

Microcrystalline Cellulose from Plant Wastes through Sodium Hydroxide-Anthraquinone-Ethanol Pulping

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Microcrystalline cellulose was prepared from wastes of *Tithonia diversifolia*, inflorescence stems of *Musa sapientum*, and *Musa paradisiaca* by soda-anthraquinone–ethanol pulping method. They were bleached by sodium chlorite and then alpha-cellulose was isolated, followed by preparation of microcrystalline cellulose. The study revealed the effect of various processing stages on the properties of the cellulose obtained. Yields of more than 80% of microcrystalline cellulose were obtained. Fourier transform infrared (FTIR) and solid state ^{13}C Nuclear magnetic resonance (^{13}C NMR) confirmed the presence of the major expected peaks in microcrystalline cellulose. Scanning electron microscopy (SEM) revealed that *Musa* species had short fiber length and mixtures of non-aggregated spherical, rod-shaped and thread like microcrystalline cellulose, but *Tithonia diversifolia* had aggregate crystal packed formation. The results compared well with those of other authors and were able to meet most of the requirements specified in British Pharmacopoeia. The study revealed that a drug excipient like microcrystalline cellulose that could protect thermo-labile active ingredients could be successfully obtained from abundant non-woody agricultural wastes.

Keywords: Alpha-cellulose; Bleaching; Instrumental analysis; Microcrystalline cellulose; *Musa paradisiaca*; *Musa sapientum*; Pulping; *Tithonia diversifolia*; Waste

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INTRODUCTION

Solid waste management is a challenge to the whole world. This could be attributed to various factors such as high rates of urbanization, increasing income per capita, and huge volumes of agricultural wastes, *etc.* (Franz and Welle 2003; de Vlioger 2003; Kanagaraj *et al.* 2006; Robertson 2008; Azahari *et al.* 2011). In Nigeria, enormous volumes of unutilized and underutilized agricultural plants and biomass wastes that contain cellulosic fibers are generated annually. According to the Food and Agricultural Organization (FAO), each year farmers harvest around 35 million tons of natural fibers from a wide range of plant and animals (Siqueira *et al.* 2010). For example, tons of maize stalks, maize cobs and husks, plantain stem, raffia from *Raphia hookeri*, elephant grass, sunflowers, empty bunches of palm oil fruits, coconut husk, just to mention a few, are allowed to rot away yearly, constituting an environmental nuisance.

Tithonia diversifolia (syn. *Mirasolia diversifolia*), is an aggressive weed growing to a height of about 2.5 m and adaptable to most soils. It is a shrub belonging to the family Asteraceae and originated from Mexico. It is now widely distributed throughout the humid and subhumid tropics in Central and South America, Asia, and Africa (Sonke

1997). It has been reported in Kenya (Niang *et al.* 1996), Malawi (Ganunga *et al.* 1998), Nigeria (Ayeni *et al.* 1997), Rwanda, Cameroon and Uganda (Drechsel and Reck 1998), and Zimbabwe (Jiri and Waddington 1998). The abundance and adaptability of this weed species to various environments coupled with its rapid growth rate and very high vegetative matter has aroused research interest (Nagaraj and Nizar 1982). The reported uses of *Tithonia* include fodder (Roothaert and Patterson 1997; Roothaert *et al.* 1997), poultry feed (Odunsi *et al.* 1996), compost (Drechsel and Reck 1998; Ng'inja *et al.* 1998), land demarcation, soil erosion control, fuelwood (Ng'inja *et al.* 1998), and building materials and shelter for poultry (Otuma *et al.* 1998). In addition, extracts from *tithonia* plant parts reportedly protect crops from termites (Adoyo *et al.* 1997) and contain chemicals that inhibit plant growth (Baruah *et al.* 1994; Tongma *et al.* 1997) and control insects (Carino and Rejestes 1982; Dutta *et al.* 1993).

The general term 'banana' is used to encompass cultivated varieties of the genus *Musa* that fall into one of two subgroups (Pillay *et al.* 2002): the sweet or dessert banana which makes up approximately 43% of world production, and the cooking banana which makes up approximately 57%. The general term plantain is applied to a specific subgroup of cooking bananas (Valmayor *et al.* 2000). Bananas/plantains are a major food crop globally and are grown and consumed in more than 100 countries throughout the tropics and sub-tropics. In developing countries they are the fourth most important food crop after rice, wheat, and maize (INIBAP 2000). The banana plant is a tall arborescent monocotyledon with a false stem (pseudostem) consisting of leaf sheaths and an underground true stem (corm) that is able to produce suckers by which the plant can reproduce vegetatively. Each pseudostem produces a single inflorescence, the female flowers of which give rise to the banana/plantain fruits (Espino *et al.* 1992). The bananas/plantains fruit can be eaten raw or cooked (*e.g.* deep fried, dehydrated, baked in the skin, steamed), can be processed into flour and can be fermented for the production of beverages such as banana juice, beer, vinegar, and wine (Morton 1987; Pillay *et al.* 2002; Nelson *et al.* 2006; Edmeades *et al.* 2006; Pillay and Tripathi 2007). All parts of sweet banana/plantain plants, but particularly the fruits, have also been used to feed livestock in those parts of the world where there is excess production (Babatunde 1992). Banana leaves have a variety of practical uses including wrapping for food, plates for serving food, and polishing floors. Fibres obtained from the pseudostem are used for making cloth (Espino *et al.* 1992; Nelson *et al.* 2006) and leaf fibres are utilised in string, cordage, and rope (Nelson *et al.* 2006). Today it is used mainly in the paper making industry where its long staple length, strength, and cellulose content, make it useful in specialised papers including tea and coffee bags, sausage casing paper, currency notes, cigarette filter papers, medical/ disposal papers, and some high-quality writing paper (Wigglesworth 2007).

In all these agricultural wastes there is a lot of cellulose, the most abundant and cheapest natural polymer with annual production of about one trillion tons, making it virtually an exhaustible source of raw materials (Ioeiovich 1999; Dutta *et al.* 2004; Kim *et al.* 2007; Israel *et al.* 2007). Cellulose fiber is a potential raw material not only for the pulp and paper industries, but also for modern eco-friendly composites such as building materials, particle boards, insulation boards, textile, cosmetics, medicine and pharmaceutical products, bio-polymers, fine chemicals, *etc.* (Bledzki and Gassan 1999; Edgar *et al.* 2001; Zhang 2001; Belgacem and Gandini 2005; Klemm *et al.* 2005; Kontturi *et al.* 2006; Czaja *et al.* 2007; O'Connell *et al.* 2008; Reid *et al.* 2008; Kalia *et*

al. 2009; Eichhorn *et al.* 2010; Tizzotti *et al.* 2010; Eichhorn 2011; Mashkour *et al.* 2011; Spence *et al.* 2011; Huber *et al.* 2012; Qiu and Hu 2013). Being biodegradable, recycling of these products gives its application another advantage over the petroleum-based products (Ryszard 2000).

Through acid hydrolysis, cellulose can be converted to microcrystalline cellulose (MCC), which has been used especially in food, cosmetics, and medical industries as a water-retainer, a suspension stabilizer, a flow characteristics controllers in the systems used for final products, and as reinforcing agent for final products such as medical tablets (Mohamed and Mohammad 2007). Microcrystalline cellulose has enjoyed extensive usage in pharmaceutical industry because of its numerous derivatives which are multifunctional excipients in drug formulations. The growing nature of pharmaceutical industry and abundant availability of agricultural waste has motivated various researchers to examine those waste as alternative source of cellulose for the production of MCC, besides the cotton and wood materials currently being used (Barba *et al.* 2002; Rocha *et al.* 2002; Ohwoavworhua *et al.* 2004; Bhimte and Pralhad 2006; Ilindra and Dhake 2008).

Pulping methods have been modified these days by addition of certain chemicals, *e.g.* ethanol and anthraquinone, to the basic pulping chemicals (soda and kraft). The choice of pulping chemicals is informed by literature indicating that addition of ethanol and anthraquinone to soda pulping would have better advantages. For example, adding ethanol to a caustic soda cook greatly improves its selectivity and degradation of lignin (McLaughan 2000) and prevents lignin condensation (Marton and Granzow 1982), while the presence of sodium hydroxide improves the delignifying ability of ethanol (Muurinen 2000). Adding anthraquinone as catalyst in sodium hydroxide system increases the pulp yields, decreases the kappa numbers, and improves the strength properties (Valladares *et al.* 1984). It also increases lignin removal by promoting cleavage of inter-unit bonds in the lignin molecules that are not cleaved in the absence of AQ. It also helps minimize recondensation of lignin reactions by reacting with the carbohydrates to increase lignin removal during pulping process and produced cellulose with high yield (Venica *et al.* 1989; Suckling 1989; Chai *et al.* 2007).

