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Studies of Dimensional Stability, Thermal Stability and Biodegradation Resistance Capacity of Chemically Treated Bamboo

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ABSTRACT

Bamboo is the world's fast growing and widely used lignocellulosic versatile material. Like other biological materials, bamboo is susceptible to environmental degradation such as moisture, heat, rain, insects etc. as well as dimensional variation when exposed to natural environmental conditions although, with the use of appropriate treatments, the shelf-life of these materials can be prolonged. Chemical modification of split bamboo with a bulk Monomer like Methyl Methacrylate (MMA) or butyl methacrylate (BMA) in the presence of a dithioligand was studied. Polymerisation of MMA and BMA was possible by heat treatment as the catalyst. The dimensional stability efficiency of the treated sample in terms of Anti-shrink Efficiency (ASE%) was improved compared to untreated samples, as were the mechanical strength properties modulus of rupture and modulus of elasticity. The efficacy of the preservative chemicals for the treatment of bamboo samples was evaluated in ground contact against termite and fungus and found to be improved on treatment. The penetration of the preservative chemicals and deterioration of the bamboo samples after burial test was evident from Scanning Electron Micrograph (SEM). From the thermal analysis data of the treated and untreated samples, it was observed that the percentage of weight loss decreases in treated samples compared to untreated sample. The interaction of the bamboo component and chemicals was confirmed by FTIR spectroscopy.

Key words: Bamboo, modification, dimensional stability, biodegradation, polymerization

INTRODUCTION

Bamboo, commonly termed "poor man's timber" in India, is a fast growing lignocellulosic and versatile bio-resource with numerous uses in rural and urban sectors as a raw material due to its fibrous nature, excellent strength, easy workability and availability (Liese and Kumar 2003). Bamboos are also used as craft and building materials in many parts of the world apart from their use as raw material for paper industry. However, some properties restrict the wider use of the lignocellulosic material like dimensional changes due to fluctuating humidity and moisture, biodegradability and photosensitivity etc. (Ozmen, 2007).

Even though bamboos have a hard and highly refractive outer rind, the presence of rich source of potential nutrients and lack of resistive chemical characteristics makes it highly biodegradable. The main components of bamboo are cellulose as a skeleton (40%), hemicellulose as a matrix (~25%) and lignin as an incrusting material (20-25%) (Tamolang *et al*., 1980; Kumar *et al*., 1994; Liese and Kumar, 2003). Most of the physical and chemical properties exhibited by lignocellulosics

are attributed to these natural polymers and their matrix arrangement. All these polymers are hydrophilic in nature due to the presence of hydroxyl and other oxygen-containing groups (Pandey *et al.*, 2009). Since these groups absorb moisture through hydrogen bonding, the moisture content of lignocellulosic material changes as the surrounding humidity and temperature change, which causes swelling of the cell wall of bamboo. On drying, the cell wall shrinks again causing dimensional instability (Ozmen, 2007; Das and Chakrabarty, 2008). Bamboo is also susceptible to degradation by fungus, bamboo borer and certain types of termites. Termites with their cellulose-decomposing bacteria in the gut can easily digest the cellulosic part of bamboo and as a result, the strength of the bamboo reduced drastically which in turn makes it unfit as a construction material. The dimensional instability and biodegradation of bamboo and other lignocellulosic materials can be minimized by modifying the hydroxyl groups found in cell wall polymers by using suitable preservatives (Deka *et al.*, 2003; Singh *et al.*, 1979; Rowell, 1975). To protect the lignocellulosic material from degradation and enhance its service life, various treatment methods have been employed during last few decades such as treating with mineral oil, coal tar ; heating in hydrocarbon oil, smoking, treating with various etherifying and esterifying agents, acetals, alkylene oxide and alkoxysilane-coupling agents and have been documented by several researchers (Rowell, 1975, 2005; Rowell and Gutzmer, 1975; Kumar *et al.*, 1991; Singh *et al.*, 1992; Deka *et al.*, 2003). Studies were also carried out to improve physical strength and other performance properties of wood was done by impregnated with suitable polymers for end use (Hill, 2006). Improvement of moisture resistance and hardness of wood using vinyl monomers followed by curing (radiation or catalyst) was described (Meyer, 1984). Polymer system cross linked by Methyl Methacrylate (MMA) alone or in combination with other monomers for enhanced the dimensional stability was reported (Ng *et al.*, 1999). It has been reported that a combined treatment of boron and MMA could extend the durability of treated wood (Musta *et al.*, 1999). Wood Polymer Composite (WPC) had improved physical and mechanical properties more than untreated wood (Rowell and Konkol, 1987). Improved dimensional stability, strength and stiffness of the wood was achieved when rubber wood is impregnated with styrene in combination with a crosslinker (glycidal methacrylate) (Devi *et al.*, 2003). However, studies on polymer (MMA and BMA) treated bamboo for improving its dimensional and other performance properties are lacking. Therefore, the present study was carried out by treating bamboo with conventional chemicals such as boric acid, copper acetate, trimethyltetramine dithiocarbamate prior to impregnation with Methyl Metha Crylate (MMA) and Butyl methacrylate (BMA) polymer for arresting dimensional change and biodegradation.

