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Technical report

The mechanical properties, deformation and thermomechanical properties of alkali treated and untreated Agave continuous fibre reinforced epoxy composites

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ABSTRACT

The mechanical properties such as tensile, compressive, flexural, impact strength and water absorption of the alkali treated continuous Agave fibre reinforced epoxy composite (TCEC) and untreated continuous Agave fibre reinforced epoxy composite (UTCEC) were analysed. A comparison of the surfaces of TCEC and UTCEC composites was carried out by dynamic mechanical analysis (DMA), Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM). The thermomechanical properties of the composite reinforced with sodium hydroxide (NaOH) treated Agave fibres were considerably good as the shrinkage of the fibre during alkali treatment had facilitated more points of fibre resin interface. The SEM micrograph and FTIR spectra of the impact fracture surfaces of TCEC clearly demonstrate the better interfacial adhesion between fibre and the matrix. In both analyses the TCEC gave good performance than UTCEC and, thus, there is a scope for its application in light weight manufacture in future.

1. Introduction

In the recent years, due to environmental concerns developed, the nature fibre composites have shown a growth of interest because of their recyclability and biodegradability. Natural fibres can be obtained at a low price using locally available manual labor and eco friendly material in nature. Natural fibres are readily available in large quantities in many countries and they represent a continuous renewable source [1]. The specific properties of natural fibre composite are such as light weight, low cost, renewable in nature, high specific strength and modulus [2,3]. A major advantage is that they can be easily disposed of at the end of their life cycle by compositing or by recovery of their calorific value in a furnace, which is not possible in glass fibre [1,3]. However, natural fibres also exhibit some undesirable characteristics such as high moisture absorption and low thermal resistance [4]. The mechanical, physical and chemical properties of plant fibres are strongly harvest dependent, influenced by climate, location, soil characteristics, weather circumstances, etc. In addition, the properties are affected by fibre processing (retting, scotching, bleaching, spinning, etc.) and by their incorporation into composites: handling, impregnation and consolidation will introduce supplementary changes [5].

Different types of natural fibres such as flax, jute, sisal, banana, hemp, ramie, coir, pineapple leaf and others as possible reinforcement have been investigated [6–16]. The mechanical and other physical properties of natural fibres are influenced by their growing conditions, fibre processing technique and, as for other fibre types, by the fineness of the fibre and sample test length. The mechanical properties of jute fibre reinforced epoxy composites, as a result of optimization of the properties of raw, alkali treated jute fibre composites, and also shrinkage of the fibres during treatment had significant effects on the structure [17].

The properties of composites depend on the matrix, fibres, and on their interfacial bonding. The adhesion between the reinforcing fibres and the matrix in composite materials plays an important role in the materials [1]. When natural fibre is used as reinforcement in composite materials, many problems occur at the interface due to incompatibility [18]. Therefore, surface modification of the natural fibres by means of some treatments is one of the major areas of current research to develop compatibility and interfacial bond strength. The surface of natural fibres is influenced by the fibre morphology, chemicals used in treatment and processing conditions [19]. Alkali and acetylation treatment process are cost effective and have been widely used to improve natural fibre surface properties [20]. In a study by Herrera Franco et al. [21,22], the surface treatment of henequen fibre with an alkali and a silane coupling agent almost doubled the interface strength by changing the mechanical interaction and the chemical interaction between fibre and matrix. The tensile strength of the high density polyethylene (HDPE) sand composite does not seem to be affected by the processing temperature, but tensile modulus shows similar behavior for filler content.

Alawar et al. [23] investigated the effect of different treatment process on the data palm fibre (DPF) and results showed that the

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treatment conditions of 1% NaOH for 1 h at 100 °C is the optimum treatment that gives the maximum tensile strength. An alkaline treatment was used to enhance both the matrix fibre wetting and the chemical surface modification in order to improve the physicochemical interaction at the fibre matrix interface [24]. Xue et al. [25] investigated experimentally and numerically newly developed aspen fibre polypropylene composites at the temperature and humidity conditions and the result shown that the tensile moduli, flexural moduli and the flexural strength increased as the wood fibre content increased in the composites. Biocomposites from kenaf fibre and soy based bioplastic were fabricated by extrusion, followed by compression moulding and their impact of fibre length and the processing method on the thermal and mechanical properties of the composites were characterized with dynamic mechanical analysis and mechanical properties measurements [26]. Singleton [27] studied the natural flax fibre, recycled high density polyethylene composite was manufactured by a hand-lay up, and compression moulding technique and experiment carried out under tensile and impact loading. Dynamic mechanical behaviour of natural rubber and its composites reinforced with short coir fibres has been studied by Geethamma et al. [28]. It's observed that as frequency increases the values of tan δ (loss tangent or damping factor) and E'' (loss modulus) decreases whereas the value of E' (storage modulus) increases in the case of both gum and the composites.

Moisture absorption of natural fibre polymer composites is a major concern in their outdoor applications. Akil et al. [29] studied the water absorption in jute fibre reinforced with unsaturated polyester composites by immersing in distilled water, sea water, and acidic solution at room temperature for a period of 3 weeks. Dhakal et al. [30] investigated water absorption, tensile and flexural properties of hemp fibre reinforced unsaturated polyester composite specimens containing various fibres volume and the specimen are immersed in a de-ionized water bath at 25 °C and 100 °C for different time durations. The percentage of moisture uptake increased as the fibre volume fraction increased due to the high cellulose content. Corrales et al. [31] studied the chemical modification of jute fibre using fatty acid to confer hydrophobicity and resistance to biofibres. Viksne et al. [32] investigated the water uptake and the influence of moisture on the flexural strength and modulus of polypropylene wood fibre composites by immersing in water at 20 °C, 50 °C and 90 °C. Sabeel Ahmed and Vijayarangan [33] investigated experimentally the effect of stacking sequence on tensile, flexural and interlaminar shear properties of untreated woven jute and glass fabric reinforced polyester hybrid composites. The layer sequence has greater effect on flexural and interlaminar shear properties than tensile properties. Bogren [34] studied wood fibre reinforced polylactide a biodegradable composite, where both fibres and matrix are from renewable resources and measured the dynamic stress transfer between the fibres and the matrix using DMA and FTIR.

