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Effect of temperature and durations of heating on coir fibers

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ABSTRACT

Biocomposites derived from polymeric resin and lignocellulosic fibers may be processed at temperatures ranging from 100 °C to 230 °C for durations of up to 30 min. These processing parameters normally lead to the degradation of the fiber's mechanical properties such as Young's modulus (*E*), ultimate tensile strength (UTS) and percentage elongation at break (%EB). In this study, the effect of processing temperature and duration of heating on the mechanical properties of coir fibers were examined by heating the fibers in an oven at 150 °C and 200 °C for 10, 20 and 30 min to simulate processing conditions. Degradation of mechanical properties was evaluated based on the tensile properties. It was observed that the UTS and %EB of heat treated fibers decreased by 1.17–44.00% and 15.28–81.93%, respectively, compared to untreated fibers. However, the stiffness or *E* of the fibers increased by 6.3–25.0%. Infra red spectroscopy (FTIR), thermogravimetric analysis (TGA) and scanning electron microscopy (SEM) were used to elucidate further the influence of chemical, thermal and microstructural degradation on the resulting tensile properties of the fibers. The main chemical changes observed at 2922, 2851, 1733, 1651, 1460, 1421 and1370 cm^{-1} absorption bands were attributed to oxidation, dehydration and depolymerization as well as volatization of the fiber components. These phenomena were also attributed to in the TGA, and in addition the TGA showed increased thermal stability of the heat treated coir fibers with reference to the untreated counterparts which was most probably due to increased recrystallization and cross linking. The microstructural features including microcracks, micropores, collapsed microfibrils and sort of cooled molten liquid observed on the surface of heat treated coir fibers from the scanning electron microscope (SEM) could not directly be linked to the effect of temperature and durations of heating although such features may have largely account for the lower tensile properties of heat treated coir fibers with reference to untreated ones.

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1. Introduction

As a potential biodegradable and renewable lignocellulosic fiber, coir fibers have been increasingly used in making biocomposites (Geethamma et al., 2005; Monteiro et al., 2005, 2008) for various applications in automobiles, construction, packaging and insulation. However, in order to achieve this, lignocellulosic fibers need to be modified and processed to make them match with the properties of the matrix or binder. Some of these processes involve exposing the fibers to high temperatures (>150 °C), high pressures (~30 MPa) and sometimes for longer period (>30 min) in order to optimize the properties. The conventional polymeric binders that are normally employed for composite manufacture require also some heating for melting, curing or vulcanization which varies depending on the type of polymer used. For conventional polymeric binders such as polystyrene, polypropylene, polyethylene and polyvinyl chloride, usually the required processing temperatures ranges from 60 °C to 230 °C (Van de Velde and Baetens, 2001).

However, lignocellulosic fibers thermally degrade through dehydration, depolymerization, and oxidation when heated (LeVan, 1989; Mamleev et al., 2007; Silva et al., 2000; Tomczak et al., 2007; Van Dam et al., 2004; Varma et al., 1986). For example, it was reported that lignin begins to undergo thermal degradation around 100 °C (Maiti et al., 2007; Yang et al., 2007). The thermal stability of hemicelluloses is also below this temperature, and it is possible that since this polymer is amorphous it will become soft (Silva et al., 2000) and flow, whereas cellulose crystalline structure is beginning to be destabilized around the same temperature (Mamleev et al., 2007). Mechanistically, this involves the breakdown of β-1,4-glycosidic chains from celluloses and hemicelluloses (LeVan, 1989; Mamleev et al., 2007) as well as the cleavage of β-O-4-alkyl-aryl ether linkages from lignin (Evans et al., 1986). Generally, the binding components of microfibrillated celluloses are removed when

