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Improving the thermal stability and impact strength of leather wastes-ABS composites via robust experimental design

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- ✓ *Thermal stability.*

Abstract

The epoxy-coated and uncoated chrome-tanned leather wastes-ABS composites were fabricated using a twin-screw extruder and compression moulding machines processed at temperature of 240 °C at different fibre loadings of 5, 10, 15 and 20 wt. %. Surface morphology, impact strength and thermal properties were investigated on both the uncoated and epoxy-coated chrome-tanned leather waste-ABS composite samples, using field emission scanning electron microscope (FESEM), drop dart impact tester and differential scanning calorimeter (DSC), respectively. The obtained results showed that the epoxy-coated composites exhibited highest impact strength of 0.126 KN/mm^2 and better thermal stability with glass transition and melting temperatures of 75.8 and 256 $^{\circ}$C, respectively, at 5 wt.% filler loading. This implies that the epoxy-coated chrome-tanned leather waste-ABS composites was ductility over a wide range of temperature, and thus could be utilized for robust engineering application where high impact strength and thermal stability properties are required.

1. Introduction

In order to find a lasting solution to the problem of agricultural wastes, their use as fillers for preparation of polymer composites has received tremendous attention. Of all the wastes from natural origin, those from plant resources have witnessed extensive use, mainly because of their volume. Thus, several studies have reported on the use of plant fibres in polymer composites. Among the studies reported so far on natural-fibre reinforced high temperature engineering plastics include empty fruit bunch fibre/recycled poly(ethylene terephthalate) [1]; date palm leaf fibre/ recycled poly(ethylene terephthalate) [2], PET/Hemp [3, 4], wood flour with high temperature thermoplastic poly (phenylene ether) (PPE) polymers [5], Jute fabric/polyamide [6] and poly (acrylonitrile-butadiene-styrene)/kenaf [7]. In all these, the observed major drawback of natural fibres which is their low thermal processing temperatures that border around 200 °C has become a matter of concern. Hence, other natural fibres such as leather wastes are being considered.

Leather is processed animal skin or hides whose main constituent is collagen. Basic collagen structure consists of twined triple units of peptide chains and the triple helices are held together by hydrogen bonds [8]. Large quantity of leather wastes generated from tanning industries are mostly disposed in landfills or discharged into the natural water bodies without any treatment, causing environmental problems [9]. Chrome-tanned leather waste is one of the wastes of leather tanneries. Under certain pH and temperature conditions, Cr (III) can undergo valence changes to Cr (VI) which is a highly toxic and carcinogenic and undergoes high water solubility, hence, mobile. In contrast, Cr(III) is insoluble and possess essential nutrient which serves as mineral supplement [10-12].

In the industry, leathers are tanned with chromium, and thus chrome-tanned leathers have shown improved physical properties in addition to chemical and biological resistances. However, the process leads to emission of solid and liquid wastes into the environment [13]. Furthermore, during leather processing, operations such as trimming, shaving and cutting often result to more than 50 % of the raw hide being rejected as wastes. For example, processing of one ton wet salted hide yields only 200 kg of leather with over 600 kg being rejected as solid wastes [14]. Of the total wastes, more than 50 % is considered as potentially hazardous because of the presence of chromium. As a result, there is much concerns on environmental waste problem caused by chrome-tanned leather wastes which find their destinations in the oceans, agricultural soils and atmosphere when incinerated [15

Among the several techniques which have been tried out to address this problem, it was discovered that utilization of leather wastes as fillers in polymer composite preparation has demonstrated to be benign and more sustainable. For example, Senthil *et al.* [16] reported the successful preparation and application of epoxy/leather fibre composites. Ramaraj [15] also reported the excellent performance of mechanical and thermal properties of leather waste/acrylonitrile–butadiene–styrene (ABS) composites. In a related study, Mohamed *et al.* [17] reported that addition of leather waste into polyamide resulted in enhancement of physical properties of the polymer matrix. Furthermore, Babanas *et al.* [18] and Andreopoulos and Tarantili[19] had demonstrated that chromium treated finished waste leather was useful as filler material for PVC and therefore showed improved wear resistance and hardness.

More so, Ambrósio *et al.* [20] had reported increase in elastic modulus and reduction in tensile strength of PVB/leather fibre composites with increasing leather fibre content. In their own study, Salwa *et al.* [21] showed improved mechanical properties for acrylonitrile butadiene rubber (NBR) composite loaded with treated leather wastes. Madera-Santana *et al.* [22] produced Leather-like composites using short leather fibres. Joseph *et al.* [23] developed waste leather buff filled polycaprolactone (PCL) biocomposites which could be used to develop low cost materials suitable for applications in footwear, bags and suitcase industries. Ambone *et al.* [10] developed eco-biocomposites using waste leather buff (WLB) as filler in polylactic acid (PLA) matrix aimed at reducing the environmental issues and providing sustainable solution. The reason for the observed decrease in mechanical properties was reported to be due to poor wetting and dispersion of the fibres within the matrix.