The present study aims to develop natural fibre composite from Agave (*Agave americana*) fibre, one of the most widely used natural fibres in yarns, ropes, twines, carpets, mats and handicrafts. Agaves are succulent plants of a large botanical genus of the same name, belonging to the family Agavaceae. Chiefly Mexican, they occur also in Asia, North America and in central and tropical South America. In India it is available in wild. The main objective of this work is to analyse the mechanical and thermal behaviours of raw and alkali treated Agave continuous fibre reinforced epoxy composites.

2. Materials and methods

2.1. Fibre preparation

Agave leaves were harvested and their margins were trimmed to avoid the thorns. Then the leaves were sun dried for 2 days to remove excess moisture. Retting of the leaves was carried out by immersing them in water for minimum 2 weeks. This facilitates maceration of the fleshy layers of the leaves. The retted leaves were then manually beaten to remove the flesh. The cured fibres were then thoroughly washed and combed to free the flesh thoroughly and was air dried for two to three days at room temperature. The dried fibres were thinned by ramming it in order to remove the unwanted short and broken fibres. The entire fibre extraction process takes 20–25 days. The diameter and length of the fibres were measured by air wedge and conventional methods respectively. The average diameter and length of fibre are 0.334 mm and 1200 mm respectively [35].

2.2. Alkali treatment of fibre

The raw Agave fibre was washed with water for three to four times for complete removal of the plant debris and dried at room temperature for 48 h. The raw fibre were immersed in 5% sodium hydroxide solution for 30 min and then washed with very dilute hydrochloric acid (HCl) to remove the excess alkali. Then, the fibre was rinsed with cold water twice or thrice. The rinsed fibres were dried at room temperature for 2–3 days. The mechanical properties as well as the chemical properties of the alkali treated and untreated Agave fibre were analysed [35] and the results are shown in Tables 1 and 2.

2.3. Matrix preparation

The matrix chosen is epoxy and has good mechanical property when compared with other resins [36]. Epoxy resin of the grade 3554A with a density of 1.1–1.5 g/cm^3 was used to fabricate the composite. To favor the viscosity, a slow hardener is mixed with the resin in a ratio of 4:1.

Table 1
Chemical properties of raw and alkali treated Agave fibre [33].

Types of fibre	Cellulose (wt.%)	Lignin (wt.%)	Hemicelluloses (wt.%)	Wax content (wt.%)	Moisture content (wt.%)	Density (g/cm^3)
Raw fibre	68.42	4.85	15.67	0.26	7.69	1.20
5%NaOH treated fibre	86.27	4.05	Nil	0.14	8.74	1.30

Table 2
Mechanical properties of raw and alkali treated Agave fibre [33].

Types of fibre	Tensile strength (N)	CV% of strength	% of Elongation	CV% of elongation	Tenacity (N/den)	B-work (N m)	Yarn count (Denier)
Un treated	5.69	38.53	7.07	29.28	0.0165	0.049	344
5%NaOH treated fibre	7.03	48.51	6.35	38.39	0.0261	0.048	269.1

2.4. Preparation of specimens

The mould used for Agave fibre composite materials is a rectangular mild steel plate with a dimension of 240 × 90 × 15 mm assembled with a top plate, side plate and a base plate. To help complete removal of the composite from the mould, a polythene sheet is laid on the clean and dry mould before fabrication of the composites. Then, the epoxy mixture and the Agave fibres (unidirectional) are evenly layered in the mould successively thrice by hand-lay up technique. Then, the setup is transferred to an oven at 60 °C for twenty minutes to prevent void content in the composites. Finally it is closed and compression molded at a uniform pressure of 0.1 ton for 24 h at room temperature for curing. The composite is removed from the mould and the ratio of fibre and epoxy is found to be 35:65. The density of composite materials determined by water displacement method is found to be 1.3 g/cm^3. This procedure is used to prepare both untreated Agave continuous fibre epoxy composites (UTCEC) and alkali treated Agave continuous fibre reinforced epoxy composites (TCEC) specimens.

2.5. Tensile test

The tensile test specimen was prepared according to ASTM D 638-03 [37] type I sample. The test was carried out using an extenso meter machine equipped with a 20 kN load cell, PC 2000 with software of digital load, extension microprocessor based elongation measurement, and the cross head speed of the specimen was 5 mm/min. The initial length, width and thickness of specimen were measured and as shown in Fig. 1. The tensile test was carried out at 28 °C with 40 ± 2% relative humidity. Five samples were tested for each specimen. The specimen was placed in the grip of the testing machine to align the long axis and the grips with an imaginary line joining the points of attachment of the grips to the machine. Simultaneous measurement of load and strain were made and data were recorded.

2.6. Compression test

For compression test, the specimen was placed within the grip of compressometer. The strain gauge readings and the load cell readings were obtained, while the axial cross head movement rate imposed on the specimen was 5 mm/s. The test was carried out according to ASTM D 695-02a [38] and size of the specimen is 12.7 × 12.7 × 50.8 mm^3. The compression test was carried out at 28 °C with 40 ± 2% relative humidity and five samples were tested for each specimen.

2.7. Flexural test

The three point flexural tests of composites were carried out using the Lloyd instrument LR 100 kN. The flexural test was carried out according to ASTM D 790-03 [39] at 28 °C with 40 ± 2% relative humidity and cross head speeds of 1 mm/min. The specimen is 127 × 12.7 × 5 mm^3, five specimens were tested, and the average was calculated. The bar of rectangular cross section rests on two supports and is loaded by means of a loading nose midway between the supports. The loading nose and supports have cylindrical surface. The flexural strength are calculated using the formula

$$\sigma_f = \left(\frac{3PL}{2bd^2}\right)[1 + 6(D/L)^2 - 4(d/L)(D/L)] \quad (1)$$

where σ_f = stress in the outer fibres at midspan, MPa, P = load at a given point on the load deflection curve, N, L = support span, mm, b = width of beam tested, mm, d = depth of beam tested, mm, D = deflection of the centerline of the specimen at the middle of the support span, mm.