This present study was aimed at obtaining alpha-cellulose through pulping using a sodium hydroxide-anthraquinone-ethanol mixture, hydrolyzing alpha cellulose that would be obtained from the stem of Sunflower (*Tithonia diversifolia*), inflorescence flower stems of both plantain (*Musa paradisiaca*) and banana (*Musa sapientum*), to possibly produce high yield microcrystalline cellulose (based on pulping chemical mixture strength) and to characterize the material obtained for possible use as pharmaceutical excipient.

EXPERIMENTAL

Materials

The plant materials used in this study were inflorescence flower stems of banana (*Musa sapientum*) and plantain (*Musa paradisiaca*) with sunflower (*Tithonia diversifolia*) stalks, obtained respectively from Oke–Asoo Farm settlement in Ilawe-Ekiti, in Ekiti State, the Demonstration Farm of the Federal University of Technology Akure, Ondo State, and the Staff quarters (Obakekere) of the same University, all in Nigeria. All the

samples were authenticated at the Department of Crop, Soil and Pest Management, Federal University of Technology Akure, Ondo State, Nigeria. Analytical grade chemical reagents used were NaClO₂ (Sigma-Aldrich), sodium hydroxide (BDH), cupriethylenediamine (Sigma-Aldrich), acetic acid (Sigma-Aldrich), ethanol (95% BDH), hydrochloric acid (BDH), and anthraquinone (BDH).

Methods

Pre-treatment of Materials

Sunflower stalks were harvested at 10 cm above the ground level. All samples were cleaned to remove dust, sand, dirt, and contaminations. They were cut into chips of about 2 to 4 cm and sun-dried. The sample was milled using Wiley mill, screened through 425 µm, and stored separately in labeled polyethylene bags for subsequent experiments.

Pulping

Pulping experiments were performed in a 15-litre reconstructed (THR -280B) thermostatically controlled electrically heated oil bath autoclave digester with an inner stainless steel cooking container. The following cooking conditions were employed: liquor to fiber ratio was 25:1 (v/w), temperature 170 °C, pressure 15 psi, cooking time of 90 min, anthraquinone 0.1% based on the dry weight content and 40% ethanol (95%) based on the liquor content. The alkali charge was 15% (w/w) NaOH. After digestion, the pulp obtained through filtration was thoroughly washed with water until free of residue alkali. The pulp yield was determined after oven drying at 105 °C to constant weight gravimetrically as percentage of oven-dry raw materials. Also, properties such as moisture, ash, silica, and kappa number were determined, respectively, using TAPPI standards T550om-03, T211om-93, T224om-93, and T236cm-85.

Bleaching

The pulps were bleached with sodium chlorite (NaClO₂) and sodium hydroxide (NaOH) solution sequences. In this process, 20 g of oven-dry pulp sample in a 2 L Erlenmeyer flask was added to 1000 mL of hot distilled water, 12 g of sodium chlorite, and 3 mL of acetic acid. The flask was covered with watch glass and the mixture heated in a water bath at 70 °C for 60 min, with intermittent stirring. After the treatment, the sample was drained followed by extraction with 1000 mL of 5% NaOH performed at 70 °C for 30 min. After alkali extraction, the sample was washed free of alkali with distilled water. This treatment sequence was carried out three times, but the sample was not washed after the third 60 min, whereupon 12 g of sodium chlorite and 3 mL of acetic acid were added and the sample was allowed to stand undisturbed for the next 60 min. After the final 60 min, the water bath power source was turned off and the sample was left inside the bath for 24 h. The pulp was filtered and washed to obtain filtrate pH of 7, then oven-dried at 105 °C to constant weight and the yield, moisture, ash, silica, and kappa number were determined using standard TAPPI methods. The beta-, gamma-, and alpha-cellulose were determined using TAPPI method T203cm-99.

Pulp Viscosity

Pulp viscosity was determined using a modified capillary viscometer method (TAPPI T230om-99) as follows; the intrinsic viscosity was determined using an Ubbelohde viscometer by weighing 0.25 g of oven-dry sample into a dissolving bottle containing eight 6 mm glass beads, and 25 mL of distilled water was added from a burette. The vial was closed tightly and shaken for 15 min. The bottle was allowed to stand for about 2 min, after which exactly 25 mL of 1M cupriethylenediamine solution was added. The bottle was capped and shaken for about 30 min until the fiber was completely dissolved. A washed, clean, and dry Ubbelohde viscometer was then prepared with its other end closed, and 25 mL of the solution was measured into the capillary viscometer. The closed end was then opened, and the efflux time estimated in seconds. Five measurements were made, and the average of three measurements was taken after discarding both the lowest and highest readings. The viscometer was carefully cleaned with nitric acid, water, and acetone and dried between measurements. The viscosity $[\eta]$ in centipoise (cP) was calculated from the efflux time of the sample solution (t) and the blank solution (t_0), using the equation of Solomon and Gatesman (Ibrahim *et al.* 2010) represented by Eq. 1,

$$[\eta] = \frac{[2(\eta_{sp} - t_0 \eta_r)]^{1/2}}{c} \quad (1)$$

where $[\eta]$ is the intrinsic viscosity (cP), η_{sp} is the specific viscosity $[\frac{(\eta_{\text{solution}} - \eta_{\text{solvent}})}{\eta_{\text{solvent}}}]$, η_{solution} is the product $t_{\text{solution}} \times \rho_{\text{solution}}$, η_{solvent} is $t_{\text{solvent}} \times \rho_{\text{solvent}}$, η_r is the relative viscosity ($t_{\text{solution}}/t_{\text{solvent}}$), C is the concentration of the sample (1.052 g/cm³), ρ_{solution} is the density of solution (g/cm³), ρ_{solvent} is the density of solvent (g/cm³), t_{solution} is the solution flow time (s), and t_{solvent} is solvent flow time (s)

Degree of Polymerization (D.P)

The viscosity was converted to the degree of polymerization (Morton 1996) as follows;

$$\text{D.P} = 598.4 \ln[\eta] + 118.02(\ln[\eta])^2 - 449 \quad (2)$$

Molecular Weight

The molecular weight was calculated using Eq. 3 (Hong *et al.* 1978),

$$\text{DP} = \frac{162}{M} \quad (3)$$

where 162 is the molecular weight of an anhydroglucose unit (AGU).

Isolation of Alpha-Cellulose

A 30 g oven-dried sample of bleached cellulose was placed in a 5000 mL flask with a cover. This was placed in a water bath maintained at 20 °C. Next, 1500 mL of 17.5 % NaOH in a 5000 mL flask was added, and mixed thoroughly for one minute. After 30 min, 500 mL of distilled water was added and mixed well for another one minute. The reaction was continued for five more minutes. The content of the flask was filtered and

the residue was washed with 500 mL of 8.3 percent NaOH, then with 400 mL of 10% acetic acid. The residue was washed free of acid with water. The content was oven-dried at 105 °C, until a constant weight was reached and cooled in desiccators. The isolated cellulose was then characterized using standard methods.

Preparation of Microcrystalline Cellulose (MCC)

The method for the preparation of the microcrystalline cellulose was a slight modification of Bhimte and Tayade (2007). The α -cellulose was hydrolyzed with 2 M hydrochloric acid (HCl), at a solid:liquor ratio of 1:20 and refluxing at 105 °C \pm 2 °C for 30 min (which is able to specifically cleave 1–4 glycosidic linkages). After hydrolysis, the microcrystalline cellulose was collected by filtration and was washed thoroughly with distilled water and then treated with 1% ammonium hydroxide solution followed by washing with distilled water to neutral pH, and then oven dried at 40 °C to constant weight. This final material was MCC, obtained as dried cake, which was powdered and stored in an airtight container until further evaluation.

