

# Absorption of Steam and Water at Ambient Temperature in Wood Polymer Composites Prepared from Agro-Waste and Novolac

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**ABSTRACT:** Banana (*Musa paradisiaca*), Hemp (*Hibiscus cannabinus*), and Agave (*Agave jourcroydes*) fibers were treated with Novolac resin for the formation of their composites in the ratio of 50 : 50 (wt/wt). These fibers were also treated with maleic anhydride, and it was found that composites based on treated fibers showed higher absorption of steam (at 100°C) up to 12 h; and beyond 18 h, it is less than the untreated fiber composites. However, at ambient temperature, the absorption of water is lesser for composites based on maleic anhydride-treated fiber than for composites based on untreated fibers. The SHORE-D hardness was commonly higher for composites based on maleic-anhydride-treated fibers. © 1998 John Wiley & Sons, Inc. *J Appl Polym Sci* 68: 1417–1421, 1998

**Key words:** wood polymer composites; Novolac; banana; hemp; agave; fibers; maleic anhydride; absorption; steam; water

## INTRODUCTION

The use of renewable fibers like jute, pulp, bagasse, and wood has increased many fold in recent years in wood polymer composites (WPCs) because of low cost and high performance characteristics of these fibers. However, several important problems are associated with the natural fiber plastic composites; notable among them are poor compatibility between them and synthetic resin materials, their poor dimensional stability, particularly in damp/wet environments and under tension, and their plasticity.<sup>1</sup>

The adsorption and desorption of water in wood is accompanied by volume changes. In fact, changes in volume or shape may occur because of the development of the moisture gradients and stresses.<sup>2</sup> Normal wood polymer composite contains polymer only in void spaces and little, if any,

in the cell walls; the result is a lowering in the rate of water vapor diffusion into the cell walls. Given enough time at high humidity, water eventually reaches the cell walls and causes substantially the same volume swelling as in untreated wood.<sup>3</sup>

Carel Caesar et al.<sup>4</sup> studied the swelling of medium density fiber board (MDF) from oil palm fiber with varying amount of fiber. George and Ramakrishnan<sup>5</sup> have also studied the swelling properties of rice husk particle board and found that the swelling property decreases with an increase in the density of particle board. The present work relates to studies on swelling behavior of fiber-reinforced polymer composites prepared separately from untreated and maleic anhydride (MA)-treated banana, hemp, agave fibers, and Novolac resin.

## EXPERIMENTAL

Banana, hemp, and agave fibers were obtained from the raw material by the process of retting. In

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the retting process, the sun-dried banana trunk, hemp stalk, and agave leaves were immersed in water for a week. The pulp of banana trunk, hemp stalk, and agave leaves was removed manually by a scrapper. The fibers obtained were washed with water and dried in sunlight. These fibers were further dried in an oven and cut into pieces 2.0 to 2.5 mm in length. Novolac resin was prepared in the laboratory by reacting phenol with formaldehyde in the mole ratio of 1:0.83 in the presence of oxalic acid (1.5% of phenol) as the catalyst; the resin was then ground as fine powder. Selected fiber and Novolac resin, 50:50 wt/wt, were mixed in a powder blender for about 10 min. The blended material was worked at 100°C for 5 min on to a heated two-roll mill. The fiber-resin mix was then cooled and pulverized. Hexamethylene tetramine (12% of resin) and calcium oxide (3% of resin), used as the curing agent and activator, respectively, were then intimately mixed with the pulverized material.<sup>6</sup>

The pulverized material was molded to 2-mm-thick sheets in a compression-molding machine. The molding was carried out at  $165 \pm 5^\circ\text{C}$  for 4 min employing pressure in the sequence of 4.8,

9.8, 14.7, and 19.6 MPa with a duration of 1 min each. The mold was then cooled under pressure by circulating cold water, and the molded sheet was ejected from the mold after releasing the pressure.