MATERIALS AND METHODS

Raw material: The experiment was carried out during 2007-2010 to study the durability and dimensional stability of bamboo. *Bambusa balcooa* Roxb. (Bhaluka) was selected for this experiment due to its wider application in construction of houses, bridge, fencing, agricultural implements and easily available bamboo species of Assam. The internodes portion of a four years old bamboo was collected for the experiments from a homestead garden of Assam, India. Rectangular strips of approximately 10.00×2.15×1.23 cm^3 (length×breadth×thickness) were prepared from bamboo culms for treatment. The samples were pretreated by refluxing in acidic methanol for three hours to elute some lignin, starch, waxes and soluble compounds, thereby facilitating the increase penetration of chemicals. The pretreated samples were then oven dried at 100±5°C so that at least 20% moisture remains inside the samples because microbial degradation can occur if moisture is more than 20% of the dry weight. The remaining 20% moisture is essential

to facilitate movement of water soluble chemical preservatives. The treatment was carried out as per BIS (BIS, 1977).

Treatment of the samples: To carry out this study two polymer namely Methyl Methacrylate (MMA) and butyl methacrylate (BMA) are used. Other chemicals used in this study are Boric Acid (BA), Copper Acetate (CA), trimethyltetramine dithiocarbamate (Triendtc). The treatment of the bamboo samples were carried out as follows:

- The samples were first treated with (5% each) boric acid followed by copper acetate and triethylenetetramine dithiocarbamate (Triendtc)
- The chemically treated samples (a) were soaked in (5%) MMA and BMA solutions and subsequent in situ polymerization were carried out by heat treatment using benzoyl peroxide as catalyst

After completion of chemical treatments, all the samples were treated with kerosene for seven days and air dried.

Evaluation of properties of treated and untreated bamboo: The dimensional parameters such as Anti Shrink Efficiency (ASE),volumetric swelling coefficient (S), Bulk Coefficient (BC) were determined by repeated water soaking method (Rowell and Ellis, 1978) using the following relations.

Wight percent gain (WPG): The WPG or chemical loading was calculated as:

$$WPG = \frac{(W_t - W_u)}{W_u} \times 100$$

where, W_t is OD weight of the treated sample in g. W_u is OD weight of the untreated sample in g.

Volumetric swelling co-efficient (S) and anti shrink efficiency (ASE): The S and ASE were calculated following the relations:

$$S_{ut} = \frac{(V_w - V_{ut})}{V_{ut}} \times 100$$

where, V_w is swollen volume of bamboo sample after treatment in cm^3, $V_{u,t}$ is OD volume of either untreated and treated samples in cm^3:

$$ASE = \frac{(S_u - S_t)}{S_u} \times 100$$

where, S_u is volumetric swelling of the untreated sample, S_t is volumetric swelling of the treated sample.

Bulk co-efficient (BC): Bulk (BC) was calculated as:

$$BC = \frac{(V_t - V_u)}{V_u} \times 100$$

where V_u and V_t are oven-drying volumes of untreated and treated samples, respectively.