In another study, leather waste from footwear industry was utilized as filler in unsaturated polyester composite (UPC) by Satariah Talib *et al.* [24]. It was found that the leather waste filled composites showed lower tensile properties than the unfilled composite. Satariah Talib *et al.* [25] also investigated the effect of filler loading on the flexural and compressive properties of untreated and treated chrome-tanned leather waste (CTLW) short fibre filled unsaturated polyester composites. Their results showed that the mechanical properties of the composite reduced as the amount of CTLW increased. Dalita *et al.* [26] developed a novel composite material from natural rubber and leather waste for use in the textile and footwear industries, while El-Sabbagh *et al.* [27] reported the recycling of chrome-tanned leather waste in acrylonitrile butadiene rubber. Their observations showed similar trend of decreasing composite properties.

Despite all these research advances in the preparation of leather wastes-polymer composites, epoxy-coating of chrome-tanned leather for composite preparation with ABS has not yet been reported, to the best of our knowledge. Thus, the present study seeks to develop robust leather wastes fibres-acrylonitrile butadiene styrene (ABS) composites via epoxy-coating of chrome-tanned leather wastes in order to enhance the thermal properties and achieve high impact strength performance. The idea to first coat the leather wastes with epoxy resin in order to enhance its thermal properties was derived from works of Thitithanasarn *et al.* [6] and Jana and Prieto [28] who pre-coated the fibres used in the preparation of their composites to improve the thermal resistance. Furthermore, Saliu *et al.* [7] incorporated epoxy-coated sodium hydroxide treated kenaf fibres into thermoplastics (ABS) at processing temperature of 230 °C and dynamically cured epoxy at high temperature (80 °C) in order to prevent degradation. Hence, ABS was selected because of its high melting temperature property against most of the thermoplastic polymers (such as PE, PP, etc) used for composites preparation which are limited by their lower melting temperatures [29].

2. Methodology

2.1 Materials

A commercial grade ABS in chip form was obtained from Toray Plastics Malaysia Sdn. Bnd. The chrome-tanned leather wastes (CLW) was collected from a leather manufacturing industry in Malaysia. Epoxy resin/hardener was supplied by Oriental Option Sdn. Bhd.), Acetone (C_3H_6O) was supplied by SYSTERM.

2.2 Methods

2.2.1 Epoxy-surface coating of leather fibres

The epoxy resin and hardener mixed in the ratio of 2:1 was used for surface-coating of the chrome-tanned leather wastes (CLW). The epoxy/hardener mixture was dissolved in acetone at an optimised ratio of 1:5 (10 wt %) epoxy to acetone respectively. The chrome-tanned leather wastes were neutralized and surface-coated with dilute epoxy resin in solution of acetone for 3 minutes, cured at a temperature of 80 °C for 24 hours in an air-circulated oven and then pulverized into short leather fibres using a pulverizing machine (Fritsch Power Cutting Mill Pulverisette 15) with 5 mm mesh size and oven-dried in the laboratory at 40 °C for 48 hours before being used as fillers prior to composites fabrication.

2.2.2 Melt processing and composites preparation

ABS chips, CLW and epoxy-coated chrome-tanned leather (ECLW) were dried under vacuum at 80 °C for 12 hours before compounded using a twin-screw extruder (PRISM TSE SYSTEMS 2094, UK) at different fibre loadings of 5, 10, 15 and 20 wt %, and at an optimised processing temperature, time and speed of 240 °C, 5 minutes and 50 rpm respectively. The extruded strands were pelletized, oven-dried at 80 °C for 3 hours, and compression moulded into flat sheets (3 mm thickness) using a compression moulding machine (Hung Ta Instrument, Taiwan) at temperature, pressure and time of 240 °C, 65kg/cm^2 and 5 minutes, respectively. The composite sheets were press-cut into appropriate dimensions for mechanical testing and surface morphological examination.

2.3 Characterization

2.3.1 Impact Testing

Drop dart impact test was done using impact machine type INSTRON dynatup (model 9250HV USA) impact tester based on ISO 179 and 180 standards. Five replicate samples of 5 x 5 x 3 mm dimensions each were tested for all the composite samples. The results recorded are average values of the five tests.

2.3.2 Thermal analysis by DSC

Differential scanning calorimeter (DSC) (NETZSCH DSC 200 F3 Maia Model Germany) was used to determine the thermal behaviour and stability of the neat ABS, uncoated and epoxy-coated chrome-tanned leather wastes-ABS composites via glass transition temperature (T_g), melting temperature (T_m) and cold crystallization temperature (T_{cc}) in accordance with ASTM D3418-82 standard. A heating run was performed from 30 to 300 °C at a rate of 10 °C/min. and crystallisation step from 300 °C down to 30 °C at 10 °C/min. The data analysis was done with in-built Proteus analysis software.

2.3.3 Scanning Electron Microscopy

The morphological study of the impact fractured surfaces of the composites was carried out using Field emission scanning electron microscopy (FESEM ZEISS FE-SEM SUPRA 40VP. Germany). The samples were sputter-coated with a thin layer of platinum using a Quorum Sputter-Coater machine (Quorum model Q150RS, UK). The micrographs were taken at 500x and 1000x magnifications.

3. Results and Discussion

3.1 Impact test

Impact properties of uncoated CLWABS and epoxy-coated ECLWABS composites were studied. The effect of leather fibre content and epoxy-coating on the impact strength of ABS is shown in Figure 1. The results showed that CLW had some significant effect on the strength of ABS. The impact strength of neat ABS was observed to be 0.056 KN/mm^2. When compared with the neat ABS, the impact strengths of the uncoated CLWABS and epoxy-coated ECLWABS composites were observed to be higher. This was attributed to presence of chrome-tanned leather wastes.