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the fiber is heated. A series of chemical explosions due to combustion, most probably, are also associated with chain breakdown reactions during thermal degradation, which ultimately influence mechanical properties due to the formation of defects such as cracks (Chand and Hashmi, 1993; LeVan, 1989). By heating flax and jute fibers in the temperature range from 165 °C to 240 °C for 6 h, Gassan and Bledzki (2001) showed as well that thermal degradation tend to depolymerize the fiber. The degree of polymerization (DP) decreased from 5360 to 320 (Gassan and Bledzki, 2001). In the study conducted on flax fiber (Van de Velde and Baetens, 2001), it was shown that ultimate tensile strength (UTS) and percentage elongation at break (%EB) of flax fiber decreased by 13–18% whereas the Young's modulus (E) remained unchanged when fibers were exposed at 120 °C for the duration of 120 min. When the temperature was increased furthermore to 180 °C for the same duration of heating, the value of UTS decreased more by 32–36% while %EB decreased by 40–56%. These studies suggest that thermal degradation actually has influence on chemical and mechanical properties of the fiber. However, this study is more interested on the mechanical degradation of coir fiber due to the influence of temperature and durations employed during processing of composites, which previously was not covered. Since all lignocellulosic fibers have similar major cell wall components and tend to exhibit the same degradation behavior (Munawar et al., 2007), therefore coir fibers are also expected to follow the same pattern.

The processing conditions have normally been optimized in order to improve mechanical properties of composites by enhancing the interaction between hydrophobic binders and hydrophilic reinforcements. For example, the best processing conditions for coffee husks particleboards were optimized at 160–170 °C and 4–5 min (Ogola, 2000). Xue et al. (2009) also suggested a temperature of 170 °C (or 180 °C) for processing of conventional polymeric binders. While in industrial practices, wood-based panels are processed at temperature of around 170–190 °C for the durations of up to 5 min. But, as reported previously (LeVan, 1989; Mamleev et al., 2007; Silva et al., 2000; Tomczak et al., 2007; Van Dam et al., 2004; Varma et al., 1986), components of lignocellulosic fibers will begin to thermally degrade even around 100 °C, and sometimes produces bad smell odorous compounds (Espert et al., 2005). Therefore, temperature is a crucial parameter during the manufacture of biocomposites. The effect of temperature and durations of heating on mechanical properties of coir fiber can be studied by exposing the fibers at different temperatures without actually making composites. The results conducted under this condition can be compared with those obtained previously from flax fibers (Gassan and Bledzki, 2001; Van de Velde and Baetens, 2001) to find out how they differ or agree.

Since tensile test technique has been used to establish mechanical properties such as E, UTS and %EB of lignocellulosic fibers (Munawar et al., 2007) including coir fibers (Kulkarni et al., 1981; Tomczak et al., 2007), then a similar technique can also be used to study the degradation of tensile properties of heat treated coir fibers.

Therefore, the main aim of this study is to address the effect of temperature and durations of heating on the tensile properties of coir fibers exposed to similar temperature range and durations employed during processing of composites. The influence of chemical and thermal degradation as well as microstructural degradation on the mechanical properties of fibers was further studied by using infrared spectroscopy, thermogravimetric analysis and scanning electron microscopy, respectively. It is foreseen that the results of this study can be used to explain further the effect of temperature and durations of heating during processing on mechanical properties of lignocellulosic fibers.

2. Materials and methods

2.1. Material

Coir fiber which is obtained from coconut husk by extraction process such as decortications was bought from Zanzibar, Tanzania and was used as received. The average moisture content of fibers ranged from 10% to 12%.

2.2. Sample treatment

The samples of coir fibers were grouped as CF, CF15010, CF15020, CF15030, CF20010, CF20020 and CF20030. The categorization of the samples was done according to temperature and duration of heating applied. CF stands for coir fiber, the first three digits refer to temperature in degree centigrade and the last two digits indicate the duration of heating in minutes. Coir fibers were heated in air in an oven for 10, 20 and 30 min at 150 °C and 200 °C. The sample denoted by CF alone is representing the untreated coir fibers.

2.3. Tensile tests

In order to carry out the tensile test, the diameter of the fiber was established using density, mass and volume relationship shown in Eq. (1) based on the assumption that fibers are cylindrical as suggested by Gassan et al. (2001). The diameter of the lumen is also assumed to be relative very small compared to the diameter of the fiber, therefore the whole fiber may be considered as a solid material.

$$D = 20\sqrt{\frac{10m}{\rho\pi h}} \quad (1)$$

where, D is diameter (mm), m is mass (g), ρ is density (g/cm^3), h is length (mm) of the coir fiber and π is a constant, equals to 3.14.

