

## **Studies on dimensional stability, thermal degradation and termite resistant properties of bamboo (*Bambusa tulda* Roxb.) treated with thermosetting resins**

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**Abstract**—Experiments were carried out to study dimensional stability, thermal degradation and termite (*Odontotermes* spp) resistant properties of thermosetting resin treated bamboo (*Bambusa tulda* Roxb.) in comparison to an untreated one. The dimensional stability of treated samples in terms of anti-shrink efficiency (ASE) was increased by 53.80–61.18% at levels of 29.73–30.14% weight percent gain (WPG) of the resins over the untreated one. The treated samples also retained bulking coefficient (BC; 7.56–8.74%) and moisture excluding efficiency (MEE; 23.51–33.42%) after three cycles of repeated wetting and drying at these levels of WPG. The treatment also resulted in an improvement of static bending in terms of modulus of rupture (MOR) by 18.49% for PF (phenol formaldehyde), 14.21% for MF (melamine formaldehyde) and 10.34% for UF (urea formaldehyde) and modulus of elasticity (MOE) by 8.15% for PF, 7.18% for MF and 6.98% for UF at 29.73, 30.14 and 29.81 levels of WPG, respectively, over the untreated samples without any remarkable effect on specific gravity. The values of MOR and MOE of the treated samples showed negligible change after twelve months' exposure to termite attack under ambient environmental conditions, while the untreated sample was badly damaged by termite. The thermal behaviour of untreated and treated samples was studied using thermogravimetric (TG) and differential thermogravimetric (DTG) techniques at heating rate 20 and 30°C min<sup>-1</sup> in temperature range 30–650°C. The kinetic parameters of untreated and treated samples were evaluated using the Coats and Redfern method. The treated samples were found to be thermally more stable than the untreated one.

*Key words:* Thermosetting resins; modulus of rupture; modulus of elasticity; thermal degradation; activation energy; frequency factor; termite resistant capacity.

### **ABBREVIATIONS**

ASE anti-shrink efficiency  
BC bulking co-efficient  
DTG differential thermogravimetric technique

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MEE	moisture excluding efficiency
MF	melamine formaldehyde
MOE	modulus of elasticity
MOR	modulus of rupture
PF	phenol formaldehyde
TG	thermogravimetric technique
UF	urea formaldehyde
WPG	weight percent gain

## INTRODUCTION

Bamboos, a group of giant arborescent evergreen grasses, are abundant in tropical and subtropical regions. Bamboos are used as craft and building materials in many parts of the world apart from their use as raw material for paper industries. Cellulose, being the chief component of bamboo, governs most of its physical and chemical properties. For use as constructional material, bamboo has some troublesome inherent properties, such as change in dimension with time due to varying atmospheric moisture and degradation by biotic and abiotic agents [1–7]. The degradation involves physical as well as chemical changes in its constituents, thereby making it unfit for use in permanent constructions.

The dimensional changes due to varying atmospheric moisture can be minimized by reducing moisture absorption and swelling, bulking the fibres to reduce water holding capacity or by cross-linking the cellulose chains of the component fibres with some chemicals to keep the wood materials in swollen state [8–10]. Such treatments prevent the woody material from expansion and contraction on further contact with moisture [11, 12]. Since the major constituent of bamboo is cellulose, it also serves as a good source of foodstuff for termites. Termites, with their cellulose decomposing bacteria in the gut, can easily digest the cellulosic part of the cell wall and, as a result, the strength of the bamboo is reduced drastically [1, 13]. Again, natural weathering factors such as heat, light, oxygen, relative humidity and such other variables also undoubtedly cause physical as well as chemical changes. To reduce degradation of cellulosic components by these biotic and abiotic factors, studies were conducted on treatment of wood materials with various etherifying and esterifying agents, acetals, alkylene oxide and alkoxysilane-coupling agents [2, 3, 5, 14–16]. Studies were also carried out to improve physical strength and other performance properties of wood by treating with thermosetting resins [8, 9]. However, reports on chemical treatment of bamboo for improving its performance properties as constructional material are very limited. The present investigation was therefore carried out to study the improvements in properties, like dimensional stability, thermal and termite resistant capacities, etc. of bamboo by treating with thermosetting resins.

