase from 7.584MPa to 7.861MPa when 10 gm of soy flour was added. Further addition of soy flour brought about decrease in the flexural strength as it can be seen with the addition of 20gms and 30gms of soy flour.

3.3. IMPACT DROP TEST

The samples made with EPON and EAS were drop tested. The sample was placed on end supports and the plum bob was dropped by pulling the pin. The 60 EPON + 40 Lindride 56V sample gave the best results. Table 3.12 shows the height at which the samples broke during the impact drop test.

Composition	Height (inches) at which Sample Broke
30 gm EPON + 30 gm EAS + 40 gm Lindride 56 V	10.5
42 gm EPON + 18 gm EAS + 40 gm Lindride 56 V	12
60 gm EPON + 40 gm Lindride 56 V	16

 Table 3.12: Impact Drop Test Results

4. CONCLUSIONS

Composite materials were fabricated from EAS, a soybean oil derived epoxy resin and different materials including glass fiber, aluminum screen and stainless steel mesh. The results showed that EAS formulations with suitable materials including steel wires recovered from scrapes can be tailored to obtain composites with good tensile and flexural strengths. Addition of EPON, a bis phenolepichlorohydrine based epoxy resin to these formulations adds toughness and increases the rigidity thereby improving the performance of these materials. Figure 4.1 shows the tensile and flexural strength comparison.

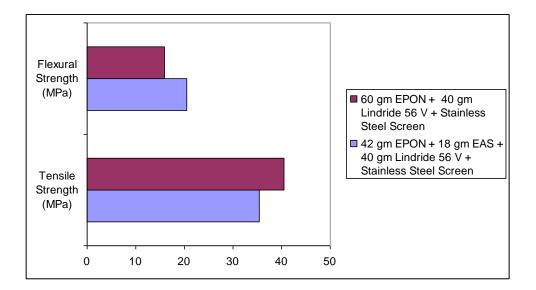


Figure 4.1: Tensile and Flexural Strength Comparison

The formulations can be tweaked to obtain materials of required strengths. 60% EPON and 40% Lindride 56V with stainless steel screen combination can be used whenever high tensile strength is desired and there are no bending loads involved. 42% EPON, 18% EAS and 40% Lindride 56V combination exhibited good tensile and flexural strength. This combination can be used when there are bending loads involved.

5. RECOMMENDATIONS

It is seen that plastics and composites of high tensile and flexural strengths can be made from EAS, a soybean oil derived epoxy resin. This research work concentrated on preparing composites by using EAS and aluminum screen, stainless steel screen, glass fiber and recovered steel wires from scrap tires. Future research work in this area can be carried out by making use of Carbon Fiber and Kevlar. Using these fibers would certainly push the cost of the material up, but at the same time more high strength and tougher materials can be obtained.

BIBLIOGRAPHY

- 1. Lannace, S., J. Macromol. Sci., Pure Appl. Chem., A32 (4), 771 (1995).
- 2. Flieger, M., Kantorova, M., Prell, A., Rezanka, T. and Votriba, J., Folia Microbiol., 48 (1), 27 (2003).
- 3. Van Soest, J.J.G., Benes, K., De Wit, D., Vliegenthart, J.F.G. Polymer, **37** (16), 3543 (1996).
- 4. Van Soest, J.J.G., Kortleve, P.M., Journal of Applied Polymer Science, **74** (9), 2207 (1999).
- Chandrashekhara, K., Sundararaman, S., Flanigan, V., Kapila, S. Materials Science 7 Engineering, A: Structure Material Properties, Microstructure and Processing, A412 (1-2) 2 (2005).
- 6. Sundararaman, S. Ahamed, S., Garg, A., Chandrashekhara, K., SAMPE International Symposium, **50**, 691 (2005).
- 7. Zheng, H., Tan, Z., Zhan, Y.R. and Huang, J., Journal of Applied Polymer Science, **90**, 3676 (2003).
- 8. Wang, S.; Sue, H.J. and Jane, J., Journal of Macromolecular Science, Pure and Applied Chemistry, A33 (5), 557 (1996).
- Graiver, D., Waikul, L.H., Berger, C., Narayan, R., Journal of Applied Polymer Science, 92, 3231 (2004).
- 10. Mo, X. and Sun, X.S., Journal of American Oil Chemists' Society, **78** (8), 867 (2001).
- 11. Wang, S.; Sue, H.J. and Jane, J., Journal of Macromolecular Science, Pure and Applied Chemistry, A33 (5), 557 (1996).
- 12. Wang, S.; Zhang, S.; Jane, J.; Sue, H.-J., Polymer Material Science and Engineering, **72**, 88(1995).

