Effect of Steam Pretreatment of Jute Fiber on Dimensional Stability of Jute Composite

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Received 3 June 1999; accepted 15 October 1999

ABSTRACT: Dimensional stability of fiber board from lignocellulosic materials is a prime concern for efficient utility of the product. A number of methods have been used to improve the dimensional stability. These include the application of coating, oil, and wax treatments and chemical modification of lignocellulosic materials. A new process has been developed to minimize irreversible swelling (i.e., permanent fixation of compressive deformation of wood fiber through a hygrothermal treatment using in-built steam from moisture of compressed fiber at high temperature). This process has been applied on jute fiber for the evaluation of dimensional stability and vis-&-vis the mechanical and thermal properties of the fiber board made from the modified jute fiber.
 J Appl Polym Sci 76: 1652–1661,2000

Key words: jute fiber; fiber board; steam stabilization; water absorption; thickness swelling

INTRODUCTION

Fiber boards, like other densified lignocellulosic-based composites, is subject to swelling forces when it sorbs water in either liquid or vapor phase. Jute and other lignocellulosic materials change dimensions with changing moisture content because the cell wall polymers contain hydroxyl and other oxygen-containing groups that attract moisture through hydrogen bonding.[1–4] The hemicelluloses are mainly responsible for moisture sorption, but the accessible cellulose, noncrystalline cellulose, and lignin also play major roles. Moisture swells the cell wall, and the jute fiber swells until the cell wall is saturated with water. Beyond this point, moisture exists as free water in the void spaces of the fiber and does not swell the fiber further; but the jute fiber shrinks when it loses the moisture below equilibrium moisture content (EMC). This process is called reversible swelling.

When jute fibers are pressed into fiber board, the fibers are compressed and flattened. So the fibers are deformed even though it has a memory of its original configuration. When the compressed fiber absorbs water, not only does swelling take place, but also the compressive force imparting to the fiber board during processing is relieved due to the recovery of the original configuration of the fiber through the old memory (the spring-back action of fiber). The rupture of the adhesive bonds between the jute fibers is due to this spring-back action of compressed jute fiber.

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Contract grant sponsor: IJIRA and FPL.

Journal of Applied Polymer Science, Vol. 76, 1652–1661 (2000)

This process is irreversible swelling. To minimize this irreversible swelling, special measures are needed for the production of a highly stable fiber board. Effective preventive solution of reducing swelling of fiber board should be as follows:

- Bind fibers together so that moisture cannot separate them.
- Prevent the build up of internal stress within the board.
- Relieve internal stress in the board prior to use.
- Prevent adsorption and absorption of moisture.

So the irreversible swelling can be marginalized if the internal stress can be somehow reduced. One way to do this is to plasticize the cell wall matrix (mainly hemicellulose–lignin matrix) by steaming during compression so that the fiber cannot recover its original shape through its old memory. This treated fiber only then undergoes the reversible swelling on absorption of water.[5–8]

The objective of this study was to evaluate the effectiveness of high-pressure steam treatment on jute fiber for production of dimensionally stable fiber board.

EXPERIMENTAL

Materials

Tossa jute (*Corchorus Olitorious*) of TD7 grade (Jute Corporation of India, Calcutta, India) as per Bureau of Indian Standards specification IS:271–1975 and water soluble phenol formaldehyde resin (procured from Hindusthan Adhesives Phenoset 5001, Calcutta, India) were used in the study. A mold of 30 × 30 × 12 cm contains one flat gasket of silicon rubber, a stainless steel square flat ring, and two stainless steel caul plates.

Fiber Preparation

Raw jute fiber was chopped through a hask chopper and the chopped fiber was then passed though a hammer mill to get fibers of an approximate length of 1 cm each.

Steam Stabilization of Fiber and Fabrication of Board without Resin

Additional water was sprayed to the fiber to get 15% moisture in the fiber on a dry weight basis. A mat (20 × 20 cm) of chopped jute fiber was made by hand and was placed in the pressing mold. The mold and the mat were placed in a hydraulic press heated to 200°C. The press was closed and held at 250 psi pressure for 4 and 8 min, respectively, for two different retention periods to observe the effect of this treatment on jute fiber board. The samples were referred to as SB-4 and SB-8, respectively.

Some boards were run through a blowing blade to break into the fibers for the fabricating of boards with the addition of extra binder (i.e., phenol formaldehyde resin). Others were used for the evaluation of dimensional stability and mechanical properties.

Resin Application and Board Fabrication with Resin

Phenol formaldehyde resin was sprayed to untreated milled jute fiber as well as to steam-stabilized jute fiber to achieve a resin level of 7% on a dry-weight basis. Thus, three types of boards were obtained with resin, including two boards with jute fibers stabilized with steam for 4 (SRB-4) and 8 min (SRB-8). The process parameters of fabrication of all the above boards were the same (i.e., 8 min at 180°C under 250 psi). The specific gravity of all the boards was 0.7.