## MATERIALS AND METHODS

### *Raw materials*

The internode portions of a 6.5 year old bamboo (*Bambusa tulda* Roxb.) were collected for the experiments from a social forest of Assam, India. Rectangular strips (without epidermal layer) of approximately  $0.70 \times 2.19 \times 10.00$  cm<sup>3</sup> (thickness  $\times$  breadth  $\times$  length) were prepared from air-dried bamboo logs (12% moisture) for the treatment as set out by the Bureau of Indian Standards [17]. The samples were then soxhlet extracted with each of dichloromethane, benzene-ethanol (2:1 v/v), acetone and methanol in sequence for at least 12 h and then oven dried. The dried samples were used for resin impregnation [12]. The soxhlet extraction removes some of the waxes, fats, resins and nonvolatile hydrocarbons, thereby facilitating the increased penetration of polymers into woody substrate [12, 18].

The resins, PF, MF and UF, the characteristic properties of which are shown in Table 1, were prepared in the laboratory for the treatment [8, 9].

### *Treatment of samples*

The resins were first diluted with water to desired concentrations. The experimental samples along with the required quantity of resins were taken in a stainless steel reactor and the treatments were carried out at a wide range of temperature and pressure for different reaction times. The treatment conditions were first standardized and then further treatments under optimum conditions were carried out to obtain sufficient polymer loading [8, 9]. The polymer-treated samples were cured at 115°C for 1 h and then evaluated for various properties.

### *Evaluation of properties of treated and untreated samples*

The properties of treated and untreated wood samples were evaluated using the following relationships [4], and the average of three test readings for identically treated specimens was reported.

The WPG or chemical retention was determined using the relation,

$$\text{WPG} = (W_t - W_u) \times 100 / W_u,$$

**Table 1.**

Characteristic properties of the resins phenol formaldehyde (PF), melamine formaldehyde (MF) and urea formaldehyde (UF)

Properties	PF	MF	UF
Specific gravity (g/cm <sup>3</sup> )	1.15–1.18	1.17–1.18	1.19–1.21
Viscosity at 30°C (Cp)	23.5–25.2	26.3–28.1	30.3–31.0
pH	9.0–9.5	8.5–9.5	9.0–9.3
Solid content (%)	58–60	62–63	50–52
Molecular weight	12431	12353	11892

where  $W_t$  and  $W_u$  are oven dry (OD) weights of treated and untreated samples, respectively.

The dimensions of treated and untreated samples were monitored with vernier calipers after subjecting the samples to three cycles of repeated wetting and drying. The test samples were wetted for 24 h under running water and then dried by putting the samples in an oven at  $100 \pm 5^\circ\text{C}$  till the samples attained constant weights. This process was repeated thrice. The anti-shrink efficiency (ASE) was calculated from the following relationship [1]:

$$\text{ASE} = (S_u - S_t) \times 100/S_u,$$

where  $S_u$  and  $S_t$  are the volumetric swelling co-efficient based on the OD volume of untreated and treated samples, respectively.

Moisture excluding efficiency (MEE) and bulking co-efficient (BC) were determined by using the relations,

$$\text{MEE} = (M_u - M_t) \times 100/M_u,$$

where  $M_u$  and  $M_t$  are equilibrium moisture contents of untreated and treated samples, respectively at  $28^\circ\text{C}$  and 75% RH, and

$$\text{BC} = (V_t - V_u) \times 100/V_u,$$

where  $V_t$  and  $V_u$  are OD volumes of treated and untreated samples, respectively.

Modulus of rupture (MOR) and modulus of elasticity (MOE) were determined according to IS-2380, 1977 [19].

$$\text{MOR} = 3PL/2bd^2,$$

where  $P$  is the maximum load in kg,  $L$  is the length of span in cm,  $b$  is the width of the specimen in cm and  $d$  is the depth of the specimen in cm.

$$\text{MOE} = P_1L^3/4bd^3Y,$$

where  $P_1$  is the load in kg at the proportionality limit,  $Y$  is the central deflection at limit of proportionality load in cm.