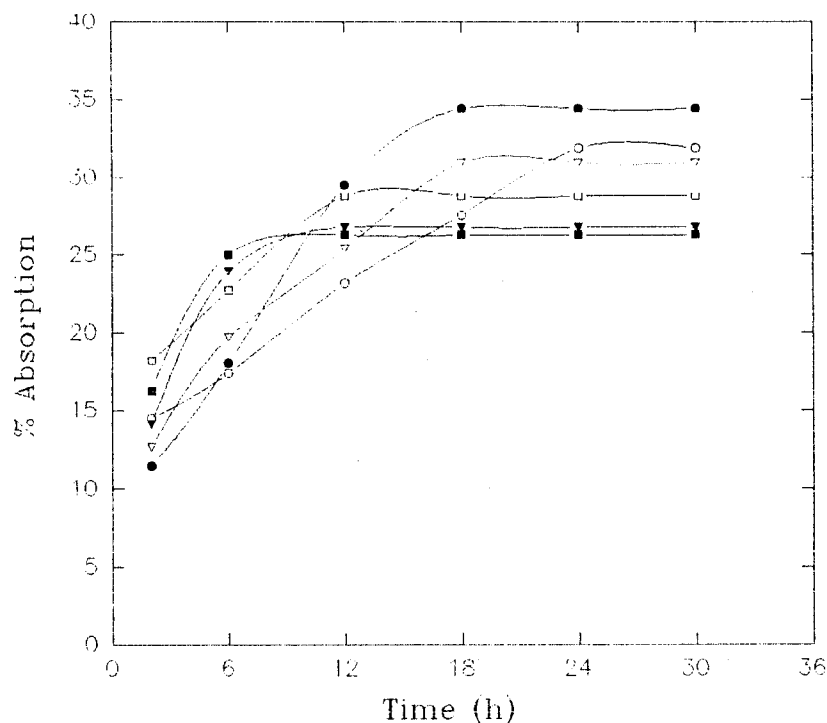
The fiber was esterified by using 2% maleic anhydride in xylene, keeping the fiber-to-solvent ratio at 1:20 (wt/v). The soaking of maleic anhydride was allowed for 18 h at 65°C. The fibers were filtered out and dried in oven at 60°C until constant weight of fibers was achieved.

The hardness of untreated and treated with maleic anhydride fiber-reinforced polymer composites was measured by a SHORE-D hardness tester. The WPCs were allowed to swell for 2 to 30 h in water at ambient temperature and in steam (at 100°C) at atmospheric pressure.

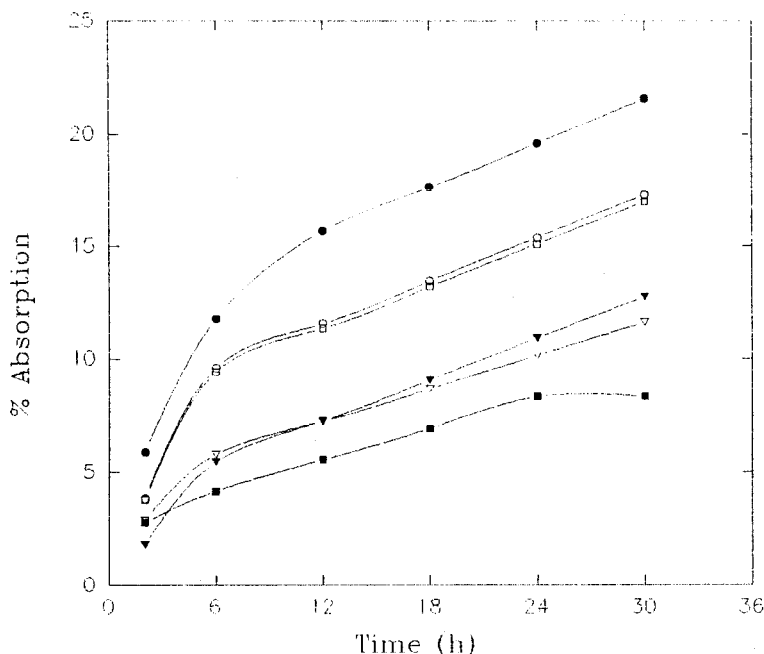
## RESULTS

### Absorption of Steam by Composites Based on Maleic Anhydride-Treated and Untreated Fibers

The composites based on MA-untreated banana, hemp, and agave fibers with Novolac resin are



