Vol. 14(1), pp. 9-23, January-March 2020
DOI: 10.5897/AJPAC2019.0821
Article Number: 81D2AC963350
ISSN: 1996 - 0840

http://www.academicjournals.org/AJPAC

African Journal of Pure and Applied Chemistry

Full Length Research Paper

Synthesis and characterization of carboxymethyl cellulose from *Musa paradisiaca* and *Tithonia diversifolia*

Alabi F. M.[1*], Lajide L.[1], Ajayi O. O.[1], Adebayo A. O.[1], Emmanuel S.[2] and Fadeyi A. E.[2]

[1]Department of Chemistry, Federal University of Technology, P. M. B. 704, Akure, Ondo State, Nigeria.
[2]Chemistry Advance Research Centre, Sheda Science and Technology Complex, P. M. B. 186, Garki, Abuja, Nigeria.

Received 4 November 2019; Accepted 11 Macrh 2020

Cellulose is the most abundant biomass in nature with properties that have enabled its application in different industrial processes. Its derivative, sodium carboxymethyl cellulose serves as an additive in food and non-food products such as desserts, detergents, paints etc. In this study, carboxymethyl cellulose (CMC) was synthesized from cellulose isolated from three ligno-cellulosic biomass, *Tithonia diversifolia* stalk (TDS), *Musa parasidiaca* stem (MPS) and unripe peel of *Musa parasidiaca* fruit (MPP). The isolation of cellulose was done by soda pulping and bleached using sodium hypochlorite, hydrogen peroxide, sodium hydroxide sequencing, followed by synthesis and purification of CMC. The physicochemical properties of the plant samples, isolated cellulose and bleached pulps including the synthesized CMC were determined. The effects of various processing stages on the properties of the cellulose and synthesized CMC were revealed in the study. CMC yield ranged from 62.57, 41.37 and 33.21% and the degree of substitution ranged from 0.33, 0.28 and 0.17 for TDS, MPS and MPP respectively. Further characterization of CMC using Fourier Transform Infrared (FTIR) confirmed the presence of major expected peaks that showed differences in terms of carboxymethyl substitution as compared to that of commercial CMC. The study revealed the potential of these plants for production of industrial grade CMC.

Key words: Lignocellulosic biomass, cellulose, carboxymethyl cellulose, soda pulping, bleaching, etherification, *Tithonia diversifolia*, *Musa paradisiaca.*

INTRODUCTION

Unutilized and underutilized agricultural plants and waste are a major environmental challenge in Nigeria because of pollution and its attendant health risk. However, most of these agricultural plants and waste are sources of lignocellulose biomass which can be converted to useful industrial raw materials (Oluwasina et al*.,* 2014; Fang et al*.,* 2016). With the current diversification policy of the government from the petroleum to the agricultural resources, vast tonnes of agricultural waste are disposed of indiscriminately and others are burnt leading to the destruction of critical infrastructures such as transformers, electricity poles, electricity sub-stations and also causing environmental pollution and its attendant health factors.

*Corresponding author. E-mail: alabifortune72@gmail.com. Tel: +2348037103284.

Some of these unutilized and underutilized agricultural waste and plants include rice husk, plantain stem and peel, corn cob, sorghum stalk, groundnut husk, Mexican sunflower, etc. These biomasses are rich sources of lignocellulose materials which could be converted to wealth through various processing techniques into useful bio-based raw materials such as cellulose, gum, polymer, carboxymethyl cellulose etc.

Carboxymethyl cellulose is one of the major derivatives of cellulose and best renewable resource available to mankind that has received a lot of attention by researchers. Major known sources of cellulose for CMC production are wood and cotton, but researchers have discovered many other sources such as Palm kernel cake (Huang et al*.,* 2017), *Tithonia diversifolia* (Oluwasina et al*.,* 2014), water hyacinth (Saputra et al*.,* 2014), pod husk of cacao (Hutomo et al*.,* 2012), banana waste (Arafat et al*.,* 2008), *Musa paradisiaca* mid-rib (Ogunsile et al*.,* 2006) and banana pseudo stem (Adinugraha et al*.,* 2005). However, there are insufficient data on the use of unripe *M. paradisiaca* peel and fruit stem in CMC production. Most studies on *M. paradisiaca* peel waste have been centered on its nutritional and medicinal properties (Akubor and Ishiwu 2013; Mohammed and Saleha, 2011; Abbas et al*.,* 2015; Akinsanmi et al*.,* 2015).

Plantain (*Musa paradisiaca*) is an evergreen tropical monoherbacious plant that belongs to the musaceae family. It is reported that over 2.11 million metric tonnes of plantain are produced and consumed in Nigeria annually (Arisa et al*.,* 2013) because it is regarded as a staple food by most families. This huge volume produced and consumed also results in the generation of large volume of waste from various parts of the plant such as the pseudo stem, fruit stem, peel and leaves. Adinugraha et al*.* (2005) synthesized CMC from banana pseudo stem with a degree of substitution of 0.75, viscosity of 4033 Cps, purity of 98.63% and a crystallinity of 38.33%. Plantain peel which is regarded as a waste very often discarded has been reported to be rich in minerals and vitamins (Arun et al*.,* 2015). Several potential applications of plantain waste are in the production of biogas, local soap, starch, bio-plastics, etc (Uhuegbu and Onuorah, 2014; Rana et al*.,* 2018; Akinyele and Agbro, 2007; Padam et al*.,* 2014).