### *Infra red (IR) study*

The IR spectra of treated and untreated samples were recorded in a Perkin Elmer Spectrometer (Model 580 B) using KBr as solvent in the range of  $4000\text{--}400\text{ cm}^{-1}$ .

### *Thermal analyses*

Simultaneous thermogravimetry (TG) and differential thermogravimetry (DTG) studies were carried out using a Shimadzu Thermal Analyzer (Model 30) at heating rates of  $20$  and  $30^\circ\text{C min}^{-1}$  in the temperature range  $30\text{--}650^\circ\text{C}$  in static atmosphere. The weight of the sample was in the range  $9.52\text{--}15.03\text{ mg}$  and  $\alpha$  alumina was used as reference material.

*Termite resistant capacity*

The treated samples with the highest dimensional and thermal stabilities were subjected to termite attack by putting the samples in soil infested with termite along with the untreated one for a period of twelve months under natural environmental conditions (average temperature 26°C and RH 72%). The termite resistant capacity was evaluated visually. The effects of termite attack on MOR and MOE were also evaluated [8, 9].

*Statistical analyses*

The experimental results were analyzed statistically using the technique of analysis of variance in which each treatment was replicated three times in a completely randomised design (CRD). The significance of the treatment differences was judged by 'F' test as outlined by Bhattacharyya, 1999 [19].

The standard error of differences (SEd $\pm$ ) was calculated using the expression,

$$\text{SEd}(\pm) = \sqrt{(2 \cdot \text{error mean square} / \text{number of replication})}$$

The critical difference (CD) was calculated to test the differences amongst the treatments as,

$$\text{CD} (5\%) = \text{SEd}(\pm) \cdot t,$$

where  $t = 5\%$  tabulated value of Student's  $t$ -variable at error degree of freedom.

**RESULTS AND DISCUSSION**

A series of experiments were carried out under varied conditions of pressure, temperature and time with different polymer concentrations to optimize the condition at which sufficient polymer loading was obtained to cause maximum level of swelling of the samples. This was achieved with 30% resin at 90–100°C in 75 psi (0.52 N/mm<sup>2</sup>) for treatment period of 1–2 h [8, 9].

*Dimensional stability*

It was apparent from Table 2 that the treated bamboo samples showed significant difference in the dimensional stability efficiency in terms of ASE (the highest values are 61.18% for PF at 29.73, 58.32% for MF at 30.10 and 53.80% for UF at 29.81 levels of WPG). The values of ASE did not change even after three cycles of repeated wetting and drying. It was also clear from Table 2 that BC of the treated samples was increased significantly with increase in WPG and reached the maximum of 8.74, 8.14 and 7.56 at 29.73, 30.10 and 29.81 levels of WPG for PF, MF and UF resin, respectively. Also, the values of MEE were increased significantly with respect to increase in WPG up to these levels. From these results, it could be inferred that the resin treatments caused the maximum level of bulking of cell wall

**Table 2.**

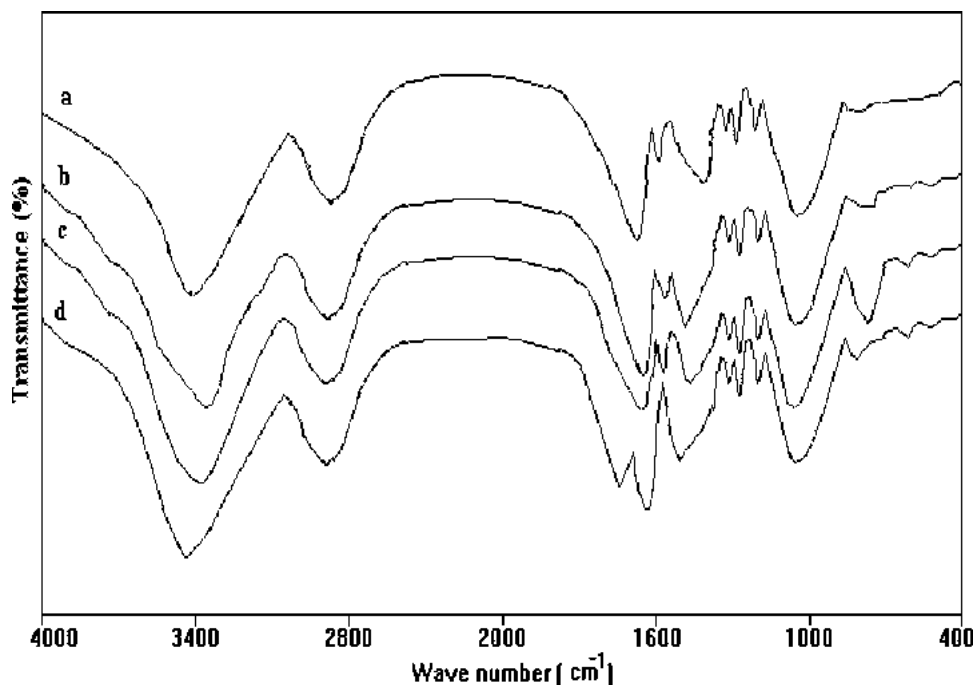
Effect of different levels of WPG on BC, MEE and ASE of bamboo

