
Surface modification of eco-friendly ligno-cellulosic fibre extracted from *Lagenaria siceraria* plant agro waste: a sustainable approach

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Abstract: Recent research on green composites addresses the environmental threats of solid waste disposal by suggesting sustainable methods. This study aims at identifying potential reinforcement of lignocellulosic fibre extracted from the solid agro waste pile of *Lagenaria siceraria* (LS) plant. The thermal stability and mechanical strength of raw *L. siceraria* fibres (LSFs) were modified by alkali treatment (2%–15% w/v of NaOH) at different temperatures (room temperature to 70°C) and time intervals (30 min to 6 h). The result showed that 4% alkali treatment at 45°C for 40 min improves the physicochemical properties of the LSFs. The higher cellulose content (86.49%) and lower hemicellulose (4.23%) and lignin (6.98%) content of optimally treated LSFs significantly improve the mechanical property with a higher tensile strength (436.8 N), elongation at break (5.47%) and thermal stability (355.5°C) compared to raw LSFs. It was found that the alkali-treated fibres with additional toughness and fatigue strength reduce environmental threats by sustainable development.

Keywords: sustainable approach; surface modification; mechanical property; thermal stability; *Lagenaria siceraria* fibre; LSF; solid waste.

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1 Introduction

The knowledge economy of this digital generation of the world has mobilised research in multiple domains. One significant landmark that has made a high impact is in the field of material science. Growing interest in materials may offer viable solutions to their metallic and synthetic counterparts like such as glass, aramid and carbon fibres. These traditional engineering fibres were have characterised by some inherent disadvantages like such as high density, high mechanical wear on mineral reinforcements, alarming health hazards and a high degree of inflexible characteristics. Hence, the industries have been on a constant quest for renewable resources. This has inevitably pushed them to explore green technology in matter and material science. This, in turn, has opened up kaleidoscopic avenues in material innovation (Rout et al., 2001; Wambua et al., 2003).

The contemporary world has almost exhausted all fossil resources and is on the verge of resource depletion. Besides, the increase in the amount of pollutants in the earth's atmosphere and the environmentally detrimental factors persuade humankind to redefine technological developments in terms of green technology. This paradigm shift has given birth to a corpus of research literature that explores the prospective applications of natural fibre composite in all possible industries. This momentum period of fibre composite alternatives has brought to limelight a host of benefits the fibre composites have over their synthetic counterparts.

Natural fibres are endowed with the required mechanical properties on reinforcement with polymer components. Their environmentally friendly characteristic, affordable pricing, abundance of availability, renewability and ease of manufacturing have made them a cut above the rest of the existing synthetic composites and render them with the required specifications for industrial and structural applications.

The industries have embarked upon researches on renewable resources such as natural fibre composite materials. Some commercially available natural fibres such as flax, jute, kenaf, hemp, coir, sisal and abaca have been extensively researched and used to the point of dwindling these resources (Symington et al., 2009). The demand for natural fibre-reinforced composites has been pressing in the current scenario in the fields of construction, energy production, sports and high-speed transports such as cars, airbuses, spacecraft due to their light weight and strength (Holbery and Houston, 2006). Hence, choosing a hitherto unexplored renewable material and a suitable combination of matrix and reinforced material was an imperative now to cater to the increasing needs of various industrial applications.

Lagenaria siceraria (LS) plant is a vegetable found to flourish on any terrestrial ecosystems for human consumption. The rest of the plant materials are discarded as a massive pile of agro waste. Quite often, they are mostly disposed of by burning, which poses environmental pollution problem. This agro-waste natural fibre is found in abundance and the environmentally detrimental factor is eliminated when it is subverted to growing industrial demands.

LS plant has been extensively used for medicinal purpose and production of biodiesel (Sokoto et al., 2013). Several observations and preliminary studies of less known LS plant fibre have hypothesised that it possesses mechanical properties that pronounce greater possibility for natural fibre-reinforced composites for targeted industrial applications (Saravanan et al., 2016). This article focuses on the feasibility of using agro waste as raw material for preparing eco-friendly composites for diverse applications.