Strength Tests

Two-point static bending tests were conducted by using an Instron universal testing machine (Model 4303) as per ASTM 790M-84 on the board specimens with a span of 160 mm and a crosshead speed of 4.3 mm/min to determine the modulus of the rupture.

Water Soaking and Thickness Swelling Tests

Thickness swelling and water absorption properties were determined both by simple water soaking test as well as by rigorous cyclic test. All samples were cut as per ASTM D 1037-96a. Water absorption and thickness swelling were measured after soaking the samples in distilled water for 24 h. Samples were also placed in boiling water for 2 h and then thickness swelling and water absorption were determined. The above treated samples were then subjected to water soaking at room temperature for 24 h, drying at 60°C for 24 h, immersion in boiling water for 2 h, and finally drying at 60°C for 24 h.

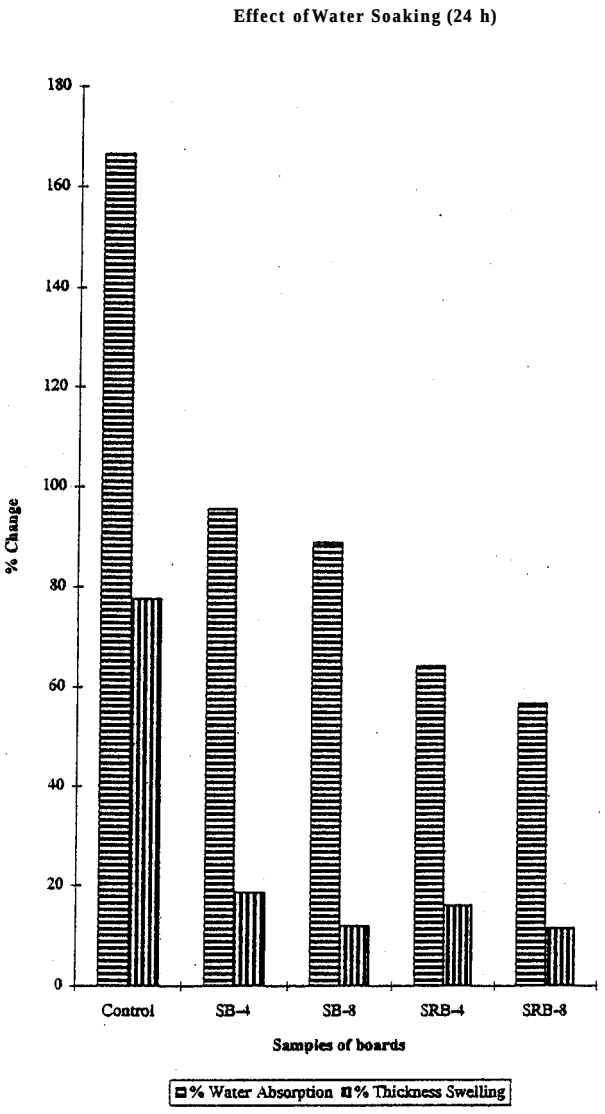

Figure 1 Effect of water soaking (24 h).

Equilibrium Moisture Content

The board was cut into 2 x 2 in. specimens and initial weights of the specimens were taken. The specimens were then dried in oven at 105°C till constant weight. The dried specimens were treated at the 65% RH (23 ± 2°C) until they reached equilibrium. EMC was calculated as follows:

$$EMC = \frac{\text{Wt. at 65\% RH} - \text{Oven-dry wt.}}{\text{Oven-dry wt.}} \times 100$$

SEM Study

The flexural fracture surfaces of the composite samples were studied with a scanning electron microscope (SEM; Hitachi scanning electron microscope, Model S-415 A) operated at 25 keV.

Thermal Analysis

Thermogravimetric analysis was carried out using a Mettler TG 50 module attached to TC-11 microprocessor of TA-4000 system. The heating rate was maintained at 10°C min^{-1} under a constant flow (150 mL min^{-1}) of nitrogen. During the evaluation of differential thermogravimetry (DTG), the maximum peak temperature, weight losses at each decomposition step, and amount of char left at 600°C were determined.

