



## DEVELOPMENT OF THERMALLY IMPROVED CHROME-TANNED WASTE LEATHER FIBRE ABS COMPOSITES



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### ABSTRACT

There is an increasing generation of solid leather wastes from the leather industry in recent times. This has become a major concern for researchers wanting to convert them into good products. In this work, chrome-tanned leather wastes were used to develop valuable composites for high temperature applications using acrylonitrile-butadiene-styrene (ABS) polymer with the aim of enhancing the interfacial adhesion and thermal stability of natural protein fibres. The chrome-tanned leather wastes were neutralized and surface-coated with dilute epoxy resin in acetone of 2:1 volume ratio, before incorporation into ABS using a twin screw extruder at different fibre loadings and finally compression moulded into composites. The epoxy-coated leather wastes (ECLW) and uncoated leather fibres (CLW) were analyzed using Fourier Transform Infrared Spectroscopy (FTIR) and Differential Scanning Calorimetry (DSC) to determine the effect of coating on the functional groups and their thermal stability respectively. Tensile and morphological tests were performed on both the epoxy-coated leather waste filled ABS (ECLWABS) and uncoated (CLWABS) composites. Result obtained showed that ECLWABS composites have improved tensile properties compared to the uncoated with maximum strength obtained at 5 wt % fibre loading which decreased with increasing fibre loading up to 20 wt%. The FTIR spectra showed significant changes in the absorption characteristics of ECLW compared to CLW samples. DSC scan revealed that the coated leather waste fibre showed higher onset and melting temperatures of 177.8 and 184.5 °C respectively than the uncoated fibres with onset and melting temperatures of 166.2 and 169.4 °C, indicating thermal stability before degradation. FESEM micrographs showed a strong bonding of the coated leather fibre and ABS matrix with delayed degradation for coated fibres. The optical micrographs of the composites showed good dispersion of fibrous structure of leather waste fillers in the ABS phase. In conclusion, the epoxy-coated, chrome-tanned leather waste composites can find applications where strength and thermal stabilities are of paramount importance thus, reducing solid wastes generated by the footwear and leather processing industries.

**Keywords:** Thermal property, epoxy coating, chrome-tanned leather wastes, tensile strength

### INTRODUCTION

Leather is a natural polymer of protein fibre origin. Their appearances are usually improved when processed but this process leads to the release of liquid and solid wastes to the environment (Pati *et al.*, 2014). The wastes generated are usually land filled, dumped into rivers or dispersed in nature thus, having serious effects on the surrounding environment (Ambone *et al.*, 2016). In order to address this problem, several applications for leather fibre reinforced composite products, which is attracting growing interests from construction, marine, automotive, etc have been developed (Dan-mallam *et al.*, 2013; Talla *et al.*, 2013). This is due to their environmental friendliness, low cost, biodegradability, renewability, recyclability and low weight. Other reported studies on leather composites include: leather fibres/recycled scrap rubber composites (Ravichandran and Natchimuthu, 2005), short leather fibres/PVC composites (Madera-santana *et al.*, 2002), chrome-tanned leather wastes/ABS composites (Ramaraj, 2006; El-Sabbagh and Mohamed, 2011; *Przepiórkowska et al.*, 2007), chrome-tanned cow leather composites (Nasr and Ismail, 2010), waste leather/PLA composites (Ambone *et al.*, 2016), leather waste/PES composites (Nahar *et al.*, 2013), and leather/unsaturated polyester composites (Talib *et al.*, 2017; Talib *et al.*, 2018). Also, Nahar *et al.* (2013) reported that pollution creating crust scrap leather can be reused and recycled into semi biodegradable biocomposite which can be used in valuable products thereby reducing environmental pollution.

Despite the above research efforts, the use of leather wastes in composite fabrication is restricted to low temperature plastics such as polypropylene, polyethylene, thermosets, among others because of their degradation at high processing temperatures. Some of the studies have been able to solve the problems of incompatibility,

moisture absorption and environmental issues yet; the problem of low thermal resistance and stability leading to degradation of leather fibres at high temperature has not been addressed. In addition, there are also limited information on composites of high temperature polymers and natural leather (of protein origin) fibres and this has further limited the potentials of these protein fibres. Ravichandran and Natchimuthu (2005) reported that when leather fibres are added to high temperature polymers, maintenance of fibre integrity at high temperature has always posed a problem as a result of leather fibre degradation.

