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Mechanical properties of sisal fibre at elevated temperatures

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Sisal fibres extracted from the leaves of *Agava sisalana* plants 3, 5, 7 and 9 years old were tested at different temperatures for tensile strength, elongation, toughness and modulus. The tensile strength, modulus and toughness values of sisal fibre decreased with increase in temperature. The effect of plant age on tensile strength, tensile modulus and toughness of sisal fibre became very much less at 100 °C as compared to 30 °C. Fractured fibres were observed by using a scanning electron microscope. The ends of fibres fractured at elevated temperature showed a failure similar to that of inorganic fibres. Elongation values at all temperatures increased with age. Elongated capillaries were observed in fibres fractured at 80 and 100 °C, due to the removal of moisture and volatiles originally present in the fibres. The fibrils are clearly observed in the form of hollow cylinders. Fractured surfaces are composed of brittle as well as ductile phases. The ductile portion increased with the increase of temperature.

1. Introduction

Plant fibres are lignocellulosic in nature. Among these lignocellulosic fibres, sisal is one of the world's most important fibres [1]. Sisal fibre is obtained from the leaves of *Agava sisalana*. It has the potential to replace synthetic fibres in various applications. This fibre has been used in making polymer composites as a reinforcing filler [1].

The agricultural and economic importance of natural fibres have been well documented, but less information is available concerning the high-temperature properties of these lignocellulosic fibres. The mechanical behaviour of the fibres at high temperature is of imporance from the industrial application point of view. The structure of the natural fibre is similar to that of naturally occurring polymeric composites [2]. These composites contain load-bearing elements held within a matrix. At room temperature the strength depends upon the cellulose content of the fibre [3].

In addition to the light weight and higher sound attenuation characteristic of sisal fibre, it does not present the problem of toxicity and waste disposal. The physical and mechanical properties of commercial sisal fibre have been measured by many investigators [4–7] at room temperature. The properties so far reported in the literature show a wide variation in the strength values [4–6]. This variation reduces the possibility of using these fibres for high-technological applications, where reliability and consistency are important factors. In order to reduce this variation the present authors [8] determined the chemical constituents and physical properties of sisal fibres from plants of different ages. It was observed that the variation in the strength values of sisal fibres could be brought down by classifying these fibres on the basis of the plant age. Adopting the same classification, the mechanical properties of sisal fibres have been evaluated at elevated temperatures.

2. Experimental procedure

The sisal fibres used in the present study were collected from Bilaspur (Madhya Pradesh, India). Leaves of *Agava sisalana* were taken from plants 3, 5, 7 and 9 years old. Fibres were extracted by using a Raspador machine. The fibres were washed in water, dried in the sun and kept in the shade for a year for seasoning purposes.

The ultimate tensile strength (UTS), elongation percentage and modulus at elevated temperatures were determined using a tensile testing machine (Instron model 1185) in which high-temperature testing facilities were provided. Test samples of sisal fibres in cardboard windows were prepared as described in our previous paper [2]. A slit 10 mm × 50 mm was cut in a rectangular piece of cardboard of size 30 mm × 90 mm. The fibre was mounted with the help of adhesive tape at both ends. After fixing the sample between two grips of the testing machine, the two wide strips of cardboard were cut with scissors before testing the fibre. Fibres were tested at a crosshead speed of 20 mm min^{-1}. The modulus of fibres was determined at 0.5% elongation by measuring the slope of the stress–strain curve. The toughness of fibres was determined by measuring the area under the stress–strain curve for each fibre. Average values of UTS, modulus and toughness of 30 samples are reported here.

An optical microscope (Leitz) was used for determining the cross-sectional area of each fibre. Fractographs of fibres were observed by using a scanning electron microscope (Jeol 35 CF).

The moisture percentage and rate of moisture removal was measured by using a Mettler balance model PE-160.