Despite a host of advantages mentioned earlier, two threats, namely resin compatibility and water absorption pose, challenges to the mechanical properties of natural fibre-reinforced composites and restrict their relevant applications. These issues can be overcome to a certain extent by surface modifications through physical or chemical treatments as they render the fibres as effective reinforcement materials (Westman et al., 2010). In this context, highlights on the modifications of physicochemical and mechanical characteristics raw *Lagenaria siceraria* fibres (LSFs) fibre by alkali treatment for preparing potential reinforcement of composite manufacturing.

2 Materials and methods

2.1 LS fibre extraction

The crude LSFs were hauled out manually from the stems of LS plant by retting process for a period of 14 days and then the adhered impurities on the surface of the fibre were got rid of by cleansing with distilled water and parched as reported earlier (Saravanan et al., 2016).

2.2 Alkali treatment of LSFs

The raw LSFs were drenched with sodium hydroxide solutions of varying concentration (2%–15% w/v) at six different temperature ranges (from room temperature to 70°C) for a period ranging from 30 min to 6 h. Ultimately, the treated fibres were washed with 1% (v/v) acetic acid solution followed by distilled water and then dried at 60°C for 24 hrs in a hot-air oven. Dried fibres were cooled and preserved in airtight container for further analysis (Van de Weyenberg et al., 2003).

2.3 Chemical composition of raw and alkali treated LSFs

The alteration encountered in the composition of the secondary cell wall components of LSFs by alkali treatment was reviewed by standard procedures (Suryanto et al., 2014). Consequently, the density (Vignesh et al., 2016), wax (Conrad and Carl, 1944) and moisture (Boopathi et al., 2012) variations of treated LSFs were also being verified as per the procedure described elsewhere.

2.4 Scanning of vibration frequencies of alkali-treated LSFs

The changes seen in the vibration frequencies of the main functional groups present in the LSFs after alkali treatment were examined by using Perkin Elmer RXI FTIR infrared spectrometer, in the wavelength range of 500 cm^{-1} to 4,000 cm^{-1} at 30 scans per minute.

2.5 Thermal stability analysis of raw and alkali treated LSFs

The thermal stability of the alkali-treated (ATLSFs) was examined, using a thermo gravimetric analyser (DSC/TG) using Jupiter simultaneous thermal analyser (model STA 449 F3; NETZSCH, Germany). The fine particles of alkali treated LSFs sample were injected into the chamber, in a nitrogen atmosphere with a flow rate of 20 ml/min, subjected to heating from room temperature to 550°C constantly, at a rate of 20°C min^{-1} .

2.6 Tensile strength of raw and alkali treated LSFs

The tensile property of the alkali treated ATLSFs was inspected, using INSTRON 5500 universal testing machine at a crosshead speed rate of 10 mm min^{-1} as per the ASTM D3822/D3822 M-14 standards (NagarajaGanesh and Muralikannan, 2016a).

3 Results and discussions

3.1 Chemical composition of raw and alkali treated LSFs

The major constituents vary for different plant fibres. The variation depends on their origin, locality, climatic condition and life span. The significant physicochemical and mechanical characteristics of any natural fibre directly depend on their type, structure and composition. The quality of the fibre was greatly influenced by the extracting procedure and preconditioning methods (Bledzki et al., 1996).

Several semi-crystalline alpha cellulosic micro-fibrils embedded in amorphous matrix of hemicellulose and lignin forms a long fibre. The inter- and intra-molecular hydrogen bonding renders not only close packing of the fibre and other constituting materials but also spiralling of fibre along the fibre axis and exerts provides strength and stiffness to the fibre. Spiral angle determines the mechanical strength of the fibre. The smaller the spiral angle, the greater will be its strength (Ugbolue, 1990).