The flexural modulus is calculated from the slope of the initial portion of the load deflection curve.

2.8. Impact test

The impact tests were carried out using an Izod digital impact tester according to ASTM D 256-05 [40] standards. The specimen is 64 × 13 × 5 mm^3, notched, five specimens were tested, to determine the impact resistance and impact strength of TCEC and UTCEC composites at room temperature.

2.9. Moisture absorption test

The specimens are cut as per the ASTM D 570-98 [41] standard for moisture absorption test. The specimens are cleaned and weighed in an electronic balance of 0.00001 g accuracy to monitor the mass during the aging process. The specimen is 76.2 × 25.4 × 5 mm^3, five specimens were tested, and the average was calculated. Moisture absorption tests were conducted by immersing specimens in a distilled water bath at room temperature (28 °C) and boiling water at 60 °C and 100 °C. At room temperature and boiling temperature, the absorption was monitored at 24 h, 2 h and 30 min interval for five repeats. During the immersion process, the specimens are allowed to rest on one edge at the bottom and entirely immersed. For each observation at each stipulated interval, the specimens are wiped with a clean, dry cloth to remove the excess moisture immediately and then weighed. In the boiling water immersion process, after each immersion, the specimen are brought to room temperature by dipping in cold water for 10 min and then processed further as mentioned above. The moisture content $M(t)$, absorbed by each specimen is calculated as follows:

$$M(t) = \left(\frac{(w_t - w_0)}{w_0}\right)x100 \quad (2)$$

where w_0 = dry weight of specimen in grams, w_t = wet weight of specimen in grams.

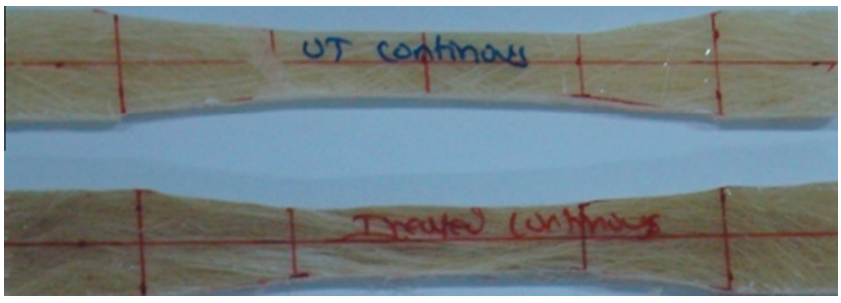

Fig. 1. Tensile test specimen of TCEC and UTCEC.

2.10. Dynamic mechanical analysis

Dynamic mechanical analysis test were carried out with a universal V4.5A TA instrument of model TA Q800 of temperature range −150–600 °C and frequency range from 1 Hz to 20 Hz. The samples were thin rectangular strips with dimension of $30 \times 12.5 \times 3\ mm^3$. The test was performed in isochronal conditions at 1 Hz, and the temperatures are varied between 30^0C and 135^0C at rate of 3^0C/min.

2.11. Fourier transform infrared spectrometry

Fourier transform infrared (FTIR) spectroscopy with a microscope was used to study the stress transfer at the molecular level. The FTIR equipment NICOLET 6700 of Thermo Electron Corporation was used to obtain the structural analysis of the specimen. The spectrometer was used in the transmission mode with a resolution of $4\ cm^{-1}$ in the range of 4000–400 cm^{-1}. FTIR can be used to detect the molecular vibration to analyse the distribution of functional groups.

2.12. Scanning electron microscope (SEM)

To illustrate the effect of alkali treatment of the fibre, the failure surfaces of the specimens subjected to test were analysed using a JEOL scanning electron microscope (SEM). In SEM a fine probe of electrons scans the surface of the sample and the signals emanating from the incident site are processed and quantified. All specimens were sputtered with 10 nm layer of gold prior to SEM observations. Each specimen was mounted on the aluminium holder of the microscope using double sided electrical conduction carbon adhesive tabs. The accelerating voltage of 5–15 kV was employed. The SEM analyses of both raw and alkali treated fibres composites were compared.

3. Results and discussion

3.1. Tensile test

The TCEC specimen is found to withstand more loads (2000.6 N) with a displacement of 7.37 mm when compared to that of UTCEC specimen (Fig. 2). Fig. 3 shows typical engineering stress–strain curves of continuous Agave fibre reinforced epoxy composites at room temperature. The tensile strength of the composite is influenced by the strength and modulus of the fibres [33]. The TCEC specimen was capable to provide some resistance to continued elongation and most of the specimen displayed brittle failure at a maximum stress of 41.2 MPa. Further the true stress–strain of the composite materials were analysed and the results are shown in Fig. 4 which indicates clearly that the treated continuous fibre composite gives better strength than untreated continuous composites. Thus, tensile strength and tensile modulus of UTCEC are

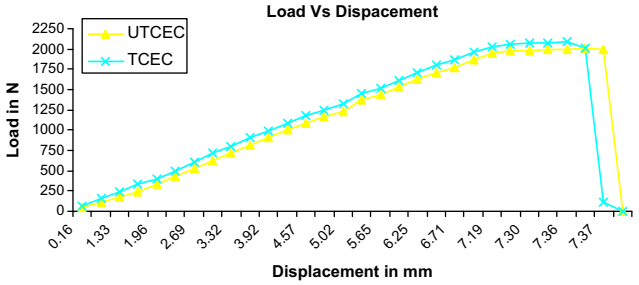

Fig. 2. Load vs. displacement of UTCEC and TCEC composites.

