

Effect of Chemical Modifications on the Thermal Stability and Degradation of Banana Fiber and Banana Fiber-Reinforced Phenol Formaldehyde Composites

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ABSTRACT: Banana fiber has been modified by treatments with sodium hydroxide, silanes, cyanoethylation, heat treatment, and latex treatment and the thermal degradation behavior of the fiber was analyzed by thermogravimetry and derivative thermogravimetry analysis. Both treated and untreated fibers showed two-stage decomposition. All the treatments were found to increase the thermal stability of the fiber due to the physical and chemical changes induced by the treatments. The thermal degradation of treated and untreated banana fiber-reinforced phenol formaldehyde composites has also been analyzed. It was found that the thermal stability of the composites was much higher than that of fibers but they are less stable compared to neat PF resin matrix. Composite samples

were found to have four-stage degradation. The NaOH treated fiber-reinforced composites have very good fiber/matrix adhesion and hence improvement in thermal stability is observed. Though both silane treatments increased the thermal stability of the composite the vinyl silane is found to be more effective. Heat treatment improves the crystallinity of the fiber and decreases the moisture content, hence an improved thermal stability. The latex treatment and cyanoethylation make the fiber surface hydrophobic, here also the composite is thermally more stable than untreated one. © 2008 Wiley Periodicals, Inc. *J Appl Polym Sci* 110: 2305–2314, 2008

Key words: biofibers; composites; thermal stability

INTRODUCTION

In recent years, there has been fast growth in using renewable natural fibers as reinforcement in polymer composite materials.^{1–3} The advantages of natural fibers such as jute, sisal, banana, flax, oil palm, etc., arise from their renewability, low cost, easy availability, and high specific strength and stiffness. When compared with synthetic fibers like glass, carbon, etc., there is a minimum health hazard in natural fibers.

To improve the interfacial bonding with polymer matrices and to decrease the moisture absorption and environmental aging of the natural fiber-reinforced composites, several surface modifications were performed. These include alkali treatment, acetylation, cyanoethylation, grafting, silane treatment, and so forth.^{4–6}

Thermal analysis of natural fiber composites is very important as these materials are increasingly

used in several aggressive environments. So, it is important to get an idea about different stages of thermal degradation in the composites. Several authors have studied the thermal degradation behavior of natural fibers.^{7–9} Ray et al.¹⁰ studied the thermal behavior of jute fibers. They found that the changes occurring in the fibers as a result of alkali treatment like dissolution of hemicellulose, splitting of fibers to filaments, increase in crystallinity, etc., influence the thermal degradation of fibers. Xiao et al.¹¹ analyzed the thermal characteristics of alkali soluble lignins, hemicelluloses, and cellulose from maize stems and rice straw. They showed that hemicelluloses degraded at first place, while lignin showed less degradation and therefore its structure was more stable. Cellulose showed an important degradation process, mainly between 250 and 330°C and its thermal stability is lower than lignin, but higher than that of hemicelluloses.

Thermogravimetric properties of PALF-LDPE composites have been studied with special reference to fiber loading and fiber treatment.¹² It was found that PALF degraded before the polyethylene matrix and the thermal stability of the composite is increased by the better fiber matrix adhesion after chemical treatments with isocyanate, peroxide, and silane treatments. Espert et al.¹³ showed that addition of cellulose improved the thermomechanical behavior of PP

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and use of PP containing a small quantity of ethyl vinyl acetate, resulted in more desirable thermal and thermo oxidative stabilities. Gassan and Bledzki¹⁴ analyzed the thermal degradation of flax and jute fibers. It was found that low temperature (<170) only slightly affects fiber properties, while temperatures above 170°C, significantly dropped tenacity and degree of polymerization.

Marcvinch et al.¹⁵ studied the thermal degradation of wood flour filled polypropylene composites. Their studies indicated a thermal interaction between fillers and matrix evidenced by the shifting of the maximum degradation peaks of the wood flour towards higher temperatures in presence of polymer matrix and the peak attributed to the matrix towards lower temperatures due to the presence of wood flour in the composite.

Thermal study of jute based polyester composites¹⁶ showed that initial thermal degradation temperature is shifted to a higher temperature while final decomposition temperature remain unaltered due to cyanoethylation of the fiber. Albano et al.¹⁷ studied the effect of acetylation of sisal fibers on the thermal degradation of blends of PP and polyolefenes. The results showed that acetylated sisal fiber exhibits a higher activation energy compared to untreated sisal fiber. They showed that thermal stability as well as thermal degradation mechanism was changed by acetylation.

Qui et al.¹⁸ studied the effect of maleation and cellulose content on the characteristics of composites of high crystalline cellulose with polypropylene. They found that both cellulose and MAPP facilitate the thermooxidative stability of PP composites in the order, MAPP/cellulose > PP/APP/cellulose > PP cellulose > PP indicating that MAPP act as an efficient compatibilizer in the system. Zhou et al.¹⁹ showed that the thermal stability of PP/sisal fiber composite is improved by grafting the sisal fiber surface with PMMA. They explained that it would be because grafting of the PMMA molecules enhances the intermolecular interaction between PP and sisal fiber.

Banana fiber, which is rich in cellulose, relatively inexpensive and abundant in India, is found to be potential reinforcement in polymer matrices.^{20,21} Several chemical modifications were performed on the banana fiber and the mechanical,²² dynamic mechanical,²³ and water sorption behavior²⁴ of these fiber-reinforced composites were analyzed earlier. In the present work, the banana fiber surface have been modified by NaOH treatment, silane treatments, cyanoethylation, heat treatment, and latex treatment and the effect of these modifications on the thermal stability of fiber was analyzed. Thermal stability of phenol formaldehyde resin and the banana fiber-reinforced phenol formaldehyde composites have also been studied. The effects of surface modifications on

the thermal degradation behavior and thermal stability of the composites were also analyzed.