For the uncoated CLWABS composites, the impact strength was observed to be highest (0.102 KN/mm^2) with 5 wt. % fibre loading, and decreased with increasing fibre loading as depicted in Figure 1. The low impact strength found with CLWABS was attributed to weak interfacial bonding between the leather fibres and ABS matrix as a result of fibre degradation in the composite system at high processing temperature (240 °C). The epoxy-coated ECLWABS composites showed highest impact strength of 0.126 KN/mm^2 at 5 wt. % fibre loading, followed by 10 wt. % and further decreased with increasing fibre loading. Comparatively, the impact strengths of the ECLWABS composites were observed to be higher than those of the uncoated CLWABS composites. This is due to the surface-coating of the leather waste fibres with epoxy resin which bound the fibres together and thus enhanced their mechanical strength.

Overall, the fact that the impact strength of the epoxy-coated ECLWABS composites were higher than those of the uncoated could further be explained on the basis of uniform dispersion and distribution of the leather fibres in the composite system. At low filler loading (5 wt. %), the CLW was well dispersed as revealed by the SEM micrographs and efficient stress transfer occurred from matrix to fillers. Above 5 wt. % filler content, CLW fillers started to aggregate, developing numerous points of stress concentration. Thus, crack initiation started from these points and created openings for inlet of degradants. Hence, the mechanical properties were affected and decreased with increasing fibre loading which created more stress concentration points when not well dispersed. This result is consistent with observations of [17, 30-32].

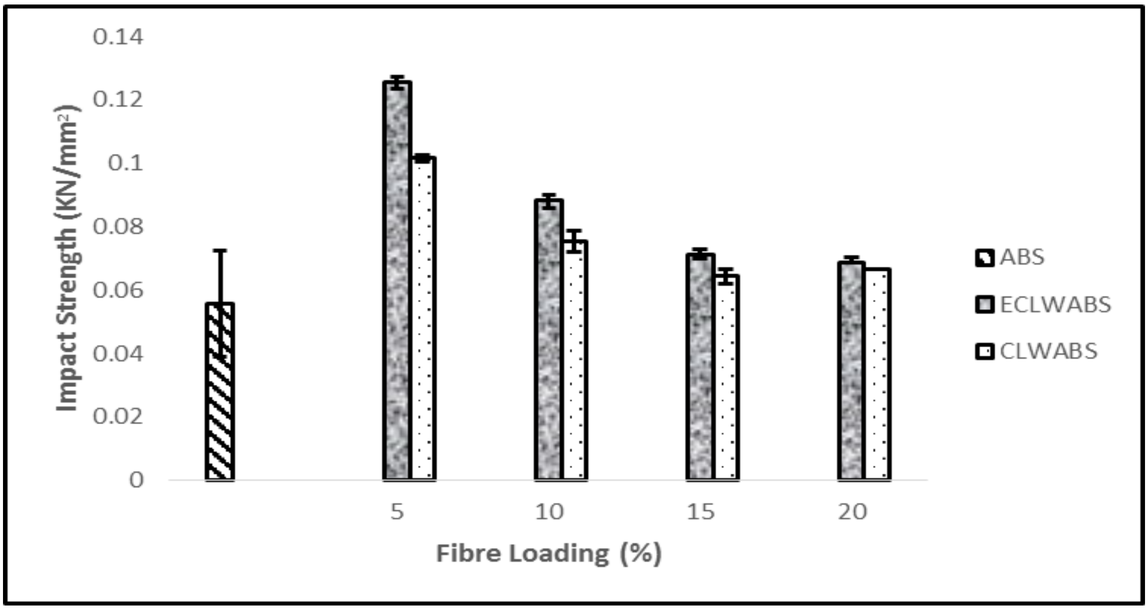

Figure 1: Effect of epoxy coating and fibre loading on the impact strength of ABS, epoxy coated and uncoated chrome tanned leather wastes short fibre filled ABS composites.

Figure 2 shows the results for the impact absorbed energy of neat ABS matrix, uncoated CLWABS and epoxy-coated ECLWABS composites at different fibre loadings. The impact maximum absorbed energy of neat ABS was found to be 1.93 J and those of uncoated CLWABS and epoxy-coated ECLWABS composites were observed to be 5.32 J and 6.39 J at 5 wt% loadings, respectively. This indicates that ABS matrix absorbed less energy as compared to uncoated and epoxy-coated ECLWABS composites. The CLWABS composites have less ability to absorb impact energy as compared to epoxy-coated ECLWABS composites due to weak interfacial bonding between the uncoated leather waste fibres and ABS matrix. The epoxy-coated ECLWABS composites were found to absorb highest energy at 5 wt. % fibre loading, and decreased with increasing filler loading.

3.2 Differential Scanning Calorimeter (DSC)

The thermal properties of ABS, uncoated CLWABS and epoxy-coated ECLWABS composites at different filler loadings are illustrated in Figure 3 and Figure 4. Figure 3 shows the thermograms for the neat ABS and uncoated CLWABS composites, whereas the thermograms for epoxy-coated chrome-tanned leather waste-ABS composites are depicted in Figure 4. The extracted values of glass transition temperature, $T_{g,}$ melting temperature, T_m, and crystallization temperature, T_{cc} of neat ABS matrix, uncoated CLWABS and epoxy-coated ECLWABS composites at different fibre loadings are given in Table 1. The results show that neat ABS recorded a T_g of 95.4 °C, T_m of 214.1 °C and T_{cc} of 227.7 °C, respectively. The T_g of uncoated CLWABS composites were observed to be 230.1 °C, 123.5 °C, 246.5 °C and 234.5 °C at 5, 10, 15 and 20 wt.% fibre loadings, respectively. While the T_m were found to be 256.0 °C , 256.4 °C , 247.2 °C and 236.5 °C at 5, 10, 15 and 20 wt.% fibre loadings, respectively.