**Figure 1** Comparison of absorption of steam in banana, hemp, and agave fiber composites with and without treatment of maleic anhydride. (●) Represents untreated hemp fibers; (○) untreated banana fibers; (▽) untreated agave fibers; (□) maleic anhydride-treated hemp fibers; (▼) maleic anhydride-treated banana fibers; (■) maleic anhydride-treated agave fibers.



**Figure 2** Comparison of absorption of water at ambient temperature in banana, hemp, and agave fiber composites with and without treatment of maleic anhydride. (●) Represents untreated hemp fibers; (○) untreated banana fibers; (▽) untreated agave fibers; (□) maleic anhydride-treated hemp fibers; (▼) maleic anhydride-treated banana fibers; (■) maleic anhydride-treated agave fibers.

taken for the steam absorption for 2–30 h. The absorption of steam in these fibers is compared in Figure 1. It is observed from the results that the absorption of steam at 2 h is least in the hemp fiber composite. Further, there is sharp increment of absorption of steam up to 18 h, and the hemp fiber composite absorbs the maximum of steam amongst the above-mentioned fiber composites. The minimum absorption of steam is observed in agave fiber, followed by banana fiber at 24 h. The order of rate of absorption of steam in MA-untreated fibers is hemp > agave > banana up to 12 h. Further, the change in the order of absorption of steam is hemp > banana > agave up to 24 h.

The composites based on MA-treated fibers show higher absorption of steam than untreated fibers up to 12 h and, further, the trend reverses up to 24 h. It is noteworthy that the isosorption of steam is obtained at 9 h for all the MA-treated fiber-based composites. The order of absorption of steam from 12 to 30 h is hemp > banana > agave.

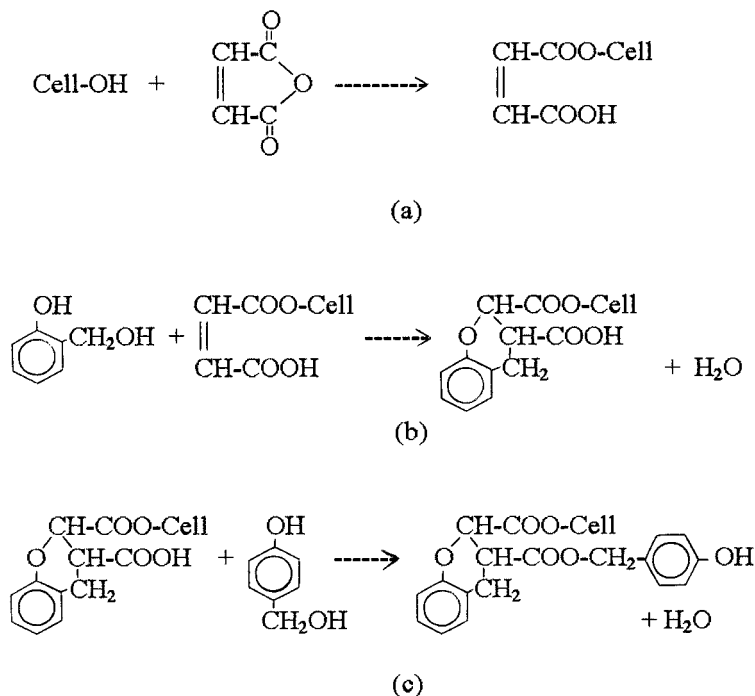
#### Absorption of Water at Ambient Temperature by Composites Based on Maleic Anhydride-Treated and Untreated Fiber

Figure 2 shows the results of water absorption at ambient temperature by composites based on MA-

treated and untreated banana, hemp, and agave fibers with Novolac resin. The order of absorption of water in different composites based on untreated fibers is hemp > banana > agave. The rate of absorption of water in all fiber based composites is maximum up to 6 h, and, further, it gets reduced in all composites. The composites based on MA-treated fibers show three times less absorption of water than the respective untreated fiber-based composites. It is also observed that the composite based on MA-treated hemp fiber absorbs more water than the composites based on MA-untreated agave fiber.

