

Green Building Materials: Hemp Oil Based Biocomposites

Nathan W. Manthey, Francisco Cardona, Gaston M. Francucci, and Thiru Aravinthan

Abstract—Novel acrylated epoxidized hemp oil (AEHO) based bioresins were successfully synthesised, characterized and applied to biocomposites reinforced with woven jute fibre. Characterisation of the synthesised AEHO consisted of acid number titrations and FTIR spectroscopy to assess the success of the acrylation reaction. Three different matrices were produced (vinylester (VE), 50/50 blend of AEHO/VE and 100% AEHO) and reinforced with jute fibre to form three different types of biocomposite samples. Mechanical properties in the form of flexural and interlaminar shear strength (ILSS) were investigated and compared for the different samples. Results from the mechanical tests showed that AEHO and 50/50 based neat bioresins displayed lower flexural properties compared with the VE samples. However when applied to biocomposites and compared with VE based samples, AEHO biocomposites demonstrated comparable flexural performance and improved ILSS. These results are attributed to improved fibre-matrix interfacial adhesion due to surface-chemical compatibility between the natural fibres and bioresin.

Keywords—Biocomposite, hemp oil based bioresin, green building materials, mechanical properties.

I. INTRODUCTION

IN the search for high performance construction materials, fibre composites are now being more widely utilized than in the past. Fibre composites generally use petro-chemically derived resins, for example; epoxy, polyester and vinylester because of their advantageous material properties such as high stiffness and strength. However, these resins also have serious shortcomings in terms of sustainability, initial processing cost, recyclability, energy consumption, and processing health hazards. Increasing environmental awareness throughout society and now within the civil engineering and construction industries is driving the research, development and utilization of more 'green' building materials, specifically the development of bioresins and biocomposites based on renewable, natural materials.

Bioresins are an emerging sustainable thermoset technology often derived from vegetable oils and are termed epoxidized vegetable oils (EVO) and when further functionalized, acrylated epoxidized vegetable oils (AEVO). Due to their

biological origin they represent a sustainable, low environmental impact option to existing petro-chemically derived resins. These bioresins may be reinforced with natural or synthetic fibres thereby creating a class of materials termed, natural fibre composites or biocomposites. The majority of biocomposite research has focused on using natural fibres in petroleum derived matrices [1-8]. Although a step in the right direction in terms of sustainability, these hybrid biocomposites are only partially based on renewable constituents. The production of truly 100% 'green' biocomposites requires the synthesis and characterisation of sustainable bioresins and biocomposites such as those presented within.

At the Centre of Excellence in Engineered Fibre Composites (CEEFC) research into the development of novel hemp oil based bioresins, such as epoxidized hemp oil (EHO) and acrylated epoxidized hemp oil (AEHO) for biocomposite applications is being conducted [9-11]. Traditionally hemp oil has been considered a by-product of hemp fibre production, with its main uses being in livestock food and pharmaceutical applications. Due to its unique fatty acid profile, high in both linoleic and linolenic acid, hemp oil shows considerable potential as a bioresin feedstock.

In this study, AEHO was synthesised, characterized and applied to jute fibre reinforced biocomposites. Comparisons with commercial vinylester (VE) based resin and AEHO/VE 50/50 blended bioresin systems both in neat resin form and applied to biocomposites was performed. Mechanical properties, flexural and interlaminar shear, were investigated and compared for all three bioresin and biocomposite sample types.

II. MATERIALS

Cold pressed raw industrial hemp oil supplied by Ecofibre (Maleny, Queensland, Australia) with a fatty acid profile consisting of the following acids; palmitic = 6.0%; stearic = 2.0%; oleic = 12.0%; linoleic = 57.0%; linolenic = 20.7%; other = 2.3% was used to synthesize EHO and AEHO. Analytical grade glacial acetic acid and hydrogen peroxide with minimum concentrations of 99.7% and 30% respectively were used as received from LabServ (Biolab, Australia). Amberlite IR-120 was used as received from Fluka (Sigma-Aldrich, Australia) and was of the ionic H⁺ form. For the synthesis and characterisation of AEHO; hydroquinone inhibitor ($\geq 99\%$, Sigma-Aldrich), acrylic acid ($\geq 99.5\%$, Acros Organics), AMC-2 catalyst (Ampac Fine Chemicals), 0.5% solution phenolphthalein (Chem Supply) and sodium hydroxide (0.5 N) were used as received. Woven jute fibre, 90°/0°, 550 g/m² was used as the natural fibre reinforcement. The fibre was cut to size, washed with warm water to remove any dust particles and other such contaminants and was dried

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for 12 h at 110 °C. No chemical treatments were performed on the fibre.