Out of different parts of plant fibre, stem fibre (bast fibre) is very hard because pectin cements the bundle of cellulosic fibres and strongly binds them to the stem (Pickering, 2008). During retting process, most of this pectin has been removed. Table 1 represents the changes in the density and chemical composition of the raw LSFs and ATLSF compared with other fibres.

Table 1 Chemical properties of raw and alkali-treated LSFs compared with other fibres

<i>Fibre</i>	<i>Cellulose (%)</i>	<i>Hemicellulose (%)</i>	<i>Lignin (%)</i>	<i>Wax (%)</i>	<i>Moisture (%)</i>	<i>Density (g/cm³)</i>
Raw LSF	79.91	12.69	7.62	0.31	9.96	1.216
2% ATLSF	82.63	8.41	7.37	0.18	8.94	1.219
4% ATLSF	86.49	4.23	6.98	0.2	8.23	1.223
6% ATLSF	84.21	3.18	6.08	0.08	8.56	1.227
8% ATLSF	85.33	2.2	5.42	0.02	9.12	1.232
10% ATLSF	83.98	-	5.13	-	9.47	1.238
15% ATLSF	80.12	-	4.42	-	9.84	1.237
Jute	64.4	12.0	11.8	0.7	1.1	1.3
Flax	64.1	16.7	2.0	1.5–3.3	3.9	1.5
Hemp	68.0	15	10.0	0.8	-	1.47
Kenaf	31–72	20.3–21.5	8–19	-	-	-
Ramie	68.6–85	13–16.7	0.5–0.7	0.3	7.5–17	1.5
<i>P. foetida</i>	77.96	12.63	10.47	0.35	9.54	1.328

Alkali treatment significantly removes the amorphous hemicellulose. Elimination of hemicellulose 12.69 to 4.23 wt. % alleviates inter-fibril connections of the fibrils and makes them undergo rearrangement themselves to the direction of tensile deformation, improves uniform load distribution in the fibres, thus reducing the stress and makes them less dense and more rigid (Symington et al., 2009).

Lignin is an amorphous polymer that mainly comprises of aromatics and has hydrophobic nature. In bast fibres, lignin plays an imperative role in determining its structure, properties (especially thermal stability) and morphology. Alkali treatment offers only partial purging of lignin (7.62–6.98 wt. %), reduces the micro-voids and makes the fibre more homogeneous and increases the fibre quality (Sawpan et al., 2011).

In general, in alkali treatment, removing non-cellulosic matter significantly increases the cellulose content from 79.91 to 86.49 wt. %. However, the cellulose fibrils are made to open and they are visible, which enhances the binding characteristics of the fibre with polymer matrix in the fabrication of composites, ensuring the better tensile strength of the composite.

The presence of wax in the fibre, grounds the adhesion and wettability characteristics, which trim down the interfacial binding in the composite preparation. Significant

reduction (0.31–0.2 wt. %) of wax by alkali treatment, augments the binding of fibre with polymer matrix (Arthanarieswaran et al., 2015).

The density of the alkali-treated fibre was increased from 1.216 to 1.238 g/cm³ due to removal of wax, fat and other non-cellulosic materials that facilitate rearrangements of fibres as well as sealing of pores with grafted molecules. However, the density of treated LSFs was found to be smaller than that of synthetic fibres, making them suitable as good reinforcements for preparing lightweight composite structures (Varma et al., 1989).