The density of coir fiber used was 1.15 g/cm^3 from literature (Bisanda and Ansell, 1992; Bledzki et al., 1996; Monteiro et al., 2005; Satyanarayana et al., 1986). Mass and length was measured by using a Mettler Toledo weighing machine and a meter rule. The length of coir fiber samples measured was about 90 mm long. The diameter of the fiber used ranged from 0.200 mm to 0.225 mm (Tomczak et al., 2007). Each coir fiber sample was mounted on a cardboard box and glued with epoxy to prevent slippage and damage. The size of the cardboard box was 50 mm to 100 mm. About 25 coir fibers were prepared for each sample group. Since lignocellulosic fibers are sensitive to moisture, the samples were then conditioned overnight at 24 °C and 50% relative humidity to ensure uniform stabilization of the mechanical properties. The tensile test was carried out by using Instron 5566 Universal Tensile Machine Model. The value of gauge length and strain rate was set at 50 mm and 20 mm/min, respectively (Kulkarni et al., 1981). The tensile properties measured include E, UTS and %EB.

2.4. Infra-red spectroscopic analysis

Surface chemical properties of untreated and heat treated coir fibers were analyzed by infrared spectroscopy using PerkinElmer (Spectrum, 2000) in attenuated total reflectance mode. The fibers were scanned between 600 cm^{-1} and 4000 cm^{-1}, and other specifications used include number of scans which was set at 16, the interval of 1 cm^{-1}, a gain of −1 and a resolution of 4 cm^{-1}.

2.5. Thermogravimetric analysis

The effect of temperature and duration of heating on heat treated coir fibers with respect to untreated coir fibers were stud-

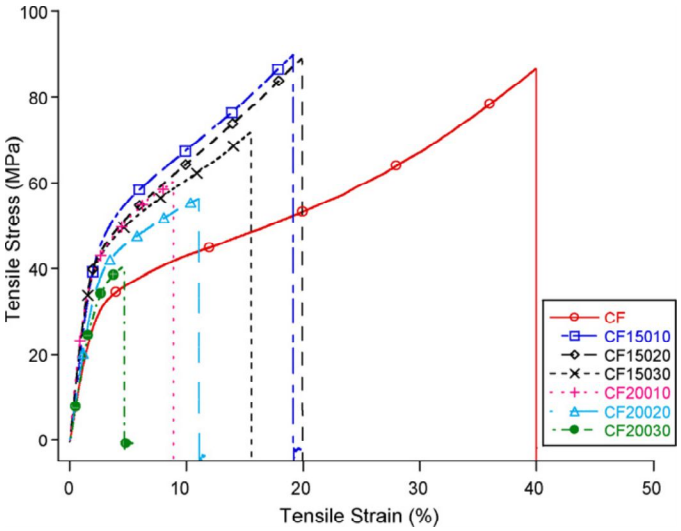


Fig. 1. Stress–strain curves of heat treated and untreated coir fibers.

ied by using a Mettler Toledo TGA/SDTA851 thermogravimetric analyzer. The samples were crushed into small pieces before testing. The weight was ranged from 11 mg to 13 mg and each sample was placed into the aluminum crucibles (70 μl). The samples were dynamically heated from 25 °C up to 800 °C at the heating rate of 10 °C/min in a nitrogen flow of 50 ml/min.

2.6. Scanning electron microscopic analysis

The microstructural degradation of coir fibers was studied using a scanning electron microscope (SEM). The fibers were cut into small sizes of approximately 1 mm long. The fibers were conditioned in a desiccator for 24 h before the experiment to dry the samples. All specimens were gold sputtered prior to testing in the S-4800 scanning electron microscope.