3. Results and discussion

Curves (a) to (c) in Fig. 1 show the effect of plant age on tensile strength of sisal fibre at temperature 30, 80 and 100 °C, respectively. Sisal fibres extracted from plants 3, 5, 7, and 9 years old were examined. At 30 °C the UTS increased with plant age. The fibres from plants of the four different ages showed average UTS values in the range 452.0 to 581.2 MPa, as shown in curve (a) of Fig. 1, with standard deviations of 55.3, 54.8, 60.2 and 58.9 MPa, respectively. The average UTS of sisal fibre at 80 °C decreased from 350.2 to 316.3 MPa with increase of plant age as shown in curve (b) of Fig. 1. The standard deviation of UTS at 80 °C was calculated as 46.3, 41.0, 51.9 and 49.2 MPa for plants 3, 5, 7 and 9 years old, respectively. Curve (c) of Fig. 1 shows a straight-line plot for the UTS of sisal fibres extracted from plants of different ages at 100 °C; UTS values ranged from 302.5 to 338.7 MPa with standard deviations 52.4, 56.9, 48.2 and 42.5 MPa, which indicates that the UTS is nearly constant at 100 °C.

Curves (a) to (c) of Fig. 2 show the effect of plant age on elongation of sisal fibres at 30, 80 and 100 °C, respectively. These results clearly show that the elongation percentage increased with plant age. The values of elongation percentage at 80 °C are higher than those at 30 °C. The increase in elongation may be due to the softening of cellulose chains at elevated temperature which allows more freedom of movement of cellulose molecules to slip over each other at low stresses. The elongation percentage decreased at 100 °C as compared to 80 °C. This may be due to the fast removal at 100 °C, of water or volatiles, originally present in the fibres, and which act as plasticizing agents in the different chains of the cellulose molecules.

Experiments were carried out to confirm that at higher temperatures, removal of water and volatiles from sisal fibres became faster. Fig. 3 shows that the rate of water removal is faster at 100 °C as compared to 80 and 60 °C. This means that when a fibre is tested at elevated temperature the time and temperature of the environment in which the fibre is tested is of importance, and affects the final results as discussed earlier. Similar observations were reported by Greenberg *et al.* [3]. In the present system the oven reaches the set temperature in 6 min, *i.e.* the fibres under test remain in the oven for 6 min before the test is carried out. The amount of moisture removed in 6 min from the fibres at 60, 80 and 100 °C was 5.9, 7.7 and 10.2%, respectively, which confirms the above assumption.

Fig. 4 shows the changes in tensile modulus with plant age of sisal fibres at different temperatures. The highest value of modulus was observed at 30 °C for the fibre from a plant of age 9 years. A decrease in modulus at a given temperature was observed with increase of plant age. At 80 °C the tensile modulus decreased from 28.6 to 17.5 GPa with increase of plant age from 3 to 9 years. Similarly at 100 °C the modulus

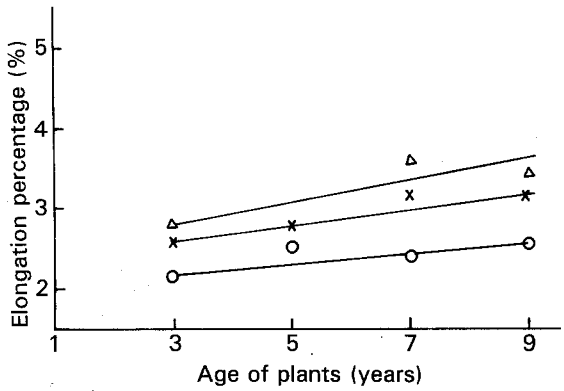

Figure 2 Change in elongation percentage of sisal fibre with plant age at (○) 30 °C, (△) 80 °C, (×) 100 °C.