Infra-Red (IR) spectroscopic studies of treated and untreated bamboos: The IR spectra of treated and untreated bamboo powder were recorded as KBr (potassium bromide) pallet in a Perkin Elmer Spectrophotometer, Model 580 B, in the range of 4000-200 cm^{-1}.

Scanning electron micrograph (SEM) study: To study the adherence of chemicals onto bamboo cell wall, the SEM of treated and untreated samples were taken in Scanning Electron Microscope. The observations were made in JEOL, JSM-35M-35 CF electron microscope at Indian Institute of Technology, Guwahati, Assam, India.

Thermal analyses: For thermal analyses of treated and untreated bamboo samples, the samples were first ground to powder form (+40 and-60 British Standard Sieve) and then simultaneous thermogravemetric (TG) and Differential Thermogravemetric (DTG) analyses were carried out using Perkin Elmer Pyris Diamond Instrument at heating rates of 20°C min^{-1} in the temperature range 40-650°C in air atmosphere. The weight of the sample powder was in the range of 5.88-8.44 mg and α alumina was used as reference material.

Evaluation of physical and mechanical properties of the bamboo samples: The physical and mechanical properties of the treated and untreated bamboo samples were evaluated by using Universal Testing Machine at NEIST, Jorhat, Assam, India.

Determination of modulus of rupture (MOR): The MOR of the specimens was reported using the following mathematical expression:

$$MOR\ (kg\ cm^{-2}) = \frac{3\,PL}{2bd^2}$$

where, P = Maximum load applied in kg., L = Length span of the tested specimen in cm., b = Width of the specimen in cm, d = Depth of the specimen in cm.

Determination of modulus of elasticity (MOE): The modulus of elasticity of the test specimen were determined using the relation:

$$MOE\ (1000\ kg\ cm^{-2}) = \frac{P1\,L^3}{4bd^3Y}$$

where, P_1 is load in kg at proportionality limit, Y is central deflection at the limit of proportionality load in cm, L is length span of the test specimen in cm, b is width of the test specimen in cm, d is depth of the specimen in cm.

Determination of termite resistance capacity: The termite resistance capacities of treated and untreated bamboo samples were determined visually. For this, the treated bamboo samples along

with untreated control samples were subjected to termite attack in a termite colony under ambient environmental condition (average temperature of 28°C and average relative humidity of 72%). After six months of exposure to termite, the samples were exhumed for visual observation and recorded the damage level.

Cellulase inhibition test: To carry out the cellulase inhibition test, five 250 mL beaker was taken (marked 1 to 5). In each of five 250 mL beakers 60 mL of phosphate buffer (pH 6.47) were taken, to each of which 1 gm of bamboo powder (BP) was added. In each of the beaker (No. 2-5), 5% (w/v) of Boric Acid (BA), Copper Acetate (CA), sodium salt of triethylenetetramine dithiocarbamate (Triendtc.) and a mixture of 5% (w/v) each of BA, CA, Triendtc. were added. To each of the five beakers 6 mL (300 units) of cellulase enzyme (Aldrich) were added. After adding chemicals and cellulase enzyme, the beakers were covered for 24 h at room temperature along with remaining beaker without adding the chemicals. After which they were heated at 60°C for 1 h in water bath to stop the cellulase activity. After cooling at room temperature, the solution were filtered and made up the volume to 100 mL and glucose was estimated by Fehling solution using methylene blue indicator relative to a standard glucose solution.