3. Results and discussion

3.1. Tensile properties of coir fibers

Fig. 1 presents stress against strain curve for the heat treated and untreated coir fibers. The value of *E* was calculated from the slope of the linear portion of the stress–strain curve. The values of UTS and %EB were obtained as average values of the individual fiber samples. It was observed that during tensile loading the untreated coir fibers deformed more before breaking compared to the heat treated coir fibers. The premature failure of the heat treated coir fibers was attributed to the increase in the number of defects as previously observed by Chand and Hashmi (1993) for sisal fibers, and this is due to the chemical breakdown of the fiber's components and subsequently, to the removal of degradation products which lead to the formation of cracks, holes or voids (LeVan, 1989).

Table 1 shows the average tensile properties of the coir fiber samples investigated. By contrast with coir fibers from Brazil (Silva et al., 2000; Tomczak et al., 2007) and India (Kulkarni et al., 1981; Satyanarayana et al., 1986), it was observed that, tensile properties of the untreated coir fibers investigated are similar to coir fibers from Brazil. The findings by Harries (1978) showed that actually coconut palm from South America was once spread to East Africa during ancient days, suggesting that these fibers have similar tensile properties because they are probably of the same species, assuming that the influence of geographical location is small. For the heat treated coir fibers, the value of *E* increased slightly by 6.3–25.0% when referring to *E* of the untreated fibers. When referring to the value of UTS of heat treated fibers, the value of UTS of untreated coir fibers decreased by 1.17% at 150 °C and 44.00% at 200 °C. The value of %EB also decreased significantly by 15.28% at 150 °C and 81.93% at 200 °C compared to the value of %EB of untreated fibers. It was observed that the highest values of *E*, UTS and %EB were obtained when the fiber was heat treated at 150 °C for 20 min. Apart from showing higher values of *E* (2.0 GPa) and UTS (72.4 MPa) than the untreated fibers, but in addition, the value of %EB was the highest with reference to other heat treated fibers, and with only 11.32% reduction of %EB compared to untreated fibers.

The decrease of *E* by a small margin with respect to the duration of heating showed that *E* is less affected with this parameter compared to UTS and %EB at the same temperature condition. At 120 °C and for the duration of 120 min, flax fibers showed no change at all on the value of *E* (Van de Velde and Baetens, 2001). The decreased values of *E* as well as UTS and %EB were very significant with respect to the durations of heating for fibers heat treated at 200 °C. This is because at longer durations (6 h) and high temperature (165–240 °C) heat treatments of lignocellulosic fibers have more detrimental effect on DP (Gassan and Bledzki, 2001). This will be illustrated better later on thermal degradation curve of coir fibers. A critical value may however be observed whereby the value of *E* increases and then decreases with temperatures and durations of heating and this can be noticed from sample CF15020 in comparison to sample CF and CF20020 with respect to temperature, as well as by comparing sample CF, CF15020 and CF15030 with respect to durations. This may be interesting for composite processing. It is obvious from this study that, exposing lignocellulosic fibers to temperature of 200 °C or above is not suitable for composite processing. However, the results of this study may probably not be conclusive as the influences of other processing parameters such as pressure are not taken into accounts. The statistical analysis is also not discussed here. Since composites are produced from more than one component, mechanical properties even of the same material may show wide variations and therefore the statistical analysis of the influence of the processing parameters may somehow be necessary.

Table 1
Tensile properties of heat treated and untreated coir fibers.

S/no.	Samples	Average *E* (GPa)	Average UTS (MPa)	Average %EB (%)
1	CF	1.6 (0.6)	68.4 (23.2)	21.2 (4.8)
2	CF15010	2.0 (0.8)	68.0 (12.5)	18.0 (8.5)
3	CF15020	2.0 (0.6)	72.4 (24.5)	18.8 (1.6)
4	CF15030	1.9 (0.5)	67.6 (8.9)	18.4 (6.1)
5	CF20010	2.0 (0.7)	40.8 (16.6)	05.1 (1.5)
6	CF20020	1.8 (0.6)	38.8 (11.4)	04.8 (4.4)
7	CF20030	1.7 (0.5)	38.0 (2.0)	03.8 (1.0)
8	Brazil coir	2.1	75.0	29.5 (Silva et al. (2000))
9	Indian coir	5.0	175.6	34.0 (Kulkarni et al. (1981))

Note: The number in brackets indicate standard deviation.