Reagents used	WPG	BC (%)	MEE (%)	ASE (%)
Untreated	0	0	0	0
PF treated	7.31	1.11	12.31	12.44
	18.42	4.83	23.91	42.35
	29.73	8.74	33.42	61.18
	36.80	8.72	33.49	60.98
MF treated	8.12	1.05	10.94	12.08
	17.90	4.45	18.46	40.54
	30.14	8.14	25.85	58.32
	37.22	8.11	25.86	58.18
UF treated	7.82	0.83	5.21	10.51
	18.24	4.31	14.23	38.92
	29.81	7.56	23.51	53.80
	37.01	7.47	23.57	53.64
SEd ( $\pm$ ) for,				
Chemical (C)		0.03765	0.53721	0.01393
WPG (G)		0.92314	1.32152	3.25617
C $\times$ G		1.00278	1.73251	3.91342
CD (5%) for				
Chemical (C)		0.07982	1.13889	0.02953
WPG (G)		1.95706	2.80162	6.90308
C $\times$ G		2.12589	3.67292	8.29645

at these levels of WPG, thereby facilitating easy penetration of chemicals into the woody substrate. This was due to the fact that once the cell wall of the treated samples bulked fully with chemicals no further expansion or contraction occurred in contact with moisture [3]. The highest values of MEE of the treated wood at 29.73, 30.10 and 29.81 levels of WPG indicate the hydrophobicity of cell wall that ultimately induced dimensional stability [8, 9]. The chemicals loaded onto cell wall beyond these levels of WPG might be present in the lumens of the samples without taking part in reaction with cell wall components. As a result of this, ASE, BC and MEE remained more or less unaffected above these levels of WPG (Table 2).

### IR studies

The IR spectra of untreated and treated samples showed the reaction of chemicals with OH groups of the cell wall components (Fig. 1). The IR spectra of untreated specimens (Fig. 1a) showed a peak near  $3432\text{ cm}^{-1}$ , which was due to the well-known hydrogen bond of  $-\text{OH}$  group vibration. The peaks at  $2889\text{ cm}^{-1}$  and  $1033\text{ cm}^{-1}$  were due to  $-\text{C}-\text{H}$  and  $-\text{C}-\text{O}-\text{C}-$ stretching vibrations, respectively. Again, the peak at  $1634\text{ cm}^{-1}$  indicated the well-known stretching mode of vibration of the benzene ring, while the peak at  $1456\text{ cm}^{-1}$  showed  $-\text{CH}_2$  group deformation vibration of lignin. In Fig. 1b, the peak at  $1634\text{ cm}^{-1}$  was due to benzene ring stretching vibration in lignin and that at  $1511\text{ cm}^{-1}$  was due to benzene



**Figure 1.** IR spectra of (a) untreated sample and sample treated with (b) PF, (c) MF and (d) UF at 29.73, 30.10 and 29.81 levels of WPG respectively.

ring stretching vibration of PF resin [20]. In Fig. 1c, the peak at  $3393\text{ cm}^{-1}$  was due to  $\text{—N—H}$  stretching mode of vibration of MF resin. The sharp peak at  $1563\text{ cm}^{-1}$  was due to bending and the one at  $815\text{ cm}^{-1}$  was due to wagging mode of vibration of  $\text{—N—H}$  group of MF resin. The UF resin treated sample showed a peak at  $1736\text{ cm}^{-1}$ , which was due to stretching vibration of  $\text{C=O}$  group of UF resin (Fig. 1d).

