

## Characteristics of Banana Fibers and Banana Fiber Reinforced Phenol Formaldehyde Composites-Macroscale to Nanoscale

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**ABSTRACT:** Microfibers and nanofibers were prepared from macro banana fibers by a steam explosion process. The fiber surface of chemically modified and unmodified banana fibers was investigated by atomic force microscopy, the studies revealed a reduction in fiber diameter during steam explosion followed by acid treatments. Zeta potential measurements were carried out to measure the acidic property of the fiber surface; the surface acidity was found to be increased from macrofibers to nanofibers. The thermal behavior of macrofibers, microfibers, and nanofibers were compared. Substantial increase in thermal stability was observed from macroscale to nanoscale, which proved the high thermal stability of nanofibers to processing conditions of biocomposite preparation. The composition of the fibers before and after steam explosion and acid hydrolysis were also analyzed using FT-IR. It was found that the isolation of cellulose nanofibres occurs in the final step of the processing stage. Further macrocomposites, microcomposites, and nanocomposites were prepared and mechanical properties such as tensile, flexural and impact properties were measured and compared. The composites with small amount of nanofibers induces a significant increase in tensile strength (142%), flexural strength (280%), and impact strength (133%) of the phenol formaldehyde (PF) matrix, this increase is due to the interconnected web like structure of the nanofibers. © 2013 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* 130: 1239–1246, 2013

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### INTRODUCTION

Fiber reinforced thermosetting composites are highly beneficial because the reinforced materials improve the strength and toughness of the plastics.<sup>1–3</sup> Natural fibers which are rich in cellulose, the most abundant biopolymer, is sustainable, biodegradable, and have low density. Additionally these materials have low toxicity and abrasiveness. Natural fibers can find application in the production of aerospace and automotive parts, building applications etc. In our previous studies we have examined the effectiveness of various natural fibers like banana, pineapple, and so forth, from macro, to nanoscale as potential reinforcing agents in various polymeric matrices.<sup>4–9</sup> Nanofibrils separated from the natural fibers have also been used for processing nanocomposites so that the mechanical properties may be improved.<sup>10,11</sup> The reinforcement is regarded as a nano when at least one of the dimensions is lower than 100 nm. The nano-

composites assume extemporary and novel properties, unseen in conventional macrocomposites, when this particular feature is attained.<sup>12</sup> Gandini and Belgacem<sup>12,13</sup> illustrated that the use of cellulose nanocrystals as a reinforcing phase in nanocomposites and has numerous well known advantages.

Electrokinetic phenomena can be observed by contacting a solid surface with a polar liquid medium, because of the existence of an electrical double layer at the solid–liquid interface.<sup>14</sup> Bismarck et al.<sup>15</sup> reported on the characterization of modified jute fibers using zeta potential measurements. Pothen et al.<sup>16</sup> studied the influence of chemical treatments on the surface polarity of cellulose fibers. Bellmann et al.<sup>17</sup> investigated the electrokinetic properties of natural fibers and concluded that this is suitable to analyze the swelling characteristics of fibers. Very recently, the surface polarity of coconut cellulose fibers has been reported from our laboratory.<sup>18</sup>

**Table I.** Properties of Banana Fiber

Tensile strength (MPa)	Tensile modulus (GPa)	Elongation at break (%)	Diameter of the fiber ( $\mu\text{m}$ )	Density ( $\text{g}/\text{cm}^3$ )	Cellulose content (%)	Hemi-cellulose (%)	Lignin (%)	Moisture (%)
$540 \pm 9$	$29 \pm 2$	$3 \pm 0.2$	$80 \pm 2$	$1.3 \pm 0$	$63 \pm 1$	$19 \pm 1.2$	$5 \pm 0.5$	$11 \pm 0.1$

Plant based cellulose nanofibers have attracted more and more interest as a source of nanometer sized fillers because of their sustainability, easy availability, low cost and the related characteristics such as large surface to volume ratio, high stiffness, high flexibility, good water resistance and superior thermo-mechanical properties as compared with other commercial fibers.<sup>19–23</sup> Treating various biomass resources by steam explosion has been studied by many researchers.<sup>23–25</sup> During steam explosion process the raw material is exposed to pressurized steam followed by rapid reduction in pressure resulting in substantial break down of the lignocellulosic structure, hydrolysis of the hemicelluloses fraction, depolymerisation of the lignin components and defibrillation.<sup>26</sup> The use of nanoreinforcements in the polymer matrix has been predicted to give improved properties compared to the neat polymer and microcomposites based on the same fibers. Therefore, it is of great interest to examine the possibilities of cellulose based nanofibers as reinforcing elements. The present work highlights the investigation results of the morphological and surface properties of banana fibers in macrofibrillated, microfibrillated and nanostructured forms and their impact on mechanical properties of the fiber reinforced PF composites. Careful analysis of the literature indicates that no study has been reported on the systematic comparison between these properties of macrofibrillated, microfibrillated, and nanostructured cellulose fibers.