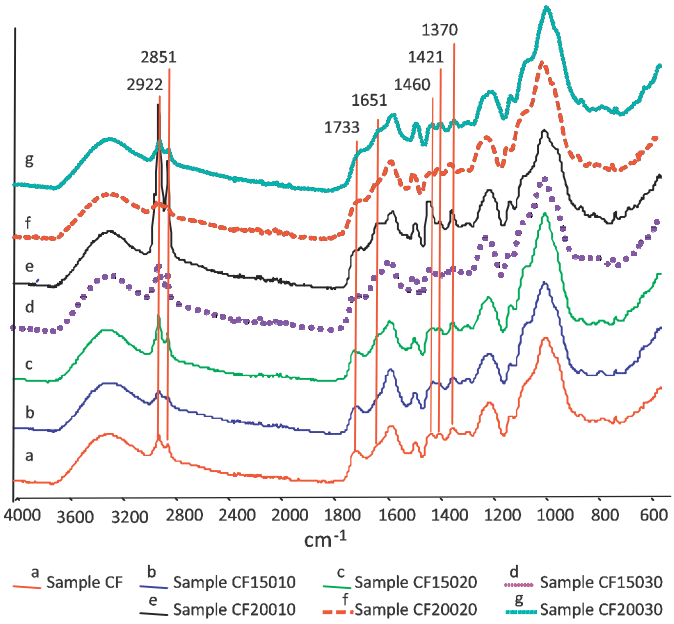


Fig. 2. FTIR of heat treated and untreated coir fibers.

3.2. FTIR analysis of coir fibers

Fig. 2 presents the FTIR results of untreated and heat treated coir fibers. The observed increase of vibrational intensity at 2922 cm^{-1} and 2851 cm^{-1} from C–H stretching and shifting of absorption band from 1733 cm^{-1} towards 1651 cm^{-1} with durations of heating indicate the formation of carboxylic acids and was mainly associated with dehydration, oxidation and depolymerization, which normally takes place in parallel with volatization of the fiber components (Silva et al., 2000). These changes were accompanied with the increase of vibrational intensity of C–H and O–H deformation bonds at 1460, 1421 and 1370 cm^{-1} from lignin. The thermal degradation is intensified further with the durations of heating when the fiber was heat treated at 200 °C, whereby the vibrational intensity not just reduced but collapsed after 20 min and 30 min heat treatment. From several studies (Chand and Hashmi, 1993; LeVan, 1989; Mamleev et al., 2007), which discussed and presented thermal properties of lignocellulosic fibers, the collapsing of such vibrational intensity may be attributed to removal or evolution of degradation products. In another perspective, it may as well conceptually be viewed as the formation of holes, voids and cracks as described elsewhere (Chand and Hashmi, 1993; LeVan, 1989; Mamleev et al., 2007), which literally are detrimental to the fiber.

3.3. Thermogravimetric analysis of coir fibers

Fig. 3 shows the TGA of heat treated and untreated coir fibers under nitrogen atmosphere. It should be noted that the initial weight loss over the temperature range from 30 °C to 100 °C is due to dehydration and can be considered beneficial during processing as it makes the fiber more flexible and ease to buckle (Ndazi et al., 2006) as well as support heat transfer (Kelly, 1977). The TGA curves of the heat treated fibers under nitrogen atmosphere also exhibited a similar pattern as the TGA curves of untreated coir fibers shown by Tomczak et al. (2007) under oxygen atmosphere, which have been attributed to the thermo-oxidative effect induced earlier by oxygen from air during isothermal heating of the fibers in the oven. The residual weight of heat treated coir fibers was also higher than that of the untreated fibers over the temperature range from 100 °C to 250 °C, which, apart from thermo-oxidative effect, may also be linked to volatization of low molecular weight components in lignocellulosic fibers as well as to the cleavage of polyuronic acids bonds (Sreenivasan et al., 1996; Van de Velde and Baetens, 2001; Varma et al., 1986). This might be the reason why the crystalline phase of the fiber would increase and leading to the increase of the thermal stability as well. Since thermal stability is influenced by chemical interaction inside the fiber, it can be linked to the effect of heat treatment to the tensile properties of the fiber. The existence of

