

INFLUENCE OF ALKALI TREATMENT ON CREEP PROPERTIES AND CRYSTALLINITY OF JUTE FIBRES

Dipa Ray,^a Mahuya Das,^{b*} and Debarati Mitra^c

In this work, the effect of the alkali-treatment and its variables viz., time and concentration of alkali, on the creep properties of jute fibre were studied. It was demonstrated that this kind of treatment leads to several changes in fine structure, such as voids creation and fibre fibrillation. The creep behaviour was measured for the alkali treated as well as the dewaxed fibres. Creep value was much higher in the 17.5% NaOH treated fibres compared to the dewaxed fibres. In the 8 hrs treated fibres, the creep was slightly more than the 20 mins treated fibres. XRD study revealed that experimental alkali treatment conditions resulted in closer packing of cellulose chains or rather increased crystallinity. Hence closer arrangement of the molecular chains (higher crystallinity) will reduce the creep extension, as expected.

Keywords: Jute Fibres; Alkali Treatment; Creep Behaviour

*Contact information: a & b: Department of Polymer Science & Technology, University of Calcutta, 92, A. P. C. Road, Kolkata-700009, West Bengal, India; b ; c: Department of Chemical Technology, University of Calcutta, 92, A. P. C. Road, Kolkata-700009, West Bengal, India. *Corresponding Author: d_mahuya@yahoo.com.*

INTRODUCTION

The modern trend is to utilize natural fibres such as jute, cotton, sisal, coir, bamboo, PALF, (Karmaker et al. 1996; Chand et al. 1998; Varma et al. 1984; Jain et al. 1992; Jain et al. 1993; Umadevi et al. 1997; Avella et al. 1998) etc. for various diversified applications. These natural fibres have a special advantage of easy availability in comparison to their synthetic opponents. In addition, they are biodegradable, renewable in nature, and have a CO₂ neutral life cycle. There are many techniques that can be employed successfully for the chemical modification of natural fibres to make them suitable for various applications. Among these techniques, mercerization or alkali treatment is a versatile one, which is often used for chemical modification. It brings about changes in dimensions, fine structure, and morphology of natural fibres (Bledzki et al. 1999).

Ray et al. (2001) and Samal et al. (1995) carried out alkali treatment of jute fibres with varying time, concentrations, and treatment conditions. Jute fibre with modified tenacity, tensile modulus, etc. was obtained. Furthermore, some authors reported changes in crystallinity in the case of coir, flax, cotton, and bamboo fibres due to alkali treatment (Gossan and Bledzki 1999; Das and Chakroborty 2006a). Improved wettability of coir and sisal fibres were observed by Prasad et al. (1983) and Chand et al. (1986), respectively. Bisanda et al. (1991) also noticed improved resin pick up by sisal fibres after mercerization. Das and Chakroborty (2006a, 2006b, 2007) have investigated the

effect of alkali treatment on the mechanical, thermal, and dynamic mechanical properties of bamboo fibre. Available literature reveals that alkali treatment removes one of its cementing materials, hemicellulose, depending on the concentration of the alkali used, time and temperature of treatment, liquor ratio, etc. Thereby, alkali treatment renders the changes in fibre properties by altering the fine structure and morphology of fibres.

Many workers have reported their studies on the alkali treatment of the jute fibres. Ray et al. (2001) reported 41% loss of the hemicellulose content of the jute fibres on treatment with 5% NaOH solution at 30°C for 2 hours.

There have been many studies related to mechanical, thermal properties of jute fibre with or without any treatment, which are enough to give insight into the real properties of fibres. Recent advances in the area of the fibre science have resulted in the development of theories, which permit interpretation of fibre properties in terms of molecular interactions. Jute fibres being used extensively for various applications, such as textile material, reinforcement for composites, etc., need to be investigated in detail for their viscoelastic properties. Therefore, long-term properties, such as stress relaxation, creep, and dynamic mechanical properties, need to be analyzed to get a complete idea about a fibre.