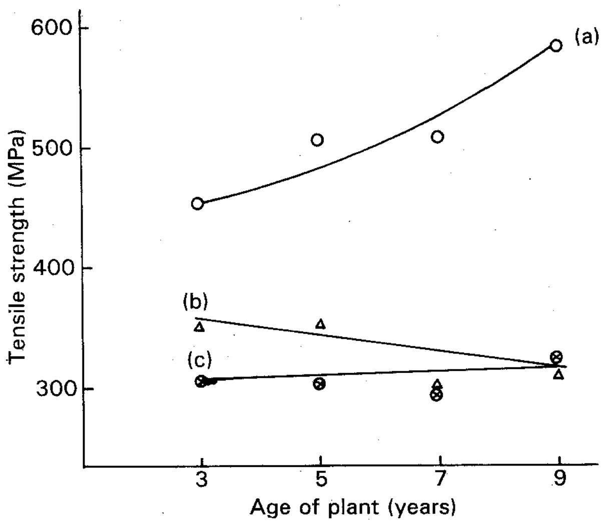

Figure 1 Variation of tensile strength of sisal fibre with plant age at (a) 30 °C, (b) 80 °C, (c) 100 °C.

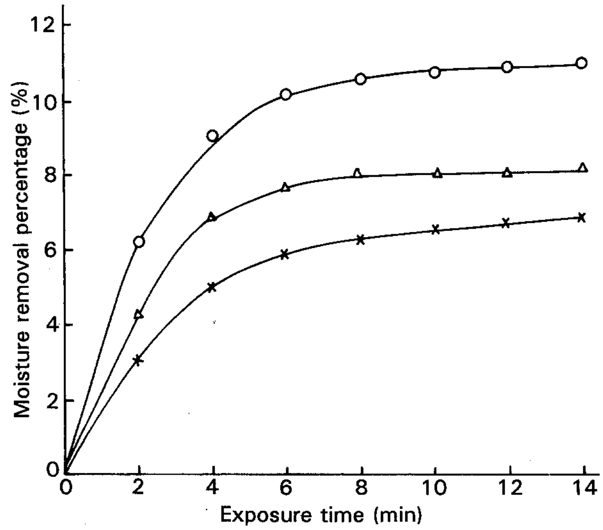

Figure 3 Moisture removal percentage of sisal fibres from plant 7 years old versus time of exposure at (*) 60 °C, (△) 80 °C, (○) 100 °C.

decreased with increase of plant age. Natural fibres like sisal fibres are composed of many chemical constituents such as cellulose, hemicellulose, lignin and pectin. Each of these components is present in the fibre in a definite ratio as reported in the literature [8]. The coefficients of thermal expansion of celllulose, hemicellulose, lignin and pectin are different. At higher temperatures these difference become large. This causes a mismatch between various components and consequently creates voids or flaws. Secondly, at elevated temperatures the moisture content and volatiles of the fibre are reduced, leaving behind cavities and voids and thus reducing the tensile strength and modulus of the fibre.

The tensile strength and modulus decreased of elevated temperatures. The differences in tensile strength between sisal fibre extracted from plants of different ages (Fig. 5) were also reduced significantly at 100 °C. The difference in tensile strength between the fibres of plants aged 3 and 9 years was 129.2 MPa at 30 °C, which reduced to a level of 23.6 MPa at 100 °C. The same is true for the modulus also. The difference in modulus of sisal fibres narrowed down to 3.9 GPa at 100 °C from 10.6 GPa at 30 °C. This result suggests that sisal fibres can be used as a reinforcing filler for various application around 100 °C.

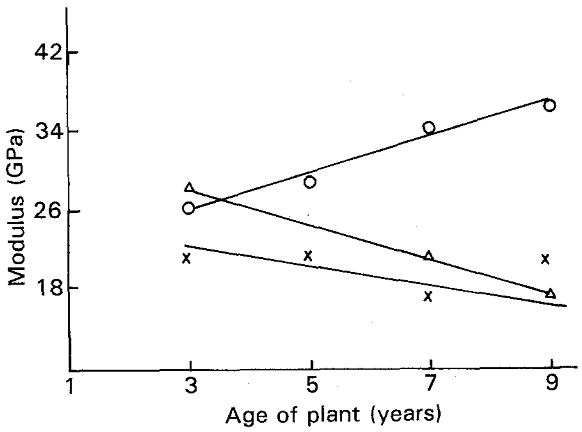

Figure 4 Change in modulus of sisal fibre with plant age at (○) 30 °C, (△) 80 °C, (×) 100 °C.