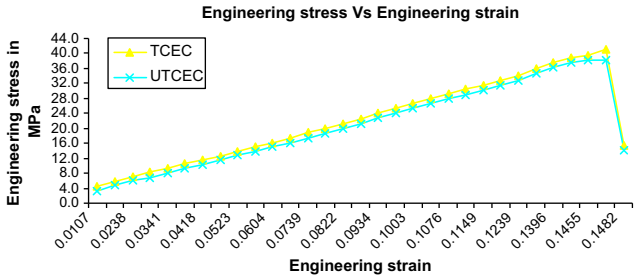

Fig. 3. Engineering stress vs. engineering strain of UTCEC and TCEC composites.

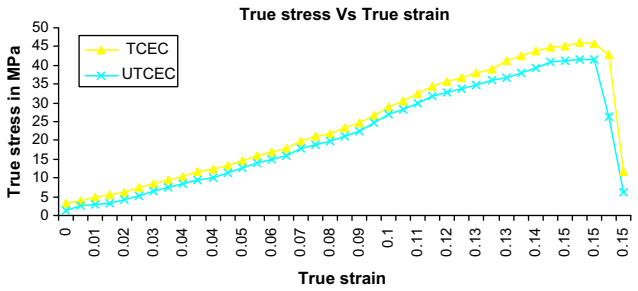

Fig. 4. True stress vs. true strain of UTCEC and TCEC composites.

15% less than that of the TCEC specimen .The tensile modulus of the composite increased to a value 263 MPa for UTCEC and 270 MPa for TCEC respectively. In the case of the alkali treated fibre, the fibre pull-out length is considerably shorter than that noticed for the untreated fibre, due to the fibre failed by tearing. The deprived tensile strength exhibited by the untreated fibre reinforced epoxy composite specimen may have resulted from poor adhesion between fibre and matrix. The mechanical properties concern with the bonds existing between the different components of the fibre [22]. Alkali treatment causes removal of hemicellulose and swelling of the fibre thus helps stress transfer between ultimate fibre cells [2]. In addition, the formation of hydrogen bonds of the cellulose chains improves chemical bonding between the fibres in composites. Thus the alkali treatment of Agave fibres leads to changes in mechanical properties like tensile strength, displacement, tensile load and tensile modulus.

3.2. Compression properties

The compression strength of the Agave fibre–epoxy composites are shown in Figs. 5 and 6. The compression strength increases 10% because of the alkali treatment thus enhancing the mechanical interlocking. Fig. 5 shows a comparison of the compressive force by varied deflection at 20%, 30%, 40% and 50% respectively. When the alkali treated fibre is used, a gradual increase in the compression force upon varying percentage of deflection was observed.

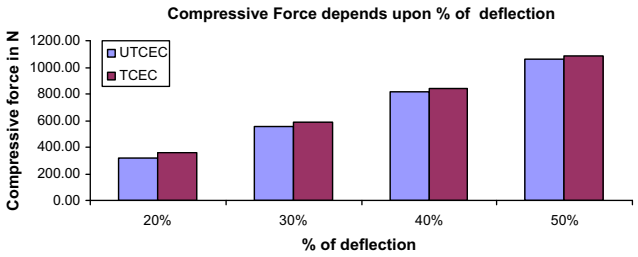

Fig. 5. Compression force depends upon percentage of deflection.

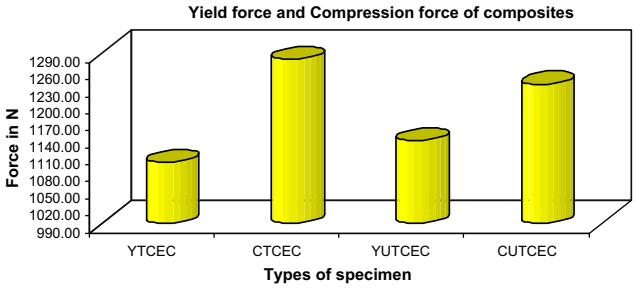

Fig. 6. Yield force (TCEC and UTCEC) and compression force (TCEC and UTCEC).

The NaOH reacts with hydroxyl groups of the natural fibre hemicellulose, and it brings on the destruction of the cellular structure and the fibrillation increases effective surface area available for contact with the matrix [20]. Fig. 6 shows an evaluation of the yield force and compression force of samples and the TCEC shows better result than UTCEC composites. This increment is attributed to chemical interaction between the fibre and the matrix. It is observed in this study that the compression moduli of TCEC samples are 39.4 MPa and UTCEC samples are 30 MPa. It is well known that the fibre reinforcing effect is most efficient along the fibre axis orientation.

3.3. Flexural properties

The flexural strength of the Agave fibre reinforced epoxy composites are shown in Figs. 7 and 8. It is observed that the flexural load increased from 420 N to 447 N for UTCEC to TCEC composites. Flexural strength increased in the TCEC samples from 10% to 15%

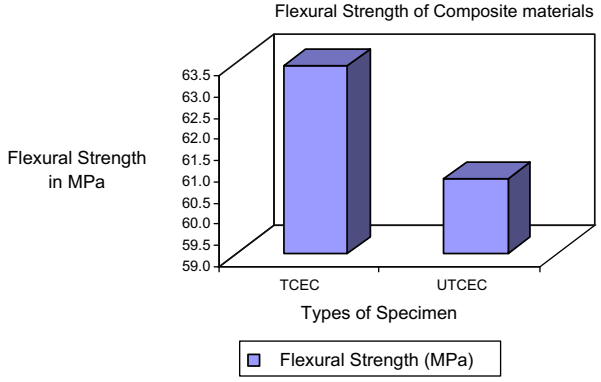

Fig. 7. Flexural strength of TCEC and UTCEC samples.