Although some long-term property investigations have been carried out on different textile fibres (Das and Chakroborty 2006a), such investigations on the viscoelastic behaviour of the jute fibres have been few. Creep, an especially important property, was measured by Sett et al. (2000) in the case of the rotor and friction spun jute blended yarn. They observed that in case of the rotor spun jute blends, creep extension values were lowered with the increase in the jute fibre concentration in the yarn as the fibre mobility was decreased. They found that the creep extension of the friction spun blends was higher than the rotor spun yarns due to less fibre consolidation, allowing greater fibre mobility. Shaughnessy (1948) studied the creep behaviour of textile rayon and textile cordura.

Here an attempt has been made to study the effect of different concentrations of alkali on the creep behaviour of jute fibres. The effect of treatment time on fibrillar rearrangement, which influences the creep behaviour of the fibres, has also been investigated.

EXPERIMENTAL

Materials

Jute fibres of grade Td4 purified through dewaxing with alcohol and benzene mixture (2:1) were used for the study. The fibres were wrapped in black paper (to avoid photo-degradation) and kept in polyethene bags (to avoid moisture degradation) at 25°C and 65% RH. NaOH pellets were used for mercerization.

Methods

Alkali treatment

50 gms of fibres were dewaxed with methanol: benzene solution (2:1) to prepare the control sample. Three bundles of fibres, containing 50 gms of fibres each and cut to

20 cm length, were dipped in three separate beakers containing NaOH solutions of concentrations 1%, 5%, and 17.5% (weight%) respectively at room temperature, maintaining a liquor ratio of 15:1 (liquor: fiber). After 20 minutes, the fibres were taken out of the beakers and washed thoroughly with water to remove any NaOH sticking to the surface. The fibres were then treated with dilute acetic acid to remove any trace of NaOH remaining and finally washed again with water and dried.

In the second set, four bundles of fibres, containing 50 gms of fibres each and cut to 20 cm length, were treated with 5% NaOH solution for four different time periods, 2 hrs, 4 hrs, 6 hrs, and 8 hrs respectively. The washing procedure was the same as mentioned earlier.

Creep measurement

The creep behaviour of the alkali treated jute fibres were measured with an Instron 5567 device. A gauge length of 20 mm was employed with a crosshead speed of 0.5 mm/min. The creep behaviour of the dewaxed untreated Td4 (control) and alkali treated jute fibres were determined at 5% NaOH treated for 20 mins and 8 hrs, respectively, and 17.5% NaOH treated for 20 mins. The creep behaviour was observed for 2 minutes at 50% of the breaking load, 50 samples were tested for each set of fibres, and the average value was reported.

X-Ray diffraction study

The XRD patterns of the dewaxed and the alkali treated jute fibres were examined with a Philips PW1710 diffractometer employing Ni filtered CuK α radiation.

RESULTS AND DISCUSSION

From the data of X-ray analysis (Ray and Sarkar 2001), the change in the 002 peak height and width of the fibres due to treatment with different concentration of alkali solutions are shown in Figs. 1 and 2, respectively. It is also evident from Fig. 1 that the intensity for I_{crys} was maximized at 1% alkali treatment, which also indicates maximum crystallinity of the jute fiber at that concentration. Maximum peak width in the X-ray diffractogram is indicative of low resolution of peaks with less crystalline perfection. On alkali treatment, two opposing phenomena were operative. Removal of hemicellulose caused interfibrillar matrix softening and also, the release of the initial strains helped the cellulose chains to arrange themselves in a compact manner, facilitating the development of a highly ordered arrangement of cellulose chains. Interplay of the two factors and predominance of one factor over another controlled the crystallinity of the fibres. At low concentration of alkali, such as 1%, the extent of removal of hemicellulose was low and did not produce a compact structure with high crystallinity and low peak width. On the other hand a decrease in peak width is an indication of improved resolution of the peaks after alkali treatment, and the diffractogram showed signs of better crystalline order than the control.