### Thermal analyses

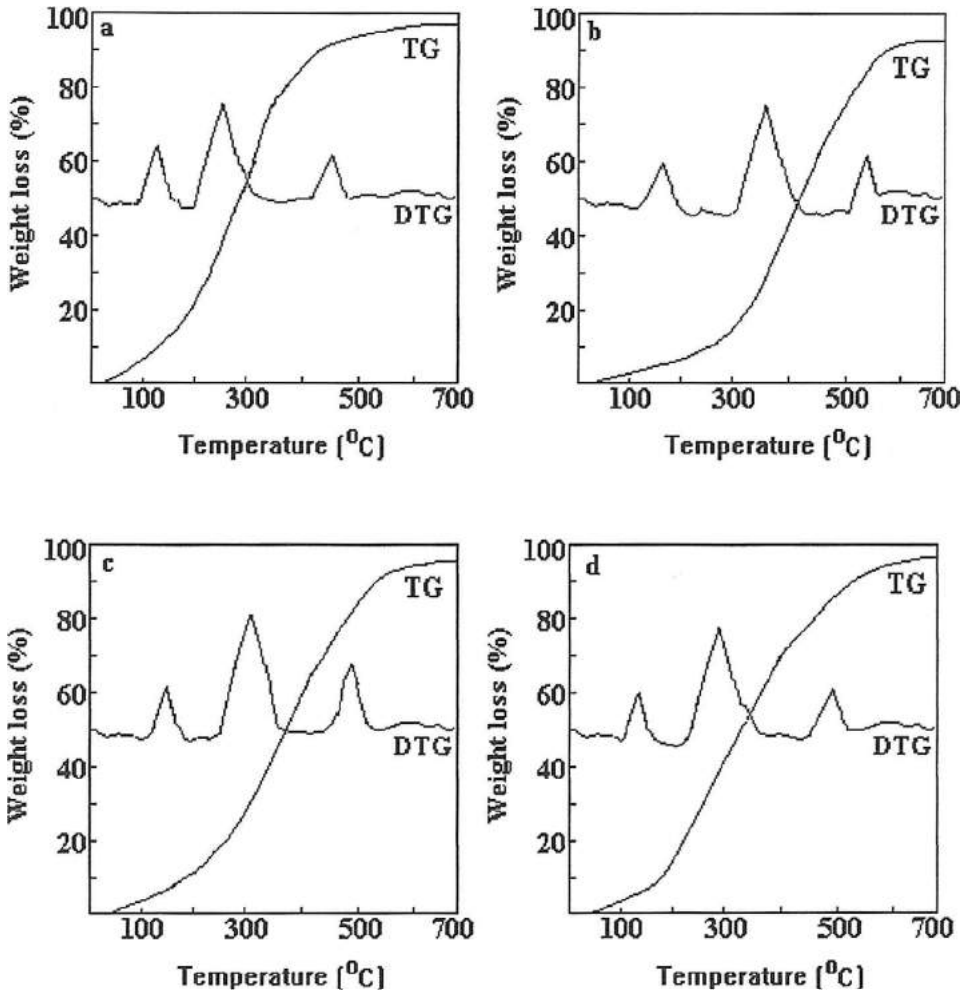
The thermal analyses data of treated and untreated samples are summarized in Table 3, which showed significant difference in weight loss and active decomposition temperature between treated and untreated samples. The data from all TG curves (Figs 2 and 3) showed an initial small weight loss up to  $100^\circ\text{C}$ , which was due mainly to evaporation of water from the samples. The major weight losses started from  $120^\circ\text{C}$  for untreated,  $163^\circ\text{C}$  for PF,  $143^\circ\text{C}$  for MF and  $132^\circ\text{C}$  for UF treated samples at heating rate  $20^\circ\text{C min}^{-1}$  and  $127^\circ\text{C}$  for untreated,  $171^\circ\text{C}$  for PF,  $150^\circ\text{C}$  for MF and  $137^\circ\text{C}$  for UF treated samples at heating rate  $30^\circ\text{C min}^{-1}$ . The active decomposition temperatures for the treated samples were found to be higher than that of untreated one (Table 3). The active decomposition temperatures that caused the major weight loss were  $258$  and  $263^\circ\text{C}$  for untreated,  $361$  and  $375^\circ\text{C}$  for PF,  $301$