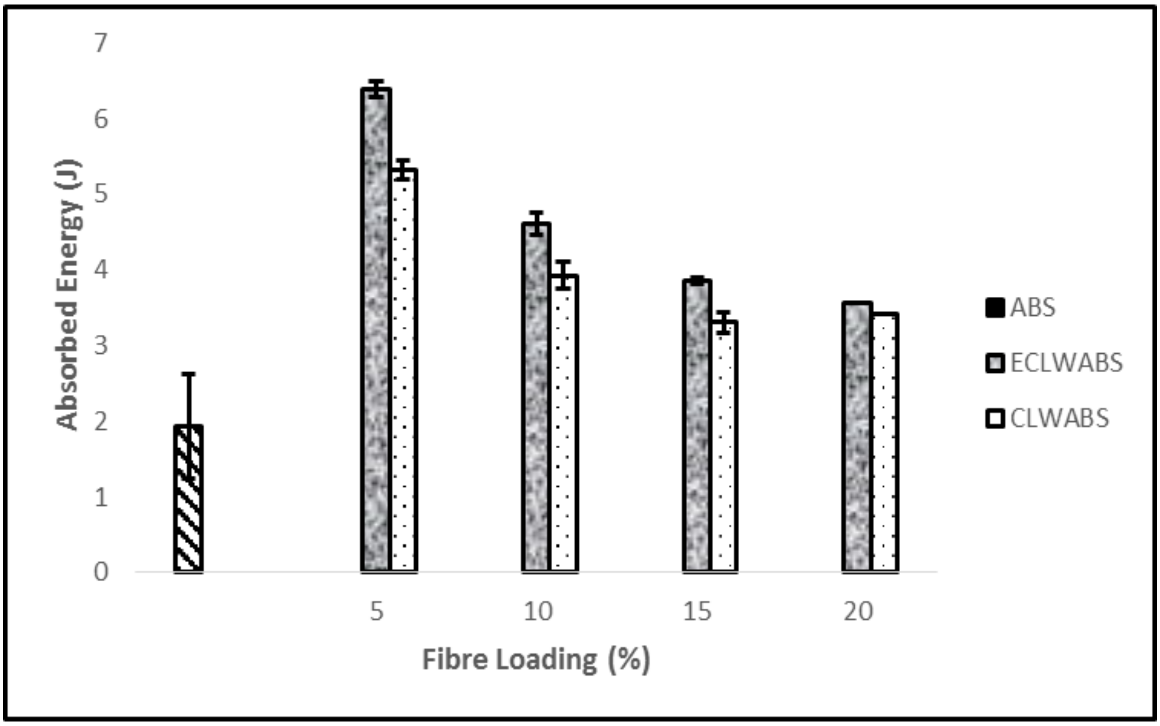

Figure 2: Effect of epoxy coating and fibre loading on the absorbed energy of epoxy coated and uncoated chrome leather wastes short fibre filled ABS composites.

When the T_g values of the neat ABS and uncoated leather waste-ABS composites were compared to those of the epoxy-coated chrome-tanned leather waste-ABS composites (ECLWABS) listed in Table 2, T_g values of the ECLWABS composites were observed to be lower than the former. Further observation of Table 1 and table 2 revealed that the melting temperatures (T_m) of both the uncoated and epoxy-coated chrome-tanned leather waste-ABS composites were not significantly different. However, the T_m values of both composites were higher than that of the neat ABS. On the other hand, the crystallization temperatures (T_{cc}) of the composites were affected but without any definite order.

From the results, it could be observed that both T_g and T_m of ABS increased with the incorporation of the fibres which further increased with increase in fibre loading, an indication of compatibility between the fibres and the ABS matrix, and shows that the addition of CLW fibres have significantly affected the T_g of the resultant CLWABS composites. However, T_m of the composites increased marginally with increasing fibre loading up to 10 wt % and decreased from 15 to 20 wt %. The highest melting temperature values were observed to be about 256 °C at filler loading of 5 wt. % and 10 wt. %, indicating that the optimum processing temperature is 250 °C which could be obtained with filler loading of 5 wt. % or 10 wt. %.

Similar observation was reported in our previous work [35] on epoxy coated kenaf fibre reinforced recycled PET in which the values of T_m increased with increase in filler loading up to 10 wt. % and a decreased as the filler loading increases to 15 wt%. The decreased in T_m at high fibre loading of 20 wt % suggests that the CLW may have reduced the cohesive force of attraction between ABS polymer chains thereby affecting the segmental mobility and consequently resulting in slight decrease in T_g and T_m as observed. In addition, the observed decrease in T_m is because of the increasing filler loading of CLW in ABS matrix component which may have reduced the free volume of the ABS phase, thus restricting the growth of ABS spherulite and reducing the high amorphous nature of ABS component as also reported by Paschoal *et al.* [12]. They concluded that leather fibre is amorphous in nature and the presence of leather fibre retarded the crystal growth of lamellar crystallites of PCL matrix and reduced the crystallinity which further decreased with increase in fibre content.