RESULTS AND DISCUSSION

The dimensional variations of bamboo samples treated with different chemicals and their combinations in terms of antishrink efficiency (ASE), bulk coefficient (BC) and Weight Percent Gain (WPG) shows in Table 1. The bamboo samples treated with BA, CA, Triendtc followed by MMA and BMA showed comparatively higher antishrink efficiency (ASE) with increase in Bulk Coefficient (BC). In the present study maximum ASE (59.39%) value at the level of 26.11% WPG shown by the sample treated with boric acid, copper acetate, triendtc followed by MMA and kerosene. An ASE of 46% was reported when oil palm wood treated with MMA without cross linking agents (Ibrahim, 1989). Musta *et al.* (1999) reported improved dimensional stability and moisture excluding efficiency of boric acid and MMA treated Japanese cedar wood. Geraldes *et al.* (2004) did a series of studies and reported that acrylates crosslinking agents capable to impart a more effective cross linking on poly (MMA) matrix. Hill (2006) studied the dimensional stability of Mahang wood treated with MMA without cross linking agents and reported that though there is no significant changes in dimension of the wood after MMA treatment, this treatment showed very effective in reducing water or moisture uptake. Similar results have been observed in the present study also. In this study MMA and BMA treatment without any cross linker showed very effective in reducing water or moisture uptake. The results of the present study suggest that in presence of polymer and chemicals, the cell wall of bamboo showed less shrinkage and swelling in contact with moisture than the untreated samples. The added polymer MMA and BMA absorbed by the remaining pores of the cell in bamboo after the impregnation of the chemicals such as BA, CA, Triendtc. The presence of kerosene creates a hydrophobic environment inside and on the surface of the samples as well as prevents the leaching of preservatives by moisture penetration. This treatment also showed higher weight percent gain (WPG 26.11%) value than the other treatments as well as untreated samples. Increase of WPG indicates the presence of added chemicals in to the bamboo samples. The dimensional stability in terms of antishrink efficiency was determined and found to be improved on treatment. However, these samples also exhibits total protection from termite compared to simple MMA and BMA treatment (Table 1).

The assignment of the major FTIR absorption bands of modified and unmodified bamboo samples were shown in Fig. 1. The appearance of a strong absorption band at 3425-3340 cm^{-1}

Table 1: Dimensional characteristics of the treated and untreated bamboo sample

Chemical used	WPG (%)	BC (%)	ASE (%)
Untreated	0	0	0
MMA	17.65	4.93	44.67
BMA	17.04	4.62	48.14
BA, CA, Triendtc, MMA, Kerosene	26.11*	6.42*	59.39*
BA, CA, Triendtc, BMA, Kerosene	24.91	6.23	54.71

*Combined treatment of BA, CA, Triendtc, MMA and Kerosene showing highest WPG, BC and ASE, value

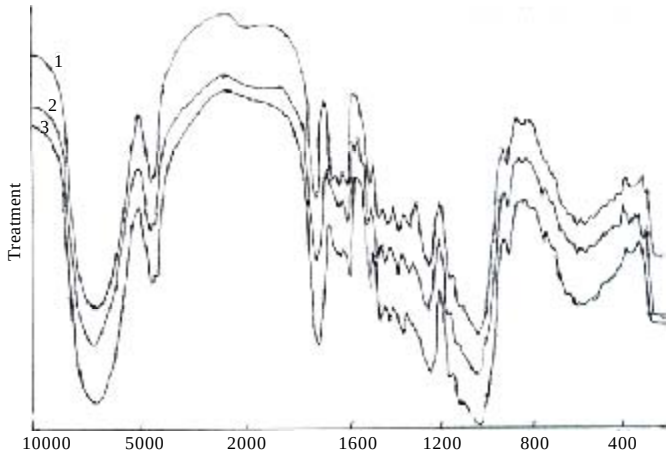

Fig. 1: IR spectra of (1) MMA, (2) BMA and (3) Untreated bamboo

ascribed due to the v_{O-H} as well as the presence of H- bonding network. However, two bands in the 2929-2960 cm^{-1} were also prominent in all spectra, these being attributed to C-H stretching vibration in methyl and methylene groups of lignin and extractives. Strong absorption band at around 1740 cm^{-1} ascribed due to carbonyl group C = O and 1270 cm^{-1} due to C-O group. Two small bands at 1600 and 1637 cm^{-1} are assigned to the absorbed water and β -glucosidic linkages between the sugar units, respectively (Owen and Thomas, 1989). Weak absorptions between 1500 and 1400 cm^{-1} arise from the aromatic ring vibrations and ring breathing with C-O stretching of lignin. Increase in the intensity of v_{O-H} in plane bending vibrations at 1385 cm^{-1}, a band was observed, which is specific to the bamboo components, cellulose and hemicellulose.