**Table 3.** Weight loss and active decomposition temperatures of untreated and treated bamboo at heating rate 20°C and 30°C min<sup>-1</sup>

Samples	20°C min <sup>-1</sup>						30°C min <sup>-1</sup>					
	Weight loss (%)			Active decomposition temperature (°C)			Weight loss (%)			Active decomposition temperature (°C)		
	I	II	III	I	II	III	I	II	III	I	II	III
Untreated	14.12	63.51	20.38	120	258	469	14.02	63.55	20.38	127	263	476
	(30–153)	(153–359)	(359–650)	(endo)	(endo)	(endo)	(30–159)	(159–365)	(365–650)	(endo)	(endo)	(endo)
PF treated	10.53	52.31	30.16	163	361	523	10.49	52.21	30.30	171	375	529
	(30–201)	(201–453)	(453–650)	(endo)	(endo)	(endo)	(30–210)	(210–465)	(465–650)	(endo)	(endo)	(endo)
MF treated	11.63	57.55	25.82	143	301	497	11.51	57.49	25.97	150	313	501
	(30–192)	(192–428)	(428–650)	(endo)	(endo)	(endo)	(30–202)	(202–433)	(433–650)	(endo)	(endo)	(endo)
UF treated	12.31	58.91	24.28	132	295	488	12.26	58.81	24.48	137	302	491
	(30–178)	(178–401)	(401–650)	(endo)	(endo)	(endo)	(30–189)	(189–407)	(407–650)	(endo)	(endo)	(endo)
SEd (±)	0.0512	0.0295	0.0584	2.1153	3.1221	2.0415	0.0435	0.0359	0.0219	2.0912	3.1162	2.9257
CD (5%)	0.1091	0.0628	0.1244	4.5056	6.6501	4.3484	0.0927	0.0765	0.0466	4.4543	6.6375	6.2317

I, II and III — Pre, second and third stage, respectively; temperature ranges (in °C) are in parentheses; endo = endothermic reaction.





**Figure 2.** TG and DTG curves at heating rate  $20^{\circ}\text{C min}^{-1}$  of (a) untreated sample and treated sample with (b) PF, (c) MF and (d) UF at 29.73, 30.10 and 29.81 levels of WPG respectively.