EXPERIMENTAL

Materials

Banana fiber obtained from Sheeba fibers and handicrafts, Poovancode, Tamilnadu was used for the study. Phenol formaldehyde resole type resin obtained from M/S West Coast Polymers Pvt., Kanner, Kerala was used as matrix. The basic characteristics of phenol formaldehyde resin are given in Table I.

Fiber modifications

NaOH treatment

Fibers were immersed in 4% NaOH solution for 2 h at room temperature. The fiber was washed many times with distilled water and finally washed with water containing little acid and dried in an air oven.

Silane treatments

The NaOH pretreated fibers were dipped in 0.6% silane solution in alcohol water mixture (60 : 40) for 1 h at room temperature. Triethoxy vinyl silane and amino propyl silane were used as coupling agents. The pH of the solution was maintained between 3.5 and 4. The fibers were washed in distilled water and dried. The reaction between silanol of silane and hydroxyl group of cellulose is evidenced by absorptions related to Si—O and Si—O—Si observed at 1285, 1075, and 1018 cm^{-1} , respectively. Physical and chemical changes induced by the chemical treatments on banana fiber have been reported earlier.²⁵

Heat treatment

Banana fiber was heated at 150°C in an air-circulating oven for 4 h. The fiber was then cooled to room temperature.

Cyanoethylation

Banana fiber was immersed in a 4% solution of NaOH saturated with sodium thiocyanate for 30 min. Subsequently, the fiber was pressed to drain of the excess water and removed quickly into round-bottomed flask allowing a reaction with acrylonitrile

TABLE I
Characteristics of Phenol Formaldehyde Resole

Appearance	Deep brown color
Viscosity (CPS)	18–22
Water tolerance	1 : 18
Solid content %	50

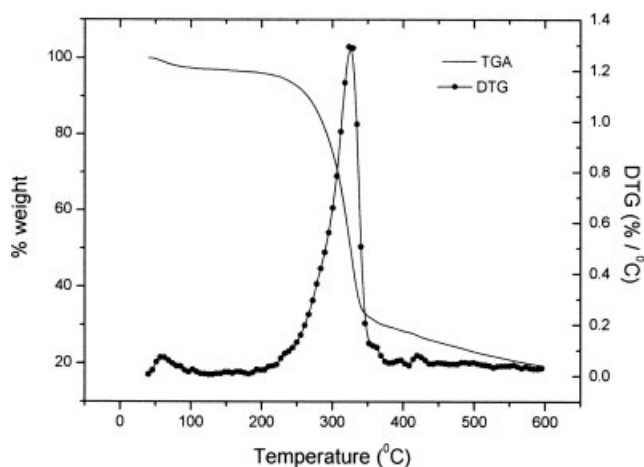


Figure 1 Thermogravimetric (TG) and the derivative thermogravimetric (DTG) curves of untreated banana fiber.

(banana fiber to AN ratio 1 : 3) for 1 h at 40–45°C. After the reaction, a solution of acetic acid was used to neutralize the alkali catalyst. The product was well washed with water and alcohol, respectively. The chemical structure of fiber was changed by cyanoethylation.²⁵ In cyanoethylated fiber, a strong absorption was observed at 2240 cm^{-1} due to nitrile stretching. Vibrations of amino and carboxyl groups are also perceivable as a result of hydrolysis of nitrile groups.

Latex treatment

The NaOH pretreated fibers were dipped in natural rubber latex having 10% dry rubber content for 1 h at room temperature. It was then taken out and dried in an air oven. This treatment gives a latex coating on the fiber surface.

Preparation of composites

Prepreg route was followed for the preparation of composites. Hand lay-up method followed by compression molding was adopted for composite fabrication. Randomly oriented fiber mats of uniform thickness were prepared from chopped banana fibers (30 mm). The fiber mats were dried and impregnated in PF resin and the prepreg was kept at room temperature up to semicured stage. It was then pressed at 100°C in a mold measuring 150 mm \times 150 mm \times 3 mm to get a three-dimensional crosslinked network. Volume fraction of the fiber in all the treated and untreated fiber/composites was 30%.

RESULTS AND DISCUSSIONS

Degradation of fibers

The chemical constituents of lignocellulosic banana fiber are cellulose (63–64%), hemicellulose (19%),

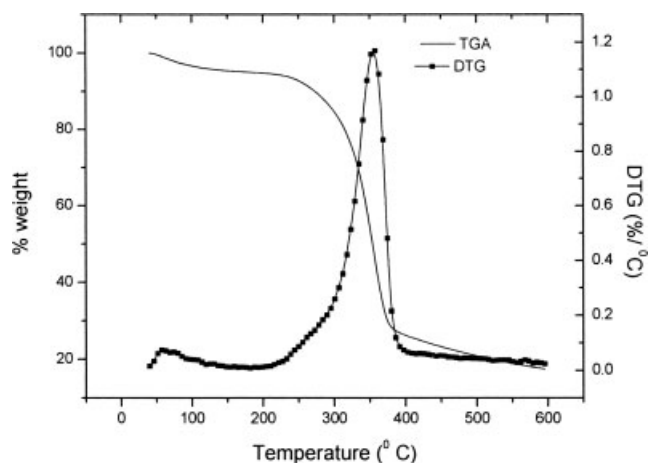


Figure 2 Thermogravimetric (TG) and the derivative thermogravimetric (DTG) curves of alkali treated banana fiber.

and lignin (5%). The moisture content of banana fiber is 10–11%.²⁶ Marcovitch et al.¹⁵ analyzed the thermal degradation behavior of cellulose and lignin, the major constituents of the fibers. They found that lignin specifically low molecular weight protolignin decomposes at slower rate than other constituents. The weight loss below 300°C was negligible for cellulose, above that temperature the cellulose begin to degrade fast and at 400°C, only residual char was obtained. This was explained due to the loss of hydroxyl groups and depolymerization of cellulose to anhydroglucose units.