## MATERIALS AND METHOD

### Materials

The reinforcement, banana fiber used in this study was obtained from Sheeba fibers and handicrafts, Poovancode, Tamilnadu, India. Phenol formaldehyde resole type resin obtained from M/S West Coast Polymers, Kannur, Kerala was used as matrix. Phenol formaldehyde resin has a dark brown colour, with a viscosity of 18CPS and a density of  $1.3 \text{ g}/\text{cm}^3$ . Sodium hydroxide (Commercial grade), acetic acid (Commercial grade), sodium hypochlorite (Commercial grade), oxalic acid (Commercial grade) obtained from Nice Chemicals, Cochin, India. Important characteristics of banana fiber are given in Table I.

### Isolation of Cellulose Fibrils

**Preparation of Microfibrils.** Microfibrils were separated from banana fibers (macro) obtained from local sources. The fibers obtained from the pseudo stem of the plant were soaked in 2% sodium hydroxide and placed in an autoclave with a pressure of  $0.0014 \text{ kg}/\text{cm}^2$  and at a temperature of  $120^\circ\text{C}$  for 1 h. After 1 h, the bottom valve of the autoclave is immediately opened to explode the steam. The fibers were taken out and washed several times with distilled water. Steam-exploded banana pulp was bleached with a solution consisting of equal amounts of sodium

hypochlorite solution [distilled water and sodium hypochlorite in the ratio (3:1)] mixed with acetate buffer (consisting of 27 g of sodium hydroxide mixed with 78.8 mL of glacial acetic acid, diluted to 1 L). The suspension of the bleached fibrils was washed several times with distilled water and homogenized, using a mechanical stirrer of type RQ-1.27 A for 45 min. The fibrils obtained were filtered and dried in an air oven at  $60^\circ\text{C}$  for 6 h. This was further powdered in a mixer. A schematic representation of steam explosion is shown in Figure 1.

**Preparation of Nanofibrils.** Banana fibers (macro) were chopped into uniform size of approximately 10-cm length. The fibers were soaked in 2% sodium hydroxide and placed in an autoclave with a pressure of  $0.0014 \text{ kg}/\text{cm}^2$  at a temperature of  $120^\circ\text{C}$  for 1 h. The pressure was released immediately. The fibers were removed from the autoclave and were washed in distilled water till it was free of alkali. The steam exploded fibers were bleached with a solution consisting of equal amounts of sodium hypochlorite solution [water and sodium hypochlorite in the ratio (3:1)] mixed with acetate buffer (consisting of 27 g of sodium hydroxide mixed with 75 mL of glacial acetic acid, diluted to 1 L). Each bleaching took 1 h and the process was repeated six times. After the bleaching, the fibers were thoroughly washed in distilled water and dried. The steam exploded bleached fibers were treated with 11% oxalic acid in an autoclave till it attained a pressure of  $0.0014 \text{ kg}/\text{cm}^2$  and the fibers were kept under that pressure for 15 min. The pressure was released immediately. The autoclave was again set to reach  $0.0014 \text{ kg}/\text{cm}^2$ , the pressure was released and the process was repeated eight times. The fibers were taken out, washed till the washings no longer decolorize  $0.05 \text{ N KMnO}_4$  solution. This was to make sure that the washings were free from acid. The nanofibrils were suspended in distilled water and stirred with a mechanical stirrer of type RQ-1.27 A at 8000 rpm for about 4 h until the fibers are dispersed uniformly. The suspension was kept in an oven at  $90^\circ\text{C}$  till it was dry. Figure 2 represents the scheme for the preparation of nanofiber from banana macrofiber.