#### DISCUSSION

From the results shown in Figure 1, it is clear that in each case, the initial absorption of steam by the MA-treated fiber-based composite is relatively high when viewed against the same for the untreated fiber-based composite; therefore, the rate of absorption declines due to the maximum penetration of steam and the attainment of a saturation effect. The initial penetrated steam forms hydrogen bonds with —OH groups on the fiber surface, and, consequently, the initial penetration



**Figure 3** Mechanism of esterification of fibers and crosslinking with phenol-formaldehyde.

of steam increases until it reaches a point of saturation. But in MA-treated fiber, the penetration effect gets restricted because the MA picked up by the fiber also partly binds the resin or the polymer by functioning as a compatibilizer. The hydrophilic natural fibers have normally low compatibility with the hydrophobic phenolic resin (Novolac). Esterification of MA with fiber leads to improvement in the compatibility between resin and fiber and draws them nearer to each other, and, due to that, the penetration of steam and/or water gets somewhat restricted. The assumptions are strengthened by the following results of the esterification.

The theoretical MA uptake values for saturation of hemp, banana, and agave fibers are 98.0, 66.2, and 48.3%, respectively, but the experimental values are 51.5, 38.3, and 29.6%, respectively, for the same fibers. These values are lesser than the theoretical values. The degree of esterification of hemp, banana, and agave fibers are 56.8, 48.0, and 37.8%, respectively. There, no acid value is obtained in untreated fibers while the same fibers show 2.90, 2.14, and 1.12% acid values, respectively, after MA treatment.

Absorption of water is much less in comparison with absorption of steam because water cannot penetrate the resin matrix as much as steam can. The absorption of water by different fiber-based

composites is largely dependent on the availability of free —OH groups on the surface of the reinforcing fiber. On MA treatment, some of these —OH groups are esterified, and, due to that, the absorption of water gets restricted. The difference in absorption of water between composites based on treated and untreated fibers is due to blocking of —OH groups in a good measure by esterification on treatment with MA [Fig. 3 (a) and (b)]. It is noteworthy that water absorption of the Novolac resin also gets reduced due to its chemical bonding with the —COOH groups of the MA-treated fibers utilizing part of the hydrophilic methylol (—CH<sub>2</sub>OH) groups in it [Fig. 3(c)]. Here, composites based on treated fibers show higher SHORE-D hardness than that shown by composites based on untreated fibers (Table I).

**Table I** SHORE-D Hardness of Composites Based on MA-Treated and Untreated Fibers

Sr. No.	Samples	Hardness	
		Untreated	MA-Treated
1	Banana fibers	66.00	73.00
2	Hemp fibers	64.00	70.00
3	Agave fibers	69.00	79.00

## CONCLUSION

The following conclusions can be drawn from these studies.

1. The steam absorption in beginning up to 6 h in MA-treated composites is very sharp than the untreated fiber composites, while, in the case of water absorption at ambient temperature, the rate of absorption is more in untreated fiber composites. The overall absorption of steam and water at ambient temperature is more in untreated fiber composites.
2. The hemp fiber composite shows the highest absorption of steam and water at ambient temperature amongst the banana and agave with and without MA-treated fiber composites.
3. The agave fiber composite shows the least absorption of steam and water at ambient temperature with respect to banana and hemp with and without MA-treated fiber composites.
4. The absorption of water in untreated banana fiber composite is more or less equal to the MA-treated hemp fiber composite.
5. The SHORE-D hardness increases with the esterification of fiber with MA, which shows that the compatibility of fiber increases with the polymer resin.

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