Determination of Properties of Microcrystalline Cellulose

The organoleptic properties such as colour, odour, and taste of the powder microcrystalline cellulose were determined physically using sense organs and the observation noted. Starch and dextrin, solubility, identification test, and loss on drying were determined by using standard methods of British Pharmacopoeia, 1993b,-2004, 1993a and 2004 respectively. The pH was determined by the method described by JECFA (1998). Ash was measured as described by Pomeranz and Meloan (1994), while moisture sorption capacity (Ohwoavworhua *et al.* 2004) and swelling capacity (Okhamafe *et al.* 1991) followed procedures given in the cited works.

True Density

The true density, (D_t), of the sample was determined by a liquid displacement method using xylene as the immersion fluid. Calculations were as follows,

$$D_t = \frac{w}{\{(a+w)-b\}} \times SG \quad (6)$$

where w is the mass of the sample, SG is the specific gravity of the solvent (xylene), a is the combined mass of the bottle + solvent, and b is the sum of the mass of the bottle, the solvent, and the sample (Alfa *et al.* 2000).

Bulk and Tapped Densities

For the bulk and tapped densities, 20 g of the sample was carefully put in a dry 100 mL graduated measuring cylinder, and the volume (V_0) occupied by the sample without tapping was noted. The sample contained in the measuring cylinder was tapped mechanically (500 taps) at the bottom of the cylinder, and the new volume (V_{500}), was also noted. The densities were determined as the ratio of the weight and volume of sample in each case (Bean *et al.* 1967). The densities were determined in triplicates. The bulk (B_d) and tapped densities (T_d) were calculated as follows,

$$B_d = w/v_n \quad (7)$$

$$T_d = w/v_{500} \quad (8)$$

where w is the weight of the sample used.

Hausner index

This was calculated as the ratio of the tap density to bulk density of the sample (Ohwoavworhua 2004).

Compressibility index (Carr's index %)

Percentage compressibility (Carr 1965) was determined using the following formula:

$$\text{Carr's index \%} = \frac{\text{Tapped density} - \text{bulk density}}{\text{Tapped density}} \times 100 \quad (9)$$

Powder Porosity

The powder porosity, PB (%), was derived from the values of true and bulk densities when fitted into the equation,

$$P_B = \left[1 - \left(\frac{B_d}{D_t} \right) \right] \times 100 \quad (10)$$

where B_d and D_t are bulk and true particle densities, respectively (Bean *et al.* 1967)

Angle of Repose

The static angle of repose a , was measured according to the fixed funnel and free standing cone method (Train 1958). A funnel was clamped with its tip 2 cm above a graph paper placed on a flat horizontal surface. The powders (50 g) were carefully poured through the funnel until the apex of the cone thus formed just reached the tip of the funnel. The mean diameters of the base of the powder cones were determined, and the tangent of the angle of repose calculated using the equation,

$$\text{Tan } a = 2h/D \quad (11)$$

where h is the height of the heap of powder and D is the diameter of the base of the heap of powder.

Instrumental Analysis

Scanning electron microscopy (SEM) was observed through the use of an SEM EVO MA-10 instrument (made by Carl Zeiss) at an acceleration voltage of 20 Kv and probe current of 227 pA. Fourier Transform Infrared (FTIR) spectroscopy was performed using a ThermoNicolet Avatar 370 FT-IR spectrometer operating in the attenuated total reflection (ATR) mode (SmartPerformer, ZnSe crystal). About 5 mg of MCC powder (vacuum dried) were analyzed. Each spectrum was taken as an average of 64 scans at a resolution of 4 cm^{-1} . Thermogravimetric analysis (TGA) was done using a Perkin-Elmer TGA 7 Thermogravimetric Analyzer. The heating was set at 20 $^{\circ}\text{C}/\text{min}$ over a temperature range of 50 to 900 $^{\circ}\text{C}$ and measurements carried out in nitrogen atmosphere.

Solid state cross polarization-magic angle spinning (CP-MAS) ^{13}C nuclear magnetic resonance (NMR) spectra were obtained on a Bruker Avance DRX-400 solid-state NMR spectrometer operating at a frequency of 100.6 MHz equipped with a Chemimagnetics solids probe (Washington State University NMR center). Samples were ground and packed into a 5 mm zirconia rotor which were spun at 5000 Hz. Spectra were recorded using a 3.75 μs proton preparation pulse followed by a 1 ms cross polarization contact time and acquisition time of 6.6 μs . Spectral analysis was performed using freeware MestRe-C v2.3a.

Statistical Analysis

The data obtained in triplicate were analysed by Probit Analysis using Duncan's Multiple Range Test (DMRT) and Analysis of Variance (ANOVA).

RESULTS AND DISCUSSION

Raw Material

The level of holocellulose listed in Table 1 indicates that the materials would be good sources of cellulose and hemicellulose. *M. sapientum* (73.43%) and *M. paradisiaca* (72.60%) had the highest values, followed by *T. diversifolia* (71.60%). Holocellulose obtained in this research work compared well with those reported by other researchers. Ates *et al.* (2008), had reported 75.74% for *P. elongata*, whereas 70.50% was reported for bamboo (Deiz and Ates 2002), rye straw was found to have 74.1% (Usta and Eroglu 1987), *T. diversifolia* stalk 77.6% (Kirci *et al.* 1998), 67.6% for tobacco stalk (Usta *et al.* 1990), and Mokhtar *et al.* (2005) reported 75.20% for pineapple leaves.

Table 1. Physicochemical Properties of Raw Material

| Parameter (%) | Sample | | |
|-----------------------|-----------------------------|----------------------------|------------------------------|
| | <i>Musa sapientum</i> | <i>Musa paradisiaca</i> | <i>Tithonia diversifolia</i> |
| Holocellulose | 73.40 ^a ±0.20 | 72.60 ^b ±0.20 | 71.60 ^c ±0.20 |
| Alpha cellulose | 55.33 ^a ±0.29 | 55.00 ^{b,c} ±0.90 | 54.00 ^{a,b} ±0.86 |
| Hemicellulose | 18.07 ^a ±1. 1.00 | 17.60 ^b ±1.10 | 17.60 ^b ±0.92 |
| Acid-soluble lignin | 22.00 ^{b,c} ±1.00 | 21.00 ^{a,b} ±1.00 | 22.97 ^c ±1.00 |
| Acid-insoluble lignin | 2.32 ^a ±0.16 | 2.95 ^c ±0.35 | 2.51 ^b ±0.05 |

Values are means of three replicate \pm standard deviation. Column means followed by different letters are significantly different at $P < 0.05$

The alpha-cellulose content followed the same ranking as that of holocellulose, with *M. sapientum* having 55.33%, *M. paradisiaca* 55.00%, and *T. diversifolia* 54.00%. The high content of cellulose in the *Musa* species could be attributed to the fact that the inflorescence flower stems from where the cellulose was extracted acts as a seed/fruit carrier, thus has the need for strong fibre to be able to carry the banana and plantain seed until maturity, and this need is met by storing up more cellulose. Other researchers have also reported similar results, while working on non-woody biomass. Mokhtar *et al.* (2005) in their research work reported 57.20%, while Ogunsile *et al.* (2006), 53.07%, 48.01%, and 40.80% respectively for mid-rib, pseudostem, and stalk of *Musa paradisiaca*. Eroglu *et al.* (1992) reported 37.4% for *Tithonia diversifolia* stalk, whereas

Gumuskaya and Usta (2006), reported 63.77% for hemp. *Eucalyptus* has 50.17% (Ayata 2008).

Evaluation of the lignin showed that *T. diversifolia* had the highest value for acid-soluble lignin, but had the second highest value for acid-insoluble lignin, while *M. sapientum* ranked second for acid-soluble lignin, but third for acid-insoluble lignin. *M. paradisiaca* had the least value for acid-soluble lignin, but the highest for acid-insoluble lignin. The lignin values obtained in this work were in the range of values that had been reported for non-woody plants acid soluble lignin. Ates *et al.* (2008), reported 20.5% for *P. elongata*. Others have reported related data for *Eucalyptus* 23.30% (Ayata, 2008), bamboo 24.5% (Deniz and Ates 2002), coniferous woods 25 to 32% (Eroglu 1998), and bagasse 23 to 32% (Rowell 1997).

Pulping

Some properties obtained from the analysis of pulp materials using combination of soda-anthraquinone and ethanol pulping liquor, are presented in Table 2.