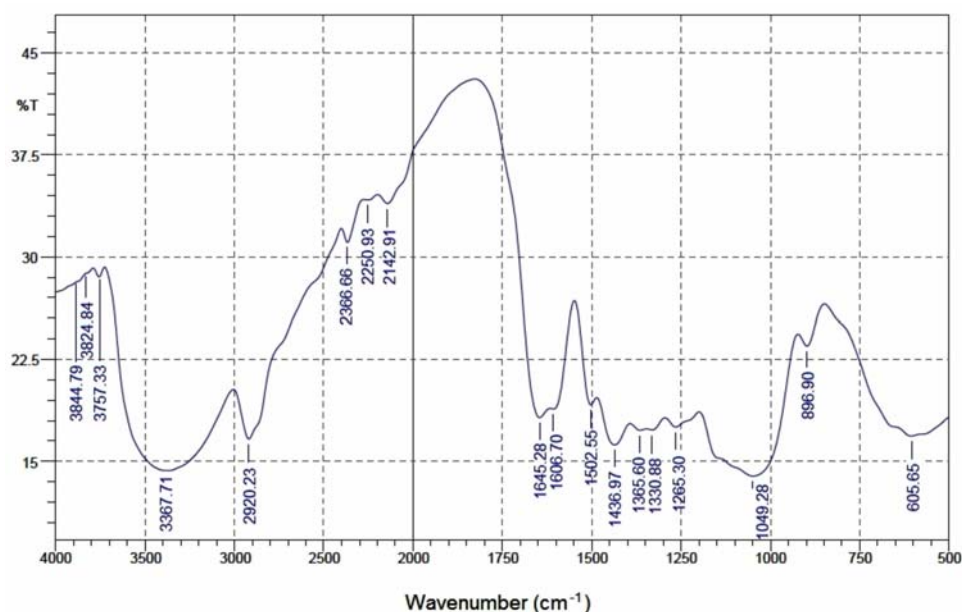
3.2 Scanning of vibration frequencies of alkali treated LSFs

The Fourier-transform infrared spectroscopy (FTIR) analyses of raw and 2% to 15% ATLSFs were conducted for observing changes in the various functional groups of the fibre constituent. The FTIR spectrum of raw LSFs was found to have major functional groups at 3,332, 2,898, 1,731, 1,636, 1,507, 1,423, 1,368, 1,315, 1,247, 1,155, 1,058 and 896 cm⁻¹.

The intense peak observed at 896 cm⁻¹ corresponds to the existence of $\beta(1-4)$ -glycosidic linkages between the monosaccharides in cellulose (Sathishkumar et al., 2013; Maepa et al., 2014). The intensity of this peak increases from 14% to 38% by alkali treatment. The maximum intensity was observed for 4% alkali treatment. The removal of impurities adhered to the cellulosic fibre and rearrangement of micro-fibrils facilitates the maximum intensity for glycosidic linkage.

The absorption peaks ranging between 2,900 and 3,800 cm⁻¹ arises due to various stretching and bending vibrations of free and hydrogen-bonded O-H, methylene groups (>CH₂), C-O and C-O-C of cellulose part of LSFs (Sun et al., 2004; NagarajaGanesh and Muralikannan, 2016b).

Figure 1 FT-IR spectra of 4% alkali treated LSFs (see online version for colours)



The intensity and relative area of the various observed characteristic peaks increase from raw fibre to alkali-treated fibre. The intensity increases gradually from 2%–4% during alkali treatment and then starts decreasing randomly. These results clearly point out that, the 4% alkali treatment is optimum for LSFs as potential reinforcement. Figure 1 shows the FTIR spectra of the 4% ATLSFs.

The raw LSFs showing characteristic absorption peaks at 1,731 and 1,247 cm^{-1} are due to C = O stretching and C-O-C stretching vibrations of acetyl group of hemicellulose constituent (El Ghali et al., 2012; Santhanam et al., 2016). The peak observed at 1,731 cm^{-1} completely vanishes whereas, the intensity of the peak in the 1,247–1,267 cm^{-1} ranges decreases gradually. These results clearly indicate that alkali treatment of the fibre removes the hemicellulose content to a large extent and reduces the water absorption characteristics of the LSFs, which improve the quality of the fibre.

The lignin fraction of raw LSFs showed the characteristic vibrational frequencies for C = C, C-O, -CH₃, -CH groups at 1,636, 1,507, 1,423, 1,368 and 1,058 cm^{-1} (Kommula et al., 2016). The intensity of the lignin peak observed at 1,058 cm^{-1} gradually increases at 2% to 4% alkali treatment and then sharply decreases. This results in a partial removal of lignin at lower concentration of alkali treatment but the rate of its removal increases at higher concentrations of treatment. The existence of lignin renders thermal stability to the fibre and it is significant at 4% alkali treatment.