Thermogravimetric (percentage of weight loss as a function of temperature) (TG) and the derivative thermogravimetric (DTG) curves of untreated and treated banana fiber are given in Figures 1–7. The step analysis of treated and untreated banana fiber scan from above figures is given in Table II. Step analysis of untreated fiber shows only a small degra-

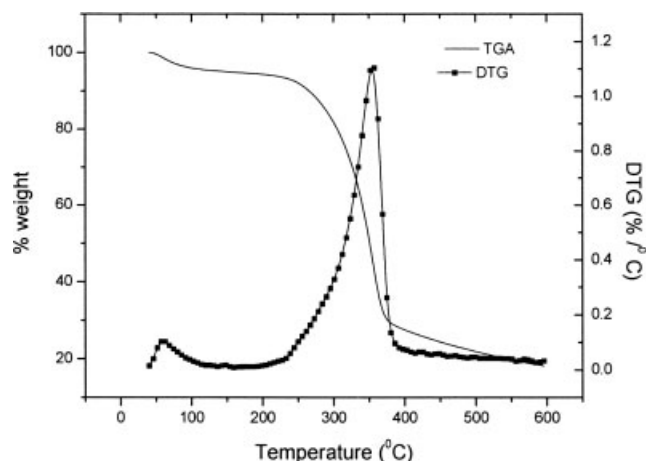


Figure 3 Thermogravimetric (TG) and the derivative thermogravimetric (DTG) curves of vinyl silane treated banana fiber.

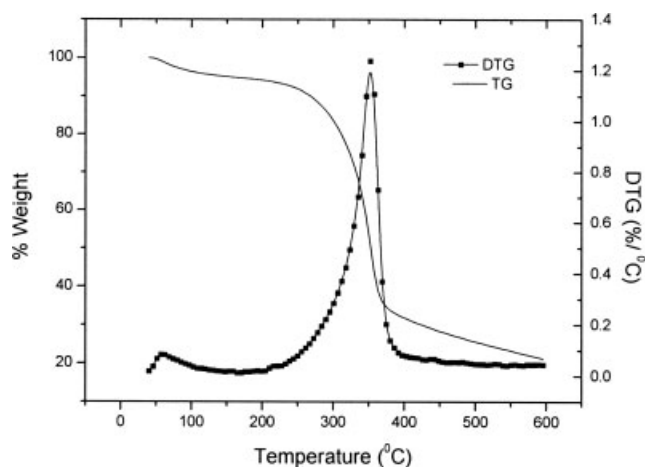


Figure 4 Thermogravimetric (TG) and the derivative thermogravimetric (DTG) curves of aminosilane treated banana fiber.

dation below 100°C. This weight loss below 100°C was the result of evaporation of moisture. Further degradation of the fiber appears to be a single step process. The peak temperatures of untreated and treated fibers obtained from DTG curves are given in Table III. In untreated fiber, the peak corresponding to moisture loss appears at 57°C with 4% degradation. The main degradation peak in untreated fiber occurs between 230 and 350°C, where about 70% of the degradation occurs. Above the main degradation stage, all the volatile materials are driven off from the sample resulting in the residual char.

In alkali treated fiber, the moisture loss peak is not found to have any considerable change compared to the untreated one, but the weight loss at 100°C is found to be slightly higher in the former. In the alkali treated fiber, the removal of alkali soluble hemicellulose results in a fibrillated structure as is clear from scanning electron micrograph (Fig. 8). So,

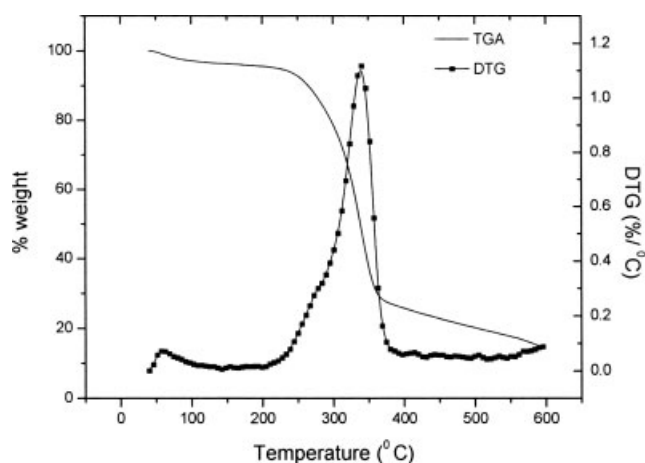


Figure 5 Thermogravimetric (TG) and the derivative thermogravimetric (DTG) curves of heat treated banana fiber.