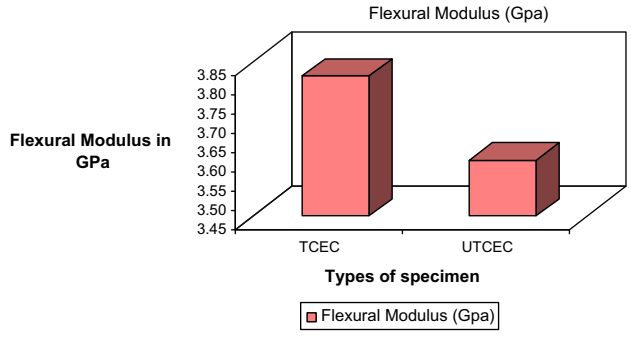

Fig. 8. Flexural modulus of TCEC and UTCEC samples.

and it is an interesting note that the alkali treated fibre showed more shrinkage and toughness that inbuilt the chemical strength between the fibre and the matrix. The TCEC sample was considered to have 15% higher flexural strength than the UTCEC sample. The fibre surface alkali treatments had a significant effect on the flexural modulus, similar to the observations made for the tensile properties [21]. The enhanced interfacial area of contact was favorable for the flexural strength [25]. The flexural modulus is used as an indication of the material's stiffness in static bending condition. The flexural modulus showed a similar behavior as the tensile modulus and the values of stiffness measured were 3.65 GPa for UTCEC and 3.85 GPa TCEC in the longitudinal direction. This indicates a better fibre/matrix contact, the increase in areas of contact between the fibre and the matrix improves the adhesion by incorporation of mechanical components of adhesion for the fibre–epoxy interfacial strength.

3.4. Impact properties

The influence of surface modification on the impact strength of composites is shown in Fig. 9. The impact strength of the TCEC sample improved to 10–15% than that of UTCEC samples. The shrinkage of the fibres during alkali treatment affects the impact strength of the composites and it helps to improve the interfacial strength between fibre and matrix. The impact resistance of TCEC and UTCEC composites is 1.53 J and 1.47 J respectively. There was significant difference in the impact strength of the composites and the alkali treated fibre reinforced composites showed slightly superior values. The improvement in the impact strength of TCEC samples is related to the chemical effect of the fibre–epoxy interface. The alkali treated fibre composites showed maximum improvement in impact strength compared with untreated fibre composites [20]. A strong interface has an effect on the impact strength as energy absorption is from the fibre–matrix debonding and fibre pullout. The impact failure of Agave fibre reinforced epoxy composites reflects a process involving crack initiation and growth in the resin matrix. It is well known that the impact response of fibre composites is highly influenced by the interfacial bond strength, the matrix and the fibre properties.

3.5. Moisture absorption properties

The percentage of moisture absorption of the Agave fibre reinforced epoxy composites was evaluated as the difference between the dry weight and wet weight of the specimen. Fig. 10 shows the percentage of weight gain as a function of time for Agave fibre reinforced epoxy composites samples (UTCEC and TCEC) immersed in distilled water at room temperature and boiled water at 30 min interval. It can be inferred that water absorption is more at boiling

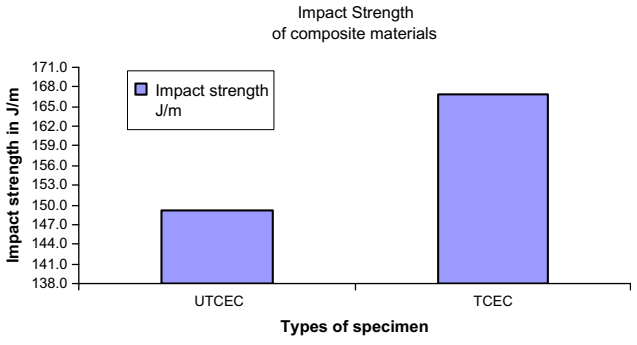

Fig. 9. Impact strength of TCEC and UTCEC samples.

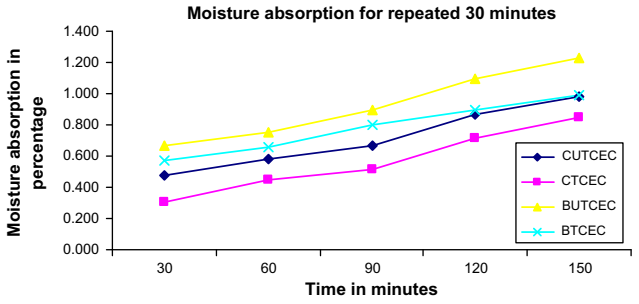

Fig. 10. Moisture absorption of cold water TCEC and UTCEC (CTCEC and CUTCEC), boiled water TCEC and UTCEC (BTCEC and BUTCEC) samples for repeated 30 min.

temperature than at room temperature. The results of moisture absorption of the samples in boiling water (100 °C) and room temperature at 2 h duration is shown in Fig. 11. When the temperature of immersion is increased, the moisture saturation time is significantly shortened. This shows that water absorption at room temperature takes fairly long period to reach equilibrium than sorption at boiling temperature. With the presence of high hydroxyl group, natural fibre to show low moisture resistance. The rigidity of the cellulose structure is destroyed by the water molecules in the cellulose network structure in which water acts as a plasticizer and it permits cellulose molecules to move freely [29]. For TCEC and UTCEC, the weight gain at moisture saturation point in boiling temperature is approximately 5% higher than that of room temperature for 24 h impression as shown in Fig. 12. When weight gain is more, water molecules interlocked in the composites is more. Thus the water molecules get chances to actively attack the interface, resulting in debonding of the fibre and the matrix internally in the composite.