3.3 Thermal stability analysis of raw and alkali treated LSFs

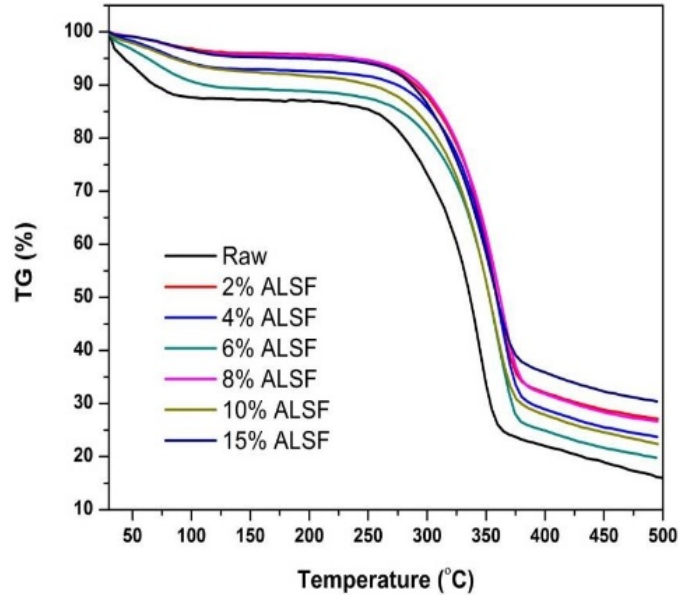
The thermal stability of fibre reinforcement is a prominent characteristic for construction of composite materials. All natural fibres are found to have less thermal stability than synthetic fibres. The thermal degradation characteristic of raw and ATLSFs were studied by primary thermogram (TG) and derivative curves of differential thermogram (DTG).

Figure 2 shows the TG and DTG derivative curves of raw and ATLSFs. The TG curve of the raw and ATLSFs have three decomposition peaks. There is an insignificant initial degradation of the fibre with weight loss of 8%–10% due to the removal of water content from the fibre in the temperature range of 80°C–100°C (Saheb and Jog, 1999). Depolymerisation of hemicelluloses content of the fibre caused the major degradation in the temperature range between 240°C and 300°C and decreases the weight of fibre from 8%–12% (Senthamaraiannan et al., 2016).

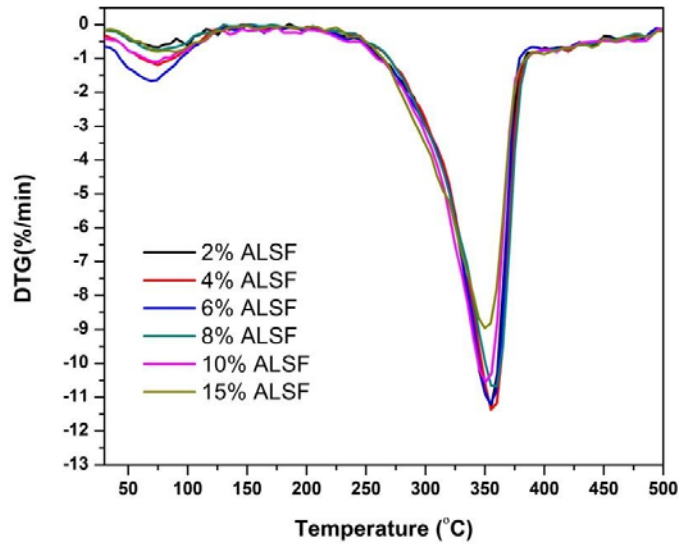
A prominent degradation peak observed in the temperature range of 330°C–370°C with a core weight loss 65%–80% was because of the significant degradation of cellulose content of the fibre (Reddy et al., 2014). DTG revealed the thermal stability of cellulose part of the LSFs fibre by a prominent peak appearing at 339.1°C (raw), 355.2°C (2%), 355.5°C (4%), 355.0°C (6%), 354.3°C (8%), 350.6°C (10%) and 349°C (15%) were obtained for raw and alkali treated fibre. Besides having a major cellulose and lignin content, the LSFs have also been attached with to less thermally stable component, which may undergo earlier thermal degradation exert a lower thermal stability on the raw fibre. Evidently, the thermal stability of LSFs was enhanced by alkali treatment, due to the decomposition or elimination or hydrolysis of such less thermally stable components adhered to cellulose and lignin (Izani et al., 2013). Of various concentrations of alkali treatment, 4% ATLSFs has the maximum thermal stability. Soon after, there was a weight loss of 8%–12% of the fibre, due to partial decomposition of lignin and cropping up of other organic content after 400°C (Kathiresan et al., 2016). Ultimately, there was a definite amount of residual mass left out because of the existence of high-energy