The fact that the epoxy-coated chrome-tanned leather waste-ABS composites exhibited lower T_g values than both the neat ABS and uncoated composites suggest that at service temperature above 75.8 ^{o}C and below about 256 ^{o}C, the epoxy-coated chrome-tanned leather waste-ABS composites would be ductile and display high impact toughness. Whereas within these temperature regimes, the uncoated composites will display high tendency for brittleness and subsequent failure. On the other hand, the T_g values of the uncoated composites suggest that at operating temperature above 230 ^{o}C and below about 256 ^{o}C, the composites would be ductile and exhibit high impact strength. Comparison of the two classes of composites indicate that the epoxy-coated chrome-tanned leather waste-ABS composites exhibited ductility over a wide range of temperature, and thus could be utilized for robust application where high impact is required. As for the uncoated composites, the result demonstrated ductility within a short range of temperature, and thus indicates that they will not be fit for robust applications.

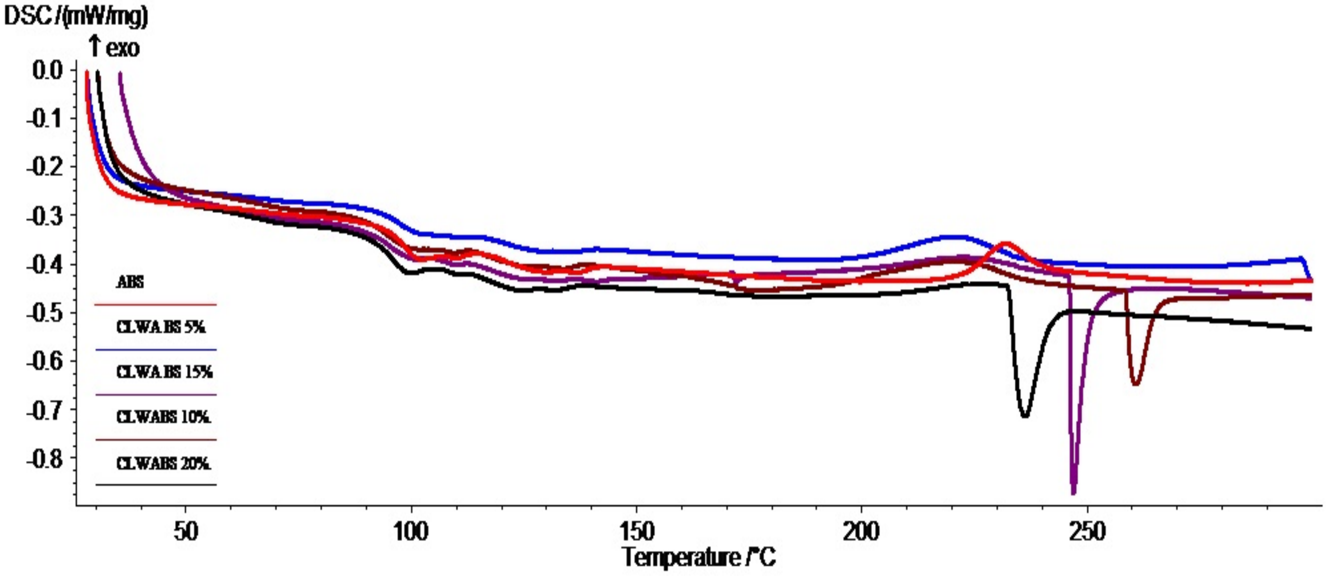

Figure 3: DSC thermograms of ABS and chrome tanned leather wastes (ECLW) at 5 -20 wt% filler loadings.

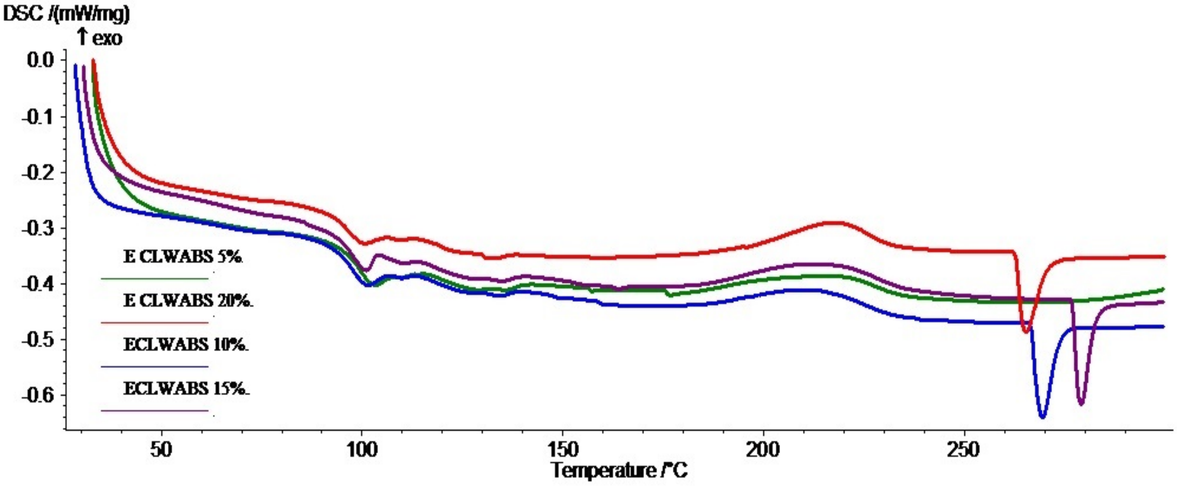

Figure 4: DSC thermograms of epoxy-coated chrome-tanned leather wastes (ECLWABS) composites at 5 -20 wt% filler loadings