There are few reports on high temperature engineering plastics with natural fibres aiming to address the problems of natural fibres degradation at high temperature. They include studies on PET/Hemp (Talla *et al.*, 2013), Jute fabric/polyamide 6 (Thitithanasarn *et al.*, 2012), ABS/kenaf (Saliu *et al.*, 2015), recycled PET/sugarcane bagasse fibres (Corradini *et al.*, 2009). In another attempt, the work of Corradini *et al.* (2009) recorded a reduced performance in tensile property of bagasse fibre due to degradation at high processing temperature. Talla *et al.* (2013) in their PET/hemp natural fibre investigation to determine the effect of processing temperature and fibre loading also reported the existence of degradation of the hemp fibre at high temperature. Owen *et al.* (2018) found that epoxy-coated kenaf filled recycled PET composite showed superior mechanical properties and were able to withstand high temperature with non-existence of degradation with respect to the composites containing uncoated fibre.

There are few studies establishing that epoxy resin surface coating and treatment are able to improved mechanical properties and degradation resistance of natural fibre composites. They include: Thitithanasarn *et al.* (2012), Nuthong *et al.* (2013), Saliu *et al.*

(2015) and Owen *et al.* (2018). However, work done specifically on epoxy coated leather fibres and high temperature thermoplastic polymers such as ABS, PET, etc., is scarcely available. The tensile properties of engineering materials are widely used for the development of processes and industrial products (Tang *et al.*, 2019; Li *et al.*, 2019; Wei *et al.*, 2019). They are important for consideration in engineering materials as strength is directly related to the load a polymer material can withstand during use.

This research seeks to develop valuable composites from chrome-tanned leather wastes with high temperature thermoplastic polymer (ABS) as matrix in order to improve the thermal stability and interfacial adhesion of the fibres. The outcome of this work is expected to provide an alternative means of handling leather wastes from tanneries through effective utilization of these wastes in the fabrication high temperature resistant composites and would also help to maintain a healthy environment.

## MATERIALS AND METHODS

### Materials

The waste chrome tanned leather shavings and trimmings were obtained from a footwear and leather manufacturing industry in Malaysia. These leather wastes were chopped into small pieces and pulverized into leather fibre (LF) using Fritsch Power Cutting Mill pulverizing machine. A commercial grade ABS polymer chips used as the matrix was obtained from Toray Plastics Malaysia Sdn. Bnd. Epoxy resin/hardener and Acetone (C<sub>3</sub>H<sub>6</sub>O) that were used for surface coating of the leather were supplied by Oriental Option Sdn. Bhd) and SYSTERM, respectively.

### Methods

#### Epoxy coating of leather fibres

The surface coating of the chrome tanned leather wastes (CLW) was done using epoxy/hardener at 2:1 volume ratio. The epoxy/hardener mixture was dissolved in acetone at a ratio of 5:1 (10 wt %) acetone to epoxy respectively. The delivered chrome tanned leathers wastes were chopped into small sizes (5 mm mesh size), washed with water, drained, neutralized and finally dried for 1 hr at 80 °C in an oven. The surface coating was done for 3 minutes after cooling with diluted resin thinned with acetone. This was followed by curing at 80 °C for 24 hr in an oven. Post curing was carried out for 48 hr at 40 °C before being used as fillers prior to composites fabrication.

#### DSC Analysis of coated leather

Differential Scanning Calorimetry (DSC) test was carried out using a NETZSCH DSC 200 F3 Maia model to study the thermal stability of the uncoated CLW and epoxy coated leather waste (ECLW) samples in accordance with ASTM D3418-82 standard on a heating run of 30 to 300 °C and crystallisation step from 300 °C down to 30 °C at 10 °C/min.

#### Melt processing and Composites fabrication

Compounding of the ABS polymer chips, CLW and ECLW were done with PRISM TSE SYSTEMS twin-screw extruder at different fibre loadings of 5, 10, 15 and 20 wt % (low fibre loading region). The processing parameters used were 5 minutes, 240 °C and 50 rpm for time, temperature and speed respectively. After the extruded and pelletized strands were dried in an oven for 3 hr at a temperature of 80 °C, it was formed into composite sheets (with thickness of 3 mm) using a compression moulding machine (Hung Ta Instrument, Taiwan) at temperature, pressure and time of 230 °C, 65 kg/cm<sup>2</sup> and 10 minutes (Preheating, hot pressing and cool pressing times were 5 min, 3 min and 2 minutes) respectively. The composite sheets were cut into appropriate dimensions for tensile and morphological characterisations.