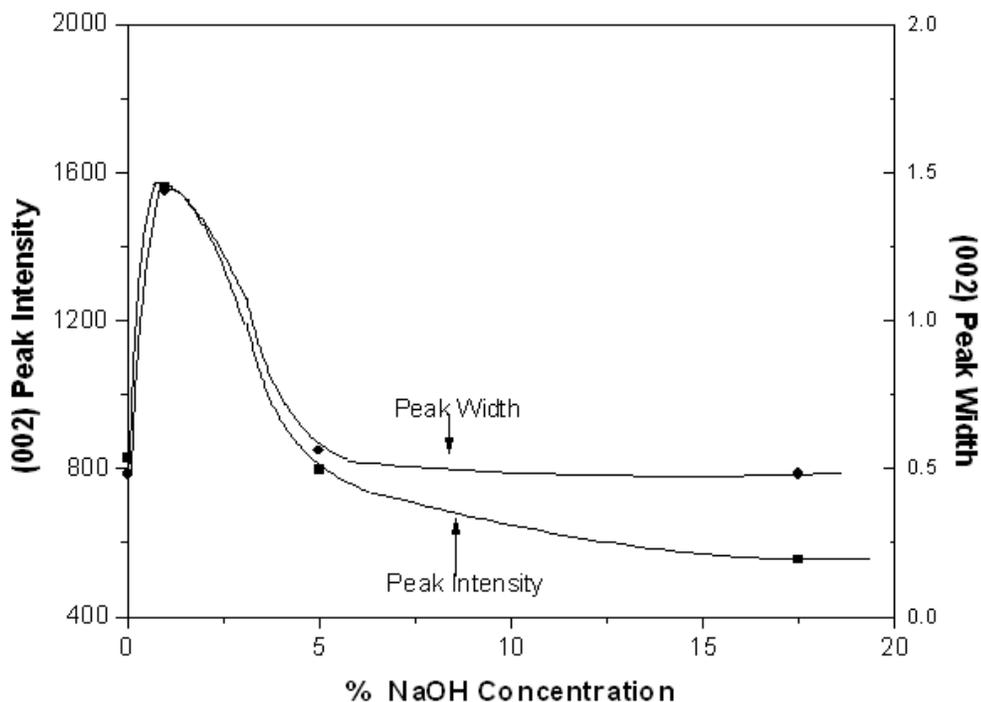


Figure 1. Change in the intensity and the width of the (002) peaks of the alkali treated jute fibres with the alkali concentration

Similarly, the variation of intensity for the (101 and 101) peak was also maximized with 1% alkali solution, whereas peak width initially showed a high value at 1% alkali concentration and then passed through a minima and again showed a continuous increasing nature. This indicates low resolution and increasing amorphous nature due to destruction of molecular chain arrangement. Sodium hydroxide then reacts with cellulose, forming a stable compound, sodium cellulosate or soda cellulose, by replacement of the ionizable hydrogen in the $-OH$ groups of cellulose with Na^+ ions. When the soda-cellulose compound is completely destroyed, by subsequent washing with distilled water and neutralization with H_2SO_4 , a lattice transformation from cellulose-I to cellulose-II results (Das and Chakroborty 2006a). The extent of conversion depends on the experimental conditions. Much of the swollen cellulose frequently does not recrystallize, resulting in a large fraction of disordered (amorphous) cellulose, which can be detected with X-ray diffraction.

The variation of the peak width and intensity for (002) peak of the jute fibres as a function of alkali treatment time are shown in Fig. 3. These also justify the changes in the rearrangement of the fibrils after treatment with alkali solution of different concentrations and after prolonged soaking in 5% alkali solution.