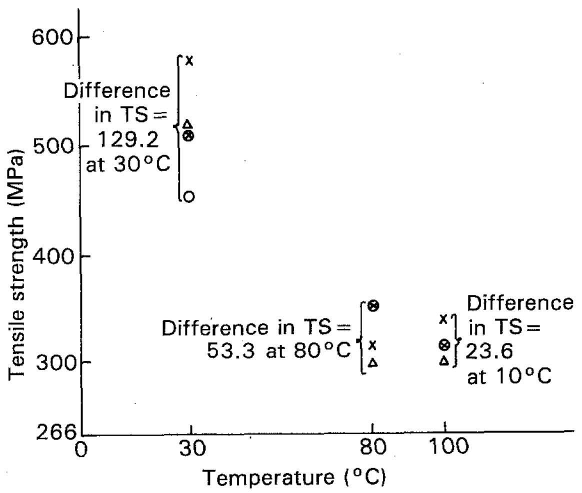

Figure 5 Tensile strength of sisal fibres extracted at different temperatures from plants of different ages: (○) 3 years, (⊗) 5 years, (△) 7 years, (×) 9 years.

The toughness of sisal fibre was determined by measuring the area under the stress–strain curve, using the following expression [9]:

$$\text{Toughness} = \int_0^L S \, dl$$

TABLE I Toughness of sisal fibre at different temperature

Age of plants (years)	Toughness per unit volume (MJ m^{-3})		
	30 °C	80 °C	100 °C
3	4.8	4.9	4.1
5	5.5	7.8	4.3
7	6.0	5.2	4.7
9	7.4	5.4	5.2

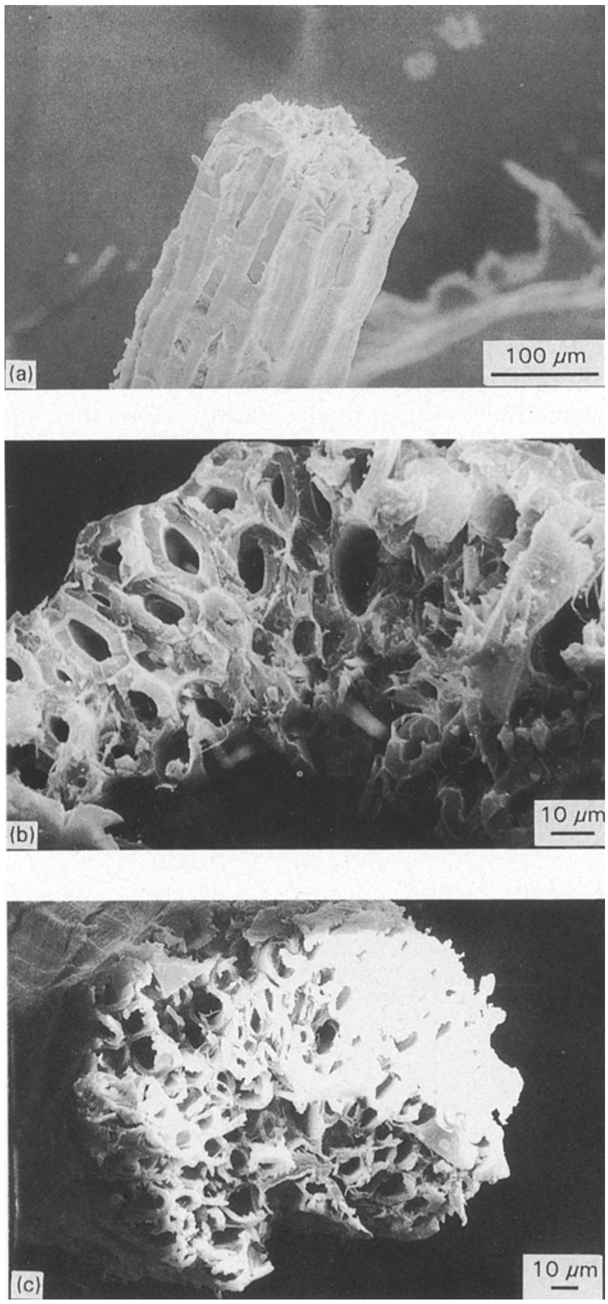

Figure 6 SEM fractograph of 3-year-old plant fibre fractured at (a) 30 °C, (b) 80 °C, (c) 100 °C.

where S is the stress, dl an infinitely small change in the length of the fibre and L the maximum elongation of the sample.