- Mo, X.; Sun, X. S.; Wang, Y., Journal of Applied Polymer Science, 73, 2595 (1999).
- 14. Lee, H. and Neville, Handbook of Epoxy Resins, McGraw-Hill, 1967, pp 5-13.
- 15. May, C. Epoxy Resins Chemistry and Technology Marcel Dekker Inc., 1988, pp 10-14.
- Zhu, J., Garg, A., Mekhissi, K., Chandrashekhara, K., Flanigan, V., Kapila, S., SAMPE International Symposium, 47, 1209 (2002).
- 17. Yadav, R.; Seemamahannop, R.; Chandrashekhara, K.; Flanigan, V.; Kapila, S., SAMPE International Symposium, **50**, 716 (2005).
- Flanigan, V., Kapila. S., Chandrashekhara, K., Garg, A., and Seemamahannop, R U.S. Patent Application submitted 2007.
- 19. <u>www.epa.gov/epaoswer/non-hw/muncpl/tires/science.htm</u> "Management of Scrap Tires." Accessed on 13 April 2007.
- 20. Eldin, N.N., Piekarski, J.A., Journal of Environmental Engineering. **119**, (6) 125 (1993).
- 21. U.S. patent 5783046 (Nov. 28, 1994), Flanigan, V.J. (to Gentech, Inc.)
- 22. Seemamahannop, R., Pai, V., Flanigan, V., Kapila, S., "Fabrication of Soy Oil Epoxy Resin based Composites." SAMPE International Symposium, 322, 53, 2008.
- 23. Seemamahannop, R., Pai, V., Flanigan, V., Kapila, S., "Fabrication of High Strength Composite Materials with Scrap Tire Recycled Steel and Soy Oil Epoxy Resin." Submitted for inclusion in the proceedings of VI International Conference on Value Added Resource Recovery, 2007, L'Aquila, Italy.

- 24. U.S. Patent 5783046, "Process and Apparatus for Destructive Distillation Of Rubber." Flanigan, V., Gentech, Inc.
- 25. Chabba, S., Mathews, G. F., Netravali, A. N., "Green composites using crosslinked soy flour and flax yarns." <u>Green Chem</u>. Volume 7, 2005, pp 576-581.
- Mo, X., Sun, X., "Thermal and mechanical properties of plastics molded from sodium dodecyl sulfate modified soy protein isolates." <u>J. Poly and Env.</u> Volume 8, 2000, pp 161-166.
- 27. Chabba, S., Netravali, A. N., "Green composites using modified soy protein concentrate resin and flax fabrics." JSME International Journal Series A. Volume 47, 2004, pp556-560.
- 28. Swain, S. N., Biswal, S. M., "Biodegradable soy based plastics; opportunities and challenges." J. Poly and Env. Volume 12, 2004, pp35-42.
- 29. Li, F., Larock, R. C., "New soy bean oil-styrene-di vinyl benzene thermosetting copolymers." J. Ceram. Soc. Japan. Volume 97, Issue 8, 1989, pp842-849.
- Shabeer, A., Garg, A., Sundararaman, S., Chandrashekara, K., Flanigan, V., Kapila, S., "Dynamic mechanical characterization of soy based epoxy resin system." J. Appl. Polym. Sci. Volume 98, 2005, pp1772-1780.
- Zhu, J., Chandrashekhara, K., Flanigan, V., and Kapila, S., "Curing and mechanical characterization of soy based epoxy resin system." <u>J. Appl. Polym.</u> <u>Sci.</u> Volume 91, 2004, pp3513-3518.
- 32. Yadav, R., Ahamed, S., Seemamahannop, R., Chandrashekara, K., Kapila, S., Flanigan, V., "Thermal and mechanical characterization of soy protein plastics." <u>SAMPE International Symposium</u>. 2005.
- 33. Zhang, J., Jiang, L., Zhu, L., "Morphology and properties of soy protein and polylactide blends." <u>Biomacromolecules</u>. Volume 7, 2006, pp1551-1561.
- 34. Swain, S, N., Rao, K. K., Nayak, P. L., "Biodegradable polymers part II: thermal degradation of biodegradable plastics crosslinked from formaldehyde soy protein concentrate." J. Thermal and Cal. Volume 79, 2005, pp33-38.