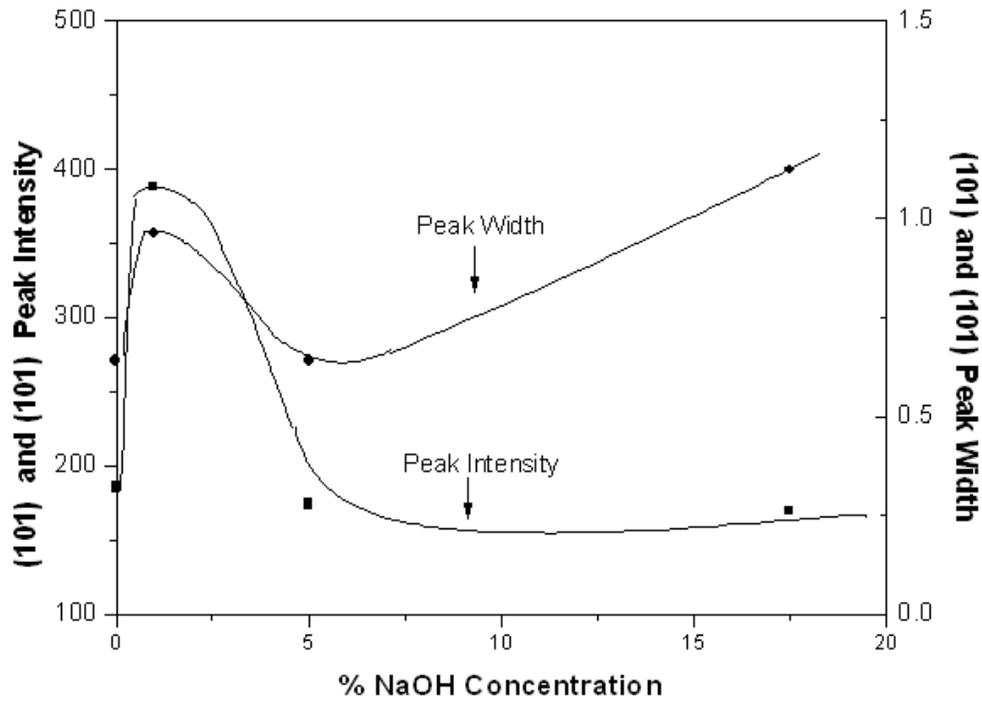


Figure 2. Change in the intensity and the width of the (101) and (101) peaks of the alkali treated jute fibres with the alkali concentration

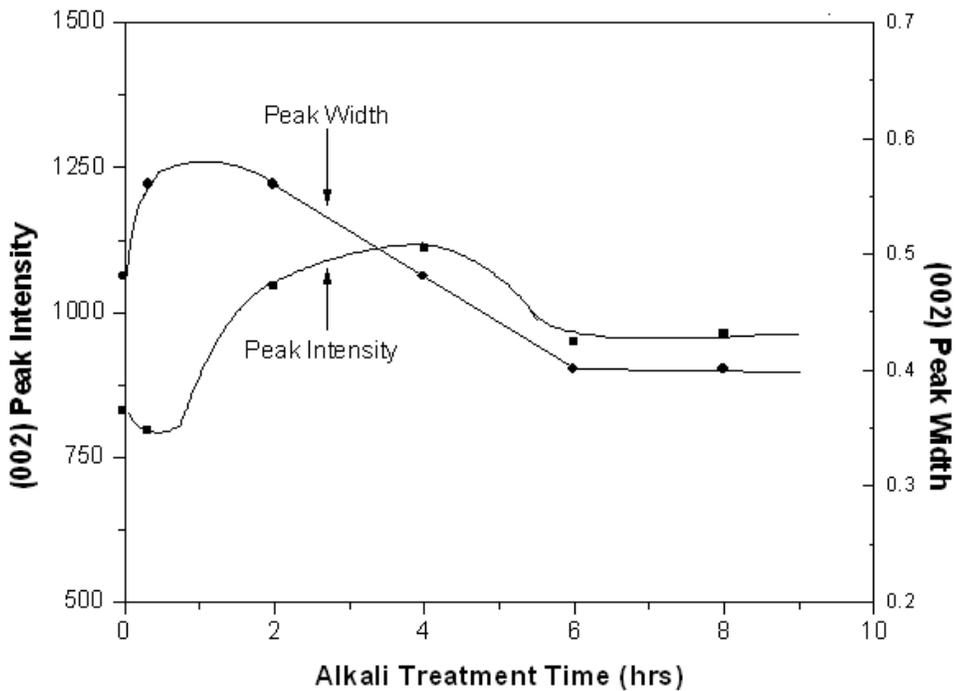


Figure 3. Change in the intensity and the width of the (002) peaks of the alkali treated jute fibres with the alkali treatment time