Table 2. Properties of Pulp Sample

| Parameter | Sample | | |
|----------------------|-----------------------|-------------------------|------------------------------|
| | <i>Musa sapientum</i> | <i>Musa paradisiaca</i> | <i>Tithonia diversifolia</i> |
| Yield (%) | 69.80 | 69.61 | 68.78 |
| Moisture content (%) | 5.00a±0.33 | 5.11a±0.19 | 5.22a±0.39 |
| Ash (%) | 3.10a±0.00 | 3.20a,b±0.01 | 3.37b±0.15 |
| Silica (%) | 1.62a±0.01 | 3.20a,b±0.01 | 1.83b±0.01 |
| Kappa number | 16.23b±0.01 | 16.16b±0.06 | 13.28a±0.69 |

Values are means of three replicate ± standard deviation. Column means followed by different letters are significantly different at P < 0.05

Table 2 indicates that *Musa sapientum* exhibited the highest yield of 69.80%, followed by *Musa paradisiaca* with 69.61% and lastly by *Tithonia diversifolia* 68.78%. The results were higher than those reported for mid-rib, pseudo-stem, and stalk of *Musa sapientum* using Soda pulping (Ogunsile *et al.* 2006), but compared favorably with the 55.2 to 69.1% yield range for jute fiber, jute cutting, and jute caddis under various pulping conditions using soda and anthraquinone mixtures as pulping liquor (Jahan *et al.* 2007). The relatively high yield obtained in this study could be attributed to the addition of ethanol and anthraquinone, because Shatalov and Pereira (2002), reported that increments in ethanol addition from 20 to 40% to soda pulping could raise the yield from 44.00 to 47.60%, while raising ethanol addition from 40 to 60% could raise yield from 47.60 to 48.90%. The moisture, ash, and silica content of *Musa sapientum* and *Musa paradisiaca* were lower than that of *Tithonia diversifolia*, which on the other hand showed a lower kappa number. The high ash and silica content of *Tithonia diversifolia* has been attributed to its grass nature (Jones and Handrick 1967). The higher kappa number in the *Musa* species could be a result of the fruit-bearing character of their inflorescence stalks, which might have built in much lignin as plant glue, which in turn assisted the toughness of the inflorescence stalks for seed-carrying capacity.

Bleaching

Bleaching, which is primarily used for purification of cellulose, had the results presented in Table 3.

Table 3. Properties of Bleached Pulp

| Parameter | Sample | | |
|--------------------------|---------------------------|--------------------------|------------------------------|
| | <i>Musa sapientum</i> | <i>Musa paradisiaca</i> | <i>Tithonia diversifolia</i> |
| Yield (%) | 61.63 | 60.70 | 60.24 |
| Moisture content (%) | 4.89 ^a ±0.19 | 4.78 ^a ±0.19 | 4.78 ^a ±.18 |
| Ash (%) | 1.03 ^a ±0.06 | 1.10 ^a ±0.04 | 1.03 ^a ±0.06 |
| Silica (%) | 0.06 ^{a,b} ±0.06 | 0.57 ^a ±0.05 | 0.77 ^b ±0.02 |
| Kappa number | 1.20 ^c ±0.02 | 1.55 ^b ±0.15 | 3.46 ^a ±0.07 |
| Beta-cellulose (%) | 2.98 ^a ±0.00 | 2.25 ^b ±0.02 | 2.20 ^c ±0.04 |
| Gamma-cellulose (%) | 4.40 ^a ±0.04 | 4.44 ^a ±0.02 | 4.22 ^b ±0.01 |
| Alpha-cellulose (%) | 54.26 ^c ±0.02 | 54.01 ^b ±0.01 | 53.79 ^a ±0.02 |
| Viscosity (cP) | 5.67 ^b ±0.22 | 5.31 ^a ±0.13 | 5.89 ^c ±0.11 |
| Degree of Polymerization | 944.33 ^b ±39 | 878.23 ^c ±23 | 982.00 ^a ±18 |
| Molecular Weight (g/mol) | 153000 ^b ±45 | 142333 ^c ±34 | 159000 ^a ±30 |

Values are means of three replicate ± standard deviation. Column means followed by different letters are significantly different at P < 0.05

Although all the samples had at least 60% yield, the values were lower than those obtained for the corresponding unbleached pulp samples. This was expected, since bleaching will remove some residual lignin and other oxidizable compounds. The bleaching action also tended to decrease the content of silica (as a component of ash) and kappa number of bleached sample as compared with the corresponding unbleached pulp samples. The reduction in these values could be attributed to the fact that the removed lignin and other oxidizable compounds might have contained both ash and silica, and the washing and squeezing action during bleaching would have also caused solubilization of ash, silica, and lignin. Alpha-cellulose content was highest in *Musa sapientum* (54.26%), followed by *Musa paradisiaca* (54.01%) and 53.79% for *Tithonia diversifolia*. The high gamma-cellulose value of the *Musa* species may be because those species are carbohydrate producers, which would have had lower molecular weight sugar in the form of hemicellulose. The viscosity value, which is an indication of degree of degradation of cellulose (Jahan *et al.* 2007) during pulping and bleaching, shows that *Tithonia diversifolia* had more resistance to chemical degradation. This can be partly attributed to a high cellulose molecular mass, *i.e.* its high degree of polymerization.

Alpha-Cellulose

The properties obtained for the alpha-cellulose are presented in Table 4.

Table 4. Properties of Alpha-Cellulose

| Parameter | Sample | | |
|--------------------------|-------------------------|-------------------------|------------------------------|
| | <i>Musa sapientum</i> | <i>Musa paradisiaca</i> | <i>Tithonia diversifolia</i> |
| Moisture (%) | 4.47 ^a ±0.06 | 4.62 ^c ±0.10 | 4.17 ^a ±0.06 |
| Ash (%) | 0.53 ^b ±0.06 | 0.53 ^b ±0.06 | 0.50 ^a ±0.10 |
| Silica (%) | 0.03 ^a ±0.00 | 0.03 ^a ±0.00 | 0.05 ^b ±0.01 |
| Viscosity (cP) | 5.42 ^b ±0.13 | 5.02 ^a ±0.11 | 5.56 ^b ±0.14 |
| Degree of Polymerization | 898.33 ^c ±23 | 822.67 ^b ±21 | 925.00 ^a ±22 |
| Molecular Weight (g/mol) | 145333 ^c ±41 | 133333 ^b ±35 | 150000 ^a ±30 |

Values are means of three replicate ± standard deviation. Column means followed by different letters are significantly different at P < 0.05

From Table 4, sample treatments pulping, bleaching, and isolation of alpha-cellulose using 17.5% NaOH had considerable effects on the properties of the alpha-

cellulose. The ashes, silica, viscosity, degree of polymerization, and molecular weight as well as moisture content of the entire sample were greatly reduced by the squeezing involved in those treatments.

Microcrystalline Cellulose

The results of the physicochemical properties of the prepared microcrystalline cellulose are shown in Table 5.

Table 5. Physicochemical Properties of the Prepared Microcrystalline Cellulose

| Parameter | Sample | | |
|--|---------------------------|---------------------------|------------------------------|
| | <i>Musa sapientum</i> | <i>Musa paradisiaca</i> | <i>Tithonia diversifolia</i> |
| Yield (%) | 85.78 | 84.95 | 82.56 |
| Starch and dextrin | Nil | Nil | Nil |
| Solubility | Complete and no residue | Complete and no residue | Complete and no residue |
| Identification | Violet-blue | Violet-blue | Violet-blue |
| pH | 6.72 ^b ±0.03 | 6.68 ^b ±0.03 | 6.36 ^a ±0.03 |
| Loss on drying (%) | 4.90 ^b ±0.04 | 4.91 ^b ±0.03 | 4.62 ^a ±0.12 |
| Ash (%) | 0.02 ^a ±0.00 | 0.03 ^b ±0.00 | 0.02 ^a ±0.02 |
| Moisture sorption (%) | 14.31 ^b ±0.22 | 13.91 ^a ±0.07 | 14.60 ^c ±0.04 |
| Swelling capacity (%) | 37.80 ^b ±0.10 | 37.27 ^a ±0.24 | 41.10 ^c ±0.26 |
| True density (g/cm ³) | 1.49 ^b ±0.01 | 1.44 ^a ±0.00 | 1.53 ^c ±0.00 |
| Bulk density (g/cm ³) | 0.25 ^a ±0.00 | 0.26 ^a ±0.00 | 0.27 ^a ±0.00 |
| Tapped density (g/cm ³) | 0.38 ^a ±0.00 | 0.38 ^a ±0.00 | 0.40 ^b ±0.00 |
| Angle of repose (°) | 48.00 ^b ±0.23 | 46.97 ^b ±0.41 | 43.57 ^a ±0.78 |
| Hausner index | 1.48 ^c ±0.01 | 1.42 ^a ±0.00 | 1.46 ^b ±0.00 |
| Carr's compressibility index | 32.47 ^c ± 0.01 | 29.61 ^a ± 0.09 | 31.35 ^b ± 0.09 |
| Flow time (g/s) | 1.67 ^a ±0.09 | 1.76 ^b ±0.00 | 3.2 ^b ±0.17 |
| Porosity (%) | 82.90 ^c ±0.00 | 81.63 ^a ±0.15 | 82.23 ^b ±0.06 |
| Viscosity (cP) | 2.95 ^b ±0.33 | 3.09 ^c ±0.16 | 2.84 ^a ±0.22 |
| Degree of polymerization (X10 ²) | 3.32 ^b ±0.95 | 3.75 ^c ±0.45 | 3.01 ^a ±0.64 |
| Molecular weight (x10 ⁴) g/mol | 5.38 ^b ±1.54 | 6.08 ^c ±0.74 | 4.88 ^a ±1.04 |