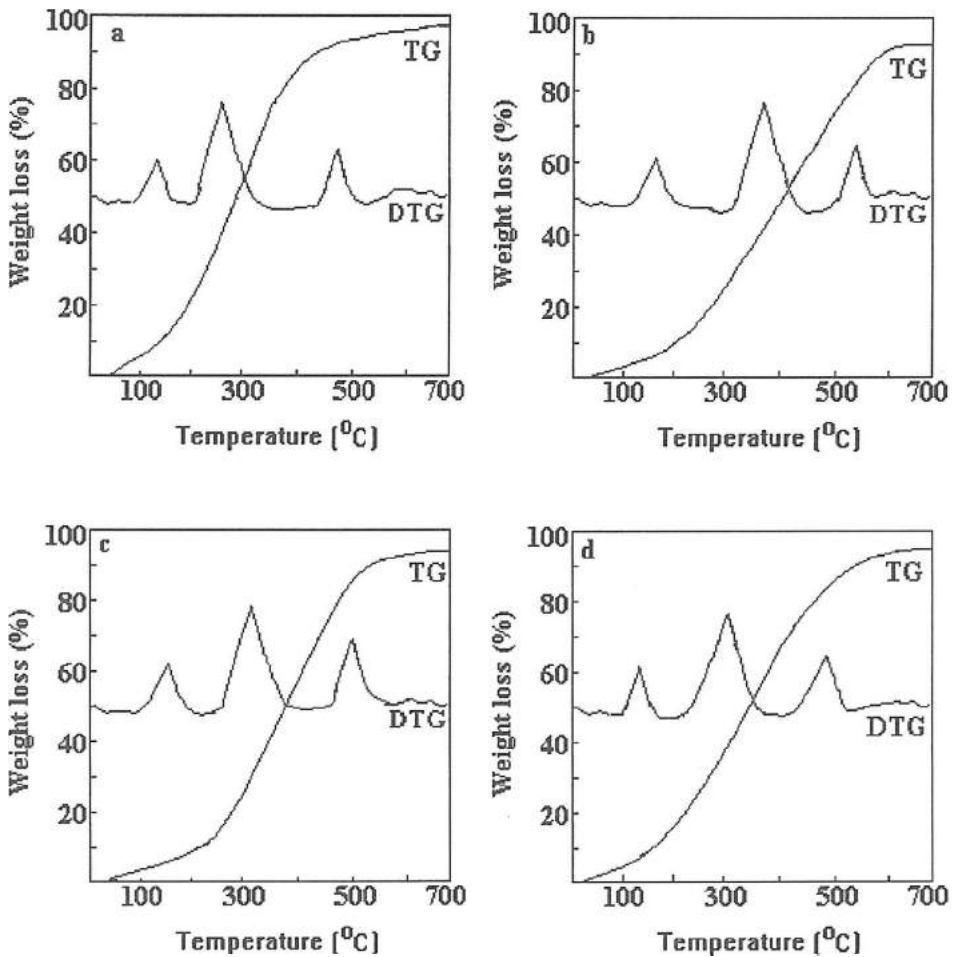
and  $313^{\circ}\text{C}$  for MF and  $295$  and  $302^{\circ}\text{C}$  for UF treated samples at heating rates  $20^{\circ}\text{C min}^{-1}$  and  $30^{\circ}\text{C min}^{-1}$  respectively (Table 3).

### Kinetic parameters

The kinetic parameters were evaluated by using the Coats and Redfern method [21]. The general correlation equation used in the Coats and Redfern method is,

$$\log_{10} \left[ 1 - (1 - \alpha)^{1-n} / T^2 (1 - n) \right] = \log_{10} [AR/aE(1 - 2RT/E)] - E/2.3RT,$$

where  $\alpha$  is the fractional conversion at temperature  $T$ ;  $n$  is the order of reaction;  $A$  is the frequency factor (in  $\text{S}^{-1}$ );  $a$  is the heating rate (in  $\text{K min}^{-1}$ );  $R$  is the gas constant (in  $\text{kJ mol}^{-1} \text{K}^{-1}$ );  $T$  is the temperature (in K) and  $E$  is the activation



**Figure 3.** TG and DTG curves at heating rate  $30^{\circ}\text{C min}^{-1}$  of (a) untreated sample and treated sample with (b) PF, (c) MF and (d) UF at 29.73, 30.10 and 29.81 levels of WPG respectively.

energy (in  $\text{kJ mol}^{-1}$ ). A computer program in FORTRAN 77 was used for the linear least square analysis with Gauss-Jordan subroutine and applied to evaluate  $n$ ,  $\Delta E$ ,  $A$  and standard error deviation (SED).

The values of activation energy ( $E_a$ ) and frequency factor ( $A$ ) are shown in Table 4. It was apparent from the data that the resin-treated samples showed higher activation energy and frequency factor than the untreated sample. The PF-treated sample was found to be thermally more stable than the others.

#### *Evaluation of physical properties*

The resin treatments caused significant increase in MOR and MOE of the samples with no remarkable effect on specific gravity. The highest values of MOR (57.8, 55.7 and  $53.8 \text{ N/mm}^2$ ) and MOE (8940, 8860 and  $8850 \text{ N/mm}^2$ ) of the treated

**Table 4.**

Dynamic thermogravimetry data and kinetic parameters of treated and untreated bamboo

Samples	$E_a$ (K cal mol <sup>-1</sup> )		$A$ (S <sup>-1</sup> )		SEd (%)	
	20°C min <sup>-1</sup>	30°C min <sup>-1</sup>	20°C min <sup>-1</sup>	30°C min <sup>-1</sup>	20°C min <sup>-1</sup>	30°C min <sup>-1</sup>
Untreated	26.251	27.312	43.921	44.012	0.00017	0.00031
PF treated	33.152	33.952	50.025	50.317	0.00021	0.00027
MF treated	29.319	29.713	48.325	48.717	0.00039	0.00072
UF treated	29.021	29.510	47.925	48.102	0.00018	0.00029
SEd (±)	0.9125	0.0751	0.0712	0.0715	ND	ND
CD (5%)	1.9801	0.1630	0.1545	0.1552	ND	ND

ND, Not determined.