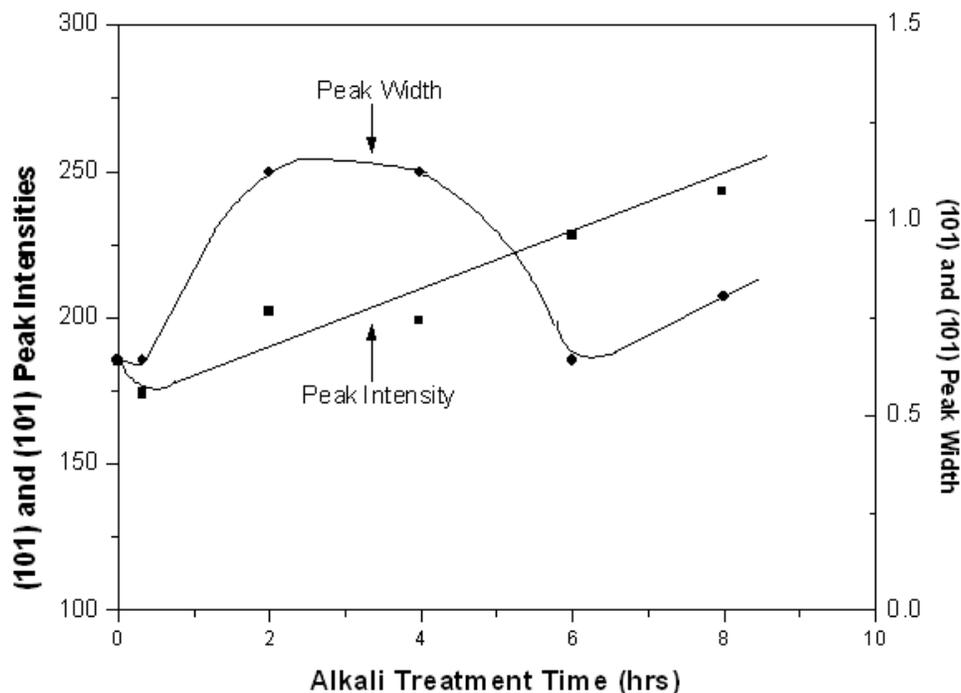


Figure 4. Change in the intensity and the width of the (101) and (101) peaks of the alkali treated jute fibres with the alkali treatment time

Similarly, the changes (101 and 101) peak heights and widths of the fibres after treatment with 5% NaOH solution for different time periods are shown in Fig. 4. It is evident from both the curves in the figure that there was no sharp value for intensity or width indicating lower resolution of the peaks. It may further be concluded that at 5% alkali concentration the fibrils were not arranged in proper crystalline order.

Creep Behaviour

The creep behaviour of the dewaxed untreated Td4 (control) and alkali treated jute fibres, 5% NaOH treated for 20 mins and 8 hrs respectively and 17.5% NaOH treated for 20 mins at 50% of the breaking load as well as the variation of $\varepsilon / \varepsilon_0$ against time (secs), where ε = strain in the fibre at time t and ε_0 = strain in the fibre at time t = 0, are shown in Fig. 5. The variation of percent creep with respect to alkali concentration and that of 5% alkali treated fiber with respect to time are shown in Fig. 6.

When a fixed load is put on a fibre, it gradually extends with time. This time-dependent extension is known as creep. On application of stress, there is an instantaneous deformation, which is called elastic deformation within elastic limit. Unlike brittle fracture, creep deformation does not occur suddenly upon the application of stress. Instead, strain accumulates as a result of long-term stress. In jute fibre the cellulose chains lie roughly parallel to the long axis of the fibre. In localized regions of the fibre, the chains are oriented with respect to each other in such a way so as to form a crystalline lattice. When load is applied on a fibre, the stress is transmitted from one chain to its

neighbour by means of intermolecular forces. These forces are strongest in the crystalline regions, where the chains lie in closest proximity to each other, whereas in non-crystalline portions, application of stress causes rearrangement of the localised cellulose chains with respect to one another. Creep depends on the fibre mobility in the stress concentration region. Hence, closer arrangement of the molecular chains (higher crystallinity) will reduce the creep deformation, whereas loosely arranged molecular chains undergo easy deformation resulting higher creep strain (O'Shaughnessy 1948).