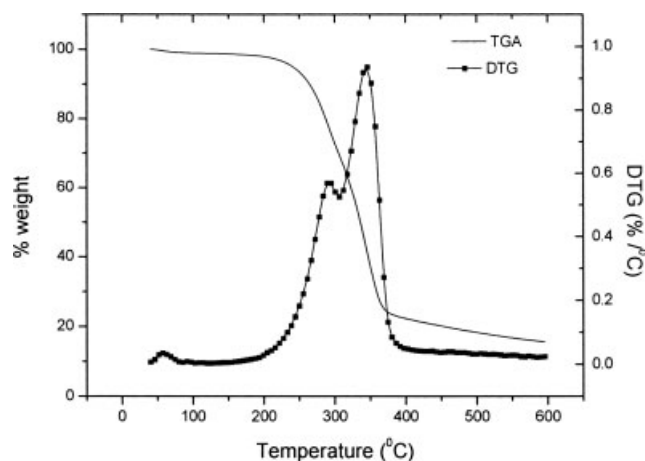


Figure 6 Thermogravimetric (TG) and the derivative thermogravimetric (DTG) curves of cyanoethylated banana fiber.

the moisture loss is easier and the weight loss is higher below 100°C, compared to untreated one. The main degradation peak temperature is shifted to higher temperatures. About 32°C increase in the degradation peak temperature and higher percentage of residue indicate better thermal stability of alkali treated fiber. Saha et al.²⁷ reported that alkali treatment reduced hemicellulose in the fiber thereby making the product thermally more stable. Ray et al.¹⁰ also reported increased thermal stability of alkali treated jute fibers.

Silane treatments both vinylsilane and aminosilane do not affect the moisture loss considerably. But the moisture loss peak is at a higher temperature (59°C) in vinyl silane compared to aminosilane (57°C). But weight loss at 100°C is higher in vinyl silane. This is because in aminosilane treated fiber, there can have H-bonding interaction with NH₂ group of amino silane treated fiber with water molecules, by which

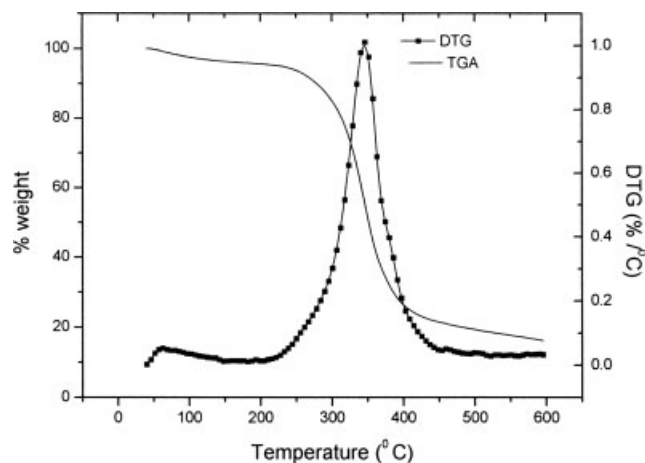


Figure 7 Thermogravimetric (TG) and the derivative thermogravimetric (DTG) curves of latex treated banana fiber.

TABLE II
The Percentage Weight Loss of Treated and Untreated Banana Fibers at Different Temperatures

Sample	100°C	200°C	300°C	400°C	500°C	600°C
Untreated	4	6	27	72.7	79.9	87
Mercerized	3.9	5.2	14.4	73.8	78.7	83.6
Vinyl silane treated	3.9	5.2	18.2	72.2	78.5	82.3
Amino silane treated	3.7	5.6	16.3	68.4	74.1	79.2
Heat treated	2.9	4.2	20.9	73.9	74	85.9
Cyanoethylated	0.2	1.9	27.9	77.2	81.7	84.6
Latex treated	1.1	4.6	15.1	73.9	80.2	83.3

the removal of water molecules is more difficult and occurs at higher temperatures compared to vinyl silane treated fiber. The main degradation peak in aminosilane also occurs at a lower temperature compared to vinyl silane treated fiber. The percentage residue is found to be higher for both the silane treated fibers compared to untreated one showing improved thermal stability of silane treated fibers.

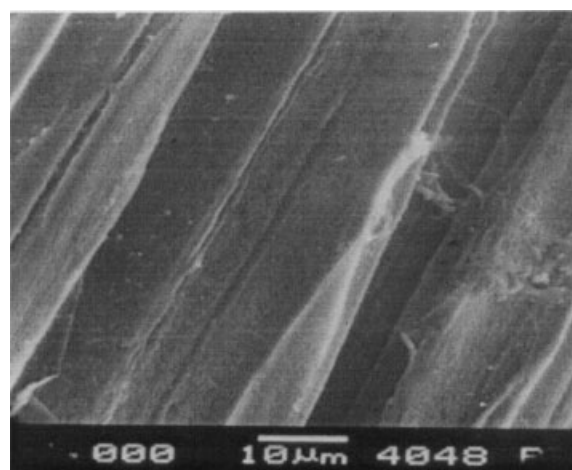
In heat-treated fiber, the weight loss at temperature range 100–500°C is found to be less than that of untreated fiber. The degradation peak is shifted to a higher temperature by 14°C indicating better thermal stability of heat-treated fiber. The improved thermal stability after heat treatment is associated with increase in crystallinity of the fiber by the treatment. Gassan and Bledzki¹⁴ have reported that thermal exposition of natural fibers leads to higher crystallinity. They explained it from the increase in size of preexisting crystallites by for example, the realignment of cellulose chain or crystalline surface or at their ends. It is also possible that crystallization occurs by the formation of completely new crystallites within the amorphous area. Rong et al.²⁸ also reported increase in crystallinity of sisal fiber by heat treatment evidenced by crystallographic studies.