III. EXPERIMENTAL

A. Synthesis of Epoxidized Hemp Oil

The optimization study performed by Cooney [12] on the epoxidation of hemp oil forms the basis of the epoxidation reaction conditions such as time, temperature and reactant concentrations used in this study. In order to produce the AEHO it is first necessary to synthesize EHO. As reported [12-17] EHO was synthesized through the epoxidation of cold pressed raw industrial hemp oil (156.25 g, 1 mol) by peroxyacetic acid, formed *in situ* by the reaction of hydrogen peroxide (113.4 g, 1 mol) and acetic acid (40.04 g, 0.67 mol) in the presence of an acidic ion exchange resin, Amberlite IR-120 H⁺ (15% by weight of hemp oil) as the catalyst, Fig. 1. The constituents were added to a four-necked reaction vessel equipped with a mechanical stirrer and thermometer. Stirring was initiated and the reactor temperature was increased until the mixture reached 40 °C whereby dropwise addition of hydrogen peroxide was performed over a period of one hour. Temperature and stirring speed were then increased to operational values of 75 °C and 110 rpm respectively. These parameters were maintained for a period of 7 h.

On completion of the reaction the catalyst was filtered off and the reactor contents were cleaned in a separation funnel by washing with water three times (cool, near-boiling, cool) to remove the aqueous phase. Next the EHO mixture was centrifuged and aerated to remove any remaining water. The bioresin was then further dried through the addition of anhydrous sodium sulphate in the proportion of 0.15 g per 1 g of bioresin. Following the addition of anhydrous sodium sulphate, the bioresin was placed in an oven at 70 °C for 12 h and subsequently filtered through Whatman No. 4 filter paper. The yield of EHO obtained from the initial hemp oil was approximately 75% by volume. At room temperature EHO is a yellow colored liquid with a slight vegetable oil odor.

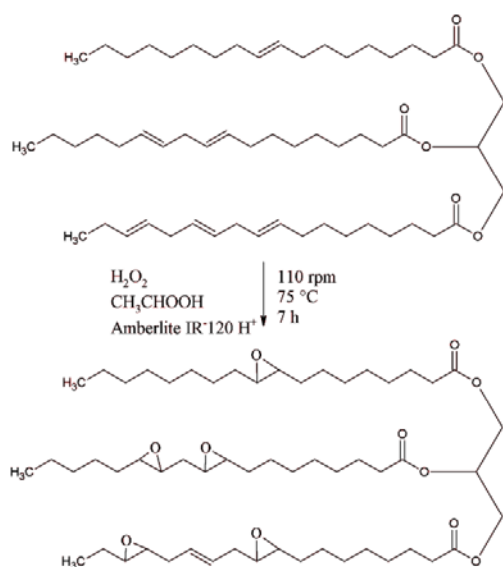


Fig. 1 *In situ* epoxidation of hemp oil

B. Synthesis of Acrylated Epoxidized Hemp Oil

AEHO was synthesized through the acrylation of EHO. EHO, hydroquinone inhibitor (0.0033 g/ml of EHO + acrylic acid) and AMC-2 catalyst (1.75% by weight of EHO + acrylic acid) were added to the Mettler Toledo LabMax automatic reactor. The constituents were mixed at 50 °C for 30 minutes whereby acrylic acid was added in a molar ratio of 1.1 moles per mol of EHO. Due to the nature of this reaction the temperature of the mixture can rapidly increase due to an exothermic reaction mechanism. To ensure a controlled reaction, the reactor was programmed to maintain a constant temperature of 90 °C over a period of 12 h at a mixing speed of 160 rpm. Fig. 2 shows schematically the *in situ* acrylation of the EHO.