Toughness values of sisal fibres were calculated and are shown in Table I. The toughness of sisal fibre increased with the age of the plant. At 30 °C the toughness increased from 4.8 to 7.4 $MJ\,m^{-3}$ of fibres for plants 3 to 9 years old, respectively; at 80 °C it increased from 4.9 to 5.4 $MJ\,m^{-3}$ and at 100 °C from 4.1 to 5.2 $MJ\,m^{-3}$. This clearly shows that with the increase of temperature there is a decrease in the toughness of fibres. The difference in the toughness of sisal fibres extracted from the differently aged plants was found to be significantly less at 100 and 80 °C, which is similar to the tensile behaviour of plant fibres.

The microstructures of fibre ends from plants 3, 5, 7 and 9 years old and fractured at 30, 80 and 100 °C are shown in Figs 6, 7, 8 and 9, respectively. A magnified view of a 3-year-old plant's fibre fractured at 30 °C is shown in Fig. 10. The size of cells increased with the age of the plant. Fractographs of sisal fibre fractured at high temperature show that there is a ductile failure, which is common for cellulosic material. 3-year-old plant fibre has more volatile and amorphous materials, while 9-year-old plant fibre has more cellulose and hemicellulose in it. It was observed that the cell wall in the case of 3-year-old plant fibre is thin as compared to 9-year-old plant fibre. At 100 °C it was found that cells elongate more in 9-year-old plant fibre due to softening of the structure and enhanced flexibility of chains, which was not observed in the fibre of 7-year-old plants. In 3-year-old plant fibre the quantity