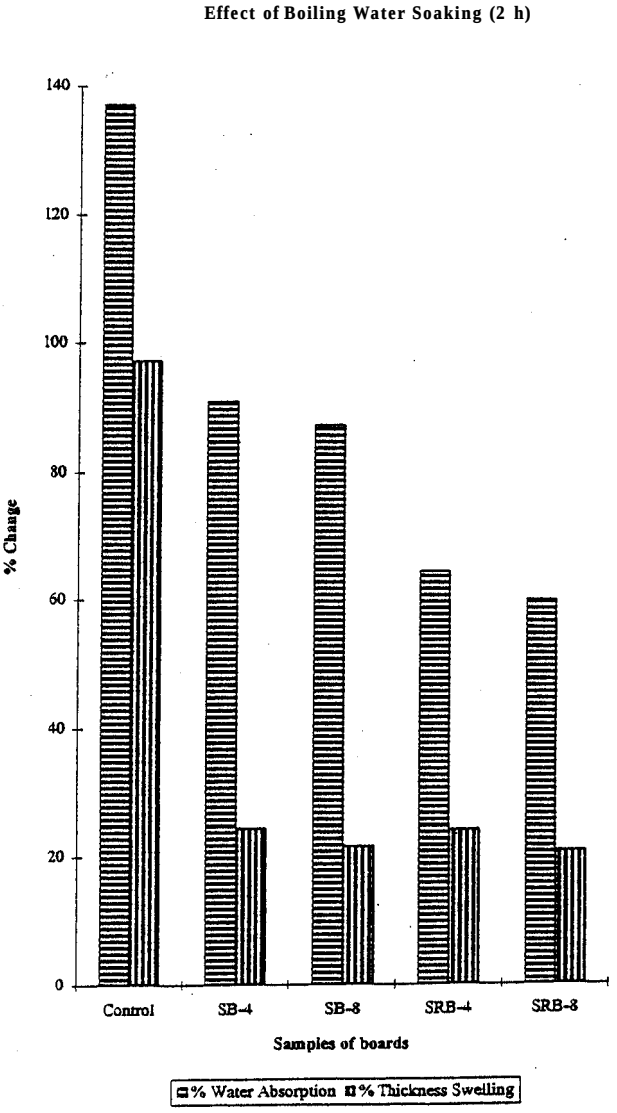

Figure 2 Effect of boiling water soaking (2 h).

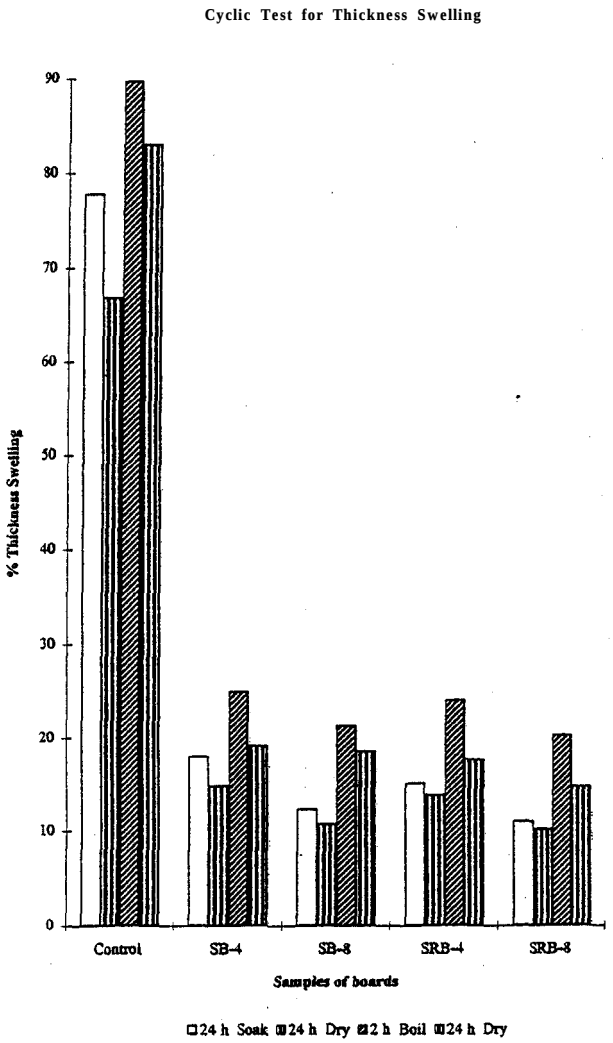

Figure 3 Cyclic test for thickness swelling.

RESULTSANDDISCUSSION

Results of 24-h soaking in water is represented in Figure 1. It was observed that the swelling of the control sample was around 77%, whereas the same was only 19 and 12%, respectively, for samples SB-4 and SB-8. When PF resin was admixed with steam-stabilized fiber, the swelling was marginally reduced. The swelling was around 16 and 11%, respectively, for SRB-4 and SRB-8.

A similar observation was also made when samples were heated in boiling water for 2 h; the results are shown in Figure 2. It was found that the swelling of the control board was around 97% and that of steam-stabilized samples reduced significantly (24 and 22%, respectively, for SB-4 and SB-8). There was little effect on the swelling behavior of the board when 7% PF resin was used So, it may be concluded that the dimensional stability of the fiber board is imparted predominantly by steam treatment, not by resin. This observation agrees with the earlier works[3,6–8] on fiber board made from wood chips.

Steam stabilization also has an effect on the water absorption property of the board. The water absorption was lowest for SRB-8. Water absorption was greatly reduced for boards made of steam-stabilized fiber without using resin.