Values are means of three replicate ± standard deviation. Column means followed by different letters are significantly different at P < 0.05

From the identification test, it was observed that the microcrystalline cellulose was soluble in an ammoniacal solution of copper teteramine in which it dissolved completely. It exhibited a violet-blue colouration with zinc chloride, confirming its composition as microcrystalline cellulose. All the materials were white in colour, crystalline powder, tasteless, and odourless. All the above results were in agreement with the specification of British Pharmacopeia, 2004.

The percentage yield showed that *Musa sapientum* had the highest value of 85.78, followed by 84.95 of *Musa paradisiaca*, and 82.56 of *Tithonia diversifolia*. This followed the pattern of their alpha-cellulose. Various yields of microcrystalline cellulose had been reported, including 65% reported for microcrystalline cellulose obtained from *Luffa cylindrical* (Ohwoavworhu *et al.* 2004), 67% for MCC obtained from *Cochlospermum*

planchonii (Ohwoavworhwa *et al.* 2005), 23.3% for MCC orange mesocarp (EjikemeA 2008), and 94.45% for MCC rice straw (Ilindra and Dhake 2008).

The low ash, which to some extent indicates the care taken to get the material to a good purity level and the loss on drying, which could be an indication of suitability of the material for usage as diluent in the formulation of hydrolysate drugs, were all within <0.05% for ash and 6 % loss on drying, as recommended by British Pharmacopeia (2004).

The pH values obtained in this study were lower than the 7.6 value reported by Ohwoavworhwa and Adalakun (2005), and were within the acceptable limit between 5 and 7 recommended by United States Pharmacopeia (2006).

The moisture sensitivity of the materials was assessed by their moisture sorption. *Tithonia diversifolia* had the highest value and *Musa sapientum* had the lowest. These values were less than 22.8 and 16.6 reported for CP-MCC and Avicel PH101 (Ohwoavworhwa and Adalakun 2005). The low moisture sorption could be suggestive that those MCC were of low amorphous cellulose content, since the crystalline portion of cellulose does not absorb as much water as the amorphous portion (Stamm 1964). This low amorphous content could be confirmed from the ¹³C NMR spectral obtained for those samples from Figs. 4, 5, and 6. On the other hand, the swelling capacity suggested that *Tithonia diversifolia* might have higher amorphous cellulose content than the rest, having recorded the highest swelling percentage of 41.10.

The relatively high true density was indicative of the crystalline nature of the prepared MCC (Stamm 1964). The bulk density with lowest values of 0.25 for *Musa sapientum* and 0.27 for *Tithonia diversifolia*, were between 0.25 and 0.31 for MCC from luffa and Avicel PH101 (Ohwoavworhwa *et al.* 2004).

The flow properties of pharmaceutical excipients are of major concern with respect to the handling and compaction of the microcrystalline cellulose. The angle of repose, Hausner index, and Carr's compressibility index are used for indirect measurement of powder flowability (Staniforth 1996). An angle of repose of up to 40° indicates reasonable flow, while greater than 50° means poor flow or the absence of flow (Bhimte and Tayade, 2007). A Carr's index value below 16 indicates good flowability, above 20 indicates not free flowing, while above 35 indicates cohesiveness (Staniforth 1996; Bhimte and Tayade 2007). A Hausner ratio greater than 1.25 indicates poor flow. Therefore, the flowability results of all the prepared MCC showed that all had poor flow properties. Thus, addition of a glidant would be needed when using these materials in solid dosage production processes. The flowability results recorded here was not in isolation, as other authors had reported similar values for various prepared MCC (Ohwoavworhwa and Adalakun 2005; Bhimte and Tayade 2007; Ejikeme 2008).

The viscosity, degree of polymerization, and molecular weight of the prepared MCC were drastically reduced compared with the values of their alpha-cellulose. This could be attributed to the hydrolytic cleavage of the 1-4 glycosidic linkages of the cellulose, thereby reducing the cellulose chain length, which directly reduced the viscosity of the cellulose. Effects of the reduction in cellulose chain length can be seen when comparing the SEM of the alpha-cellulose and that of their produced microcrystalline cellulose. The viscosity and degree of polymerization of the prepared MCC, are within the range prescribed by India standard and compared favourably with those reported by Ilindra and Dhak (2008).

Morphological Study of MCC

The fiber images of all the alpha-cellulose are presented in Fig. 1 (a, b, and c). All appeared in the form of long fibers, while the obtained microcrystalline cellulose images are presented in Fig. 1 (d, e, and f).

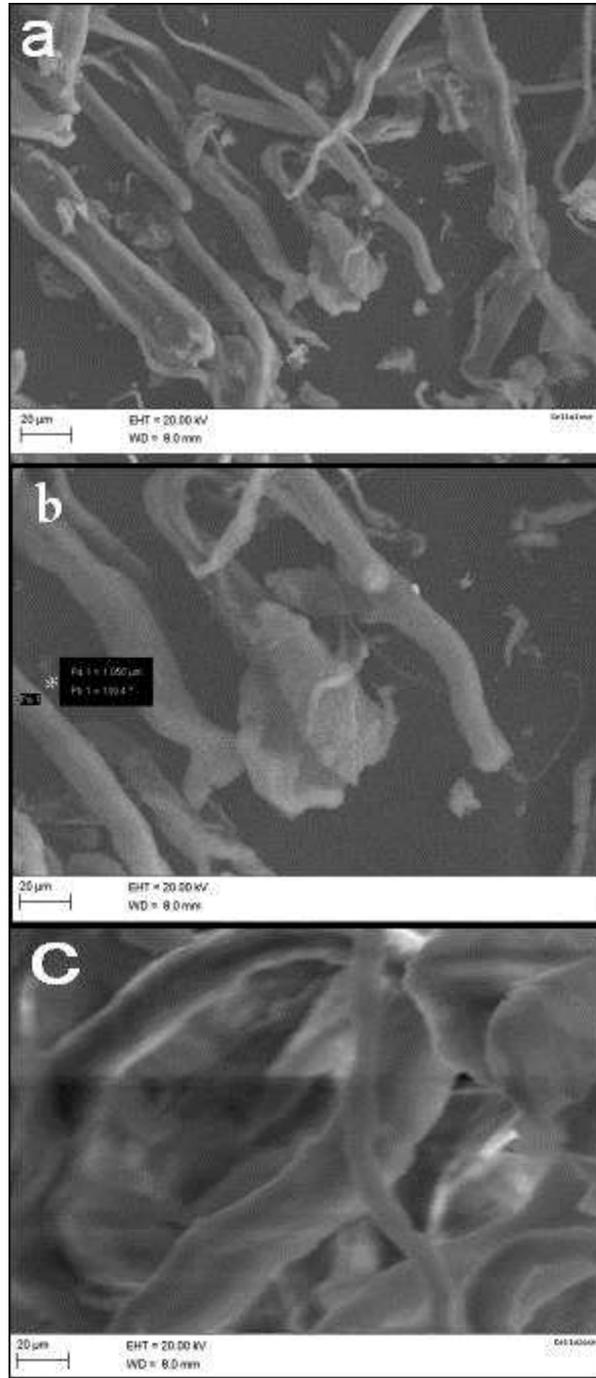


Fig. 1. SEM Photographs. a.) *Musa sapientum* alpha-cellulose; b.) *Musa paradisiaca* alpha-cellulose; c.) *Tithonia diversifolia* alpha-cellulose, while (d, e, and f) are for the MCC. d.) *Musa sapientum* MCC; e.) *Musa paradisiaca* MCC and f.) *Tithonia diversifolia* MCC.