Table 1: Summary of DSC data for neat ABS and uncoated leather waste-ABS composites

Sample	Onset Temp T_g (°C)	Melting Peak T_m (°C)	(T_{cc}) Temp (°C)
ABS	95.4	214.1	227.7
CLWABS 5wt %	230.1	256.0	205.2
CLWABS 10wt %	123.5	256.4	194.1
CLWABS 15wt %	246.5	247.2	257.5
CLWABS 20wt %	234.5	236.5	242.0

Table 2: Summary of DSC data for epoxy-surface chrome-tanned leather waste-ABS composites

Sample	Onset Temp T_g (°C)	Melting Peak T_m (°C)	(T_{cc}) Temp (°C)
ECLWABS 5wt %	75.8	257.1	211.8
ECLWABS 10wt %	75.9	257.2	211.9
ECLWABS 15wt %	72.0	256.4	210.3
ECLWABS 20wt %	73.8	256.8	211.0

It was also observed from the DSC results in Figure 3, Figure 4 and Table 1 as well as Table 2 that the thermal transition temperatures of the CLWABS composites were generally higher as compared to ABS and further increased with increase in filler loading up to 10 wt %, which is an indication of high compatibility between the leather fibres and the matrix. Satariah Talib *et al.* [24] found a decreased in glass transition temperature with increase in filler loading from 1-3 wt % with leather waste unsaturated polyester composites. However, a decrease in cold crystallisation temperature T_{cc} of the composites was found at 5 wt % and 10 wt % with values of 205.2 °C and 194.1°C respectively. The T_{cc} values increased with increasing filler content up to 20 wt% which indicates that T_{cc} of leather fibre ABS composites exhibited higher degree of amorphous regions at high filler content. Elamri *et al.,* [33] also reported an increased in the values of the cold crystallisation temperature T_{cc} of composites with increasing filler content. The results obtained in this study also indicate that fibre loading has a significant influence in thermal behaviour of the resultant composites. Khan *et al.* [34] also found that fibre loading greatly influenced thermal stability of the resulting composites in which an excellent thermal property was obtained at 50 wt. % fibre loading.

3.3 Scanning Electrons Microscopy of ABS, CLW/ABS and ECLW/ABS Composites

After the impact fracture analysis of the neat ABS, uncoated CLWABS and epoxy-coated chrome-tanned leather waste-ABS composites, the FESEM investigation was conducted on the samples to examine the fractured surfaces in order to understand the interplay between the fibres and the matrix (ABS). Thus, Figure 5 shows the FESEM micrograph of the fractured surface of unfilled ABS matrix. Observation of the micrograph at both lower magnification (Figure 5(a)) and higher magnification (Figure 5(b)) shows lack of cohesion due to absence of reinforcing leather waste fibres in the matrix. Figure 6 shows the FESEM micrographs of uncoated leather waste CLWABS composites at (a) 5 wt. %, (b) 10 wt. %, (c) 15 wt. % and (d) 20 wt. % fibre loading. When compared to Figure 5, Figure 6 micrographs revealed presence of fibre strands. Further observation of Figure 6 clearly shows the fibres pulling out of the matrix, which is an indication of matrix-fibre detachment/debonding. This is an evidence of poor interfacial adhesion of the raw leather waste fibres (CLW) with the ABS matrix, and therefore possess the propensity for quick degradation at high temperature. This observation corroborates the T_g results stated earlier.

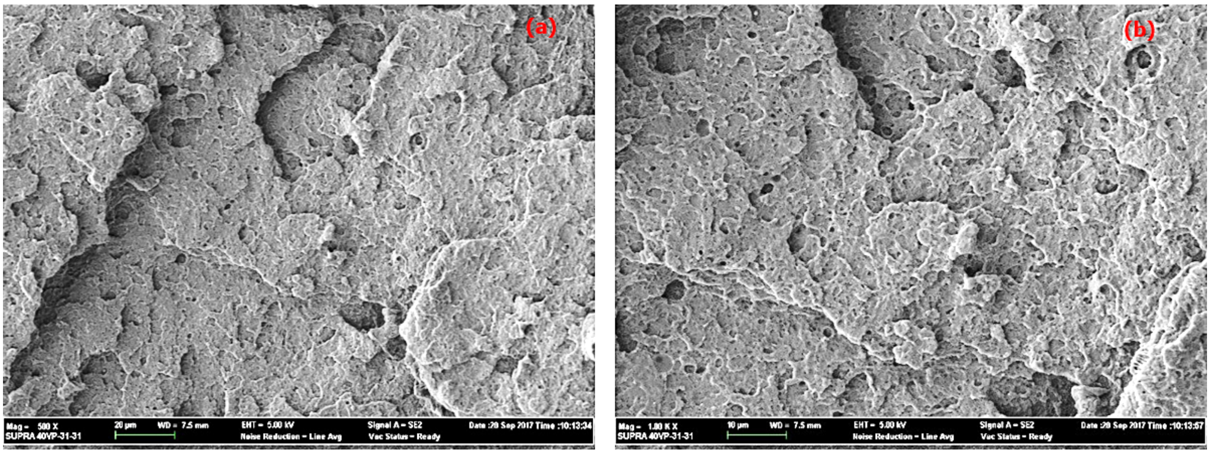

Figure 5: FESEM micrograph of fracture surface morphology of unfilled ABS at (a) 500x and (b) 1000x magnifications.