The major chemical components of wood degrade at different temperatures. Hemicelluloses and lignin are amorphous and start to degrade before cellulose (Hill, 2006). Hemicelluloses are the least thermally stable wood components due to the presence of acetyl group (Bourgois *et al.*, 1989). In the present study two significant weight losses has been observed. The first weight loss is attributed due to the decomposition of hemicelluloses (Nguyen *et al.*, 1981) and the second weight loss is attributed to cellulose decomposition (Nguyen *et al.*, 1981; Bouchard *et al.*, 1986). From the experimental results of thermo gravimetric analyses, it was observed that the major weight losses due to thermal degradation for treated and untreated bamboo samples were found at the stage II, which supports the present findings. These weight losses were followed by lignin at a temperature above 370°C. In contrast to the other weight loss, the polymerised bamboo material shows minimum weight loss (53.82). This could be due to the greater stability of the hemicellulose after

polymerisation with MMA and BMA. Amongst the treated samples BA+CA+Triendtc+MMA and kerosene showed the highest thermal stability. It was also observed that the temperature at which moisture started to be liberated was higher (235°C) for treated samples. This observation can be explained on the basis of changes occurring in the fine structure and morphology of bamboo fibers due to treatment. On modification, the tendency to liberate absorbed moisture upon heating was decreased, as moisture is strongly held within a tightly packed structure, leading to a higher finished temperature (Bouchard *et al*., 1986), similar results have been found in the present study. Variations of weight loss and decomposition temperatures of the treated and untreated bamboo samples are shown in Table 2. From TG curves, (Fig 2, 3) it was observed that the first weight loss in the range of 120-150°C corresponds to water evaporation. The active decomposition temperature that caused the major weight loss were started from 245°C and it was highest (247-359°C) for BA, CA, Triendtc followed by MMA and kerosene treated (Fig. 3) sample indicating increased bulking and -OH group modification to give good dimensional stability. The result showed that there is a substantial decrease of weight loss in the treated samples and it was minimum(53.82%) for BA, CA, Triendtc followed by MMA and kerosene treatment, which also revealed that enhanced ASE and BC gave better thermal stability of bamboo samples.

From the measurement of strength (MOR) and stiffness (MOE) of both treated and untreated samples, it was observed that there is increase in the Modulus of Rupture (MOR) and compressive stress. No significant difference in MOE was found between the simple MMA and BMA treated and untreated bamboo samples, although 14% improvement was observed in BA, CA, Triendtc,

Table 2: Weight loss and active decomposition temperature of untreated and treated bamboo sample

Chemical used	1st decomposition temperature (°C)	Weight loss (%)	2nd decomposition temperature (°C)	Weight loss (%)
Untreated	43-103	10.11	198-354	81.54
MMA	44-114	7.93	235-370	57.86
BMA	44-111	8.62	238-363	58.67
BA, CA, Triendtc, MMA Kerosene	43-113	7.30*	245-3595	3.82*
BA, CA, Triendtc, BMA, Kerosene	43-112	7.80	238-357	54.11

Combination of BA, CA, Triendtc, MMA and Kerosene treatment showing minimum weight loss