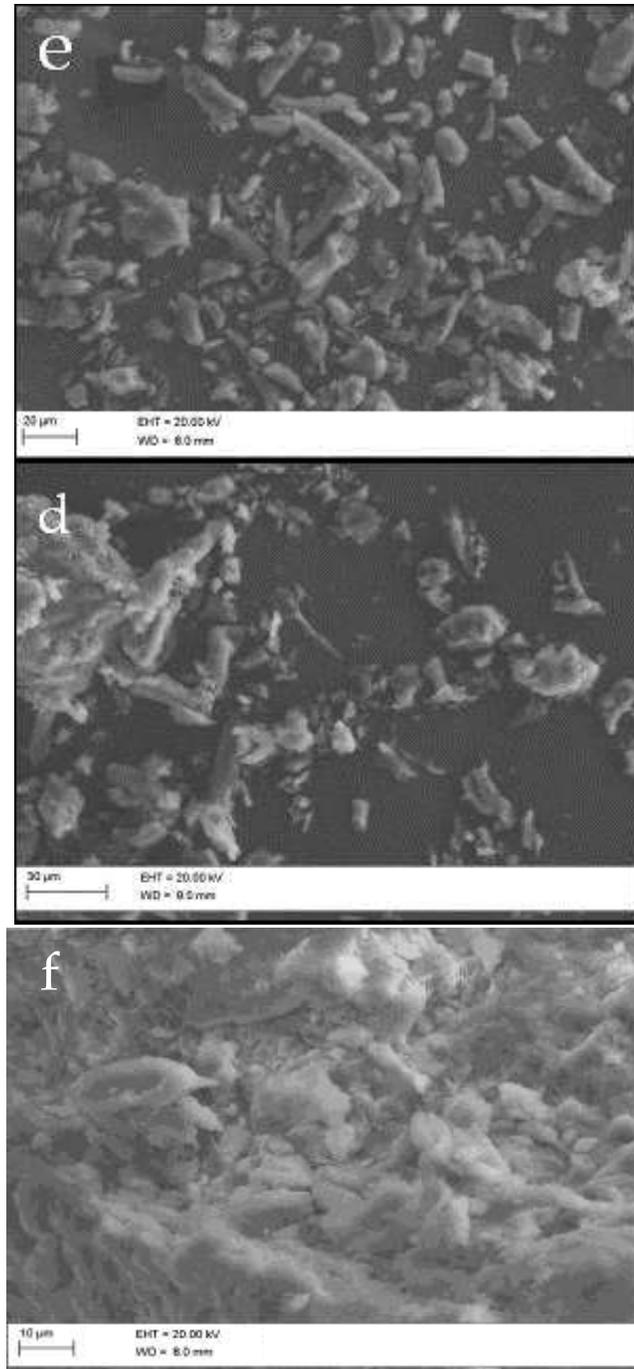


Fig. 1. SEM Photographs. a.) *Musa sapientum* alpha-cellulose; b.) *Musa paradisiaca* alpha-cellulose; c.) *Tithonia diversifolia* alpha-cellulose, while (d, e, and f) are for the MCC. d.) *Musa sapientum* MCC; e.) *Musa paradisiaca* MCC and f.) *Tithonia diversifolia* MCC .

Figure 1 (a, b, and c) indicates that the alpha-cellulose fibres appeared as long fiber threads. This was in agreement with the finding of other authors that cellulose always exhibits a long fiber structure, although the length may differ (El-Sakhawy and Hassan 2006; Adel *et al.* 2010; Ibrahim *et al.* 2010; Pereira *et al.* 2011; Morgado and

Frollini 2011). The figures d through e represent SEM micrographs of MCC from *Musa* species, which revealed that during acid treatment, cellulose chains were degraded by the hydrolysis of the glycosidic bonds, leading to shortening of fiber length and formation of mixtures of non-aggregated spherical, rod-shaped, and thread-like microcrystalline cellulose. *Tithonia diversifolia* –microcrystalline SEM (f) suffered degradation of its glycosidic bonds and also had its entire fiber structure disrupted, leading to aggregate crystal packed formation. The shape of the prepared microcrystalline cellulose of *Musa* species compared well with groundnut husk–MCC (Ohwoavworhwa *et al.* 2009), cotton stalks, and rice straw prepared by El-Sakhawy and Hassan (2006). Meanwhile, an SEM of prepared *Tithonia diversifolia* microcrystalline cellulose, Avicell PH 101 MCC (El-Sakhawy and Hassan 2006), and oil palm biomass MCC-OPEFB-MCC (Wanrosli *et al.* 2011), showed some resemblance.

Fourier Transform Infrared Spectroscopy (FTIR)

The FTIR spectra of the prepared MCC are shown in Fig. 2, which reveals the major peaks.