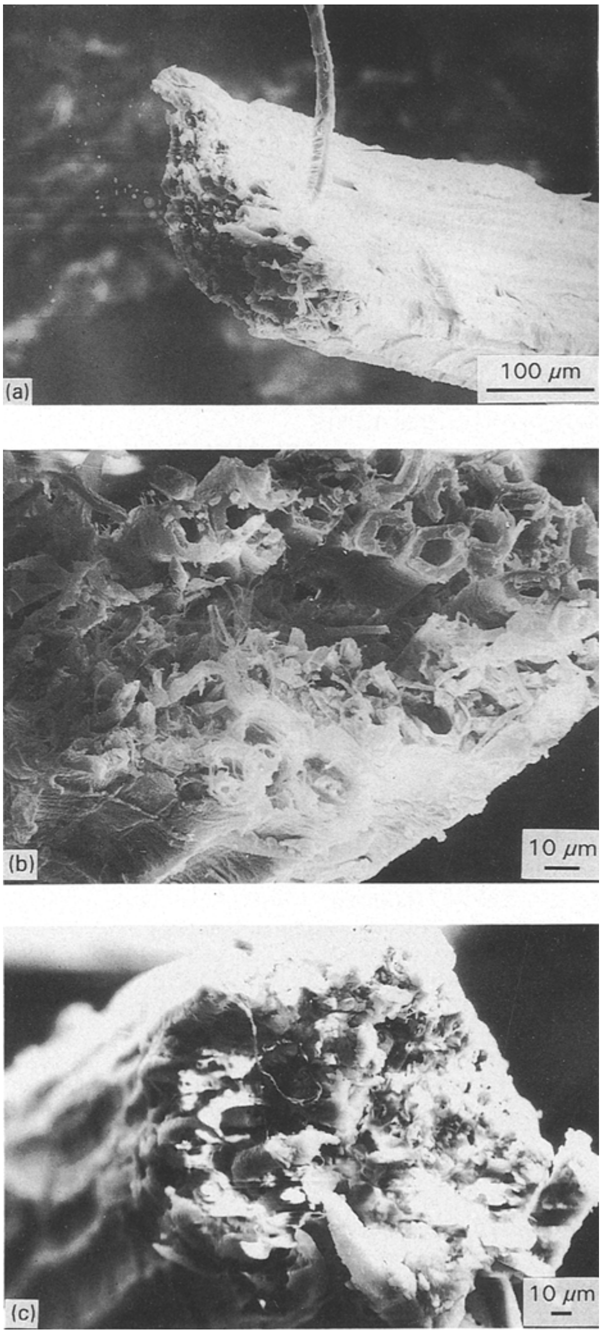

Figure 7 SEM fractograph of 5-year-old plant fibre fractured at (a) 30 °C, (b) 80 °C, (c) 100 °C.

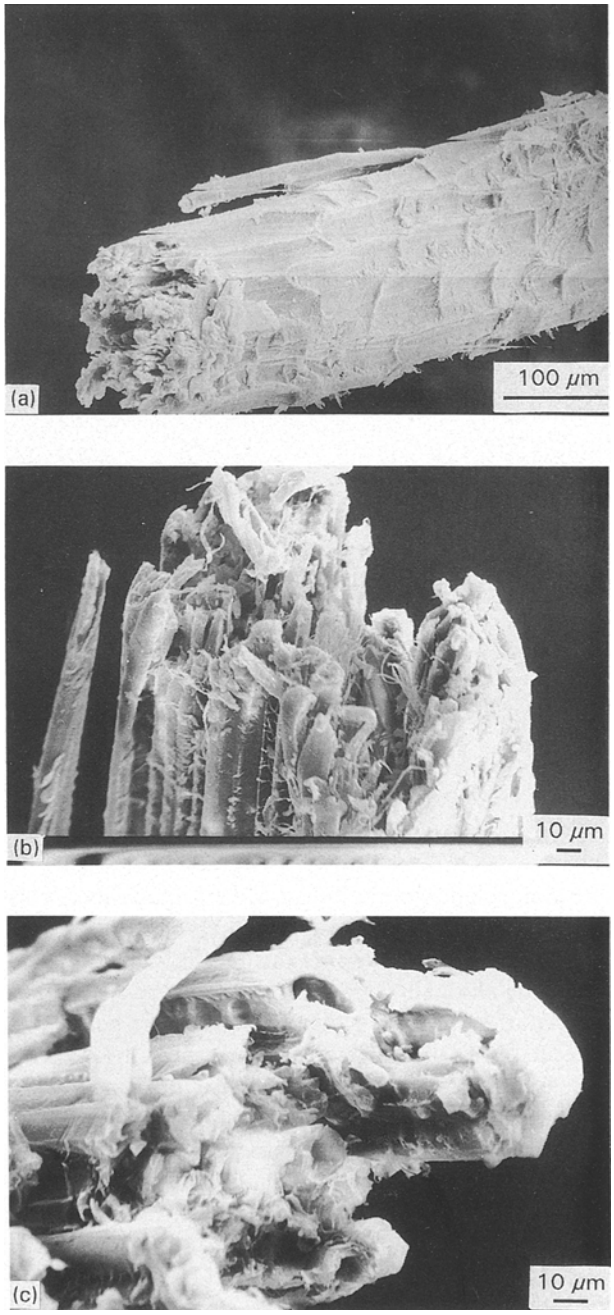

Figure 8 SEM fractograph of 7-year-old plant fibre fractured at (a) 30 °C, (b) 80 °C, (c) 100 °C.