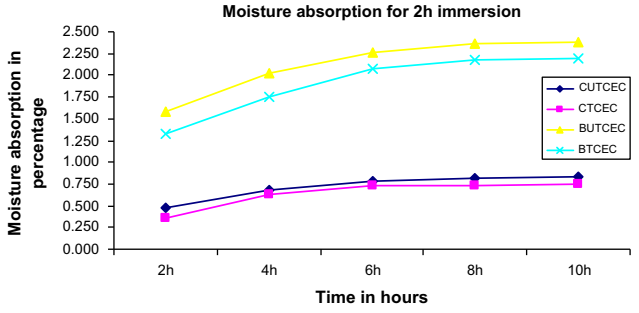

Fig. 11. Moisture absorption of cold water TCEC and UTCEC (CTCEC and CUTCEC), boiled water TCEC and UTCEC (BTCEC and BUTCEC) samples for 2 h immersion.

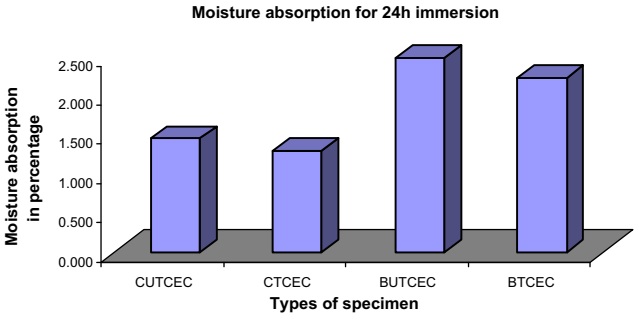

Fig. 12. Moisture absorption of cold water TCEC and UTCEC (CTCEC and CUTCEC), boiled water TCEC and UTCEC (BTCEC and BUTCEC) samples for 24 h immersion.

3.6. Dynamic mechanical analysis (DMA)

The mechanical behaviour of Agave fibre reinforced epoxy composite was investigated in the linear range using dynamic mechanical analysis (DMA) to investigate the properties of interface and the impact of the fibre treatment on the fibre matrix adhesion. Dynamic mechanical analysis is a technique where a little deformation is applied to a sample in a cyclic manner. This permits the material's response to stress, temperature, frequency and other values. DMA applies a sinusoidal force at a set frequency to the sample and measures changes in stiffness and damping, these are reported as modulus and tan delta. The in-phase component is expressed as the storage modulus (E') and an out of phase component is loss modulus (E'') and the ratio of the loss to the storage ,the tan delta ($\tan\delta$) of the TCEC and UTCEC material's is as shown in Fig. 13 and 14. Tan δ varies with the state of the material, its temperature, and with the frequency. Changes in modulus values with temperature and transition in materials can be seen as changes in the E' or tan δ curves. This includes not only the glass transition and the melt, but also other transitions that occur in the glassy plateau as shown in Figs. 13 and 14. DMA is an effective tool to determine the dynamic glass transition temperature, T_g, morphology of crystalline polymers and damping factor. The dynamic T_g is defined [17] as the temperature at which maximum of the tan δ occurs or maximum of the E'' occurs or the middle point of E' versus temperature curve. T_g values are frequently determined, by constructing a straight line through the linear viscoelastic part of the E' and a tangent to the elastic portion of the E'. It can be seen from Fig. 12 that maximum in tan δ and E'' curves and the middle point of E'' curves

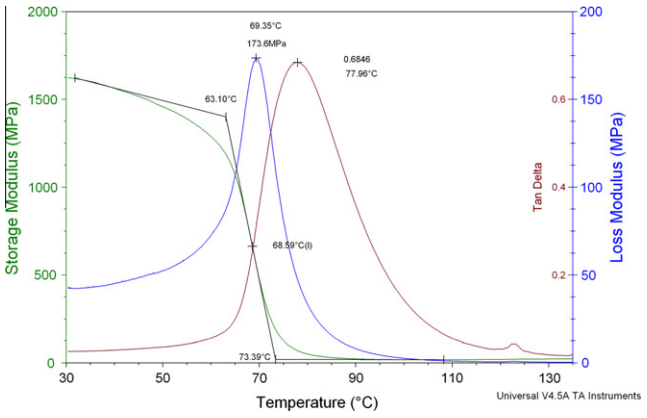

Fig. 13. Dynamic mechanical properties of TCEC composites at 1 Hz.

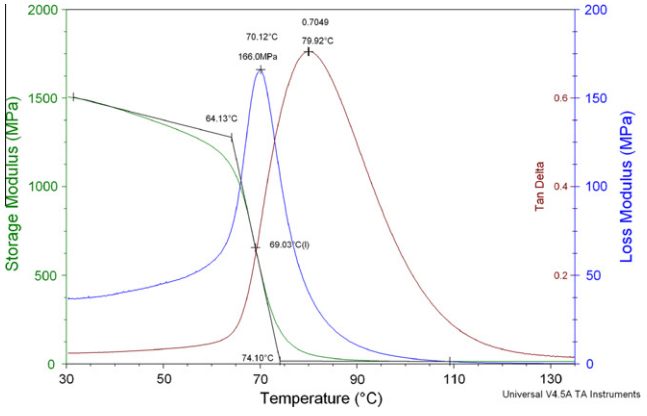

Fig. 14. Dynamic mechanical properties of UTCEC composite at 1HZ.

of the TCEC material at 1 Hz nearly coincides with one another. Likewise in the present study two prominent peaks were observed for the TCEC materials i.e. tan δ is maximum of 0.6846 to the corresponding temperature 77.96 °C and the E'' is the peak of 173.6 MPa at maximum temperature of 69.35 °C. The other peak at high temperature was due to the dynamic mechanical behavior of Agave fibre. This behavior depends mainly on the dynamic mechanical properties of epoxy resin, Agave fibre and the adhesion between these two.