degrading constituents and ash. The thermal stability of natural fibres is a fundamental feature in the development of natural fibre composites. Moreover, the optimally treated LSFs can enduring the temperature up to 270°C; which makes it suitable for the casting of thermos- or thermoset composites in this temperature range. Thus, ATLSFs can be used as a renewable potential reinforcement for composite materials.

Figure 2 TG and DTG of raw and alkali treated LSFs (see online version for colours)



(a)



(b)

3.4 Single-fibre tensile testing of raw and alkali treated LSFs

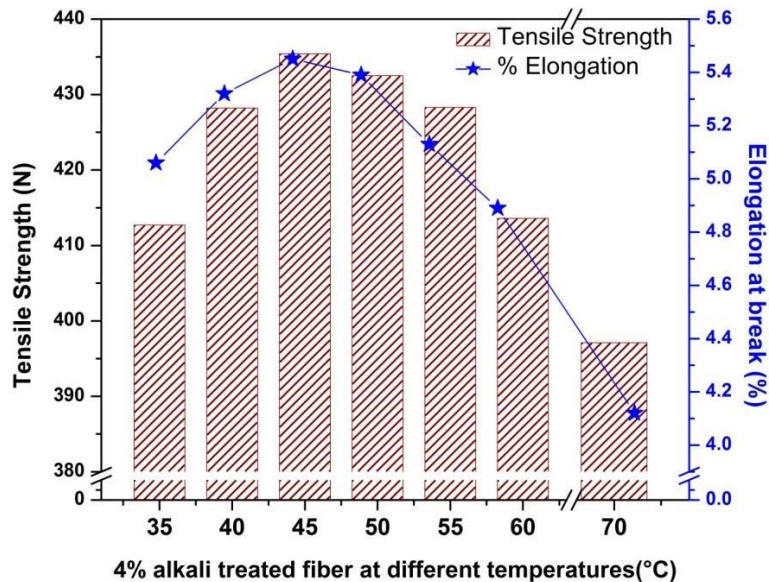
Single-fibre tensile strength is a distinctive parameter, for any fibre can be used as a reinforcement. The average tensile strength and elongation at the break of the raw LSFs and the various concentrations of alkali-treated fibres are shown in Table 2.

Table 2 Tensile strength of raw and alkali treated LSFs

Fibre	Tensile strength(N)	Elongation at break (%)
Raw LSF	415.8	3.33
2% ATLSF	422.1	3.52
4% ATLSF	436.8	5.47
6% ATLSF	430.7	5.17
8% ATLSF	422.6	4.85
10% ATLSF	416.3	4.69
15% ATLSF	399.6	3.88

The results clearly show that the average tensile strength and elongation at the break (%) are markedly increased up to 4% ATLSFs and then decreases significantly at higher concentrations. Removal of impurities by alkali treatment induced the rearrangements of the fibrils due to the reduction of fibril angle, which causes the dense packing of cellulose chains, improving the physical and mechanical properties of the fibre (Taha et al., 2007).