C. Bioresin and Biocomposite Specimen Preparation

Novel bioresins and jute reinforced biocomposites containing three different matrices; VE, a 50/50 blend of AEHO/VE and 100% AEHO were produced. To produce the AEHO bioresin system, 33 wt% of styrene was added to the AEHO and thoroughly mixed. The styrene was added to decrease viscosity and make the bioresin suitable for composite manufacturing processes. Following the addition of the styrene, the accelerator, N2-51P (0.25% by weight) and 40% MEKP based catalyst (4% by weight) was added and thoroughly stirred. The VE resin system was simply produced by mixing the accelerator, N2-51P (0.25% by weight) and 40% MEKP based catalyst (2.5% by weight) with the commercial VE. Similarly the 50/50 AEHO/VE bioresin system was produced by mixing 50 wt% of the AEHO bioresin with 50 wt% of the VE resin system. To produce the flexural testing specimens, the produced resin/bioresins were degassed and poured into waxed moulds, cured as described below and cut to size.

Biocomposite laminate panels were manufactured using the hand lay-up process using the bioresins reinforced with four layers of jute fibre. Manufactured panels measured 300×300×5 mm with fibre wt% approximately 25. Initial curing for both resin/bioresin and biocomposite samples was performed at room temperature for 4 h followed by post curing at 120 °C for a further 4 h. Subsequent to post curing, the samples were removed from the mould, cut to size, dried at 80 °C for 4 h to ensure the removal of any induced moisture and then cooled in a desiccator ready for mechanical testing.

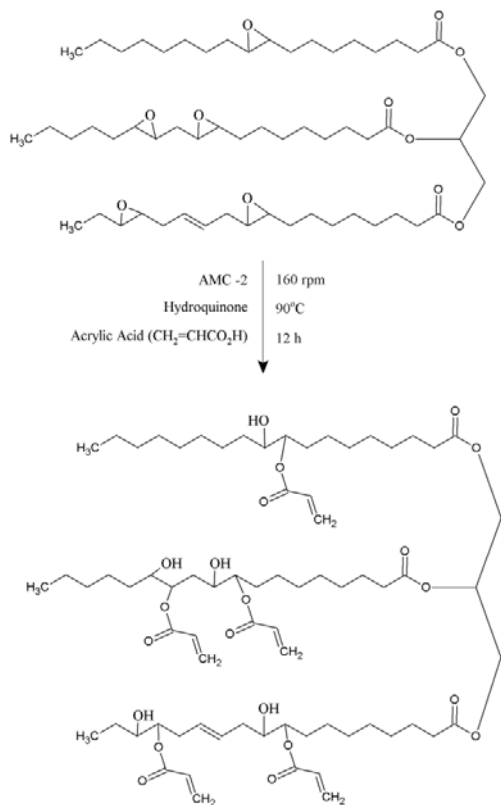


Fig. 2 *In situ* acrylation of epoxidized hemp oil

D. Mechanical Testing

Flexural testing in the form of 3-point simple beam loading was performed on neat resin/bioresin and biocomposite specimens to determine flexural behavior. Bioresin and biocomposite flexural testing was conducted in accordance with ISO 178 and ISO 14125 respectively using an MTS Alliance RT/10 10 kN machine. A cross head speed of 2 mm/min and a span depth ratio of 16:1 were used throughout the tests with specimen dimensions being 80×10×4 mm. Interlaminar shear strength (ILSS) testing was performed to determine the affect of fibre-matrix interfacial shear strength. Testing was performed using ISO 14130 on a MTS Alliance RT/10 10 kN machine with a crosshead speed of 1 mm/min. Five specimens of each sample type were used in each mechanical test.

IV. RESULTS AND DISCUSSION

A. Bioresin Characterisation

The synthesis and characterisation of EVO and AEVO bioresins is widely studied and understood [9-12, 18-35]. Therefore the focus of the characterisation in this study utilizes these reliable, well established methods and principles. The quality of EVO bioresins is largely dependent on the degree of epoxidation, measured by oxirane oxygen content and the number and consumption of carbon-carbon double bonds (C=C) measured by the iodine value. Owing to the fact that the AEHO is synthesised from the EHO it is important to ensure that the EHO is of high quality. With regards to the

characterization of the AEHO synthesis process and final product quality, several commonly used characterisation techniques are employed. To monitor the progress of the acrylation reaction, consumption of acrylic acid was determined through titrating with sodium hydroxide and an indicator. FTIR spectroscopy was also used to monitor the reaction and characterize the final AEHO bioresin. Viscosity measurements were also performed.