Dynamic mechanical properties of untreated Agave continuous fibre reinforced epoxy (UTCEC) were shown in Fig. 14 and the peaks were observed i.e., the E'' is maximum of 166 MPa at the temperature of 70.12 °C and the tan δ is 0.7049 at maximum temperature of 79.92 °C. It can be understood that the movement of polymer chains is highly influenced by the presence of fibres, which in turn influenced the elastic characteristics of the matrix epoxy. The maximum T_g of TCEC and UTCEC materials observed from DMA are 63.1 °C and 64.13 °C respectively. Hence the T_g value obtained from the E'' peaks gives more reliable and suitable index than the one based on the tan δ peak. The E'' of both TCEC increases 10%, due to alkali treatment of fibre, compared to those UTCEC materials. Therefore, the tan δ peaks in the low temperature region are larger if the material with lower T_g is the continuous phase. It can be seen that the values E' decreases harshly after 50–110 °C as a result of the glass transition phenomenon. Therefore it can be seen that, on increasing the temperature the value of E' decreases until it is steady and coincides with the elastic zone of the materials. It is interesting that the transition between the glass and elastic zone is more important for TCEC is compared that of UTCEC composites and also little modulus drop. As expected, the E' values of the TCEC is higher than the UTCEC which indicates the greater interfacial bond strength and adhesion between the resin matrix and the fibre. The efficiency of Agave fibre reinforced epoxy composites can be improved by enhancing the interfacial adhesion between fibre and matrix by alkali treatment. The variation of tan δ of UTCEC and TCEC samples indicates that there is a significant difference in height of tan δ peak between the UTCEC and TCEC samples indicating the same order of damping capabilities and its better impact properties compared to the alkali treated fibre composites.

Two prominent peaks were observed in the tan δ curve of these composites due to the dynamic mechanical behavior in matrix and fibre. Further small peak observed in the transitional region represents the dynamic mechanical behavior in the interface. It was reported that the higher the damping at the interfaces, the poorer the interfacial adhesion in the study on short coir fibre reinforced natural rubber composites [28]. The present study exposed that composite with poor interfacial bonding tend to dissipate more energy than those with good interfacial bonding. The composite containing fibres subjected to alkali treated exhibited very high tan δ values in the low temperature region but the lowest values at high temperature region. This showed that these composites are good chemical compounds at higher temperature. The treated fibre incorporation increases the E'' of these composites compared to that of the untreated fibre. The rapid rise in loss modulus in a composite indicates an increase in the structural mobility of the polymer.

3.7. Fourier transformation infrared analysis

Figs. 15 and 16 show FTIR spectra corresponding to the Agave fibre used before and after alkali treatment reinforced epoxy composites. The spectrum corresponding to composites was deduced in order to highlight the band issuing from alkali treatment. The broad shape of the peak between 4000 cm^{-1} and 3000 cm^{-1} of TCEC and UTCEC samples are showed at lower peak of 3303 cm^{-1} of TCEC and 3327 cm^{-1} of UTCEC composites. It indicates that the treated fibre stretching increased in TCEC compared with UTCEC are seen as minimum up and down peaks i.e., the chemical bonding between fibre and matrix are good [34]. Thus the poor adhesion of untreated fibre with matrix played major role to form high peak of the specimen. The broad intense peaks around 2250 cm^{-1} and 1750 cm^{-1} were assigned to maximum molecule strength of fibre of TCEC samples. The peaks near 1500 cm^{-1} and 750 cm^{-1} are related to residual unhydrolyzed groups and small intensity indicates that most of the alkaline adhered under their conditions was actually hydrolyzed. In treated fibre composites hydroxyl group on the fibre surface helps a three dimensional network which could be attributed to hydrogen bonding and thus

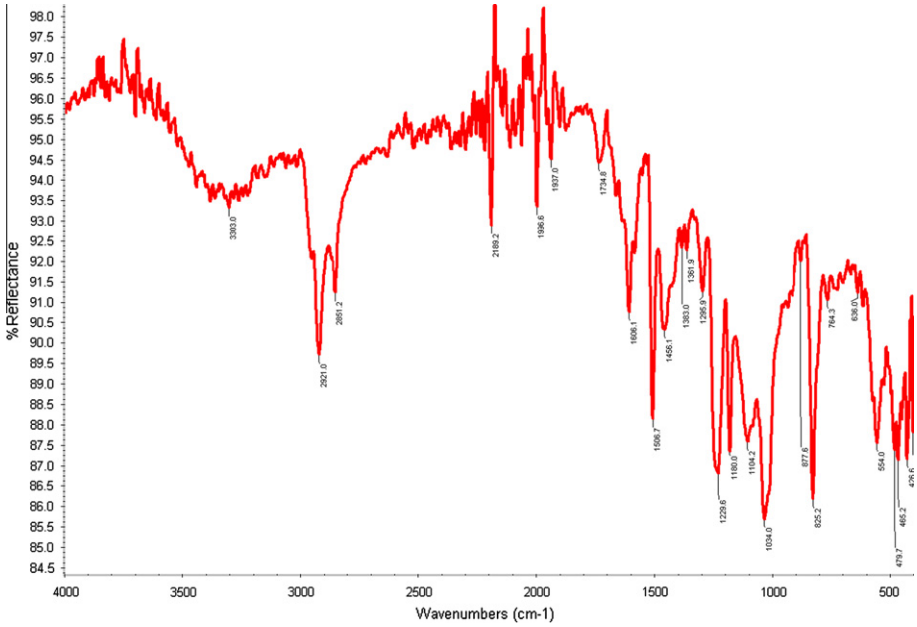

Fig. 15. FTIR spectra of TCEC sample.