Tithonia diversifolia, commonly known as tree marigold or Mexican sunflower is an underutilized, herbaceous flowering plant of the Asteracea family. Though it is native to South and Central America but has now been naturalized in Asia and Africa. Researchers have reported of its several uses such as in animal feed, insecticide, poultry feed, compost and medicinal uses (Olayinka et al, 2015; Akanbi et al., 2007; Buragohain, 2016; Drechsel and Reck, 1998; Jama et al*.*, 2000, Chukwuka and Ojo, 2014; Bisht and Joshi, 2017; Tona et al., 1998). Recent studies have shown the potentials of its stalk as a source of valuable industrial chemicals for the production of lignin-base resins (Oluwasina et al*.,* 2014; Friday and Muhammad, 2015) including Microcrystalline cellulose for drug formulation (Oluwasina et al*.,* 2014) and other derivatives for various industrial applications (Otoide et al*.,* 2018).

As a result of the potentials of these agricultural plant biomasses as sources of industrial raw materials for the manufacturing sector, this study therefore aims to provide more insight on the physicochemical properties of cellulose and carboxymethylcellulose derivable from them. It will also serve as a guide for further research on these unutilized and underutilized agricultural plants and waste.

MATERIALS AND METHODS

Sunflower stalks were obtained from the Asokoro Military Cantonment area of Abuja, Nigeria in August 2016. Plantain stem and unripe peels were obtained from traders in a local market in Nasarawa State, Nigeria in October, 2016. The plant materials 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 sodium hydroxide (BDH), acetic acid (Sigma Aldrich), ethanol (95% BDH), $NaClO_2$, Monochloroacetic acid.

Sunflower stalks were harvested 10 cm above ground level. The samples were cleaned to remove dirt and contaminants. The sunflower stalk and plantain stem were debarked using knife. All plant samples were then cut into chips of about 2-3 cm and sundried for 14 days. The dried samples were milled, screened using a 325 µm screen sieve, and stored in a ziploc polyethylene bag for subsequent analysis.

Physicochemical analysis of samples

The samples were analyzed using the following TAPPI and ASTM standard methods: Moisture content (T550om-03); Ash (T221om-93); Silica (T244om-93); water soluble matters (T207om-87); 1% sodium hydroxide (T212om-02); Ethanol-Benzene extractive (T204om-97); Holocellulose (ASTM D1104-56); α-cellulose (ASTM D1103-60); Kappa no (T236om-99).

Pulping

Pulping experiment was conducted in a 25-litre thermostatically controlled autoclave digester following the methods of Oluwasina et al*.* (2014) and Hutomo et al*.* (2012). The plant samples (200 g each) were initially pretreated with 1 L of water to de-lignify the materials for 30 min at 110°C. The pretreated plant materials were further digested using the alkaline sulphite pulping method with an alkaline active charge of 18% (w/w) NaOH. The pulping conditions of the plant sample materials are as follows: liquor-to-plant ratio for all the cooking was 20:1 (v/w), temperature 110°C, pressure 15 psi and cooking time of 120 min. After digestion, the pulp obtained through filtration was thoroughly washed 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.

Bleaching procedure

The bleaching process was conducted using the method described

by Oluwasina et al. (2014) but with a slight modification. In the bleaching procedure, 20 g of the air dried samples were placed in a 2 L Erlenmeyer flask and 500 ml of 3.5%w/v (JIK) sodium hypochlorite and 3 ml of 90%v/v acetic acid were added. The flask was covered with a watch glass and the mixture heated in water bath at 70°C for 1 h with intermittent stirring. After 1 h treatment, the sample was drained and 500 ml of hydrogen peroxide added and heated in water at 60°C for 1 h with intermittent stirring. After treatment the sample was drained followed by extraction with 500 ml of 5%w/v NaOH conducted at 70°C for 1 h. The sample was washed free of alkali after extraction using distilled water. This process sequence was conducted thrice but the sample was not washed after the third time but rather 500 ml of 3.5%w/v (JIK) sodium hypochlorite, 3 ml acetic acid and 500 ml of hydrogen peroxide were added and allowed to stand undisturbed for another 1 h. The power source was put off after final 1 h and the experimental set up left for 24 h. The pulp was filtered and washed to obtain bleached cellulose pulp of pH 7 measured using a pH meter, oven-dried to constant weight and characterized using standard method.