- Graiver, D., Waikul, L. H., Berger, C., Narayan, R., "Biodegradable soy protein polyester blends by reactive extrusion process." <u>J. Appl. Polym. Sci</u>. Volume 92, 2004, pp3231-3239.
- Wang, N., Zhang, L., Gu, J., "Mechanical properties and biodegradability of cross linked soy protein isolate/water borne polyurethane composites." <u>J. Appl.</u> <u>Polym. Sci</u>. Volume 95, 2004, pp465-473.
- Zhengh, H., Tan, Z., Zhan, Y. R., Huang J., "Morphology and properties of soy protein plastics modified with chitin." <u>J. Appl. Polym. Sci.</u>, Volume 90, 2003, pp3676-3682.
- Huang, J., Zhang, L., Wang, X., "Soy protein lignosulphonate plastics strengthened with cellulose." J. Appl. Polym. Sci. Volume 89, 2003, pp1685-1689.
- 39. Seemamahannop, R., Kafeel, M., Yadav, R., Nam, P., Chandrashekara, K., Kapila, S., Flanigan, V., "Development and characterization of soy based plastics." <u>SAMPE International Symposium</u>. 2006.
- 40. Mo, X., Sun, X., "Effects of storage time on properties of soy bean protein based plastics." J. Poly and Env. Volume 8, 2000, pp 161-166.
- 41. Mohammed Kafeel Ahamed, Dr. Virgil Flanigan, Dr. S. Kapila, Dr. K. Chandrashekara., "Part 1. Rice Hull Nano Silica as fire retardant in Epoxy Composites. Part 2. Development and Characterization of Soy based Plastics." Thesis, University of Missouri-Rolla, 2006.
- 42. Rupali Yadav, Dr.K.Chandrashekara Dr. S. Kapila, Dr. Virgil Flanigan, "Development and Characterization of Soy based materials: Plastics, Coatings and Foams." Thesis, University of Missouri-Rolla, 2005.
- 43. Gorman, Jim. "Where the rubber is the road." Audobon, Nov-Dec 1993, Pp.24-31.
- 44. Eldin, Neil N.; Julian A. Piekarski. (Epidemiological Studies), Journal of Environmental Engineering, Vol.119, No.6, Nov/Dec 1993.
- 45. EPA Document EPA-450/3-91-024 Dec.1991, "Burning tires for fuel and tire Pyrolysis: Air implications."

- 46. Thompson, W.H. "Studies of Asdes Triseriatus-the LaCross virus carrier." Wisconsin Academy Rev., Vol.31, pp. 64-66.
- 47. Korte, Richard A. "LaCross Encephalitis," Public Works, Nov.1988, p.64.
- 48. Tamura, S., Murakami, K., Chandrasekhara, K., Flanigan, V., Kapila, S., SAMPE International Symposium 47, 1209 (2002).
- 49. Yadav, R.: Seemamahannop, R., Chandrasekhara, K., Flanigan, V., Kapila, S., SAMPE International Symposium 50, 716 (2005).
- 50. Flanigan, V., Kapila, S., Chandrasekhara, K., Garg, A., and Seemamahannop, R., U.S. Patent Application submitted 2005.

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