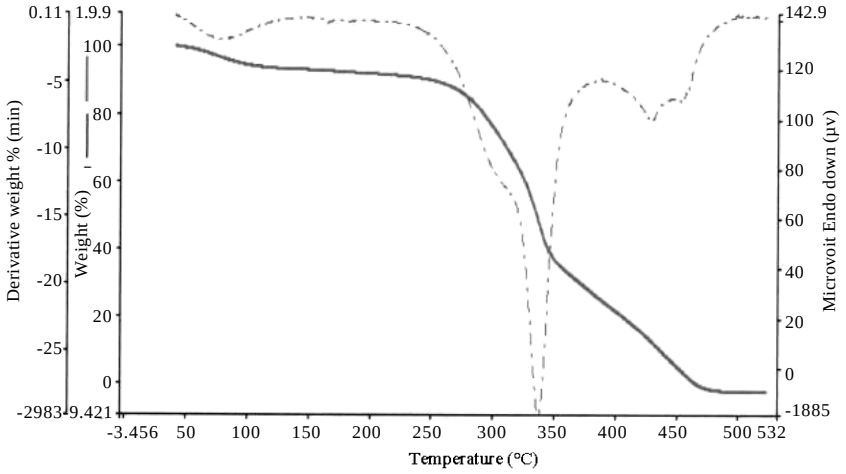

Fig. 2: Thermogram of untreated bamboo sample

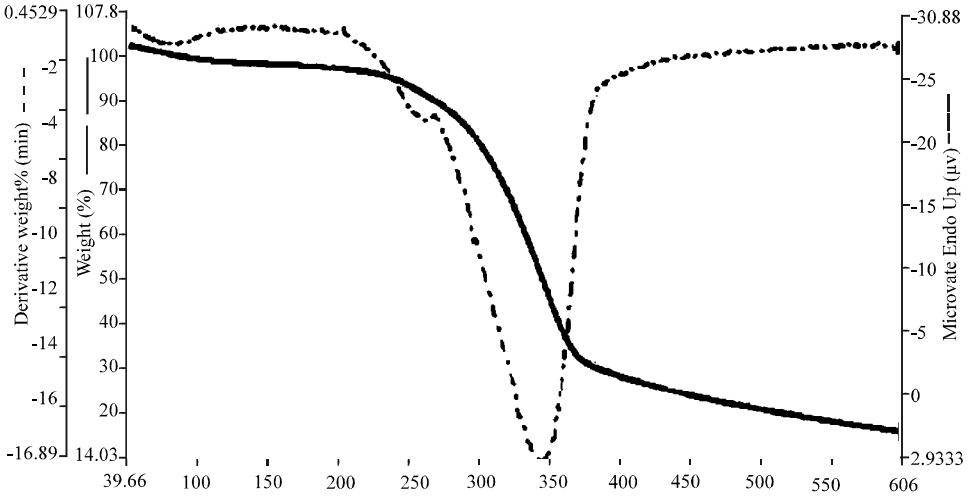

Fig. 3: Thermogram of BA, CA, Triendtc and MMA treated bamboo sample

Table 3: Effect of chemicals on strength (MOR) and stiffness (MOE) of bamboo sample

Chemical used	MOR (N mm^{-2})	MOE (N mm^{-2})
Untreated	48.70	6520
MMA	52.87	6843
BMA	51.04	6570
BA, CA, Triendtc, MMA, Kerosene	61.06*	7543*
BA, CA, Triendtc, BMA, Kerosene	58.51	7183

*Combination with BA, CA, Triendtc, BMA and Kerosene treatment showing highest strength (MOR) and stiffness (MOE)

MMA, BMA and kerosene treated samples (Table 3).The insignificant change in MOE suggest that the polymer itself was not elastic enough which could enhance the elasticity of bamboo. Schneider *et al.* (2003) also reported the same for basswood when treated with MMA and the treated material with addition of 1% TMPTMA showed highest MOR value (88.87%). In the present study bamboo samples treated with BA, CA, Triendtc, MMA and kerosene showed highest MOR (61.06 N mm^{-2}) and MOE (7543 N mm^{-2}). It could be due to the chemical modification of cellulose by MMA and physical bulking of copper acetate, boric acid, triendtc and kerosene oil. Wahab *et al.* (2006) studied the strength properties of *Gigantochloa scortechinii* treated with Ammonium Copper-Quaternary (ACQ), Copper Chrome Arsenic (CCA) and Borax Boric Acid (BBA) and reported that the strength properties of the treated bamboo dependent on the type of preservative applied, concentration and their retention in the bamboo. Impregnation of *Dyera costulata* wood with phenol formaldehyde resin mixed with urea showed higher MOR, MOE and dimensional stability (Izreen *et al.*, 2011).This treatment also rendered higher anti swelling efficiency (20%).

The incorporation of the preservatives constituent in to the internal configuration of the bamboo samples were studied by Scanning Electron Micrograph (SEM). The penetration of the chemicals resulted in bulking of the cell wall to give dimensionally stabilized bamboo. The SEM of the untreated sample (Fig. 4) showed a diffused configuration, while the white patches seen in the micrograph (Fig. 5-7) were the penetration of the chemicals observed on the cell walls of the bamboo.