### Fabrication of Phenol Formaldehyde Composites

**Macrocomposites.** Banana fibers were chopped to the desired length, dried in an air oven at  $70^\circ\text{C}$  for 4 h and then in a vacuum oven at  $60^\circ\text{C}$  for 1 h before preparation of composites. The mould was polished and mould releasing agent was applied. The cleaned fibers were arranged in the mould in the form of mats and pressed. Composites were fabricated using compression moulding (CM). The dimension of the mould in CM technique was  $150 \times 150 \times 3 \text{ mm}^3$ . The composite samples were prepared by prepreg method in CM. Here, the resin was poured

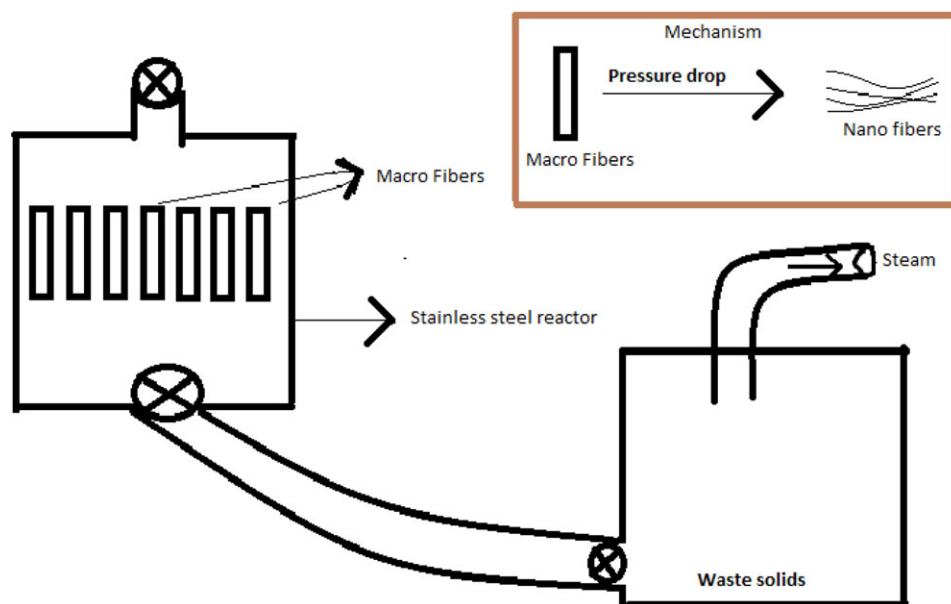


Figure 1. Schematic representation of steam explosion. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

into the mat until it was completely soaked. The mould was closed and hot pressed at a temperature of 130°C for 20 min and at a pressure 10 Kg/cm<sup>2</sup> and were slowly cooled to room temperature. The samples were subjected to post curing operation at 70°C for 1 h to ensure complete curing. Composite samples were prepared by keeping fiber length constant (30 mm) and by varying the fiber loading (0, 10, 20 wt %).

**Microcomposites.** Prepeg route was followed for the preparation of the composite. Composites were prepared using the microfibrils as reinforcement of varying loading such as 0, 4, 8, 10, 15, and 20 wt %. The microfibrils were evenly arranged in the mould in the form of mat and pressed. Then, this mat was completely impregnated with phenol formaldehyde resin. The prepeg was hot compressed at a temperature of 80°C for 20 min and at a pressure 10–15 MPa and was slowly cooled to room temperature. The samples were subjected to post curing operation at 70°C for 1 h to ensure complete curing.

**Nanocomposites.** PF resin and the prepared cellulose nanofibers were used for the composite preparation. The phenolic resin and the cellulose nanofibers were mixed well for 20 min by hand mixing. Different composite sheets were prepared with nanofiber loadings of 4, 6, 8, 10 and 12 wt %; the curing temperature was 80°C for 20 min and at a pressure 10–15 MPa and was slowly cooled to room temperature. The samples were subjected to post curing operation at 70°C for 1 h to ensure complete curing.

#### Characterization Techniques

**Atomic Force Microscopy Analysis.** Banana macrofiber, cellulose microfibrils and nanofibrils were observed using atomic force microscopy (AFM), Nanoscope IVa, Multimode SPM (Veeco Santa Barbara), in tapping mode. Calibration was performed by scanning a calibration grid with precisely known dimensions. All scans were performed in air with commercial Si