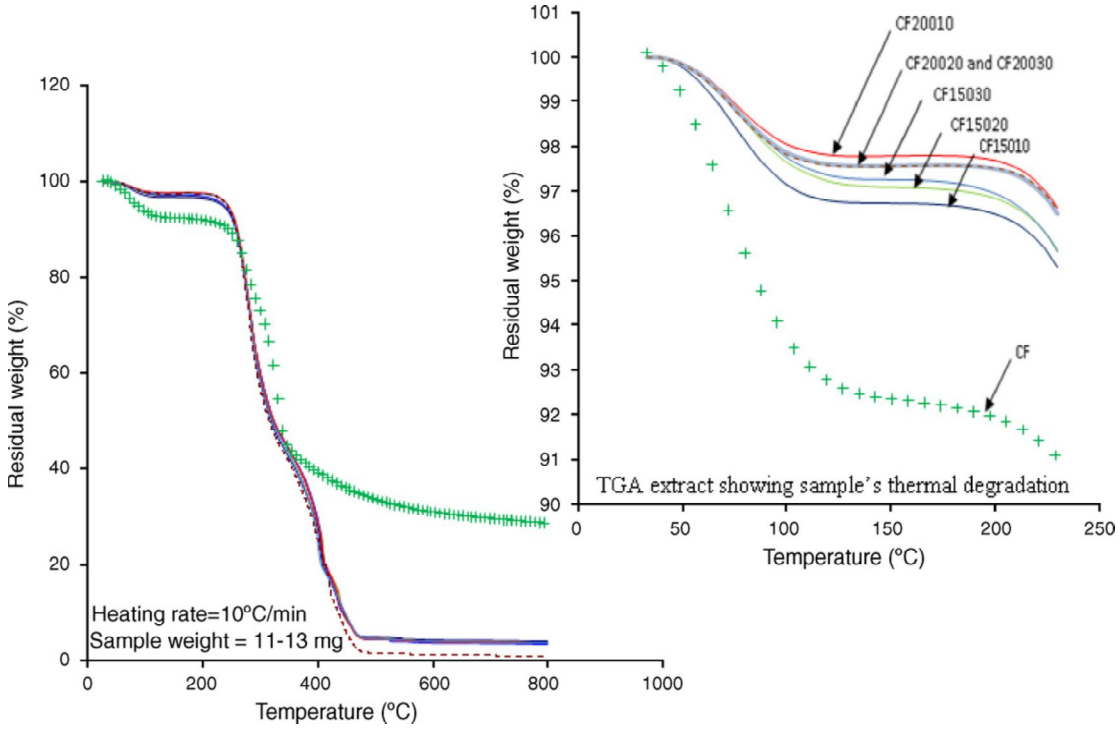


Fig. 3. TGA curves of the heated treated and untreated coir fibers under nitrogen atmosphere.