### Characterization

#### FTIR Analysis of leather fibre

Coated (ECLW) and uncoated (CLW) leather fibres were subjected to FTIR analysis using FTIR spectrometer (Perkin Elmer Spectrum 400, model. Perkin Elmer Inc., USA) to study changes in functional groups of the short leather fibre samples.

The spectra were acquired over a frequency range of 4000 cm<sup>-1</sup> to 1000 cm<sup>-1</sup>.

### Tensile testing

Tensile analyses were performed on the samples with Universal Tester (SHIMADZU Autograph Precision, AG-X Series, Japan) at standard conditions of 50% relative humidity and 23 °C according to ASTM D 638 standard procedure. The test was performed on five specimens with dimension (length, width and thickness) of 150 mm x 13 mm x 3 mm respectively at 500 mm/min crosshead speed.

### Morphological analysis

#### Field emissions scanning electrons microscopy (FESEM)

The morphological studies of CLWABS and ECLWABS composite specimens were studied using ZEISS FE-SEM Germany model of field emission scanning electron microscopy for different magnifications. Sputter-coating was done to make the samples more conductive, using thin layer of platinum on a coating machine (Quorum model Q150RS, UK). The positioning of the samples was at 30° for better viewing.

#### Optical microscope

To study the fibre distribution, images of the uncoated (CLW) and coated (ECLW) leather fibres were taken with Olympus BX51TRF model of optical microscope at 10x magnification.

## RESULTS AND DISCUSSION

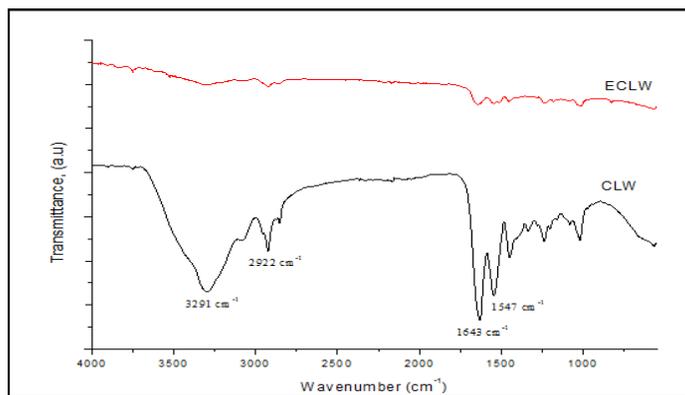
### Leather Fibre Analysis - FTIR

FTIR spectra of the uncoated leather (CLW) and epoxy coated leather wastes (ECLW) samples are shown in Figure 1. The two spectra show dissimilar peaks due to epoxy coating which led to changes in functional groups of the leather fibres. Wide bands at 3291 cm<sup>-1</sup> which are assigned to hydroxyl group can be seen in the CLW sample. Short and intense peak at 2922 cm<sup>-1</sup> is assigned to the =NH bonds asymmetrical stretching band in the CLW sample. The spectrum at 1643 cm<sup>-1</sup> of uncoated leather revealed the typical broad peak assigned to C=O group characteristic of protein fibres, while low intensity is observed with ECLW which may be considered as a proof of reduced absorption of water. The characteristic band at 1547 cm<sup>-1</sup> found in CLW showing the presence of carboxyl group is not found in ECLW.

The spectrum of raw leather fibres showed that uncoated leather fibre (CLW) is highly water soluble owing to its inherent high moisture absorption with high vibration peaks intensities as seen in Figure 1. There is a general reduction in peak intensities at 1547 cm<sup>-1</sup>, 1643 cm<sup>-1</sup>, 2922 cm<sup>-1</sup> and 3291 cm<sup>-1</sup> for the epoxy-coated leather fibres compared to CLW due to interaction between the epoxy resin and the functional group of the fibres. The hydrophobicity of the leather fibres has been improved and this will enhance the bonding of the epoxy chain segments and the leather fibres. This simply means that the water absorption characteristics of the fibre can be improved without adversely affecting the chemical composition of the fibres. Subsequently, the absorption peaks have also provided the sites for the cross-linking network between epoxy-coated fibres and the ABS chain in its constituent composites.