The results of the cyclic test of thickness swelling and water absorption are depicted in Figure 3 and 4. The control board showed (Fig. 3) thickness swelling as 77.83% after 24 h of water soaking and it decreased to about 66.92% after the first drying cycle. After 2 h in boiling water, the thick-

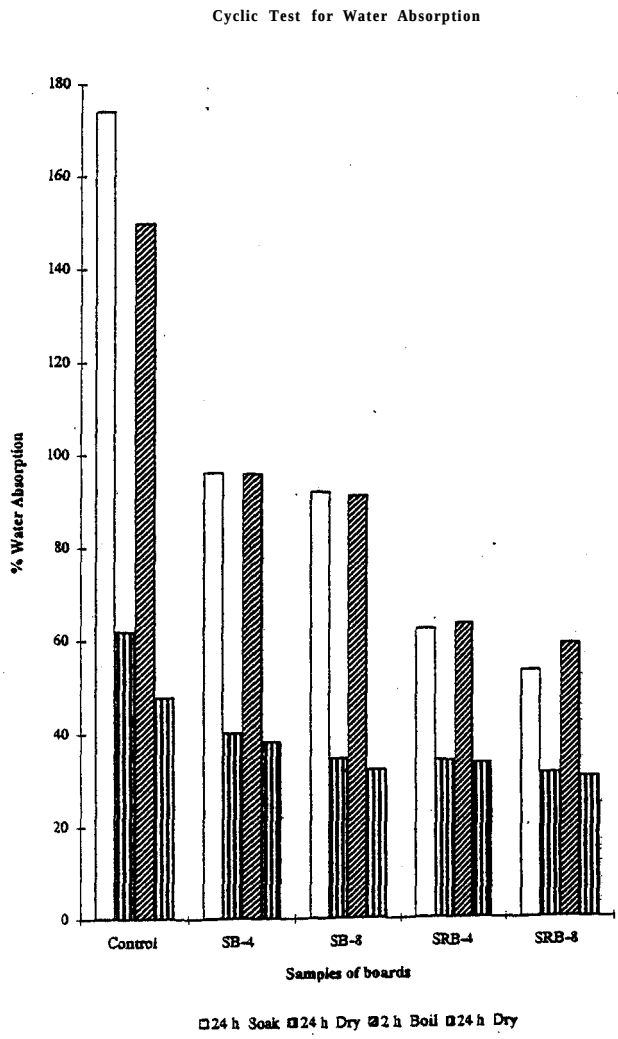

Figure 4 Cyclic test for water absorption.