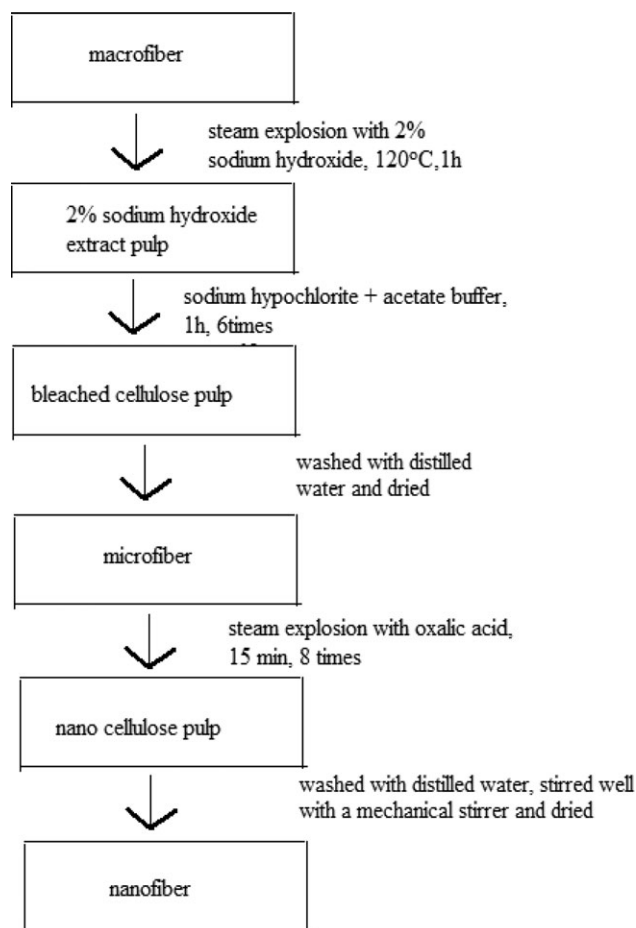
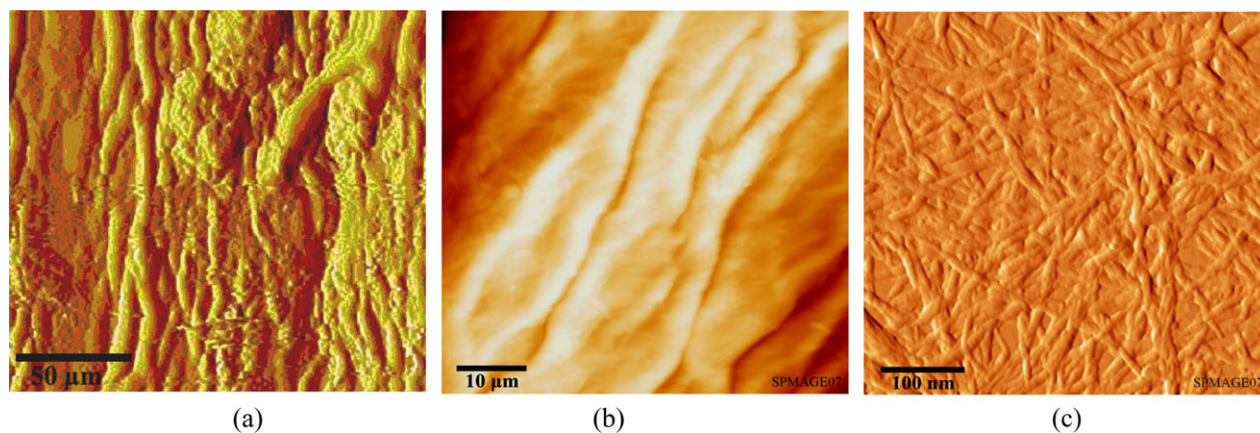


Figure 2. Schematic representation of the preparation of nanofiber from banana fiber.



**Figure 3.** AFM images of (a) macro banana fiber, (b) micro banana fiber and (c) nano banana fiber. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

nanoprobes SPM tips with a resonance frequency of about 300–330 kHz. A free amplitude ( $A_0$ ) of about 20 nm and a set-point ratio ( $r_{sp}$ ) between 0.4 and 0.6 were used. ( $r_{sp}$ ) is the ratio between the set point amplitude ( $A_{sp}$ ) and  $A_0$ . Image processing including flattening was made. Height and phase images were obtained simultaneously in tapping mode at the fundamental resonance frequency of the cantilever with a scan rate of 0.5 line/s using a j-type scanner. The free oscillating amplitude was 3.0 V, while the set point amplitude was chosen individually for each sample. For each sample images were scanned on at least five different fibers. Usually two different areas of each fiber were investigated. Only one representative image per sample is shown. For AFM analysis of cellulose nanofibril samples for characterisation were prepared by pipetting a 0.12 g/L aqueous whisker suspension and allowed to dry on a freshly cleaved mica surface. The sample was allowed to dry at room temperature overnight.

**Zeta Potential Measurements.** Zeta potential ( $\zeta$ ) measurements were made with the electrokinetic analyser EKA (Anton Paar KG, Graz, Austria) based on the streaming potential method. An electrolyte solution was forced by an external pressure through a bundle of capillaries (fiber plug). The potential  $U$  resulting from the motion of ions in the diffuse layer was measured with respect to the applied pressure. The electrokinetic potential or zeta potential was calculated from the measured streaming potential using Smoluchowski's equation ( $\Delta U/\Delta p$ ). The pH dependence of the  $\zeta$ -potential was determined in a  $1 \times 10^{-3}$  M KCl electrolyte solution and to keep the ionic strength constant, the pH value in the range 3–11 was altered by the addition of 0.1M HCl or KOH solution.