### Thermal analysis (DSC) of leather waste fibres

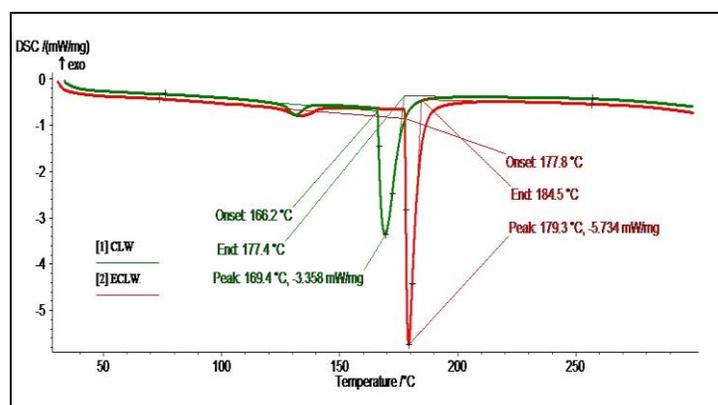
Figure 2 shows the effect of surface coating on thermal behaviour of uncoated (CLW) and epoxy coated-leather fibres (ECLW). From the DSC results, it was observed that uncoated leather fibres (CLW) generally had undergone chemical, structural and physical changes with change in temperature. This has resulted in the functional groups undergoing molecular orientation, phase transition, oxidation, dehydration and even decomposition. CLW showed reduced thermal stability because of degradation setting in at 166.2°C as against the epoxy coated chrome-tanned leather (ECLW) with onset temperature of 177.8°C. The reason for this behaviour could be the hydroxyl group in the fibre leading to increased moisture absorption. The endothermic melting (endset) temperature and melting peak for CLW and ECLW were respectively, 169.4°C and 177.4°C.



**Figure 1:** FTIR spectra of coated (ECLW) and uncoated (CLW) short waste leather fibres

The most thermally stable composite was found to be ECLW with melting and peak crystallization temperatures of 184.5°C and 179.3°C respectively. From the DSC results obtained, it is observed that ECLW fibres generally has higher thermal values as compared to uncoated CLW fibres which indicate that epoxy coating improved the thermal resistance and stability of the fibre

as compared to uncoated CLW fibres. The onset and melting peak temperatures of ECLW were improved by 7 % and 6 % respectively as compared to CLW fibres, which implies that surface coating of leather fibres with epoxy resin, can enhance the thermal properties thereby suggesting the possibility of being processed with high temperature polymers.



**Figure 2:** DSC thermograms of uncoated (CLW) and coated leather (ECLW) wastes.

### Tensile Properties

Figures 3 and 4 show the tensile strength and modulus of neat ABS, epoxy-coated and uncoated leather fibres filled ABS composites respectively. The tensile strength of neat ABS is 42.4 MPa and observed to have slightly decreased upon incorporation of leather fibres. The tensile strength of epoxy-coated ECLWABS composites was 36.9, 36.5, 35.2 and 32.9 MPa at fibre loadings of 5, 10, 15 and 20 wt% respectively, while the uncoated CLWABS composites has tensile strength values of 38.2, 34.1, 33.4 and 30.6 MPa at fibre loadings of 5, 10, 15 and 20 wt%, respectively.

From the results obtained in Figure 3, it is generally seen that the tensile strength of coated ECLWABS composites were higher as compared with uncoated CLWABS composites. This can be attributed to better interaction between the epoxy resin and the increased crosslink network chain on the leather fibre surfaces which have resulted to increased bonding for the epoxy matrix and leather fibres interface as confirmed by the SEM micrographs (Figure 6). However, the superior strength of ECLWABS composites decreased consistently with increasing fibre loading from 5-20 wt %. Maximum strength obtained within the low fibre loading region was at 5 wt % while 20 wt % yielded the lowest tensile strength and this might be attributed to fibre agglomeration caused by higher filler loading. The tensile strength results have indicated that an optimized fibre loading within the low fibre loading region is between the range of 5 wt % and 10 wt %.