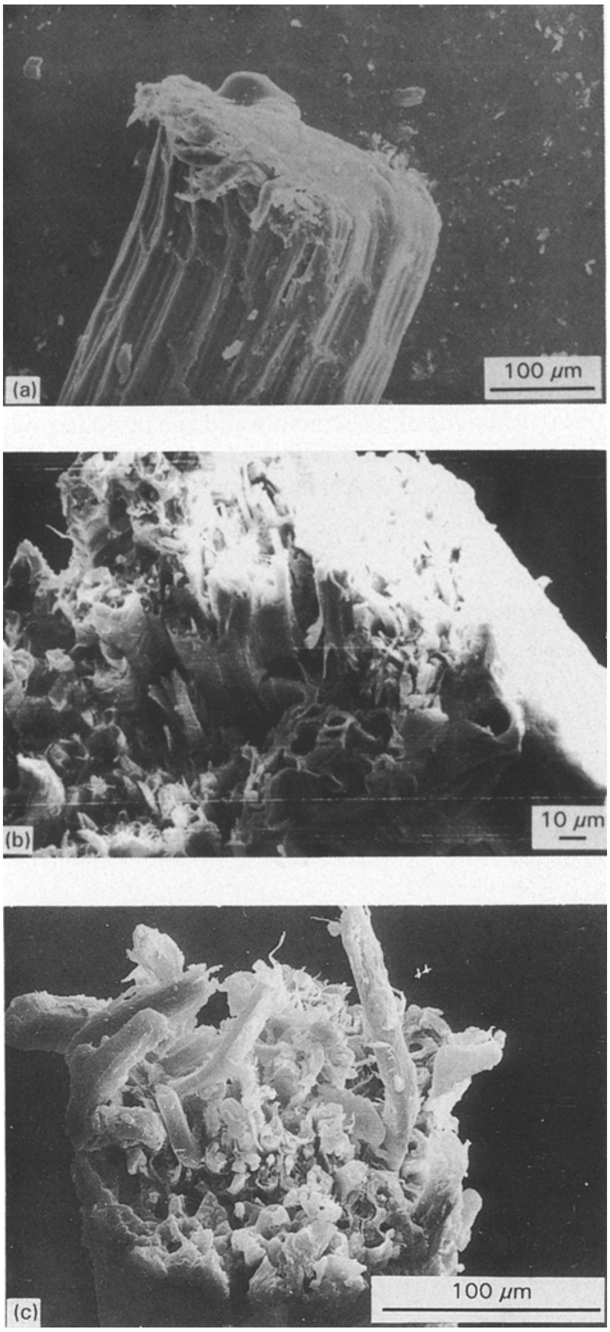

Figure 9 SEM fractograph of 9-year-old plant fibre fractured at (a) 30 °C, (b) 80 °C, (c) 100 °C.

of cellulose and hemicellulose is very much less. This observation of an increase in the elongation of cells with increasing age matches the tensile and elongation data. The elongation percentage increased with plant age. Another reason for this type of fracture behaviour in sisal fibres may be the increase of cell size with increase of plant age. An increase of temperature increased the flexibility and mobilization of the molecular chains. This is confirmed by the magnified fractograph of 3-year-old plant fibre fractured at 100 °C. This shows the complete hollow tubular structure of the cells.

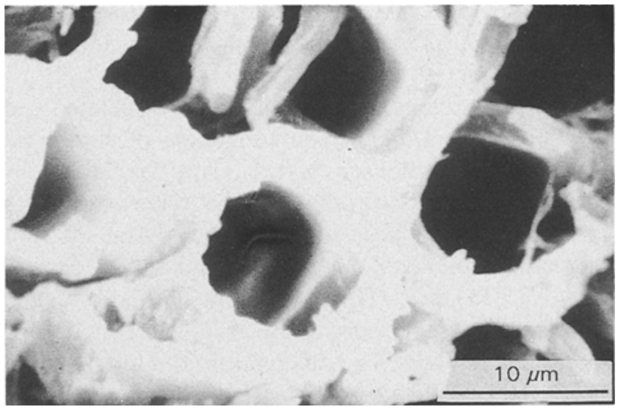

Figure 10 Magnified view of 3-year-old plant fibre fractured at 30 °C.

4. Conclusions

1. The tensile strength, tensile modulus and toughness of sisal fibre increase with increase of plant age.
2. The tensile strength, tensile modulus and toughness of sisal fibre decrease with increase of temperature.
3. The variation of tensile strength, modulus and toughness of sisal fibre obtained from plants of different ages was reduced at 100 °C.
4. Fracture of fibre ends at high temperature occurs after the pulling of microfibrills.

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