**Fourier Transform Infrared Spectroscopy.** A Bruker IFS 66 spectrophotometer, fitted with an MCT detector was used for recording absorption spectra of the fibers. Samples were scanned for characteristic absorptions in the range 4000–800  $\text{cm}^{-1}$ . The background used was dried powdered KBr.

**Thermogravimetric Analysis.** Thermogravimetric analysis (TGA) was carried out using a NETZSCH TG 209F1 in Helium environment, with a heating rate of 30°C/min. The temperature

range scanned was from 25 to 700°C. The thermal degradation behavior of untreated and treated macrofibers, microfibers, and nano banana fibers was analysed. The weight of all specimens was maintained around 10 mg.

#### Mechanical Testing

Tensile testing was carried out using FIE electronic universal testing machine TNE 500 according to ASTM D 638-76. Three point flexure properties were also tested using the same machine according to ASTM D 790. Charpy impact strength (unnotched) was measured in a WinPEN CEAST (SpA, Italy), according to ISO 179.

## RESULTS AND DISCUSSION

#### Characterization of Banana Fiber

Banana fibers obtained from local sources was subjected to steam treatment to obtain microfibers, and nanofiber. The diameters of macrofibers, microfibers, and nanofiber were compared using AFM. Figure 3(a–c) represents macro, micro and nano banana fibers, respectively. The average fiber diameter was found to be much lower for nanofibers and is around 30 nm. Steam treatment of macrofiber at high pressure reduces the fiber diameter. It is clear from the AFM micrographs that high pressure steam treatment helps in fiber separation and fibrillation. It is important to mention that the surface roughness of the fiber samples were decreased from macrofiber to nanofiber. Both microfibers and nanofibers resulted in the significant removal of noncellulosic components. These results truly indicated that steam correlated acid treatment helped to develop fibers of higher cellulosic component, and thus suggest a more effective removal of the middle lamella and the primary cell wall and therefore a more cellulose rich surface and effective reduction of fiber dimension to nanorange.<sup>18,23,27–28</sup> AFM suggested that only few lateral associations occur between adjacent nanofibers. Nanofibers are much more clearly defined probably because of the removal of amorphous zones and they seem to be more interconnected with nano-ordered chains, which may elevate the reinforcing capability of the developed nanofibrils in the composite matrix.<sup>26</sup>

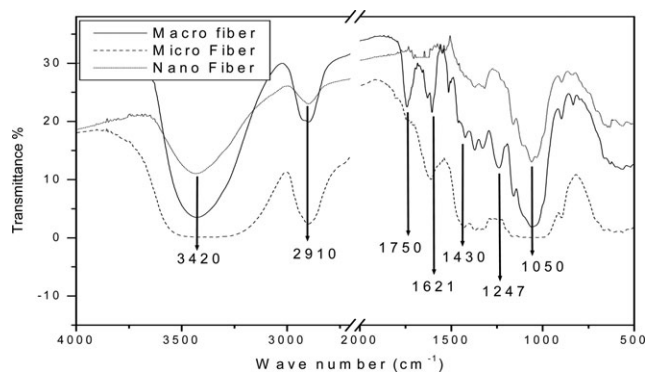


Figure 4. FTIR spectra of macrofibers, microfibers, and nanofibers.