The reasons for the low tensile strength of raw leather composites when compared with epoxy coated leather composites are likely due to the degradation of the fibres at elevated temperature

leading to reduced bonding between the natural leather fibre and ABS polymer matrix as was observed in our previous work (Owen *et al.*, 2018), an indication that a high processing temperature is critical for the leather fibres as it will be adversely affected resulting to loss of strength and decreased in properties of the resultant composites. Corradini *et al.* (2009) also mentioned decreased tensile strength and modulus on addition of bagasse sugarcane fibre, noting that it is a normal phenomenon for that behaviour especially when the fibre is a natural fibre and processed at high temperature (230°C). Talib *et al.* (2017) in their waste leather polyester composites reported reduced tensile properties with increase in fibre load from 1 wt % to 3 wt % due to severe filler agglomeration.

It is further observed that tensile modulus (Figure 4) also showed similar trend as that of tensile strength. Epoxy-coated composites gave higher modulus as compared to uncoated composites. Maximum modulus was found at 5 wt % and decreased with increasing fibre loading. Neat ABS has a tensile modulus of 2752.2 MPa which decrease upon incorporation of the fillers. The reduction in mechanical properties as the filler loading increases was also reported by Talib *et al.* However, there was improvement in the mechanical properties of the treated composites when compared to the untreated ones (Talib *et al.*, 2018).

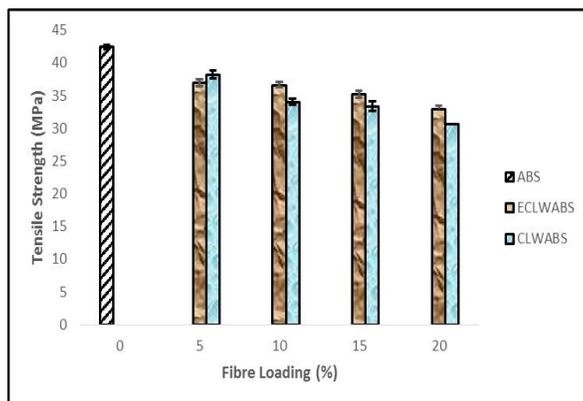
### Morphological Properties

#### Scanning Electron Microscopy (SEM)

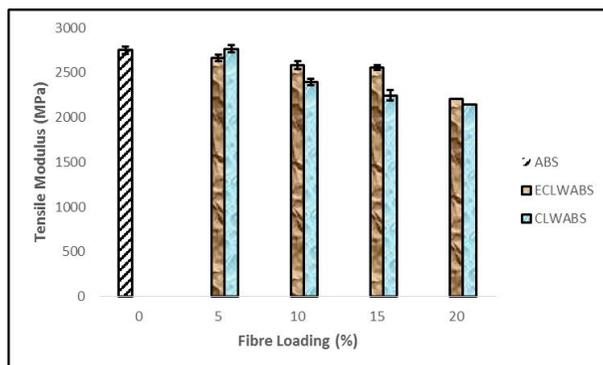
The SEM micrographs of neat ABS (Figure 5 (a-b)), uncoated CLWABS and epoxy coated chrome leather wastes filled acrylonitrile-butadiene-styrene ECLWABS (Figure 6 (a-f)

composites for different fibre loadings of 5, 10 and 15 wt %. The fractured micrographs of unfilled ABS matrix indicating the absence of reinforcing leather fibres is shown in Figure 5. Figure 6 (a, c and e) revealed evidences of fibre degradation at high temperature as the fibrous leather fibres were rarely visible in the composites system, there are evidences of fibre pullout and debonding at 10 and 15 wt % as a result of inadequate bonding between the ABS matrix and uncoated leather fibres (CLW). These might have been the cause of the inferior tensile properties of the composites with respect to the epoxy-coated leather composites (Figure 6 (b, d and f)). SEM micrograph of epoxy-coated leather composites, revealed an improved interface for the ABS matrix and the coated leather (ECLW). The fibrous structure