**Table 5.**

Effect of resin treatment on strength (MOR) and stiffness (MOE) of bamboo at different levels of WPG

Reagents used	WPG	Specific gravity (g cm <sup>-3</sup> )	MOR (N/mm <sup>2</sup> )	MOE (N/mm <sup>2</sup> )
Untreated (control)	0	0.538	48.7	8270
PF treated	7.31	0.541	55.5	8530
	18.42	0.537	56.2	8780
	29.73	0.540	57.8	8940
	36.80	0.683	57.8	8950
MF treated	8.12	0.539	53.0	8500
	17.90	0.537	53.7	8730
	30.14	0.539	55.7	8860
	37.22	0.691	55.7	8860
UF treated	7.82	0.539	52.0	8520
	18.24	0.538	52.6	8730
	29.81	0.539	53.8	8850
	37.01	0.710	53.8	8850
SEd (±) for, Chemical (C)	ND	0.1492	0.4517	0.3125
WPG (G)	ND	0.1083	0.0053	0.0042
C × G	ND	0.3209	0.8723	0.0512
CD (5%) for Chemical (C)	ND	NS	0.9345	0.6459
WPG (G)	ND	NS	0.0121	0.0062
C × G	ND	NS	1.8052	1.0351

ND, Not determined; NS, Not significant.

samples were achieved at 29.73, 30.10 and 29.81 levels of WPG for PF, MF and UF, respectively, against the MOR (48.7) and MOE (8270) of the untreated samples. At these levels of WPG, the treated samples also possessed negligible reduction of MOR (1.30% for PF, 1.90% for MF and 2.20% for UF against 17.69% for untreated sample) and MOE (0.81% for PF, 1.11% for MF and 1.32% for UF against 21.15% for untreated sample) even after twelve months' exposure to natural environmental

**Table 6.**

Effect of termite attack on physical properties of treated and untreated bamboo at different levels of WPG during twelve months' exposure

Reagents used	WPG	MOR (N/mm <sup>2</sup> )	MOE (N/mm <sup>2</sup> )
Untreated (control)	0	40.1	6520
PF treated	7.31	53.2	8360
	18.42	54.8	8640
	29.73	57.0	8870
	36.80	56.9	8860
MF treated	8.12	50.3	8330
	17.90	52.0	8590
	30.14	54.6	8770
	37.22	54.6	8770
UF treated	7.82	49.1	8290
	18.24	50.8	8550
	29.81	52.6	8730
	37.01	52.5	8730
SEd (±) for,			
Chemical (C)	ND	0.5129	0.4121
WPG (G)	ND	0.0065	0.0027
C × G	ND	0.8812	0.5002
CD (5%) for			
Chemical (C)	ND	1.0873	0.8737
WPG (G)	ND	0.0138	0.0057
C × G	ND	1.8681	1.1060

ND, Not determined.

conditions in a termite colony (Tables 5 and 6). It was also found that the treatments did not cause appreciable change in specific gravity of the samples, though there was considerable increase of WPG. This might be due to the fact that the polymer bulked the samples to normal swollen volume [8, 9], which was retained after curing. However, the reduction of specific gravity of the treated samples above 29.73–30.14 levels of WPG might be due to removal of resins deposited in the lumens.

## CONCLUSION

From the present study, it could be concluded that the bamboo when treated with thermosetting resins exhibited higher dimensional stability, strength and stiffness properties in comparison to untreated bamboo. The treated bamboo also showed termite resistant capacity as evident from the negligible change that took place in strength properties after twelve months' exposure to termite colony. The treated samples also showed high thermal stability.

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