### Fourier Transform Infrared Spectrometry

The IR spectrum of the macro, micro and nano banana fiber is shown in Figure 4. A number of peaks disappeared in the case of nanofibers, which indicates the removal of hemicelluloses, lignin, pectin, and so forth, this is in accordance with previous publications.<sup>18,28</sup> The peaks in the area  $3420\text{ cm}^{-1}$  arise due to  $[-]\text{OH}$  stretching vibrations of hydrogen bonded hydroxyl group. The narrowing of the peak at  $3420\text{ cm}^{-1}$  is due to the formation of free hydroxyl groups by breaking up of hydrogen bonds.<sup>28</sup> The hydrophilic tendency of macro, micro and nano are reflected in the broad absorption band at  $3700\text{--}3100\text{ cm}^{-1}$  region due to the presence of  $[-]\text{OH}$  groups present as main component. The peak at  $2910\text{ cm}^{-1}$  is due to aliphatic saturated  $\text{C}[-]\text{H}$  stretching vibration in hemicellulose and cellulose. The sharpening of the peak at  $2910\text{ cm}^{-1}$  reveals the increase of crystallinity and thereby the increase of cellulose in the fibers.<sup>27</sup> The peak at  $1750\text{ cm}^{-1}$  is a characteristic peak of hemicelluloses, and is absent in the banana nanofibers. This is a clear evidence of the removal of hemicellulose during steam explosion.<sup>18</sup> Similarly, the peak at  $1621\text{ cm}^{-1}$  indicates the presence of lignin by  $\text{C}[-]\text{C}$  vibration and is absent in the banana nanofibers.<sup>29</sup> The peak at  $1430\text{ cm}^{-1}$  is also due to lignin components.<sup>30</sup> The bands in the region  $1250\text{--}1050\text{ cm}^{-1}$  involve the  $\text{C}[-]\text{O}$  stretching of primary and secondary alcohols in cellulose, hemicellulose and lignin.<sup>31</sup> The  $1050\text{ cm}^{-1}$  peak is assigned to the ether linkage ( $\text{C}[-]\text{O}[-]\text{C}$ ) in lignin and hemicelluloses, the peak area are decreased for nanofibrils.<sup>27</sup> From the Fourier transform infrared (FTIR) spectra of nanofiber the prominent peaks of lignin and hemicellulose are absent or intensities reduced, this indicates that the removal of binding components present in the fibres due to the process of steam explosion and acid treatment.

### Zeta Potential Measurements

Zeta potential measurements were carried out to investigate the surface properties and the possible interactions. Nature of the surface of banana fibers and banana fibril can be understood through the studies of the influence of pH on zeta potential. The pH that agrees with the zero of the zeta potential (Iso electric point, IEP) goes to decide the acidity or the basicity of the solid surfaces qualitatively. Thus at this pH the number of negative charges equals the number of positive ones.<sup>32</sup> It is the IEP that characterises the acidity of the surface. When the IEP val-

ues are low there is dominance for the number of acidic groups. Banana macrofiber holds the IEP value 2.5, microfiber 2.1 and that of nanofiber is 1.6, thus indicating an acidic surface (Figure 5).<sup>33</sup> The increase in acidity of the nanofiber surface is due to the presence of carboxyl group of the oxalic acid. If the solid surface being investigated contains acidic functional groups, the negative zeta potential increases with PH due to increasing dissociation of these groups and remain constant above a given PH due to their complete dissociation.<sup>34</sup>

### Thermal Properties

The TGA and DTG curves of macro, microfibrils and nanofibrils are illustrated in Figure 6(a–b) respectively. Natural fibers present three main weight loss regions, that is, in the ranges around  $60\text{--}100^\circ\text{C}$ , between  $200$  and  $400^\circ\text{C}$ , and  $400\text{--}600^\circ\text{C}$  [Figure 6(a)]. The initial weight loss in the region around  $60\text{--}100^\circ\text{C}$  is mainly due to moisture evaporation.<sup>35</sup> The main degradation peak in untreated fiber occurred between  $230$  and  $370^\circ\text{C}$ , where  $65\text{--}70\%$  of the degradation occurred. Beyond the main degradation stage, all the volatile materials were driven off from the sample resulting in the residual char. The major weight loss occurred due to the degradation of cellulose, hemicellulose, and lignin.<sup>35</sup> The DTG curve of macrofiber shows a peak at  $326^\circ\text{C}$  (mass loss  $51\%$ ) which is due to the thermal decomposition of cellulose. During the formation of microfibrils, hemicellulose, lignin, and pectin get dissolved out partially in alkali and results in a fibrillated structure. The increase in the % crystallinity index of microfibrils and nanofibrils reduces the moisture absorption. The DTG curve of banana microfibril [Figure 6(b)] exhibits two peaks. The initial shoulder peak at about  $60^\circ\text{C}$  corresponds to a mass loss of absorbed moisture or low molecular weight compounds remaining from the isolation procedure and the major decomposition peak at about  $347^\circ\text{C}$  (mass loss  $51\%$ ) is attributed to cellulose decomposition. But in the case of banana nanofibrils, we can see a major decomposition peak at  $386^\circ\text{C}$  (mass loss  $52\%$ ) due to the cellulose