1) Iodine Value

The iodine value was used to quantify the degree of ethylenic unsaturation, or C=C present in the hemp oil. It is defined as the mass in grams of iodine that is consumed by 100 g of vegetable oil. That is, the higher the iodine value, the more C=C present in a vegetable oil. In terms of producing a high quality EVO, the vegetable oil should contain a large percentage of C=C. Furthermore a successful epoxidation reaction will see a high proportion of those C=C converted to epoxides. In this study the iodine value of hemp oil was theoretically determined as approximately 163 (g iodine/100 g of hemp oil) as per [12, 36] with the results presented in Table I.

Experimental iodine value were obtained using Wij's method [12, 26, 27, 30, 307-39] which involved titrating using Wij's iodine solution as per the standard methods for the analysis of fats and oils as outlined by Cooney [12]. After mixing Wij's solution with the hemp oil sample the degree of ethylenic unsaturation was calculated by examining the amount of iodine that reacted with the C=C in the oil. The iodine value was calculated as 159. This value is slightly higher than that obtained from Cooney [12] who found an iodine value of 122 for hemp oil. For comparative purposes typical iodine values for soybean and canola oils are approximately 140 and 112 respectively [36]. Following the completion of the epoxidation reaction the iodine value was determined to be approximately 21 giving a relative conversion relative to iodine value of approximately 89.4%.

2) Oxirane Oxygen Content

An oxirane ring, is highly reactive and highly strained with bond angles approximately 60° [26]. Formation of crosslinking occurs throughout the epoxy resin when cured due to the presence of the highly reactive oxirane rings. According to Mungroo et al.[26], and Goud et al. [30] higher quality polymers are correlated with high level crosslinking due to higher conversion of C=C to oxirane rings (epoxides). Oxirane oxygen content is a measure of the amount of C=C which has been transformed into epoxides through the epoxidation process. In this work the oxirane oxygen content of each sample was determined analytically by the direct titration method with hydrobromic acid solution in acetic acid as per the literature [12, 26, 27, 30, 37, 38]. The maximum theoretical oxirane oxygen content (OO) was determined from (1). Where IV_0 is the triglyceride iodine value of the vegetable oil, A_i (126.9) is the atomic mass of iodine and A_o (16.0) is the atomic mass of oxygen.

$$OO_e = \left[\frac{\left(\frac{IV_o}{2A_o} \right)}{100 + \left(\frac{IV_o}{2A_o} \right) A_o} \right] \times 100 A_o \quad (1)$$

$$\text{Conversion relative to } OO = \frac{OO_e}{OO_i} \times 100 \quad (2)$$

The hydrobromic acid serves to open the epoxide through the process of bromination, subsequently forming a hydroxyl group. A colour change of the solution typically from violet to yellow to clear indicates the reaction is complete. From the titration, the oxirane oxygen content of EHO was determined to be 8.3%. This represents a relative conversion to oxirane of 89.4% based on the theoretical oxirane oxygen content of 9.3% calculated from (1) and (2). In comparison typical oxirane oxygen content values for commercial epoxidized soybean oil (ESO) is approximately 7%.

3) Acid Value

As per [40] acid number titration was used to determine the extent of the acrylation reaction of EHO by measuring the amount of unreacted acrylic acid in the AEHO mixture. 5 g of acetone was added to 1 g of the AEHO reaction mixture and then mixed using a magnetic stirrer until the AEHO was dissolved. Three drops of 0.5 wt% solution phenolphthalein in 50% ethanol pH indicator was added to the AEHO/acetone mixture to establish the neutralization point. The solution was then titrated with 0.5 N NaOH until the end point. The reaction was deemed to be complete when the acid number was equal to or less than approximately 10 (mg NaOH/g AEHO). If the acid number was found to be greater than 10 the reaction was continued until the acid number was equal to or below 10.

TABLE I
IODINE VALUE AND COEFFICIENTS FOR HEMP OIL

Fatty acid (FA)	FA (%)	Molar mass (g/mol)	No. of DB	C _{FFA}	C _{TAG}	IV _{FFA}	IV _{TAG}
Palmitic (16:0)	6.0	256.42	0	0.00	0.00	0.00	0.000
Stearic (18:0)	2.0	284.48	0	0.00	0.00	0.00	0.000
Oleic (18:1)	12.0	282.46	1	0.90	0.86	10.78	10.32
Linoleic (18:2)	57.0	280.45	2	1.80	1.73	103.1	98.71
α-Linolenic (18:3)	19.0	278.43	3	2.70	2.62	51.96	49.70
γ-Linolenic (18:3)	1.7	278.42	3	2.70	2.62	4.64	4.44
Other	2.3	292.00	0	0.00	0.00	0.00	0.000
Total	100					170.5	163.2