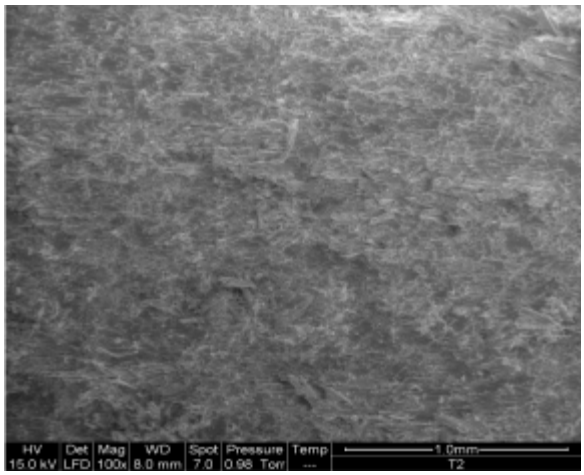

Fig. 4: SEM of untreated bamboo sample

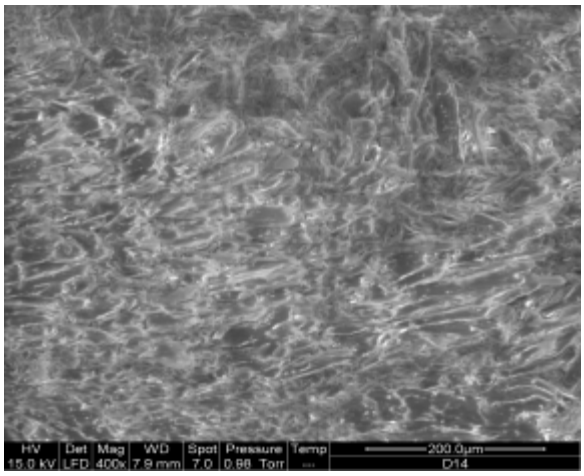

Fig. 5: SEM of MMA penetration in bamboo sample

After one year of graveyard (termite mound) test (Fig. 8) it was observed that compared to untreated samples, the samples treated with only MMA and BMA was slightly damaged by termite (Fig 9). On the other hand, the samples treated with BA, CA and triendtc showed good biodegradation inhibition but the dimensional stability was not optimum.

The bio-resisting property of this treatment may be due to the antibacterial and antifungal activity of the boric acid, copper acetate and dithiocarbamate. Boric acid has been reported as stomach poison for insects (Yamaguchi, 2003). Similarly transition metal compounds of dithiocarbamate and dithiophosphinates are known antifungal and antibacterial agents and the mode of action being of certain vital enzymes by the sulphur donors, the Cu^{2+} also can inhibit action of several biomolecules (Kalita *et al*., 2002). Dithiocompounds also inhibit the enzyme actycholinesterase (Gruzdyev *et al*., 1980). Further petroleum oil like kerosene has low toxicity to warm blooded animals but prevents metabolism in egg or insect body. The oil can easily penetrate through the wax scale and cuticles, can cause coagulation of the cytoplasma and inhibit the course of enzyme process (Gruzdyev *et al*., 1980).