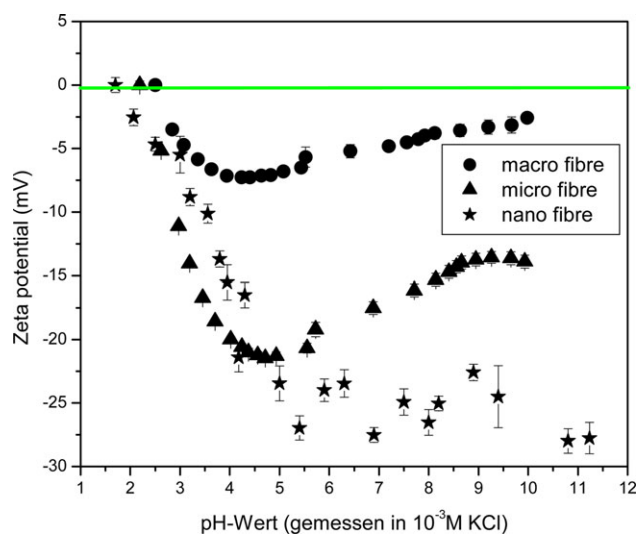
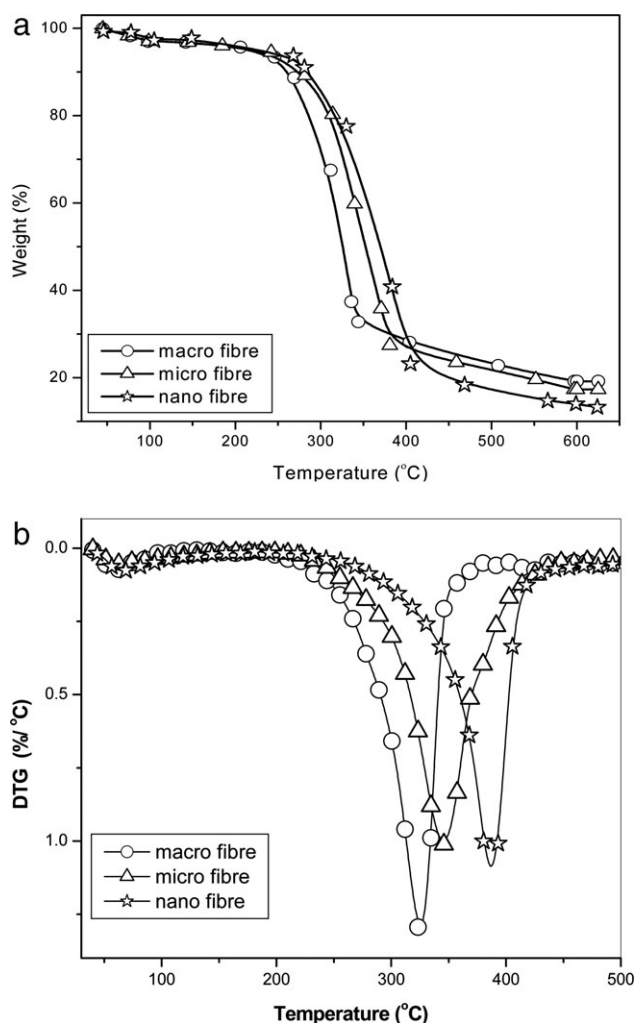


Figure 5. pH dependence of zeta potential of macro, micro and nanocellulose fibers. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]



**Figure 6.** (a) Thermograms of macro, micro and nanocellulose fibers. (b). DTG curves of macro, micro and nanocellulose fibers.

decomposition. The main degradation temperature gets shifted towards a higher temperature region. The shift has been found to be higher for nanofibrils. From the above observation it is clear that there is a shift in the major decomposition temperature from 326 to 386°C as we go from macrofiber to nanofi-

brils. Dissolution of the various components leaves cellulose as the residual material which has been reported to be crystalline.<sup>18</sup> A greater crystalline structure required to get a higher degradation temperature.<sup>36</sup> The increase in the degradation temperature in the nanofibrils occurs due to the high crystallinity of the fiber structure. Therefore, it can be concluded from these results that the developed nanofibers exhibits enhanced thermal properties compared to the macrofiber and microfiber so that it can act as a suitable reinforcing element in biocomposite preparation. The results show that at 600°C, the highest residue was obtained for macro banana fibers (20%) and the lesser residue was obtained for nanofibers (14%). The relative low amount of residue in nanofibers may be due to removal of hemicelluloses and lignin from the fibers. These results are in agreement with FTIR and AFM results.