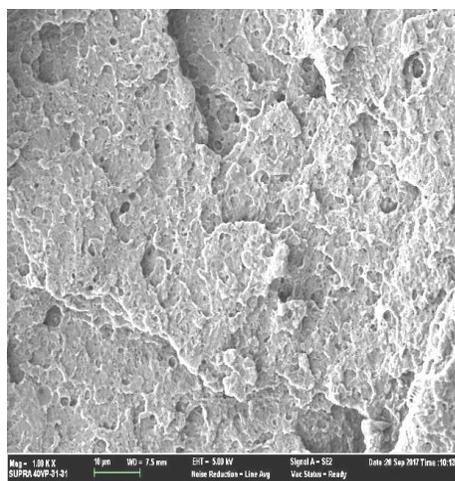
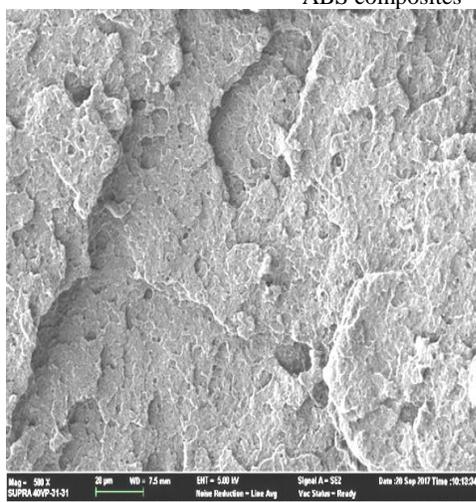
of the leather can be visibly seen, even after surface coating. The fibrous structure of the coated leather fibres are seen to be maintained without degradation. The epoxy-coated leather fibres ECLW was generally found to retain much of its fibrous structure to a major extent without degradation in the composites matrix. The epoxy-coated leather composites still retained its integrity at high temperature and have showed a strong fibre /matrix interfacial bonding preventing fibre/matrix catastrophic failure as shown for 5, 10 and 15 wt% loadings. This implies that epoxy coating has interacted well with the leather fibres thereby creating strong bonds. These might be the reason for the superior tensile properties of the epoxy coated leather composites.



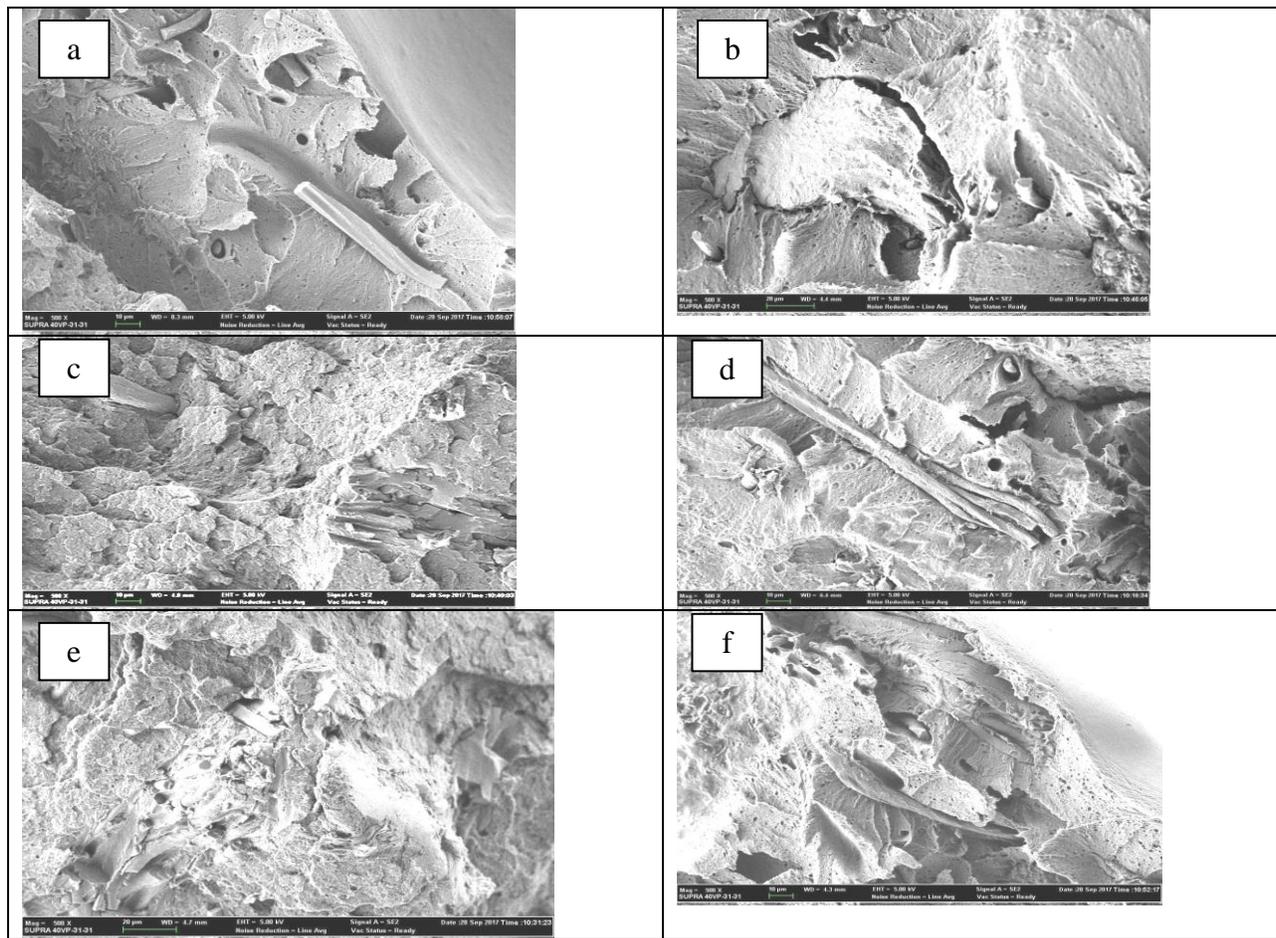
**Figure 3:** Effect of fibre loading and epoxy coating on the tensile strength of neat ABS, epoxy coated and uncoated chrome leather wastes filled ABS composites.