4) Viscosity

Viscosity measurements were performed at room temperature (25 °C) with a Brookfield DV-II+ viscometer using a plate spindle. Viscosity measurements were taken for hemp oil, EHO, AEHO and AEHO mixed with styrene. For comparative purposes the viscosities of commercial ESO, commercial synthetic epoxy and commercial VE were also

measured, Table II. EHO exhibited a value of 845 cP while AEHO and AEHO mixed with styrene displayed values of 20550 and 358 cP respectively. It can be observed that the viscosity of AEHO is too high for most composite processing techniques. Therefore, 33% by weight of styrene was added to the AEHO bioresin in order to decrease viscosity and improve processability for manufacturing composite parts by traditional composite processing techniques. For reference purposes the viscosities of the following commercial resins, Plasthall ESO, R246TX epoxy and FG SPV6003 vinylster were also measured at 340, 1490 and 425 cP respectively.

TABLE II
VISCOSITIES OF BIORESINS AND COMPARATIVE SYNTHETIC RESINS

Resin	Viscosity (cP)
EHO	845
Plasthall ESO	340
Synthetic epoxy (R246TX)	1490
AEHO	20550
AEHO + styrene (33%)	358
Vinylester (SPV6003)	425

5) FTIR Spectroscopy

The premise behind the use of FTIR spectroscopy is the ability to identify and analyse the structure of the hemp oil before and after epoxidation. Specifically FTIR spectroscopy was used to quantify the development of epoxy groups, percentage consumption of C=C in the EHO and the formation of acrylic groups [26, 41].

Fundamentally, FTIR spectroscopy operates by measuring the different frequencies present in different molecules. Different molecules absorb specific frequencies distinctive of their structure which in turn is dependent on the mass of the atoms and the varying strength of the bonds between the atoms. Therefore particular frequencies can be accurately associated with different bond types enabling identification of the structure of the sample. When a particular molecule within the structure of the sample absorbs IR radiation of a certain wavelength it is displayed in the spectrum as a peak at that particular wave number [42].

In this study a Nicolet 6700 FT-IR spectrometer (Thermo Fisher Scientific) was used to characterize the 'success' of the epoxidation and acrylation reactions. Samples were analyzed at a resolution of 4 cm⁻¹ and 64 scans. Analysis was performed using OMNIC software version 6.2. All tests were performed on room temperature samples.

Fig. 3 presents the FTIR spectra obtained from hemp oil (top curve) and EHO (bottom curve) samples. On the spectra significant areas at specific wave numbers are documented and outlined below. The peak indicated at wavenumber 3010 cm⁻¹ corresponds to the consumption of C=C in the epoxidation reaction [12, 36, 43]. From Fig. 3 it can be noted that the peak is reduced in size between the hemp oil and the EHO. Upon successful completion of the epoxidation reaction this peak is completely removed as can be seen in this case. The main peak of interest occurs at 823 cm⁻¹ and is associated with epoxide formation [12, 36, 44]. By examining Fig. 3 it can be observed that there is a noticeable peak present in the EHO spectrum compared with the hemp oil spectrum indicating the formation of epoxy groups.

Fig. 4 presents the FTIR spectra associated with the acrylation reaction of the EHO. The peak visible at 3500 cm^{-1} is attributed to hydroxyl groups which indicate opening of the epoxides [40, 45]. The peak at 1630 cm^{-1} is attributed to the stretching vibration of the C=C. The peaks of most importance regarding the acrylation reaction are visible at 1400, 985 and 810 cm^{-1} . These peaks are attributed to the formation of acrylic functional groups in AEHO [35].