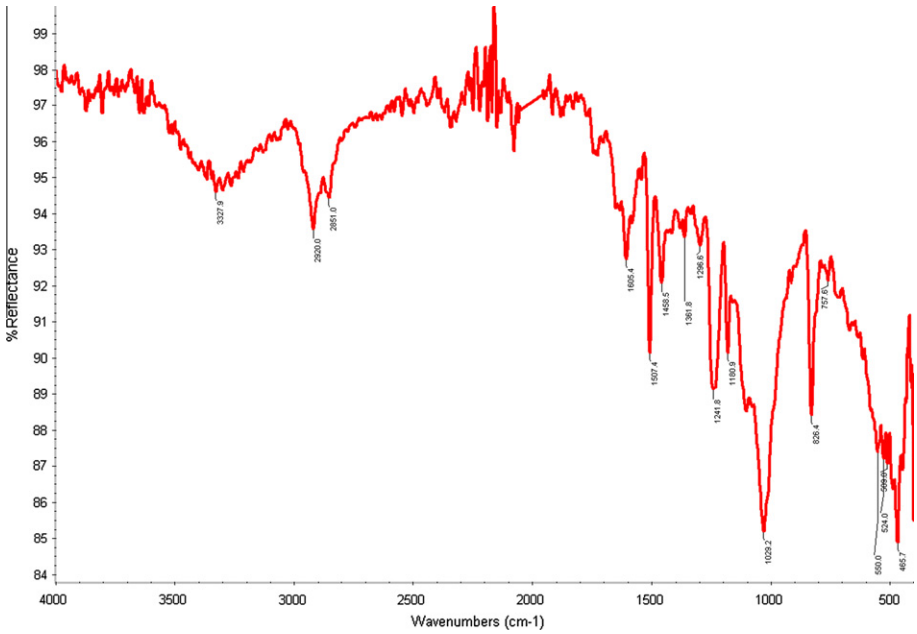

Fig. 16. FTIR spectra of UTCEC sample.

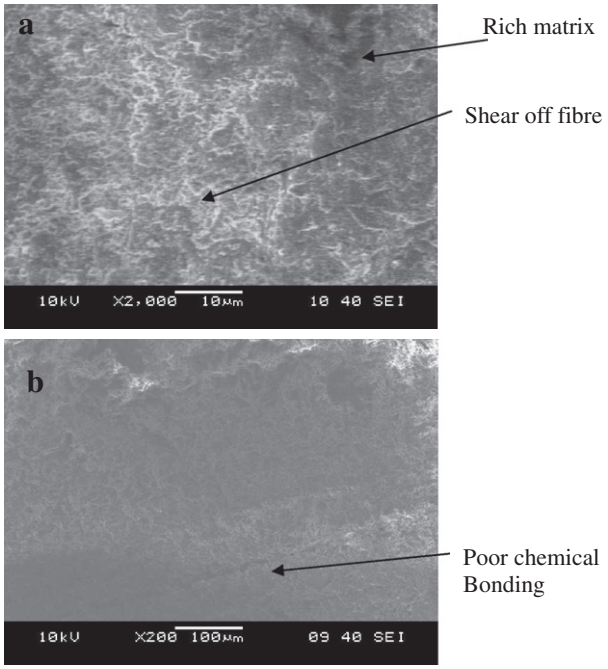

Fig. 17. (a and b) SEM image of UTCEC and TCEC sample.

better interface between the fibre and matrix. These linked zones enhance the strength and modulus of composites.

3.8. Surface morphology of tensile test composites

Observation of failure of tensile test specimens further clarifies the importance of fibre adhesion on tensile test results. A series of SEM micrographs are shown in Fig. 17a and b for the composite with 35% volume fibre content and tested under tensile loads. SEM image are evidence that the matrix bridges, thin layers that have failed in the crack wake by extensive stretching and ductile tearing by plastic flow [27]. In Fig. 17a, the untreated continuous Agave fibres (UTCEC) appear to be free of any matrix materials adhering to them, thus indicating poor fibre matrix adhesion. It exposed that the untreated fibre partly adhered to the epoxy, demonstrating a weak fibre matrix adhesion. Most fibres, whether present as composite, appear to have failed in a brittle manner and display what appears to be a considerable amount of debris on the fibre surface [17]. The presence of this debris would appear to specify that the degree of adhesion between the fibre and the matrix is poor. In Fig. 17b, the Agave fibres treated with the 5% NaOH solution (TCEC), more tearing of the fibres could be observed, together with some cavities absent by the pulled out fibres and shown many polymer particles that appear to be chemically bonded to the fibres surface. It can be incidental that mechanical interlocking and friction are responsible for the observed composite strength increment. It can be observed that fibres are evenly distributed on the composite surfaces, but rather from fibre group that are bonded at random intervals along the length of the fibres. Very few fibres can be observed on the fracture surfaces due to fibre cover up by the matrix materials, although, a few strictly damaged fibres can be seen protruding out of the matrix. These fibres appear to have split longitudinally down the fibre length, and this is through to be the result of microfibrils shearing within the fibres that gained induced stress. This micrograph also discovered that the fibre matrix adhesion of treated fibre composite was superior after alkali treatments. Appears to be strong bonding between the alkalis treated fibres and the matrix is proof by the relative lack of fibre pull-out when compared with the untreated fibre reinforced material. There is evidence of a greater amount of fibrillation at the fibre surface, indicating that a good bond existed between the fibre and matrix prior to fracture. In the case of the surface treated fibre composites, the fibre pull-out length is considerably shorter than that noticed for the untreated fibres composite material.

4. Conclusion

The results of the present study showed that useful composites with good strength could be successfully developed using Agave fibre reinforced epoxy composites. Tensile strength, tensile modulus, compression strength and compression modulus were

significantly high due to alkali treatment of the fibre. It is also proved that good chemical bond between alkali treated fibre and epoxy resin play a role in increasing the flexural strength, flexural modulus and impact strength of composites. In DMA the effect of TCEC and UTCEC was studied and it was found that composites with poor interfacial bonding tend to dissipate more energy than that with good interfacial bonding. The FTIR showed that the thermal stability composite increases with alkali treatment. The SEM micrographs of the fracture surface showed that the alkali treatment of fibres improved due to the dissolution of the hemicellulose and increased aspect ratio, which resulted in a better fibre matrix adhesion. Thus in Agave fibre reinforced epoxy composite thorough enhancement in bonding between the fibre and epoxy could be achieved by surface treatment of the fibre by alkali NaOH which could benefit its application in light weight materials industry. They are indeed biodegradable and aesthetic in nature.

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