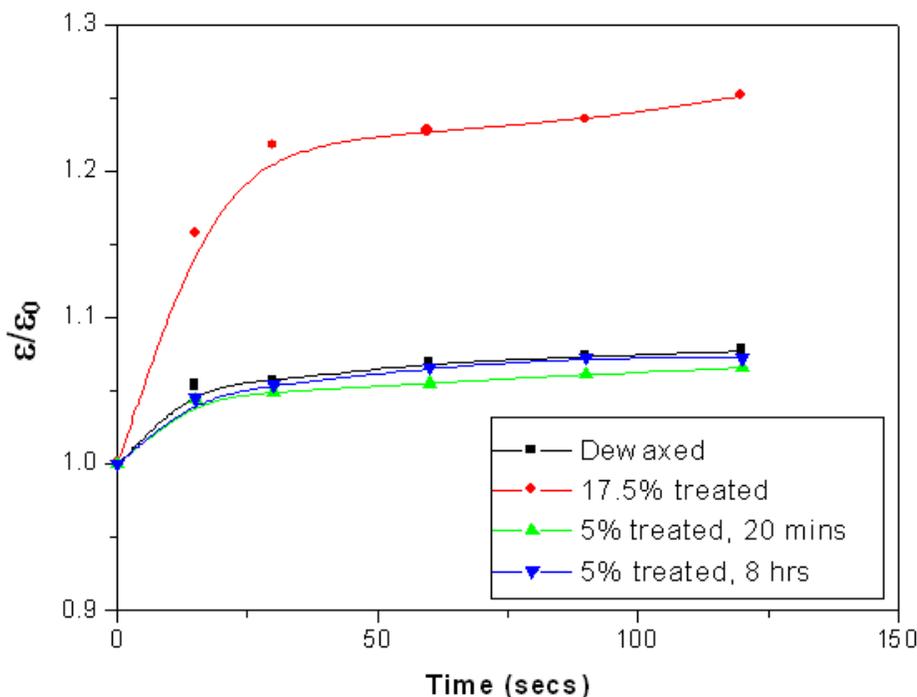


Figure 5. Variation of the $\varepsilon / \varepsilon_0$ of the dewaxed, 17.5% NaOH treated and 5% NaOH treated (20 mins and 8 hrs treatment time) against time

Any lignocellulosic material is composed of multicellular fiber. Each unit cell of fiber consists of small cellulose microfibrils, which are surrounded and cemented together with lignin and hemicellulose. Although the length of each cell is very small, they are held with each other in a longitudinal direction, thereby producing a long, continuous fiber. The neighbouring units are also attached among themselves, producing a mesh-like structure. Alkali treatment leads to destruction of the total structure, first by removing the cementing material, and it splits the fiber into finer filaments. It was observed that with the stronger alkali (17.5% NaOH solution), the destruction of the mesh structure of the jute fibres occurred rapidly, and thereby closer arrangement of molecular chain destroyed. So, the creep behaviour was much higher in the 17.5% NaOH treated fibres compared to the dewaxed and 5% alkali treated fibres due to loss in the fibrillar arrangement (Mukherjee et al. 1993) (Fig. 5). This loss in fibrillar arrangement also is supported by increase in width of (101 and 101) peak at higher concentration of alkali, as obtained from XRD data (Fig. 1).