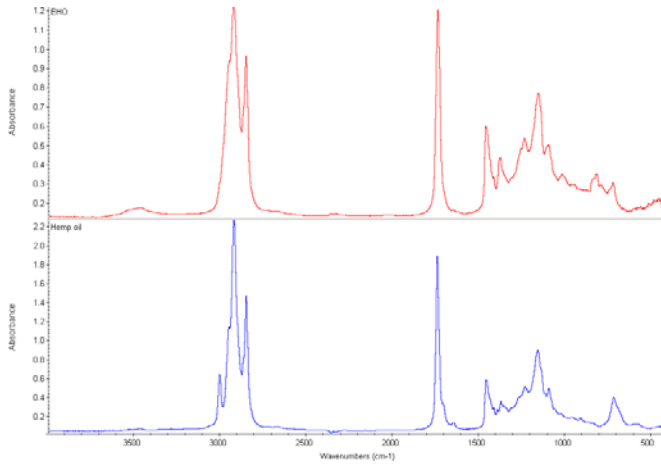


Fig. 3 FTIR spectra obtained from hemp oil and EHO

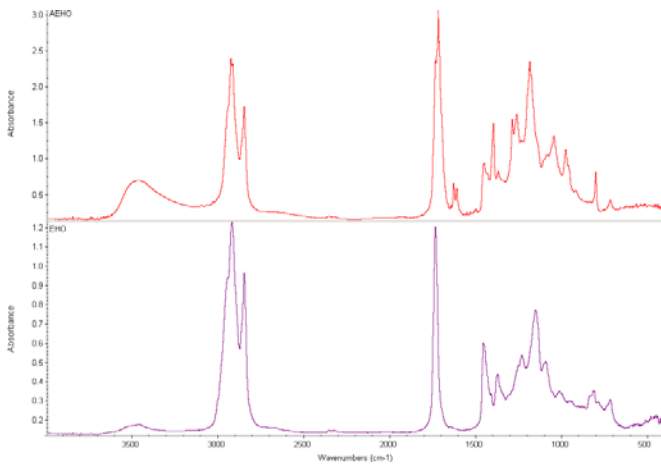


Fig. 4 FTIR spectra obtained from EHO and AEHO

B. Flexural Properties

Fig. 5 shows the flexural modulus of AEHO based neat bioresin and biocomposite samples. The commercial VE resin displayed almost 1.45 and 3.2 times the flexural modulus of the 50/50 and AEHO bioresin samples. This behavior is somewhat expected considering that the long fatty acid chains of the AEHO impart flexibility to the matrix. The addition of the jute fibre reinforcement to the matrices was found to greatly improve the flexural modulus for all sample types. Improvements in flexural modulus were found to be in the order of 1.43, 1.93 and 3.75 for the VE, 50/50 and AEHO based biocomposite samples respectively. Due to this

reinforcement and improvement in flexural modulus, the 50/50 and AEHO based samples displayed flexural moduli that were only approximately 8 and 18% lower than the commercial VE based sample respectively.

The flexural strength of the AEHO based bioresin and biocomposite samples is shown in Fig. 6. VE resin flexural strength was found to be approximately 1.4 and 2.3 times higher than that of the AEHO based bioresins. In terms of the biocomposite samples, no considerable variation of flexural strength was observed with both AEHO and 50/50 samples being comparable with the VE biocomposites. Generally this behavior is unexpected, as the tensile strength of jute fibres (approximately 600-1100 MPa [18, 46]) is higher than that of the commercial VE resin (86 MPa). However in this case, the reduction in flexural strength is attributed to poor fibre-matrix interfacial adhesion of the synthetic resin with the natural fibre reinforcement, as observed in the ILSS results. As a result of the poor bonding between fibres and matrix, the load is not able to be efficiently transferred from the matrix to the fibres. Consequently a large degree of the load is carried by the matrix itself. In the case of the AEHO based biocomposites, the improved flexural strength compared with the neat bioresin is predominately associated with improved fibre-matrix interfacial adhesion as a result of surface chemical compatibility between the natural fibres and the bioresin.