Cyanoethylated fiber is also found to have better thermal stability compared to untreated one. In cyanoethylated fiber, a new peak occurs at 300°C. This is associated with the degradation of cyanoethyl group of the cyanoethylated fiber in the composite.

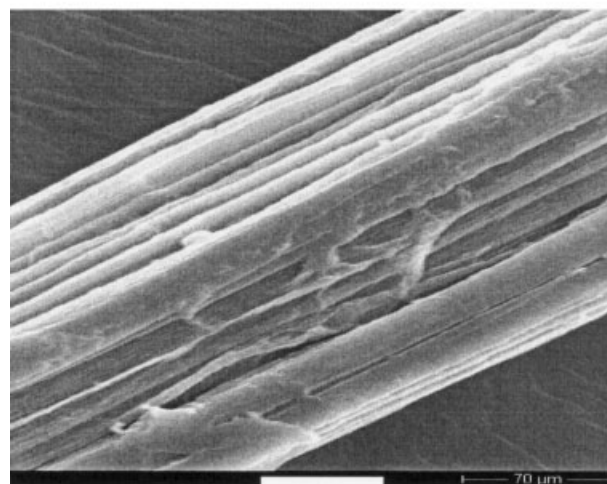
TABLE III
Peak Temperatures of Treated and Untreated Banana Fiber

Sample	Peak I (Temp., °C)	Peak II (Temp., °C)
Untreated	74	346
Mercerized	74	380
Vinyl silane treated	74	374
Amino silane treated	83	369
Heat treated	79	363
Latex treated	68	385
Cyanoethylated	53	296, 340

Similar results were obtained by Saha et al.¹⁶ They observed a peak between 299 and 310°C and believed it to be due to the degradation of the cyanoethyl group in the cyanoethylated fiber. The degradation peak temperature is shifted to a higher temperature. It is also clear from the Table I that the weight loss below 100°C associated with the moisture loss is negligibly small in cyanoethylated fiber. This is due to the hydrophobicity of the fiber surface after the treatment, which decreases the moisture absorption. Several authors have reported the increase in thermal stability of acrylonitrile grafted natural fibers. Mohanthy et al.²⁹ have reported that grafting of acrylonitrile on jute improved the thermal stability as evidenced by the increase in the degradation temperature from 170 to 280°C. Saba³⁰ has reported improved thermal stability for acrylonitrile grafted sisal fibers as evidenced by increased initial



(a)



(b)

Figure 8 SEM photograph of untreated and alkali treated banana fiber.

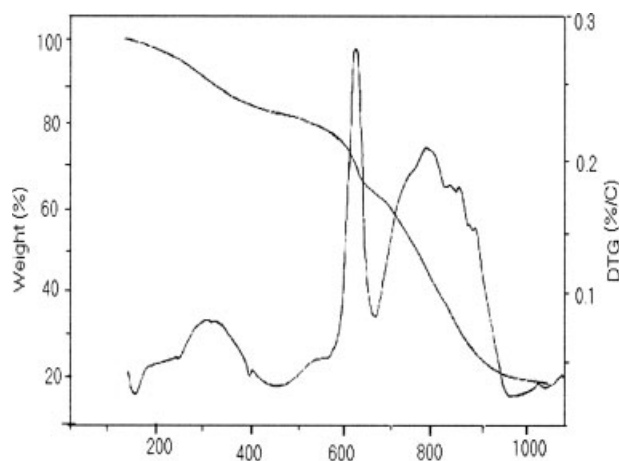


Figure 9 Thermogravimetric (TG) and the derivative thermogravimetric (DTG) curves of PF resin.

degradation temperature, lowering of rate of degradation, and total weight loss.

Latex treatment also improves the thermal stability of the fiber. The hydrophobic latex coating in the fiber reduces the moisture absorption and hence the moisture loss peak is shifted to a higher temperature. This is also clear from the percentage weight loss at 100°C which is very low compared to untreated fiber. The weight loss at 400 and 500°C are comparable to that of untreated fiber. But the percentage residue as well as the degradation peak temperature is higher in latex treated fibers indicating better thermal stability of the latex treated fiber.

Thermal degradation of composites

Figure 9 shows the TGA and DTG curves of cured phenol formaldehyde resin. The weight loss of neat PF is marginal up to 300°C. Decomposition rate becomes rapid at 490°C. The decomposition is completed at about 820°C. A total weight change of 91% occurs at a temperature of 970°C. The major decomposition of the resin occurs at 550°C. The initial weight loss is due to the escape of water and the degradation process originates from the initial bond breaking reaction. The decomposition of cured phenolic resin starts above 250°C.

In banana fiber-reinforced phenol formaldehyde composites, four degradation peaks are observed (Figs. 10–16). The first one below 100°C corresponds to the removal of moisture entrapped in the fiber as well as micro voids in the composites. The peaks at 305 and 410°C are associated with the degradation of cellulose fiber. In the case of fiber present in composites the part of fiber, which are not well impregnated by resin, can degrade very easily, while the interfacial region fully wetted by the resin degrades at a higher temperature. So, the initiation of thermal

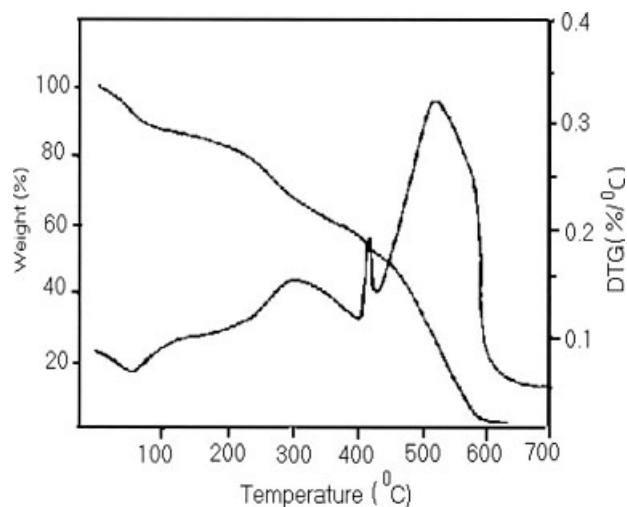


Figure 10 Thermogravimetric (TG) and the derivative thermogravimetric (DTG) curves of untreated banana fiber-reinforced PF composites.

degradation of phenol formaldehyde and the fiber portions, which are well impregnated by the resin, will be degrading at 410°C. The peak at 544°C is associated with the degradation of PF resin matrix.