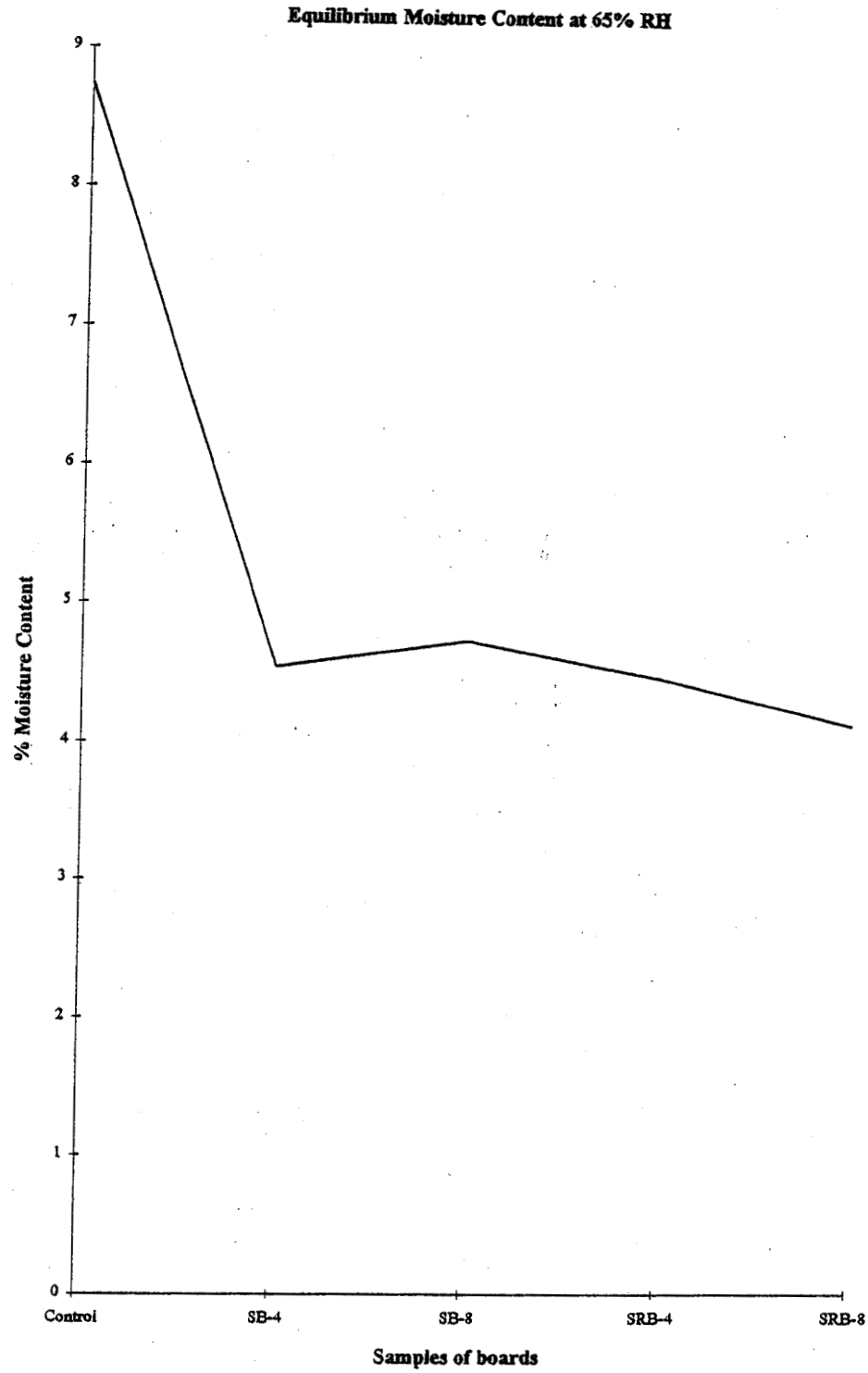

Figure 5 Equilibrium moisture content at 65% relative humidity.

ness swelling was about 89.79%, and then decreased to about 83.11% after the second drying cycle. The thickness swelling of SB-4 after 24-h water soaking was 18.02%. This was 76.84% reduction in swelling with respect to that of the control board. This swelling decreased to about

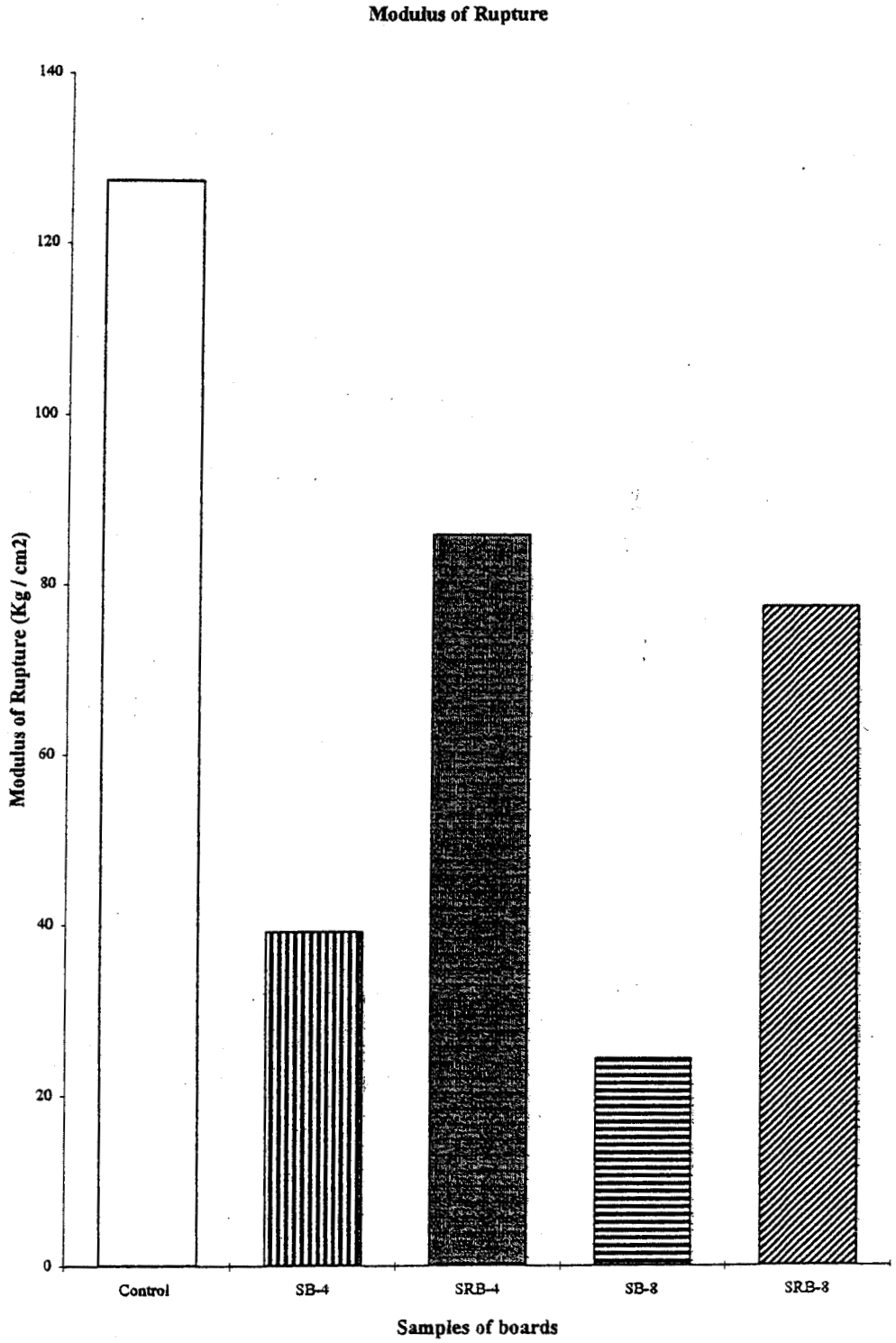

Figure 6 Modulus of rupture.