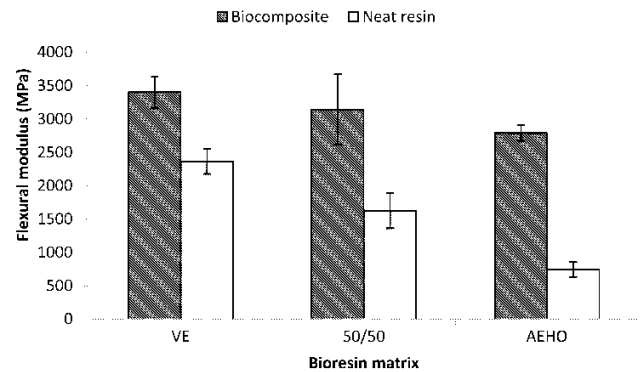


Fig. 5 Flexural modulus of bioresin and biocomposite samples

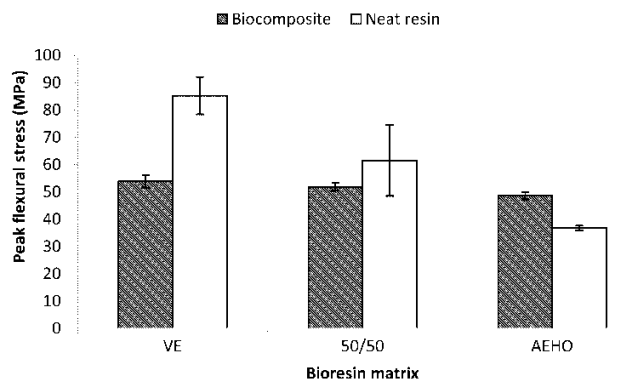


Fig. 6 Flexural strength of bioresin and biocomposite samples

C. Interlaminar Shear Strength

Interlaminar shear strength of fibre composites is largely dependent on the fibre-matrix adhesion. This property has a strong influence on the structural performance of composites, since their strength is strongly influenced by factors weakening the interface. Fig. 7 depicts the ILSS of the biocomposite samples. It was found that AEHO and 50/50 samples displayed higher ILSS than VE based samples. 100% AEHO samples displayed the highest ILSS. These results suggest that the fibre-matrix interfacial adhesion is stronger for the AEHO based samples compared with the VE based samples since the strength of the VE resin is superior to that of the AEHO bioresin as presented in the flexural properties section of this paper. As previously discussed, the surface chemical compatibility between the natural fibres and the bioresin, specifically the greater quantity of hydroxyl groups present in the AEHO bioresin compared with the VE is proposed as the reason for enhanced fibre-matrix adhesion. The hydroxyl functional groups present in the AEHO serve to interact with the hydroxyl groups present in the cellulose of the natural fibres to form strong hydrogen bonds thereby improving adhesion and ultimately flexural and ILSS performance.

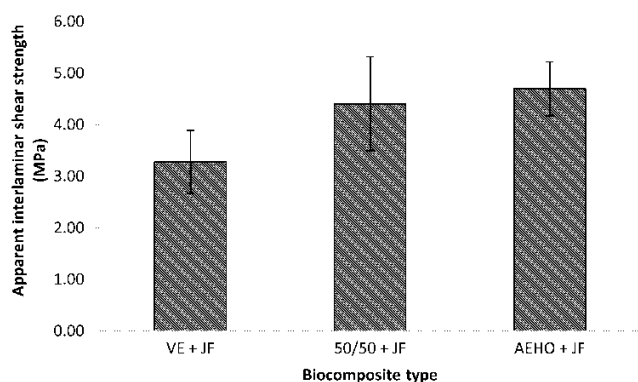


Fig. 7 Interlaminar shear strength of biocomposite samples

V. CONCLUSION

In this study, AEHO was synthesized, characterized and applied to jute fibre reinforced biocomposites. Comparisons with commercial VE based resin and AEHO/VE 50/50 blended resin systems both in neat resin form and applied to biocomposites were performed. Mechanical properties, flexural and interlaminar shear, were investigated and compared for all three bioresin and biocomposite sample types.

Results from the mechanical tests showed that AEHO and 50/50 based neat bioresins exhibited lower flexural properties compared with the VE samples. Conversely when AEHO based bioresin matrices were reinforced with jute fibre they displayed comparable flexural performance and improved ILSS. This is thought to be due to surface chemical compatibility between the natural jute fibres and the AEHO based bioresin. Specifically the greater quantity of hydroxyl

groups present in the AEHO bioresin compared with the VE is proposed as the reason for this enhanced fibre-matrix interfacial adhesion. These hydroxyl functional groups present in the AEHO provide functional groups that interact with the hydroxyl groups present in the cellulose of the natural fibres. The end result is believed to be the formation of strong hydrogen bonds thereby improving adhesion and ultimately flexural and ILSS performance.

Further work in the form of scanning electron microscopy (SEM) will be undertaken to confirm the enhanced interfacial adhesion of AEHO and natural fibres. Mechanical testing in the form of tensile and impact, and moisture absorption analysis will be undertaken. Furthermore in conjunction to this dynamic mechanical analysis (DMA) will be performed to determine the thermo-mechanical properties of the AEHO bioresin and biocomposite systems.

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