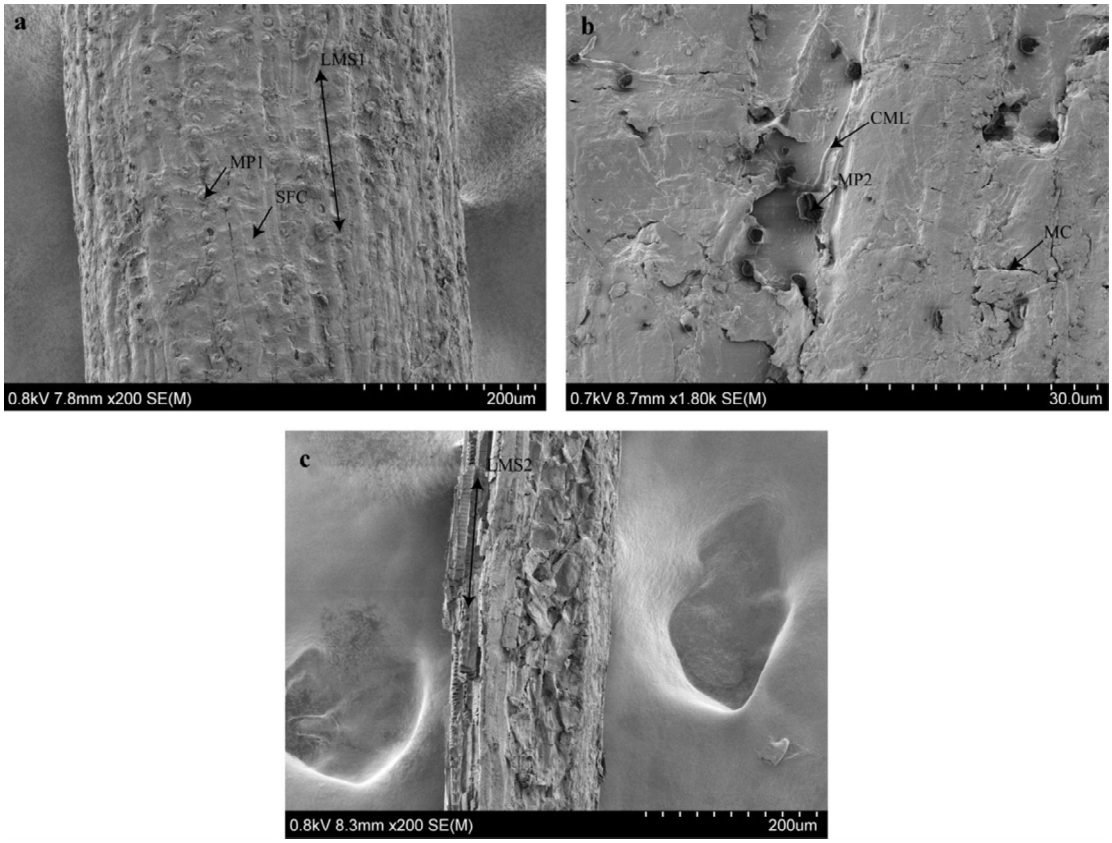


Fig. 4. SEM micrographs of heat treated and untreated coir fibers, (a) untreated coir fiber (b) heat treated coir fiber at 150 °C for 20 min and (c) heat treated coir fiber at 200 °C for 30 min (LMS1 is the longitudinal direction of microfibril strands, LMS2 is a destroyed microfibril strand, MP1 shows closed micropores and MP2 is the open micropores; SFC represent a segment of a fiber cell; MC is a microcrack and CML is sort of cooled molten liquid from fiber components).

low molecular weight components in the fiber as non-condensable components such as water vapor, pectin as well as extractives (Teeri et al., 2007), and in addition, the presence of lignin which is affected over this temperature range through free radical bond cleavage of the β-O-4-alkyl-aryl ether linkages (Evans et al., 1986), with the formation of thermally stable monomers and dimmers as explained in Rice–Herzfeld free radical model, by softening (Silva et al., 2000) or cross-linking (Van Dam et al., 2004; Vázquez-Torres et al., 1992) may help to furthermore explain the aforementioned thermal degradation phenomena much better in the context of chemical changes. The increase in thermal stability, for example, has been associated with the increase in stiffness of the fiber, and definitely, loss of the amorphous hemicelluloses and other non-condensable components responsible for the fiber's plasticization can also be the reason of not just large decrease of UTS and %EB but also of high sensitivity of the properties to temperatures and durations of heating. Apart from that, cellulose has also been associated with depolymerization, which apparently dominates over temperature ranges near 200 °C (Mamleev et al., 2007). The reduction of DP from 5360 to 320 by heating flax and jute fibers at the temperature of 165 °C and 240 °C for 6 h presented by Gassan and Bledzki (2001) gives a vivid example of how detrimental depolymerization is on the properties of the fiber. Since the temperature at which depolymerization phenomena commence is around the temperature range at which the fiber is exposed during processing, and because the phenomena itself from Fig. 3 is very fast, working around this temperature or above it could immensely affect the overall performance of lignocellulosic fibers and therefore may reduce the reinforcement potential of these fibers in composites.