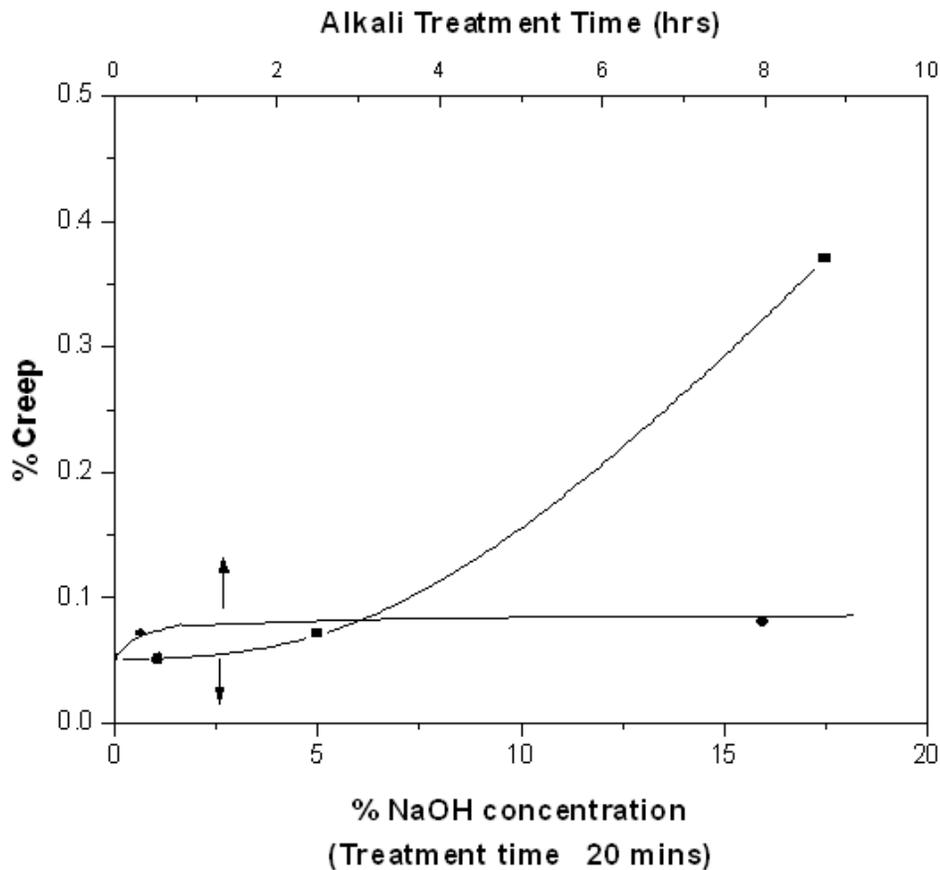


Figure 6. Creep behaviour of the dewaxed and the alkali treated (5% and 17.5% NaOH treated) jute fibres as a function of alkali concentration and alkali treatment time

Figure 6 presents the variation in percent creep [creep deformation divided by deformation due to the applied load, which is not time-dependent $\times 100$ i.e. $(\epsilon/\epsilon_0) \times 100$] with the variation in alkali concentration where immersion time is 20 mins. In 5% NaOH treated fibres, compared to higher alkali solution, a lesser amount of hemicellulose was removed (Ray and Sarkar 2001). Thus, in that case, interfibrillar matrix softening was operative without extensive destruction of molecular chain arrangement. Hence there was little increase in the creep properties compared to the dewaxed fibres. Again, for 5% alkali concentration a variation in time of immersion of fiber did not produce any significant increase in creep, due to the same reason as explained above.

CONCLUSIONS

1. On alkali treatment, two opposing phenomena were operative. Removal of hemicellulose (Ray and Sarkar 2001) caused interfibrillar matrix softening and also, the release of the initial strains helped the cellulose chains to arrange themselves in a

compact manner, facilitating the development of high stress in the fibre. Interplay of the two factors and predominance of one factor over another controlled the crystallinity and creep behaviour of the fibres.

2. Higher concentration of alkali treatments leads to reduction of crystallinity. Low concentration but longer dipping time also reduces crystallinity, but at a slower rate.

3. At 5% alkali concentration, removal of hemicellulose and consecutive interfibrillar matrix softening play a role for a little decrease in the creep deformation in comparison to the dewaxed fiber.

4. When treated with 17.5% NaOH solution, removal of hemicellulose was accompanied by swelling and shrinkage of the ultimate cells, resulting in some disorientation of the fibrils and destruction of the close arrangement of molecular chains. Such loss in fibrillar arrangement might have been responsible for such high creep behaviour.

5. Longer treatment times at 5% alkali concentration do not lead to extensive rearrangement of fibrils, so increase in creep deformation is not so prominent.

6. The results have shown distinct findings relative to previous studies (based on the effect of alkali treatment on the different mechanical properties of jute fibers) and are important with respect to the application of jute fiber under constant load.

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