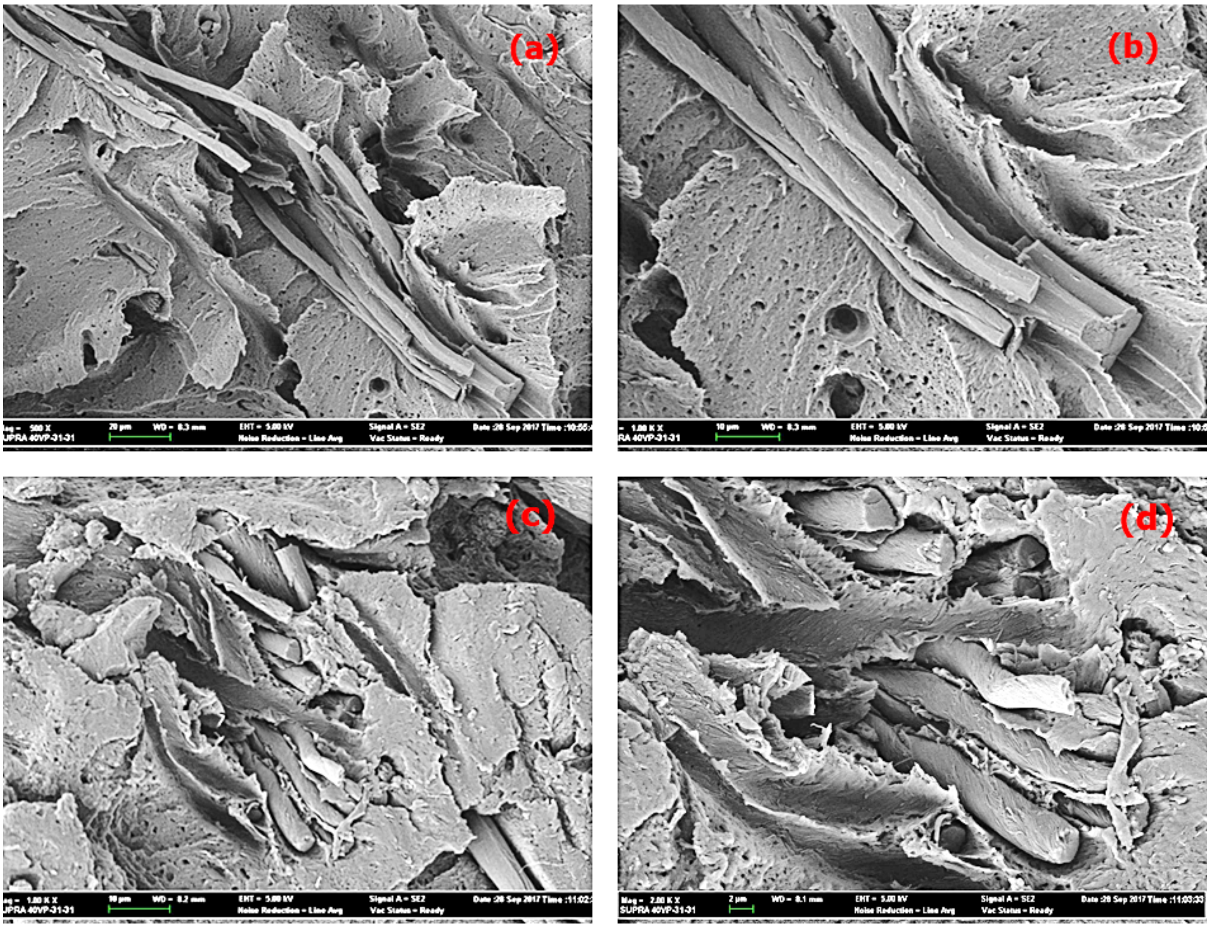

Figure 6: FESEM micrograph of fracture surface morphology of uncoated chrome leather wastes filled ABS composites at 500x and 1000x magnifications.

Further, the reason for the observed poor interfacial adhesion could be attributed to inability of the fibres to appropriately wet the ABS matrix, due to non-polar nature of the ABS matrix, hence, the weakness of the interfacial bond. Thus, the observed low mechanical behaviour of the CLWABS composites shown in Figure 1 and Figure 2 could be attributed to this poor interfacial bonding.

Interestingly, Tan *et al.* [1] also reported similar observations with untreated empty fruit bunch fibre reinforced unsaturated polyester resin synthesized from recycled polyethylene terephthalate (PET). Figures 7 shows the FESEM of fractured surface of epoxy-coated ECLWABS composites at (a) 5 wt. %, (b) 10 wt. %, (c) 15 wt. % and (d) 20 wt. % fibre loading. It can be observed that there is a clear evidence of cohesive failure of the composites due to strong interfacial bonding between ECLW and ABS, resulting from the cross-linked interaction of epoxy with the –OH groups of the leather fibre and the chemical bonding with the matrix ABS polymer in formation of strong interface. There are also evidences of the fillers retaining the integrity with non-existence of fibre degradation in the composite system. This has ensured proper load transfer from the matrix to the fillers and subsequently improved the impact performance compared to CLWABS composites. Jana and Prieto [28] also made similar observations on the effect of coupling agents on morphology of epoxy coating on wood flour particles filled poly phenylene ether composites which resulted in an improved adhesion. This result suggests that interfacial adhesion between the epoxy coated leather fibres and the ABS matrix are much more favourable and stronger upon surface coating of the fibres with epoxy before incorporation in the polymer matrix. The observed results are consistent with our previous work [35] and the report of Tan et al., [1].