**Figure 4:** Effect of fibre loading and epoxy coating on the tensile modulus of neat ABS, epoxy coated and uncoated chrome leather wastes filled ABS composites



**Figure 5:** FESEM micrographs of fracture surface morphology of unfilled ABS at 500x and 1000x magnifications.

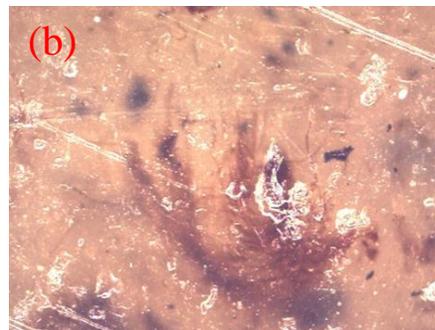


**Figure 6:** FESEM micrographs of uncoated (a, c and e) and epoxy coated (b, d and f) chrome leather wastes ABS composites at 5 wt % (a-b), 10 wt % (c-d) and 15 wt % (e-f) filler loadings.

### Optical Microscopy

The optical micrographs of fibre distribution for both ECLWABS and CLWABS composites are presented in Figure 7. In both cases, the result showed a good dispersion of fibrous structure of leather waste fillers in the ABS phase. This is an indication that

the leather fibres have fused well during melt mixing and compounding which could be linked to the effective interaction of the resin with the leather fibre that is spongy in nature. This must have played a very good part for the improved tensile properties.



**Figure 7:** Optical Micrographs of fibre distribution for (a) ECLWABS and (b) CLWABS composites

### CONCLUSION

The tensile properties of thermally-improved chrome-tanned waste leather fibre ABS composites were studied. It is found that:

- The tensile properties of epoxy coated leather fibre/ABS composites are superior to the uncoated leather composites; maximum strength was at 5 wt%, but the values are marginally lower than the unreinforced.
- FTIR spectra show changes in absorption characteristics of epoxy-coated leather waste (ECLW) which confirmed a successful coating and good interaction between the epoxy resin and the leather fibre.

- Thermal analysis also confirmed an improvement in thermal stability: onset and melting temperatures of 177.8 and 184.5°C for ECLW as against 166.2 and 169.4°C for CLW.
- FESEM micrographs showed strong bonding at the coated leather fibre and ABS matrix interface with reduced fibre degradation.

Natural leather fibres of animal origin can therefore be processed at slightly higher temperatures if surface coated with epoxy resin leading to the development of valuable composite for engineering applications especially where tensile strength is of paramount importance. This work has potential application in the reduction of solid wastes from footwear and leather processing industries.

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