Although the study did not aim to elucidate mechanistically what happen during heat treatment or the phenomenon associated with it, which lead to the degradation of mechanical properties, it is however important to understand that changes that are observed on TGA curves cannot fully explain the influence of heat treatment in this context, because TGA does not show exactly where most thermal degradation reactions begin. It is difficult to know if some detrimental chemical reactions started to take place unnoticeably within the fiber before the 150 °C or 200 °C temperatures. Wood, for example, have the tendency to cross link immediately and form an outer cage, which prevent further evolution of degradation products and thus promote charring especially in the absence of oxygen (LeVan, 1989). Technically, TGA technique generally works out well if there is weight loss, which only happens during gasification and not otherwise. By gasification it means chemical or thermal conversion of fiber components to gaseous products such as hydrogen, carbon dioxide, water vapor and other volatile organic compounds which may very often take place by combustion.

3.4. Scanning electron microscopic analysis

Fig. 4 shows SEM micrographs of untreated and heat treated coir fibers. Fig. 4(a) is untreated coir fiber revealing longitudinally fiber cells, micropores and microfibrils strands. Some microcracks and open micropores can be seen on the coir fiber that was heat treated at 150 °C for 20 min in Fig. 4(b). Untreated coir fiber also has irregular outer layer with twisted and shrink lines as observed elsewhere (Rahman and Khan, 2007). At higher magnification in Fig. 4(b), a sort of cooled molten liquid can be observed as if pouring out from the micropores. This could probably be hemicelluloses and other low molecular weight components which had softened, because they are amorphous and have low thermal stability (LeVan, 1989). The presence of collapsed microfibrils can be seen for the heat treated

coir at 200 °C for 30 min in Fig. 4(c) with reference to untreated coir fiber. However, from other SEM micrographs (not shown) it can be argued that probably degradation of microstructure is not only due to high temperature treatment but also by other harsh processes such as dry milling or decortication employed during extraction of the fibers. The retained outer layer which was observed from the SEM micrographs after different heat treatments indicates that probably this layer is very resistant to degradation at these conditions. But, it is also possible that features due to microstructural degradation as the result of heat treatment were too small to be identified using this technique, maybe by using a more powerful microscope. Otherwise, the effect of temperature and duration of heating on the microstructural degradation for the same fiber or sample should be explained after establishing microstructural features due to the effect of other processes especially extraction ones. However, such surface defects on the microstructures of heat treated and untreated coir fiber could explain why the coir fibers studied have the lower tensile properties compared to the coir fibers from India.

4. Conclusions

It was observed that coir fibers from Tanzania have to large extent similar properties to coir fibers from Brazil. The values of *E*, UTS and %EB were averaged 1.6 GPa, 68.4 MPa and 21.2%, respectively for untreated coir from Tanzania. When the coir fibers were heat treated at temperatures of 150 °C and 200 °C for the durations of 10, 20 and 30 min to simulate processing conditions, it was concluded that despite a slight increase of the value of *E*, processing temperatures around 200 °C and above and durations of heating above 30 min, even at lower temperature than this, are generally detrimental to the fiber mechanical properties. The effect of temperature and durations of heating using FTIR and TGA was attributed to dehydration, oxidation and depolymerization which have severe effect on the properties of the fibers. Increased thermal stability in TGA is associated with the loss of some fiber components as well as degradation products as it was also observed from FTIR and is related to the degradation of UTS and %EB and increased value of stiffness of heat treated with respect to untreated coir fibers. Increased thermal stability of heat treated coir fibers is also attributed to the increase of the degree of recrystallization and cross linking of the fiber's components which causes the increase in stiffness of the fiber. However, the value of stiffness decreases with durations of heating. The microstructural defects observed on the surface of the fibers such as microcracks, irregular outer layer with twisted and shrink lines could be the reasons for lower UTS, %EB and the decrease of *E* with durations of heating. Since we did not have any control over the extraction processes of the coir fibers, it may be plausible to consider the extraction effects in order to properly address the influence of processing conditions on the properties of coir fibers.

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