The percentage weight loss at different temperatures of PF resin, untreated fiber composite, and treated fiber-reinforced composites are given in Table IV. The incorporation of fiber decreases the thermal stability of the resin revealed by the weight loss at all temperatures and lower yield of the residue. The peak temperatures obtained from DTG curves are given in Table V. Upon alkali treatment, the percentage weight loss is decreased and percentage residue is increased. Alkali treatment increases the thermal stability of the composites. The resin is well penetrated into the mercerized fiber thereby resulting in a strong fiber/matrix interface. This will increase the stability of the composite.

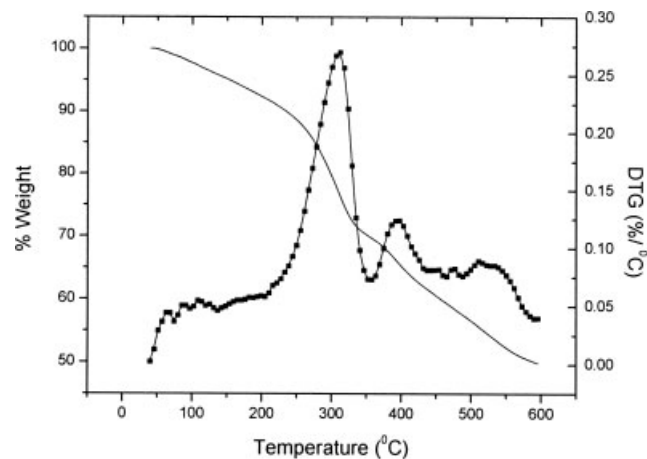


Figure 11 Thermogravimetric (TG) and the derivative thermogravimetric (DTG) curves of alkali treated banana fiber-reinforced PF composites.

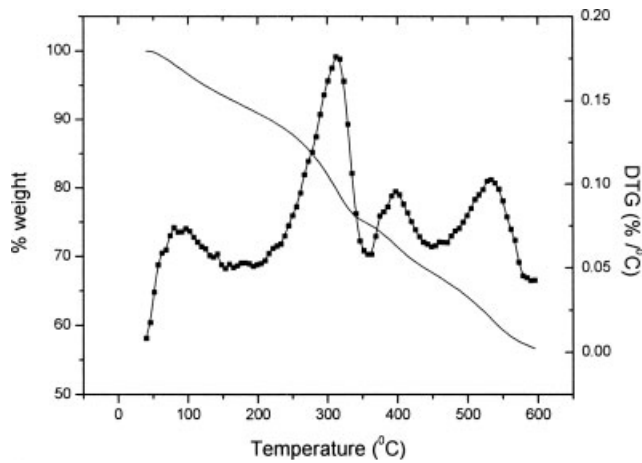


Figure 12 Thermogravimetric (TG) and the derivative thermogravimetric (DTG) curves of vinyl silane treated banana fiber-reinforced PF composites.

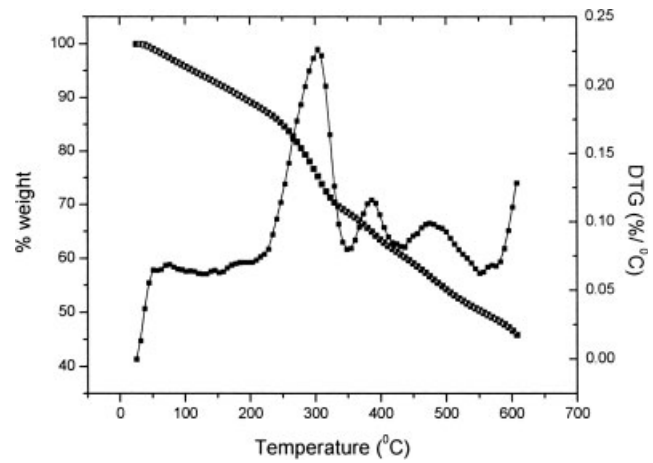
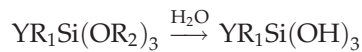


Figure 14 Thermogravimetric (TG) and the derivative thermogravimetric (DTG) curves of heat treated banana fiber-reinforced PF composites.

Treatments with silanes, both aminosilane and vinyl silane increase the thermal stability of the composite even higher than NaOH treatment. To explain the mechanism of action, silanes can be represented by $YR-Si(OR_2)_3$, where R is an aliphatic linkage that serves to attach the functional organic groups to silicon. The OR_2 is a hydrolysable alkoxy group. Traces of moisture in the fiber hydrolyze the silanes to form silanols.