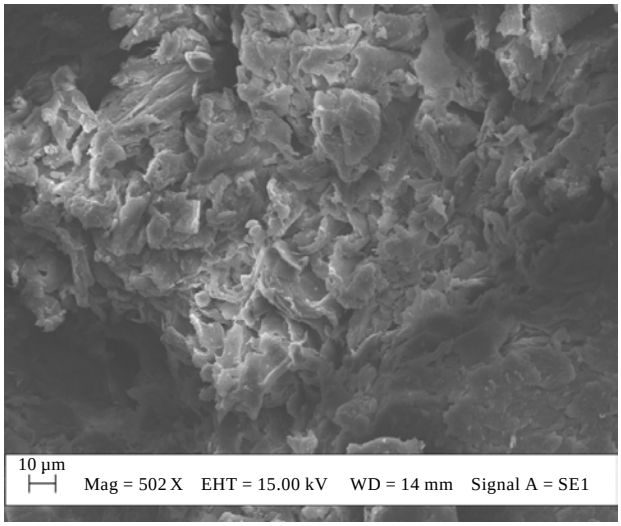

Fig. 6: SEM of BMA treated bamboo sample

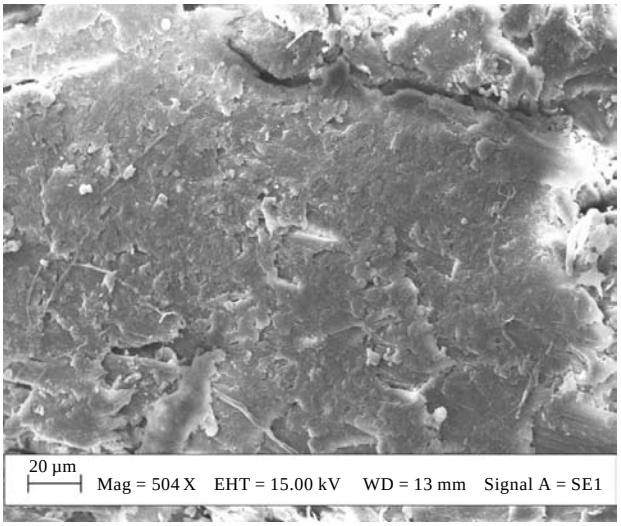

Fig. 7: SEM of BA, CA, Triendtc and MMA treated bamboo sample

Lignocellulosic materials are degraded by fungus and termites which contain cellulose enzyme. In the mechanisms of the cellulase action, it is found that the *Tricoderma* Cellulase consists of three enzymes systems, (a) endoglucanase (EG), (b) Cellulohydrolase (CBH) and (c) β-glucosidase (β-G) (White, 1982). The inhibition of cellulase activity and dimensional stabilization may be concurrently considered from two angles: (1) Application of cellulase activity inhibitor and (2) Chemical modification of the-OH group of the non reducing chain end, the site of CBH attack. The -OH groups are not only enzyme attack sites but also water absorption sites through hydrogen bonding, which facilitate swelling. In the cellulase activity inhibition experiment (Table 4) it was observed that in presence of boric acid and copper acetate the cellulase activity (amount of glucose

Fig. 8: Termite mound test of bamboo samples

Fig. 9: Deterioration of the treated (G1,G2,G3)and Untreated(C) bamboo samples after Graveyard test

released) is slightly reduced. The combined treatment of boric acid, copper acetate, triendtc and cellulose showing least amount (0.154 g $mole^{-1}$) of glucose liberation, which showed that these chemicals are effective as enzyme inhibitor. The present finding was supported by Vyas *et al.*

Table 4: Cellulose inhibition activity data in absence and in presence of chemicals

Chemical used	Amount of glncose (g mL^{-1})
Bamboo powder (BP)+cellulase	0.321
BP+BA+cellulase	0.224
BP+CA+cellulase	0.209
BP+Triendtc+cellulase	0.201
BP+BA+CA+Triendtc+cellulase	0.154*

Combination of BP+BA+CA+Triendtc+cellulase showing least amount of glucose liberation

(2005), that in alkaline media cellulase activity is enhanced compared to acidic media. The presence of boric acid and copper acetate hydrolyze to give slightly acidic medium due to the presence of acetic acid. However, the presence of boric acid, copper acetate and dithiocarbamate, there is a substantial decrease in the cellulase inhibition activity as seen from the amount of glucose released.

CONCLUSION

Dimensional stabiity and resistance to biodegradation by chemical treatment of lignocellulosic materials depends upon the type of chemicals, their penetration and how much hydroxyl groups are modified to give adequate cross linking and bulkiness. From the present studies, it may be concluded that treatment of bamboo samples with boric acid followed by copper acetate, triethylenetetramine dithiocarbamate, methyl methacrylate and kerosene can be used for achieving dimensional stability and prevent biodegradation. The strength and stiffness properties of bamboo can also be improved by this treatment. Compared to other chemical methods of treatment of bamboo, the present method appears to be better in the sense that the chemicals are cost effective, can be handle and applied easily and less toxic at low concentration and at the same time give very good results.

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