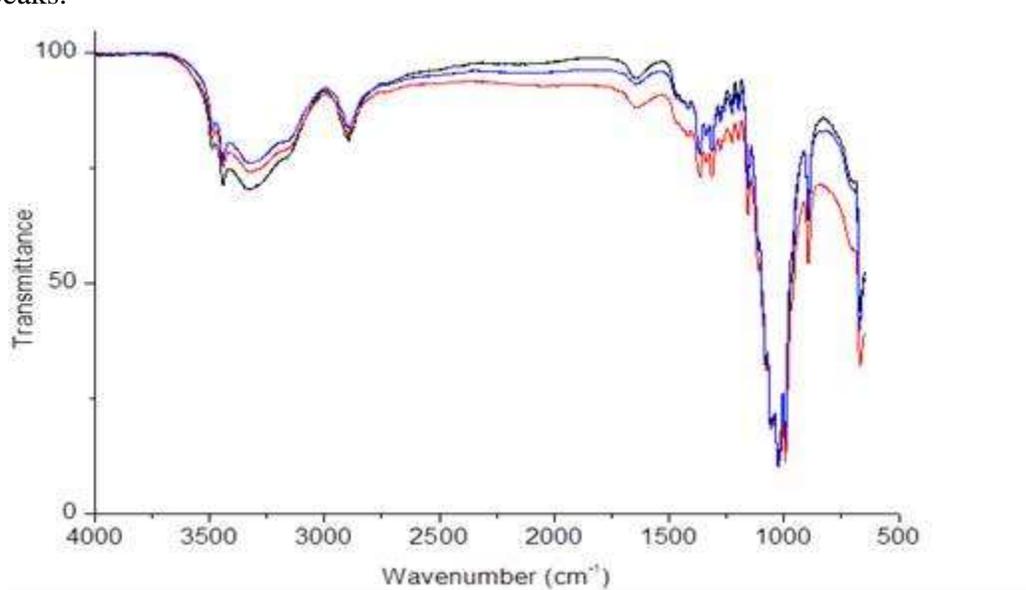


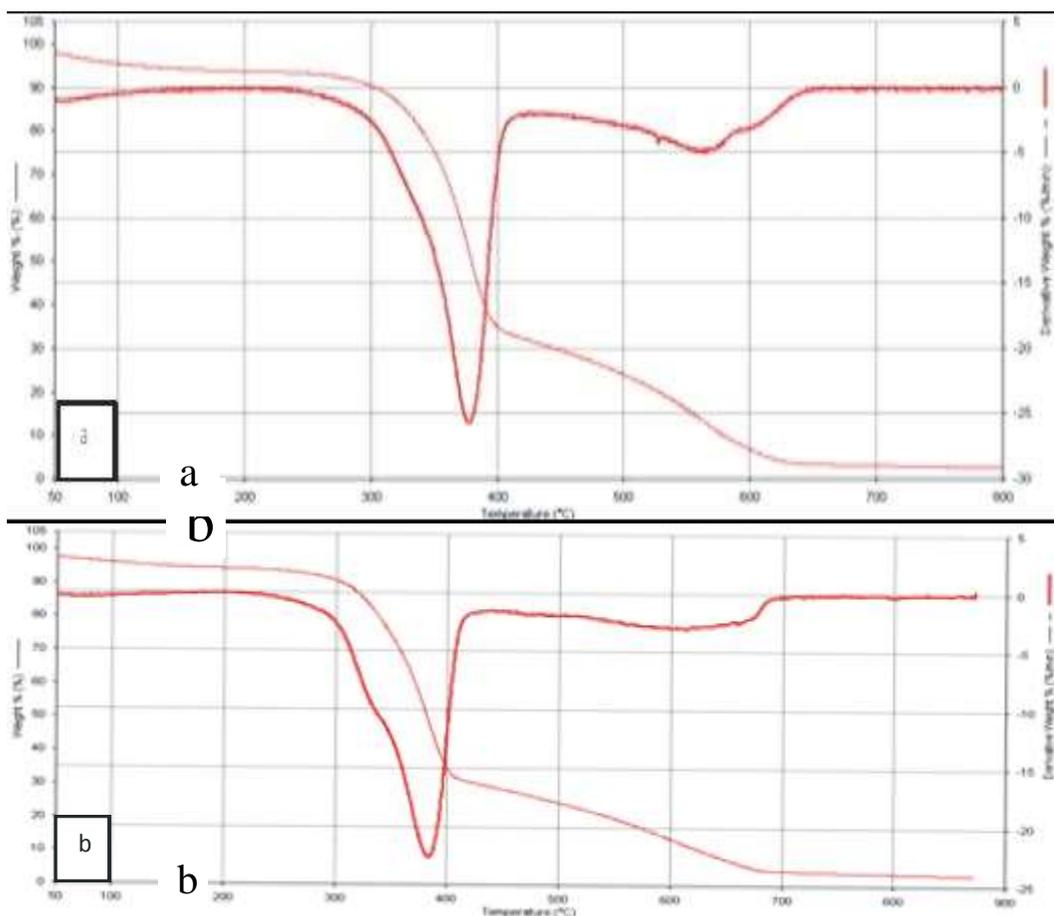
Fig. 2. IR Spectra. *Musa sapientum* -MCC (blue); *Musa paradisiaca* -MCC (black); *Tithonia diversifolia* -MCC (red)

The infrared spectra showed that there were no significant differences among all the spectra obtained and that the most representative bands of microcrystalline-cellulose can be summarized as follows: The absorption at 3486.1 to 3327.8 cm^{-1} is related to the stretching of H-bonded OH groups. The band at 2891.1 cm^{-1} is assigned to the C–H stretching of methyl and methylene groups (Liang and Marchessault 1959; Marchessault *et al.* 1962; Ivanova *et al.* 1989; Cao and Tan 2004; Pandey 2005; Wang *et al.* 2007). The band at 1630 cm^{-1} was attributed to the bending mode of the absorbed water (Cao and Tan 2004). The bands at 1417.5 cm^{-1} and 1313.1 cm^{-1} in the spectrum were assigned to the symmetric CH_2 bending and wagging (Colom *et al.* 2003; Cao and Tan 2004); the C–H bending occurs at 1364.9 cm^{-1} and 1225.8 cm^{-1} (Colom and Carrillo 2002). The absorption at 1198.4 cm^{-1} belonged to the C–O–H in-plane bending at C-6 (Marchessault *et al.* 1962; Wiley and Atalla 1987; Ivanova *et al.* 1989; Fengel and Ludwig 1991; Oh *et*

al. 2005). The absorption band at 1157.6 cm^{-1} arose from C–O–C stretching at the β -(1 \rightarrow 4)-glycosidic linkages (Hinterstoisser *et al.* 2001; Cao and Tan 2004). The peaks at 1066.2 cm^{-1} and 1025.1 cm^{-1} were indicative of C–O stretching at C-3, C–C stretching, and C–O stretching at C-6 (Wiley and Attalla 1987; Fengel and Ludwig 1991; Oh *et al.* 2005; Liu *et al.* 2006). The spectra obtained in the work and assignment of the spectral peaks were very similar to the work of other authors (Sun *et al.* 2008; Ohwoavworhwa *et al.* 2009; Nada *et al.* 2009)

Thermogravimetric Analysis of MCC

The thermal gravimetric analysis (TG) and the derivatives of thermal gravimetric curves (DTG) of samples are shown in Fig. 3.



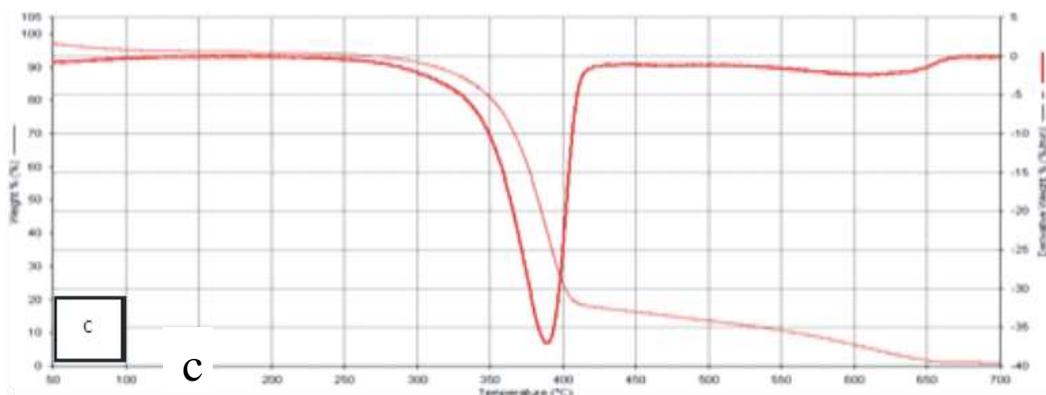


Fig. 3. TGA and DTG curves of (a) *Musa sapientum* MCC, (b) *Musa paradisiaca* MCC, and (c) *Tithonia diversifolia* MCC

The TGA and DTG curve results showed that there was small weight loss at about 100 °C, which has been attributed to loss to some retained moisture; *Musa sapientum* -MCC had 4.56%, *Musa paradisiaca*-MCC 3.68%, and *Tithonia diversifolia*-MCC had 4.59%. These results can be compared to the 4.90%, 4.91%, and 4.62% moisture contents recorded, respectively for *Musa sapientum* -MCC, *Musa paradisiaca* -MCC, and *Tithonia diversifolia* -MCC, as determined by the gravimetric method. Different authors had compared moisture content determination using TGA and gravimetric methods, and their various results showed that the same values had never been obtained (Tomasetti and Campanella 1986; Tomasetti *et al.* 1987; Tomasetti *et al.* 1989; Tomasetti *et al.* 1991).

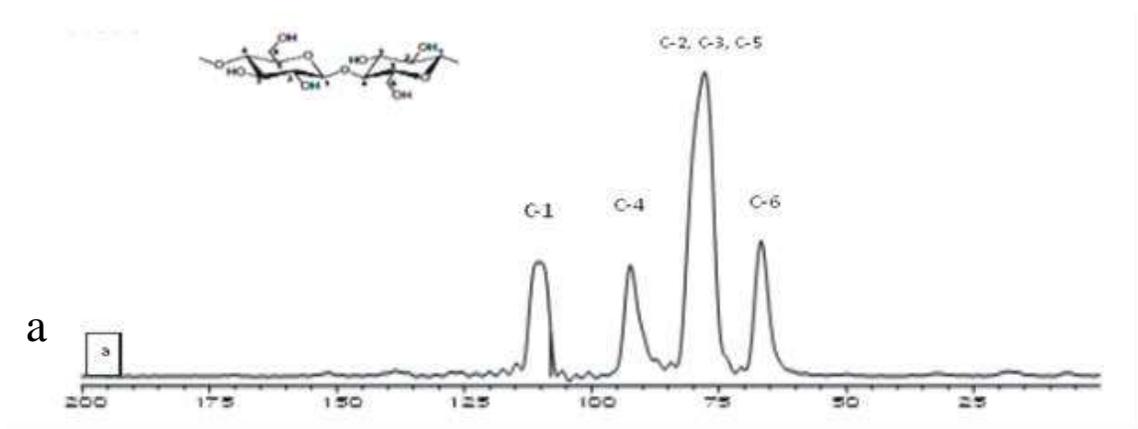
The nature of the thermogravimetric (TGA) curves of the prepared microcrystalline cellulose (MCC) were very similar to that of microcrystalline cellulose prepared from cotton rag and commercial MCC (Chauhan *et al.* 2009). The TG curves showed that the onset of decomposition of *Musa sapientum*-MCC and *Musa paradisiaca*-MCC occurred at about 330 °C, with 15.96% and 16.04% weight loss, respectively, while *Tithonia diversifolia*-MCC was about 340 °C, with 15.30% weight loss. The differences in values obtained could be attributed to differences in the morphology of those materials as confirmed by the SEM photography. The SEM results indicated that *Tithonia diversifolia* -MCC was made of granular crystal and its crystalline nature might have conferred resistance to degradation (Chauhan *et al.* 2009), such that the first derivative recorded a high value of 386 °C. The onset of degradation was attributed to the evolution of non-combustible gases such as carbon dioxide, carbon monoxide, formic acid, and acetic acid (El-Sakhawy and Hassan 2006; Nada *et al.* 2009). The weight loss of *Tithonia diversifolia* -MCC, which was the lowest even at higher temperature, shows that it had a good temperature resistance and would be a good excipient for a drug that could easily be affected by high temperature.