The hydrolyzed silanol forms strong covalent bonds or H— bonds with OH group of cellulose. The individual coupling agent molecules attached to cellulose forms a continuous link. The long hydrophobic polymer chain of polymerized silane can adhere to the matrix mainly because of Vander walls

type attractive force. As a result, silane coupling agents forms a bridge at the interface. Thus the activated surface of cellulose is represented schematically below.³¹

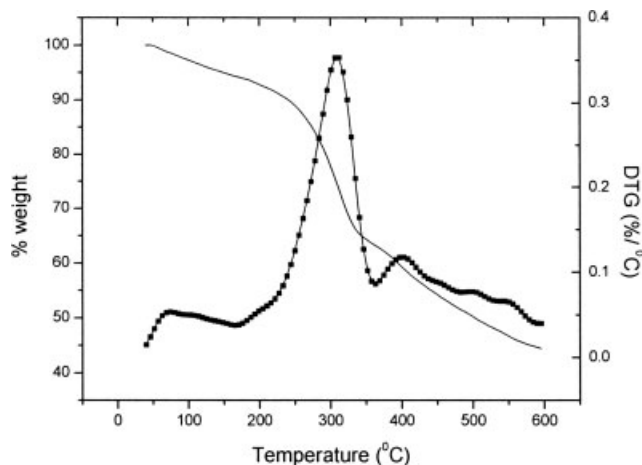
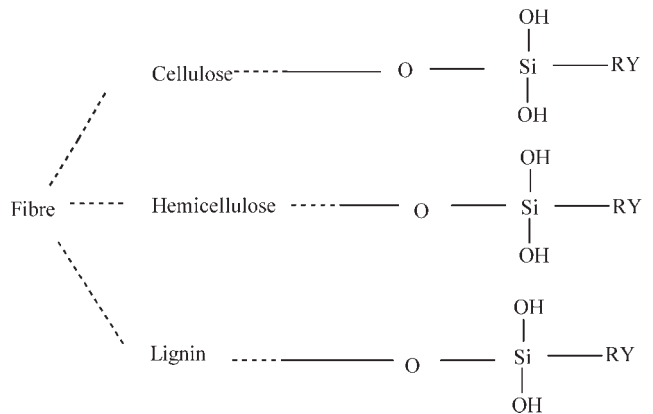


Figure 13 Thermogravimetric (TG) and the derivative thermogravimetric (DTG) curves of aminosilane treated banana fiber-reinforced PF composites.

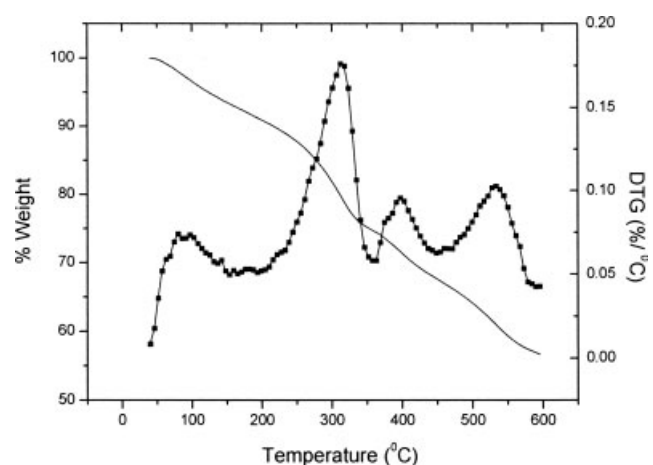


Figure 15 Thermogravimetric (TG) and the derivative thermogravimetric (DTG) curves of cyanoethylated banana fiber-reinforced PF composites.

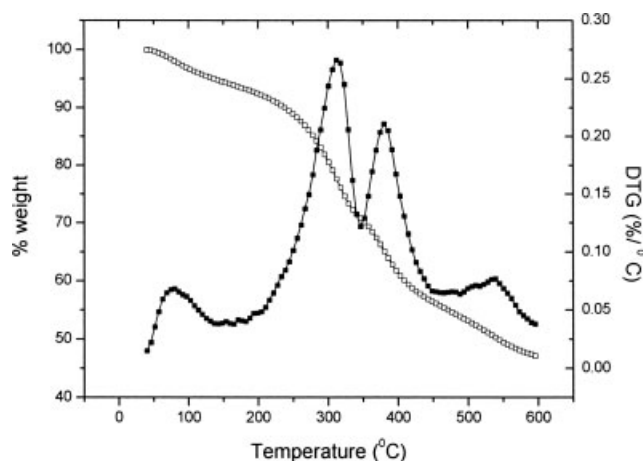
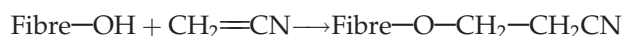


Figure 16 Thermogravimetric (TG) and the derivative thermogravimetric (DTG) curves of latex treated banana fiber-reinforced PF composites.

In aminosilane the Y unit is $-\text{NH}_2$ group, which can react with $-\text{OH}$ groups of other molecules of silanes or cellulose or OH groups of PF matrix. In vinyl (2-ethoxy methoxy) silane the Y group is the vinyl group. It is reported that the hydrophilic amino groups if present in silane coupling agent can react with resin matrix and make a strong interface. If the amino group does not take part in reaction, it will be responsible for the poor water resistance of the fiber/matrix bond.³² Though the percentage weight loss is higher in vinyl silane at lower temperatures (up to 200°C), the high temperature degradations occur at a higher rate in aminosilane treated fiber-reinforced composites with higher percentage of weight loss. The degradation peaks obtained from DTG curves also reveals this. This is due to the degradation of aminosilane by pyrolytic cleavage, which disturbs the fiber/matrix bonding and favors the composite degradation. The percentage residue is higher (by about 10%) in vinyl silane than aminosilane, indicating better thermal stability of vinyl silane treated fiber/PF composites compared to aminosilane treated one, though both of them are more stable than untreated fiber/PF composite. Heat treatment also increases the thermal stability of the

composites but to a lesser extent compared to other chemical modifications mentioned earlier. The removal of moisture and improved crystallinity as a result of heat pretreatment of the banana fiber improve the interfacial bonding in the composite as clear from the weight loss and peak temperatures.