The above morphological interpretations are in accordance with the impact properties of the composites at 5 wt% of CLWABS and ECLWABS composites. The fillers were well dispersed in the matrix system and showed improvement in impact properties, and as the filler loading increased up to 20 wt %, the fillers agglomerate and served as crack initiators which subsequently declined the impact properties.

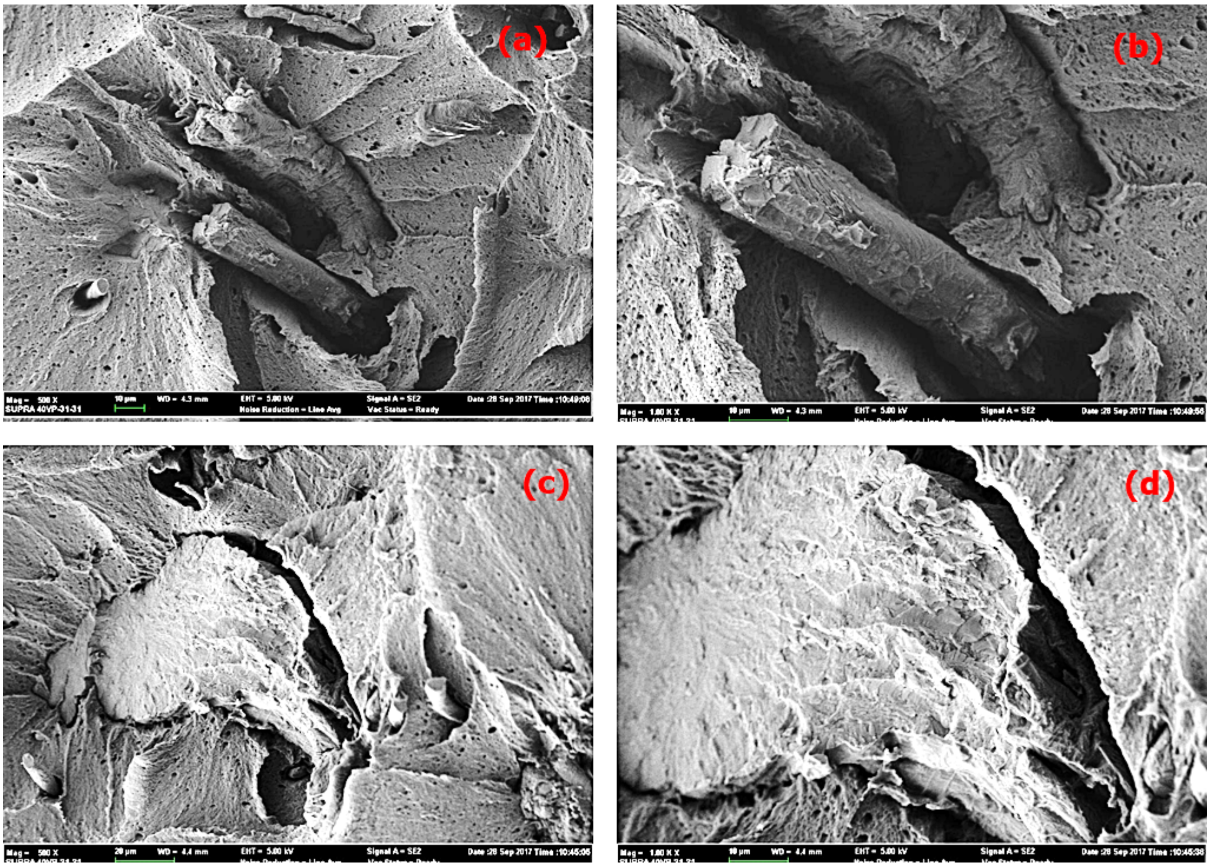

Figure 7: FESEM micrograph of fracture surface morphology of epoxy coated chrome-tanned leather wastes filled ABS composites.

Conclusions

The given study was carried out to investigate the use of recycled chrome-tanned leather wastes (CLW) as fillers in high temperature engineering ABS polymer composites and our findings have shown that CLW are potential fillers for ABS and the filler loading should not exceed 5 wt%. The study on thermal and impact strength analyses showed that ECLWABS gave an improved thermal stability and better impact properties as compared to CLWABS composites indicating that natural fibres of animal origin can be processed with high temperature polymers above its degradation temperature if surface-coated with epoxy resin. The study further has shown that the developed leather waste fibre filled ABS composites could provide solution to the environmental problems associated with waste management of the leather industry. Our results have demonstrated that the high impact and thermal stable epoxy-coated chrome-tanned leather waste-ABS composites obtained from this study could find applications in load-bearing engineering applications.

Conflict of interest-Authors declare there are no competing interests regarding the publication of this article.

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