The second derivative stage (peak of degradation) confirmed by the DTG has been attributed to pyrolysis and evolution of combustible gases (LeVan 1989). For *Musa sapientum*-MCC, this occurred at 544 °C with 82.99% weight loss; *Musa paradisiaca*-MCC at 610 °C with 87.06% weight loss; and *Tithonia diversifolia*-MCC at 614 °C 95.03% weight loss. The different values obtained might be inferred from the SEM micrography, while *Musa* species were having mixtures of non-aggregated spherical, rod shaped, and thread like particles, whereas *Tithonia diversifolia* exhibited an aggregated crystal packed structure.

The total residue obtained at the end of the experiment showed that *Tithonia diversifolia* -MCC had 0.36% residue, *Musa sapientum* -MCC 2.09%, and MPSAE-MCC 3.35% the highest, but 0.02%, 0.03%, and 0.02% ash contents were recorded, respectively, for *Musa sapientum* -MCC, *Musa sapientum* -MCC, and *Tithonia diversifolia* -MCC by gravimetric method. The variation in char residues quantitatively suggests variation in the morphology and degree of polymerization (DP) of the microcrystalline celluloses (Calahorra *et al.* 1989). However, the residues obtained from the thermogravimetric analysis and the gravimetric method were not the same. Other authors had reported higher char residues, 7.9% for Avicel PH-101, at 500 °C (Shakeri and Staiger 2010) and 9.9% for microcrystalline cellulose obtained from oil palm biomass (Wanrosli *et al.* 2011). From the work of other authors it has been suggested that to get the correct ash content using the thermogravimetric method, the determination of the ash needs to be done under an air stream at approximately 700 °C (Tomasetti and Campanella 1986; Tomasetti *et al.* 1987; Tomasetti *et al.* 1991).

Solid-state ^{13}C NMR

The solid-state ^{13}C NMR spectra of the prepared MCC are presented in Figure 4.



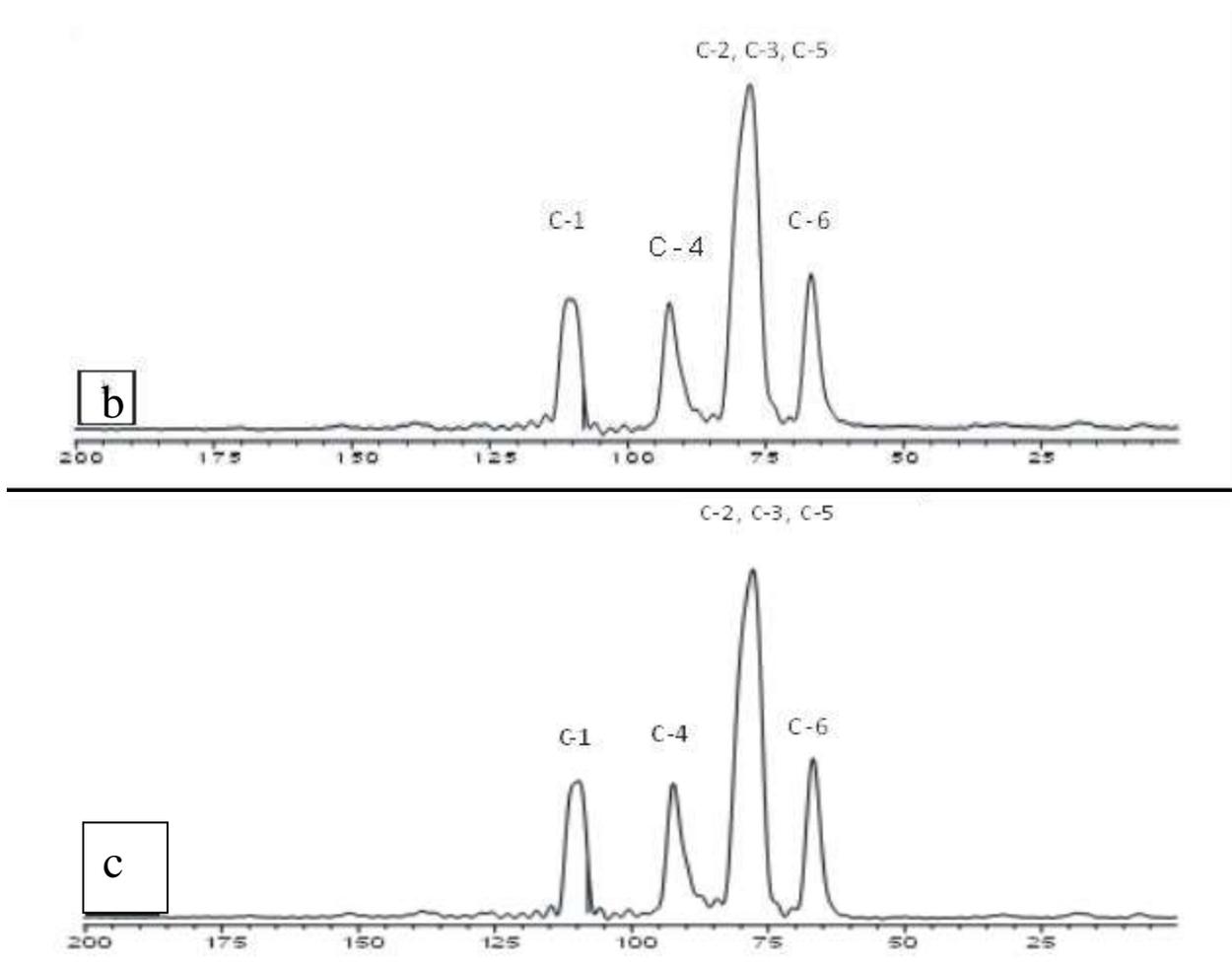


Fig. 4. Solid state ^{13}C NMR spectrum of (a) *Musa sapientum* MCC, (b) *Musa paradisiaca* MCC, and (c) *Tithonia diversifolia* MCC

As presented in Fig. 4, the obtained spectrum showed no significant difference, and the labels are the peaks assigned to the different carbon atoms of the glucopyranose repeating units in cellulose atoms. The chemical shift values indicated that the chemical shift of six carbon atoms in glucose unit of the MCC prepared was almost the same. The C-1 of the prepared MCC was assigned chemical shift of almost 110 ppm, C-4 was located approximately at 93 ppm, and C-2, 3, and 5 had a peak at about 78 ppm and spread between 75 and 83 ppm.

CONCLUSIONS

1. It is possible to obtain alpha-cellulose with good properties from various waste biomasses, thus encouraging environmental waste management.
2. Microcrystalline cellulose of relatively good yield was successfully prepared from alpha-cellulose of *Tithonia diversifolia*, inflorescence stems of *Musa sapientum*, and *Musa paradisiaca*.

3. The prepared microcrystalline cellulose, despite the poor flow properties which could be solved by addition of a glidant when use to make a solid dosage, were able to meet many of the requirement specified by British Pharmacopoeia for microcrystalline cellulose.
4. TG /DTG revealed that the prepared microcrystalline cellulose would be good excipient for drug active ingredient in high temperature region.

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