14.89% after the first drying cycle. After 2 h in boiling water, the thickness swelling was about 25.05% and this decreased to about 19.23% after the second drying cycle. For SB-8, the thickness swelling was 12.37% after 24 h water soak and decreased to about 10.78% after the first drying

a) Steam stabilized jute fiber b) Untreated jute fiber

Figure 7 Scanning electron micrographs of (a) steam stabilized jute fiber [SB-4] and (b) untreated jute fiber [control].

cycle. After 2 h in boiling water, the thickness swelling was about 21.4% and this decreased to about 18.64% after the second drying cycle. The addition of 7% PF resin to steam-stabilized jute fiber for fabrication of boards (SRB-4 and SRB-8) imparts Little effect on swelling behavior. Figure 4 shows that water absorption was also reduced significantly when the jute fiber was steam stabilized.

The same trend is also observed (Fig. 5) on equilibrium moisture content.

Moduli of rupture values for both the steam-stabilized boards (SB-4 and SB-8) were much smaller compared to that of the control board

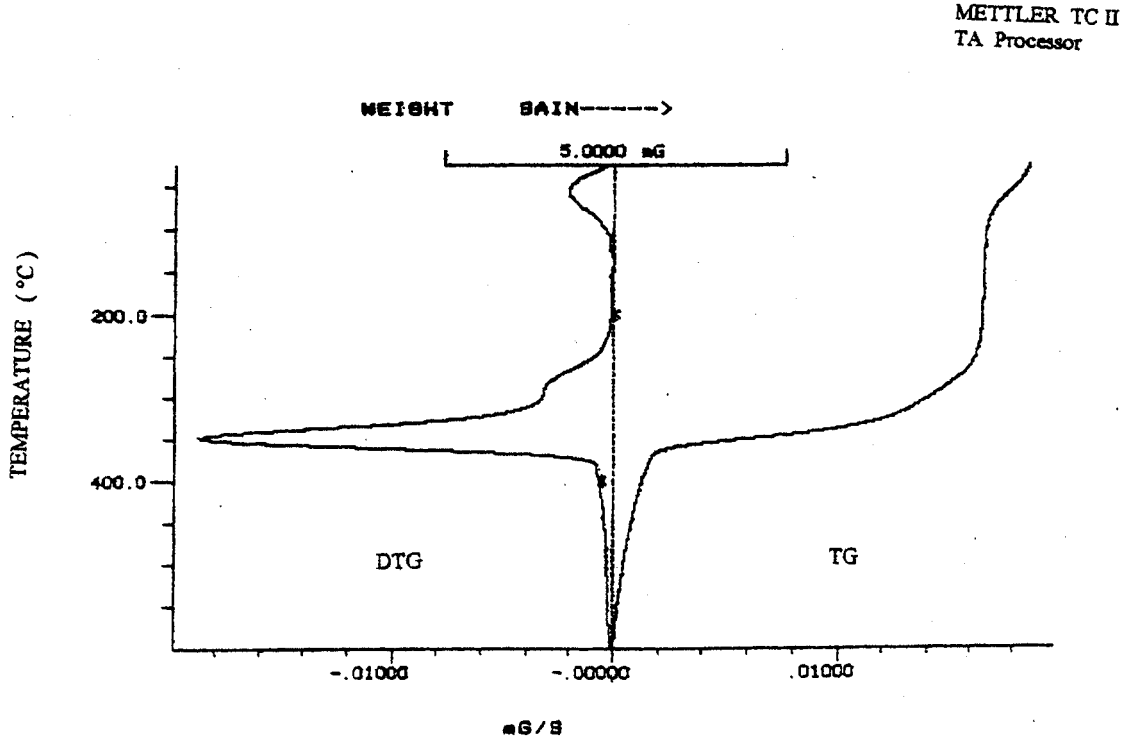

Figure 8 Thermogravimetric and differential thermogravimetric curves of jute fiber.