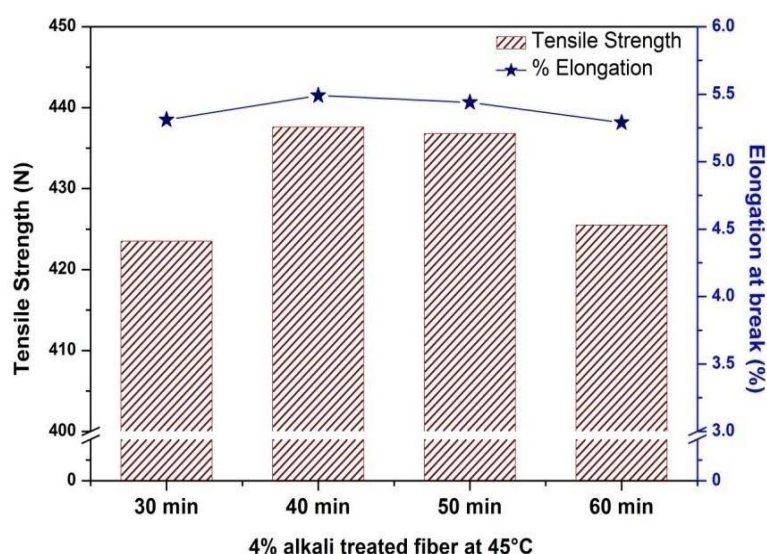
Figure 3 Tensile strength of 4% alkali treated LSFs at different temperature (see online version for colours)



Alkali treatment not only removed the adhered impurities but also made open the cellulose fibrils (Izani et al., 2013). It allows a strong interaction between the fibrils and alkali, which causes a deformation in the crystalline cellulose structure (Nishiyama and Okano, 1998). Alkali treatment induces degradation of the opened cellulose fibrils,

resulting in the chain-breaking and renders poor tensile strength (Beckermann et al, 2004). At higher temperatures, the cellulose chain undergoes depolymerisation, which results in the reduction of tensile strength (Maya and Thomas, 2008). Increase of boiling temperature and time may eliminate the impurities at a faster rate; however, this adversely affects the stiffness and the strength of the fibre. As a result, 4% ATLSFs at 45°C for 40 min have significant tensile strength. Figure 3 shows the single-fibre tensile strength of 4% ATLSFs at different temperatures. Figure 4 shows the single-fibre tensile strength of 4% ATLSFs at different time intervals.

Figure 4 Tensile strength of 4% alkali treated LSFs at different time interval (see online version for colours)



4 Conclusions

Alkali-treated LSFs would eliminate the secondary metabolites such as wax, fat, hemicellulose, lignin, pectin and other organic materials adhered to the cellulose fibre, which turns the smooth surface of the fibre into a rough surface. This improves the adhesion of fibre with matrix while fabricating the composites, ensuring a greater tensile strength of the composite material. Alkali treatments help in the rearrangements of micro-fibrils along with sealing of micro-voids with grafted molecules, which improve the physical and mechanical properties of the fibre.

The investigation illustrates that higher-concentration alkali treatment (above 4% up to 15%) damages the crystalline characteristics of the cellulose, thereby decreasing the mechanical and thermal stability. If alkali concentration is less than 4%, no significant difference in tensile strength and thermal stability of the fibre is observed. At the outset, 4% ATLSFs at 45°C for 40 min have a marked tensile strength and good thermal stability, making them a suitable lightweight composite material for structural and automobile applications.

Extraction of fibre from the agro waste reduces the environmental threats to a larger extent and serves as a potential source of natural fibre on a large-scale. It is a boon for environment and prevents human health deterioration. The consumer items produced from these bio-composites cause minimal environmental harm, which helps us to reduce our carbon footprints. We may live and produce eco-friendly and sustainable communities. In this regard, the 3-R concept of reduce; reuse and recycle may lead us to map our daily energy consumption.

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