Latex treatment and cyanoethylation decrease the hydrophilicity of the fiber. This will decrease the possible chemical interaction between the hydrophilic PF resin and OH groups of lignocellulosic fibers (Fig. 17) in the composite. Cyanoethylation results in reduction of hydroxyl groups and introduction of cyanoethyl groups by the following reaction.



The moisture loss peaks as well as degradation peaks are here also shifted to higher temperatures. The percentage weight loss is lower for both cyanoethylated and latex treated fiber/PF composites compared to untreated fiber/PF composite.

CONCLUSIONS

Effect of chemical treatments on the thermal degradation behavior of banana fiber was investigated with thermogravimetric analysis. Chemical treatments employed were sodium hydroxide treatment, silane treatments, heat treatment, cyanoethylation, and latex treatment. The DTG curves showed two stage decomposition for all the fibers first one below 100°C indicating the moisture loss and the second one between 320 and 360°C due to the major degradation of fiber. The studies indicated that all the treatments improved the thermal stability of the banana fiber indicated by lower weight loss at different temperatures, shifting of degradation peaks to higher temperatures and increase in percentage residue. The treatments with sodium hydroxide removed some hemicellulose and hence a decrease in moisture absorption was observed. The physical changes like fibrillation resulted an improved interlocking with matrix. Both vinyl silane and aminosilane treatments are found to be very effective in increasing

TABLE IV
The Percentage Weight Loss of PF Resin, Untreated Banana Fiber/PF Composite, and Treated Fiber/PF Composites at Different Temperatures

Sample	100°C	200°C	300°C	400°C	500°C	600°C
Neat PF	4	10	17	22	27	39
Untreated	8	13	33	36	49.9	72
Mercerized	1.8	7.7	20.4	34.6	43.3	50.1
Vinyl silane treated	3.6	8.8	18.1	28.7	35.8	42.3
Amino silane treated	2.1	7.1	22.2	40.7	49.7	56
Heat treated	4.1	10.9	23.9	36.4	45	52.9
Cyanoethylated	3.6	9.7	22.5	34.6	43.6	50.1
Latex treated	3.6	7.72	19.32	38.75	38.7	53

TABLE V
Peak Temperatures Obtained from the DTG Curves of PF Resin,
Untreated Banana Fiber/PF Composite, and Banana/PF Composites
After Different Surface Modifications

Sample	Peak I (Temp., °C)	Peak II (Temp., °C)	Peak III (Temp., °C)	Peak IV (Temp., °C)
PF alone	–	200	500	700
Untreated	46	305	402	510
Mercerized	84	309	392	511
Vinyl silane treated	82	311	397	535
Amino silane treated	60	312	394	502
Heat treated	63	306	388	482
Latex treated	74	314	379	535
Cyanoethylated	72	315	397	532

thermal stability of the fiber. Cyanoethylation and latex treatment make the fiber hydrophobic and hence the moisture content in the fiber is decreased and thermal stability increased. Heat treatment increased the crystallinity of the fiber, without much change in chemical structure of the fiber. Therefore the effect is less. Banana fiber-reinforced phenol formaldehyde composites are found to have much higher thermal stability compared to fiber, but lower than that of neat PF resin. NaOH treated fiber composites having improved fiber/matrix adhesion resulted in stable composites with better thermal stability than untreated fiber/PF composites. Silane treatments also improved the composite stability but vinyl silane treated fiber composites are found to be more

effective than aminosilane treated fiber. Cyanoethylated and latex treated fiber-reinforced composites are also found to have higher thermal stability, though the hydrophobicity of the fiber decreases the fiber matrix adhesion. This study reveals that composites having very good fiber/matrix adhesion showed improved thermal stability.

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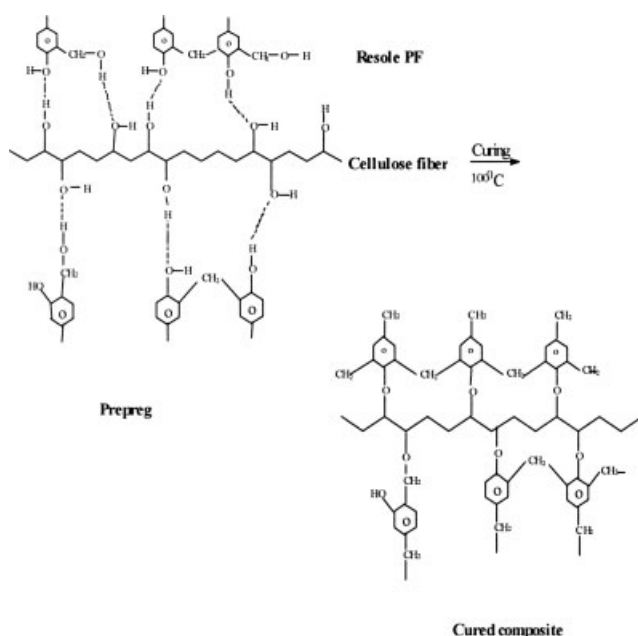


Figure 17 The possible chemical interaction between cellulose fiber and phenol formaldehyde resin matrix.

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