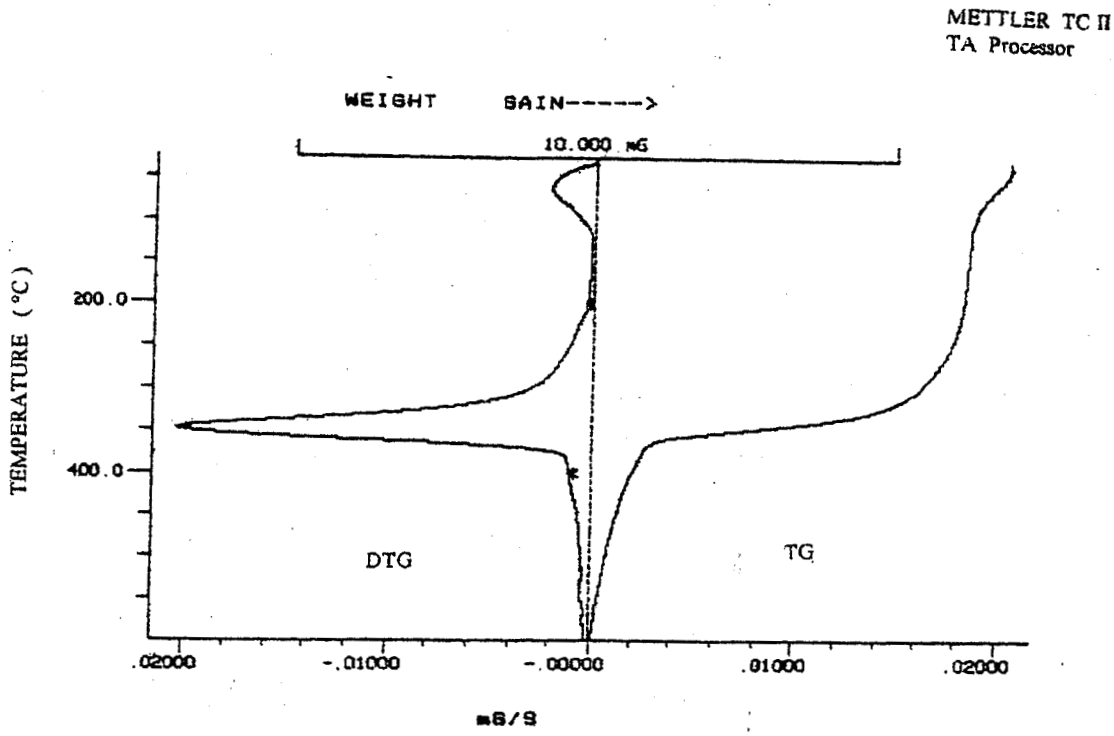

Figure 9 Thermogravimetric and differential thermogravimetric curves of SB-4.

made from untreated jute with 7% PF resin. Some improvement of modulus was noticed when resin was incorporated with the steam-stabilized fiber (SRB-4 and SRB-8), but this was below the modulus value of the control board (Fig. 6). Thus the data obtained from mechanical testing (Fig. 6)

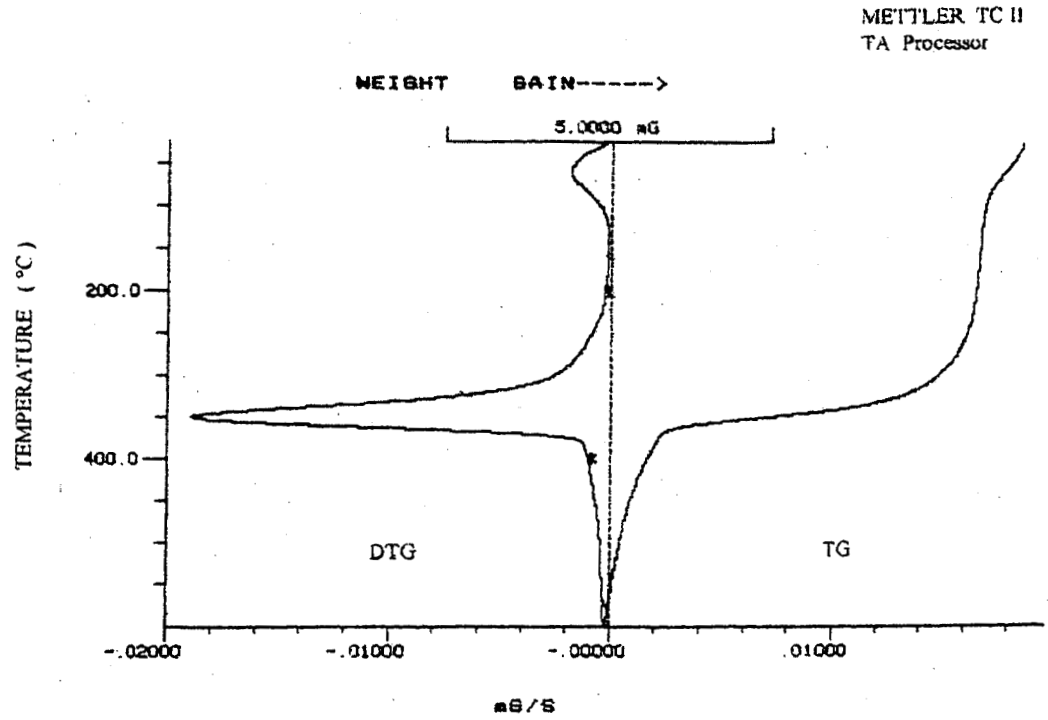

Figure 10 Thermogravimetric and differential thermogravimetric curves of SB-8.