### Characterization of Nanocomposites

Nanocomposites have better mechanical properties (tensile strength, flexural strength, and impact strength) compared to microcomposites and macrocomposites with very limited amount of filler (Table II). The brittle nature of PF resin decreased even with the addition of small quantities of nanofibers. In a previous work Joseph et al. observed that the banana fibers reinforced PF composites are as good as glass fiber reinforced PF composites. They observed little improvement in mechanical properties with glass fiber content less than 20 wt %.<sup>4</sup> However, we have significant increase in tensile strength (142%), flexural strength (280%), and impact strength (133%) of the PF matrix composites with small amount of nanofibers. Recent work by Yano and coworkers observed excellent improvement in mechanical and thermal properties of wood fiber reinforced acrylic composites.<sup>37</sup> The presence of cellulose nanocrystals in the PF matrix contributes more effectively in enhancing the mechanical properties or in other words the increase in mechanical properties is due to the efficient stress transfer between the fiber cellulose nanocrystals and the matrix.<sup>38</sup>

Generally, the interaction of cellulose fiber with PF resin is good due to the hydrophilic nature of cellulose and PF resin. Hydrophilicity of the cellulose crop up from the free cellulosic hydroxyl groups. These can easily form hydrogen bonds with phenolic hydroxyl groups of the resole in the partial curing stage at 50°C. On curing at high temperatures these groups can undergo cross condensation reaction leading to three-

**Table II.** Tensile Strength (TS), Flexural Strength (FS), and Impact Strength (IM) of Macrocellulose, Microcellulose and Nanocellulose Reinforced/Phenol Formaldehyde Composites (C) at Different Fiber Loading (wt %)

Macrocellulose reinforced				Microcellulose reinforced				Nanocellulose reinforced			
C (wt %)	TS (MPa)	FS (MPa)	IM (kJ/m <sup>2</sup> )	C (wt %)	TS (MPa)	FS (MPa)	IM (kJ/m <sup>2</sup> )	C (wt %)	TS (MPa)	FS (MPa)	IM (kJ/m <sup>2</sup> )
PF	7 ± 2	10 ± 2	12 ± 1.8	PF	7 ± 2	10 ± 2	12 ± 1.8	PF	7 ± 2	10 ± 2	12 ± 1.8
10	13 ± 1.6	25 ± 1.9	13 ± 2	4	9 ± 1.3	14 ± 1.5	13 ± 1.2	4	13 ± 1.7	25 ± 1.7	15 ± 1.2
20	16 ± 2.7	34 ± 2.3	17 ± 1.9	8	12 ± 1.4	15 ± 1.2	13.5 ± 1.3	6	15 ± 2	28 ± 1.9	18 ± 1.9
				10	14 ± 3.2	20 ± 1.7	14 ± 1.8	8	16 ± 1.8	33 ± 2.1	22 ± 2.1
				15	15 ± 2.8	23 ± 1.8	16 ± 2.3	10	18 ± 1.9	36 ± 2.2	25 ± 1.8
				20	18 ± 3	25 ± 2.3	20 ± 1.9	12	17 ± 1.5	38 ± 1.8	28 ± 2.2

dimensional network between the fiber and matrix. This increases the strength of the chemical interlocking at the hydrophilic centers of the phenol formaldehyde resin. Thus the effective stress transfer takes place at the interface which leads to the debonding of the fibers only at very high tensions causing rupture of the composite structure.<sup>39</sup> It can be seen that an approximately linear relationship between the tensile modulus and the fiber content is obtained over the whole range of fiber loading. In the case of nanofibers, cellulose fibers may be swollen in phenolics and formaldehyde. The polymerisation is then carried out, generating a molecular cell wall/PF composite in the outer layers of the nanofibers and hence strong fiber-matrix interface.<sup>40</sup> It is also important to add that since the size of the nanofibers is too small, there exist better bonding between the fiber and matrix. This supports the fact that cellulose nanofibril reinforced PF composite exhibited enhanced strength compared to microfibril-PF composites which can be attained by inducing smaller volume fraction of nanofibrils.

## CONCLUSION

Cellulose microfibrils and nanofibrils of banana fibers were isolated using high pressure hydrothermal process. Characterisation of the synthesised microfibrils and nanofibrils were done using AFM, FTIR, electrokinetic studies and TGA. The IR studies give evidence for the dissolution and chemical modification that occurred during steam explosion and further treatment of the fibers by steam explosion in acidic medium. The AFM analysis also showed that there was reduction in the size of banana fibers to the nanometer range (around 30 nm). The surface acidity was proved to be increased from macroscale to nanoscale as the IEP value decreased. Substantial increase in thermal stability was observed from macrofibers to nanofibers which proved the high thermal stability of nanofibers to processing conditions of biocomposite preparation. The mechanical properties of PF reinforced fibers such as tensile strength, flexural strength, and impact strength were compared, it was found that a small amount of nanofiber can potentially improve the composite properties by a long way.

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