Table I Results of Thermogravimetric Analysis of Steam-Stabilized Jute

Sample	Decomposition Temperature (°C)	Weight Loss (%)	Residual Weight at 600°C (%)
Jute Fiber	64.7	10.52	
	282.3	9.38	14.51
	345.2	65.31	
SB-4	64.7	8.11	
	—	—	20.34
	348.0	71.49	
SB-8	67.5	7.58	
	—	—	20.21
	348.0	72.03	

indicate that steam stabilization helps to bond fiber to form fiber board even without a binder, although the modulus value is lower than that of control. This is probably due to thermoplasticization of fiber lignin which acts as a binder and imparts good moldability.[9]

The SEMs of the untreated and steam-stabilized jute fiber are shown in Figure 7. It is evident from the figure that the untreated fiber has practically no tendency to thermal flow under pressure of the hot press, whereas the steam-stabilized fiber shows a distinct thermal flow under the identical conditions.

Steaming probably lowers the softening points of jute components and imparts thermoplasticity, and thereby fiber undergoes a change in its original structure. Thus, fiber loses its first memory of original configuration because of steaming and the internal stress induced during pressing of the mat is relieved and minimized. Lower internal stress within the pressed jute products may cause less spring-back of the original configuration. The new flattened fiber structure is stable and only undergoes normal reversible swelling when exposed to moisture. During the steam stabilization process, fiber is subjected to heat as well as pressure in the presence of air and moisture, whereby both degradation and oxidation of jute constituents may take place and this may lead to remarkable loss of fiber strength.

The jute fiber and steam-stabilized jute fiber were subjected to thermogravimetric analysis in nitrogen atmosphere to understand their thermal nature. The TG and DTG curves are shown in Figures 8–10 and the results are summarized in Table I. The DTG curve of jute fiber shows an initial weight loss (10.52%) at 64.7°C due to the loss of moisture. In the pyrolysis temperature range, the curve exhibits two decomposition peaks. The first peak (shoulder) at 282.3°C is due to the decomposition of hemicellulose (wt. loss, 9.38%) and second peak at 345.2°C is due to a-cellulose decomposition.[10] Maximum decomposition occurs at this step (wt. loss, 65.31%). In the steam-stabilized fiber (SB-4 and SB-8), the weight loss at initial moisture desorption step is slightly lower than the jute fiber. The hemicellulose decomposition peak is missing in both cases (4 and 8 min process). This means that during steam stabilization, hemicellulose may have been modified to some other form. The cellulose decomposition peak temperature is slightly higher than that of jute. Weight loss at this stage increases. Steam-stabilized fiber from both processes yields almost the same amount of char, which is higher than that of jute fiber.

CONCLUSION

Steam stabilization of jute fiber imparts the dimensional stability to the fiber board. The dimensional stability has been marginally increased with an increase in treatment time after 4 min, but the fiber loses its mechanical strength with an increase in process time. Thermal stability of steam-stabilized fiber is almost the same as that of untreated jute fiber.

The first four authors express their thanks to Professor K. Jayachandran, Director, Indian Jute Industries' Research Association, Calcutta, for his encouragement and keen interest in this work. This study is part of the collaborating research program between IJIRA, Calcutta, India and FPL, Madison, Wisconsin funded by UNDP Jute development program (1992–1997).

REFERENCES

1. Stamm, A. J. Wood and Cellulose Science; The Renold Press Co.: New York, 1964.
2. Rowell, R. M.; Banks, W. B. Water Repellency and Dimensional Stability of Wood, USDA Forest Service General Technical Report FPL 50, FPL, Madison, WI, 1985.
3. Macmillan, W. G. J Text Inst 1939, 30, 305.
4. Mitra, B. C.; Das, S.; Mandal, A.; Saha, A. K in International Symposium on Biocomposites and Blends Based on Jute and Allied Fibers; New Delhi, December 1994.

5. Hsu, W. E. in Proceedings, Stabilization of the Wood Cell Wall, Suchsland, O., Ed.; Michigan State University, East Lansing, MI, 1988.
6. Inoue, M.; Morooka, T.; Norimoto, M.; Rowell, R. M.; Egawa, G. Permanent Fixation of Compressive Deformation of Wood (II): Mechanism of Permanent Fixation; Forest Research Institute Bulletin No. 176, Plackett, D. V.; Dunnigham, E. A, Eds.; Rotorua, New Zealand, 1992, pp 181–189.
7. Inoue, M.; Tanahashi, M.; Rowell, R. M. Wood Fiber Sci 1993,25(3), 224.
8. Rowell, R M.; Kawai, S.; Inoue, M. Wood Fiber Sci 1995, 27(4), 428.
9. Shiraishi, N. Chapter 18 in Wood and Cellulosic Chemistry Hon, D. N. S.; Shiraishi, N., Eds.; Marcel Dekker: New York, 1991.
10. Basak, R. K; Saha, S. G.; Sarkar, A. K; Saha, M.; Das, N. N.; Mukherjee, A. K Text Res J 1993,63,658.