Synthesis of sodium carboxymethyl cellulose

The synthesis of carboxymethyl cellulose was conducted according to the method described by Ambjornsson et al. (2013) but with a slight modification. A sample of 5 g of oven dried cellulose pulp was placed in a 500 ml flask with 64 mL of ethanol and 5.8 ml of distilled water and covered with aluminium foil to avoid evaporation. The flask was placed in an automated mechanical shaker rotating at 120 rpm and at a temperature of 20±5°C. After 15 min, a solution of 6.7 g of NaOH and 10.2 ml of distilled water was added to the mixture maintained under the mechanical agitation for 2 h. In the next step, a solution of 7.3 ml of 87% ethanol and 7.3 g of monchloroacetic acid was added to the reaction mixture and the temperature of the mechanical shaker gradually increased from 20 to 25°C within 30 min and then maintained for 2 h at 60°C. The reaction was terminated by neutralization with the addition of 20 ml of 90% (v/v) acetic acid. The suspension was filtered and the filtrate washed repeatedly with 200 ml of 87% ethanol and 200 ml of 70% methanol, and finally washed with 250 ml of absolute methanol to remove all sodium containing by-products (NaCl and $C_2H_3NaO_3$), until the filtrate gave negative response to silver nitrate test of chloride. The slurry obtained was suspended in acetone, stirred for 30 min and dried in an oven at 50±5°C for 12 h to constant weight. The percentage yield of carboxymethyl cellulose synthezised was calculated based on the weight of oven dried sample.

Determination of the properties of carboxymethyl cellulose

The properties such as ash and moisture contents were determined by T221om-93 and T550om-03, pulp viscosity (TAPPI T230om-99). pH by method described by JECFA (1998), bulk and tapped densities were determined by a modification of the method of Kumar and Kothari (1999), true density by method of Itiola (1991), swelling capacity by Iwuagwu and Okoli (1992), degree of substitution by ASTM (2005) and Sodium Chloride content by ASTM (1995)

Pulp viscosity

The kinematic viscosity of the CMC was determined using a modified capillary viscometer method (TAPPI T230om-99). The viscosity (centipoise) was calculated using the following formulae:

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

Where $[\eta]$ is the intrinsic viscosity (cP), η_{sp} is the specific viscosity $\left[\frac{\eta\text{solution} - \eta\text{solvent}}{\eta\text{solvent}}\right]$ $\eta_{solution}$ is the product $t_{solution}$ x $\rho_{solutiont,}$ $\eta_{solvent}$ is $t_{solvent}$ x $\rho_{solvent}$, and ηr is the relative viscosity ($t_{solution}/t_{solvent}$), C is the concentration of the sample (1.052g/cm^3), $\rho_{solution}$ is the density of the solution (g/cm^3), $\rho_{solvent}$ is the density of the solvent (g/cm^3), $t_{solution}$ is the solution flow time (s) and $t_{solvent}$ is the solvent flow time (s).

Bulk density and tapped densities

The bulk and tapped densities of the CMC powder were determined by a modification of the method of Kumar and Kothari, 1991. The densities were calculated as follows,

$$\text{Bulk density} = \frac{Weight\ of\ sample\ (w)}{Volume\ of\ sample\ (Vo)}$$

$$\text{Tapped density} = \frac{Weight\ of\ sample\ (w)}{Volume\ of\ sample\ (\text{V500})}$$

Swelling capacity

The swelling capacity was determined according to the method described by Iwuagwu and Okoli, (1992) with a slight modification. The swelling capacity was calculated as:

$$S = \left[\frac{(V_s - V_t)}{V_t}\right] \times 100$$

Where: S = Percentage Swelling capacity, Vs = Volume of swollen material, Vt = Tapped volume of sample material.

True density

The true density, (D_t) of the CMC was determined by the Pycnometer method using liquid displacement technique with xylene as the immersion fluid (Itiola, 1991) and the sample density calculated as follows:

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

Where: w = weight of the sample, SG = specific gravity of the solvent (xylene), a = weight of the bottle + solvent, b = weight of the bottle + solvent + sample.

Degree of substitution

The standard method (ASTM, 2005) was used to determine the degree of substitution of the prepared CMC samples. The Degree of Substitution (DS) was calculated as follows:

$$DS = \left[\frac{(0.162 \text{ x A})}{1 - (0.058 \text{ x A}}\right]$$

Table 1. Physicochemical properties of raw materials.

Parameter (%)	*M. paradisiaca* (stalk)	*M. paradisiaca* (unripe peel)	*T. diversifolia* (stalk)
Moisture	4.75^{a}±0.01	6.44^{c}±0.01	4.94^{b}±0.02
Ash	6.71^{a}±0.69	11.19^{c}±0.62	8.67^{b}±0.45
Cold water solubility	29.86^{c}±0.10	28.15^{b}±0.06	16.82^{a}±0.02
Hot water solubility	37.70^{c}±0.06	30.76^{b}±0.05	19.24^{a}±0.03
Silica	3.02^{b}±0.9	3.36^{c}±0.50	2.13^{a}±0.20
1% NaOH solubility	43.61^{b}±0.06	59.42^{c}±0.06	36.93^{a}±0.01
Ethanol-Benzene extractives	2.33^{a}±0.08	6.22^{c}±0.20	2.56^{b}±0.02
Holocellulose	64.13^{a}±0.03	54.04^{b}±0.13	68.81^{c}±0.20
α-cellulose	46.03^{c}±0.20	29.17^{a}±.0.01	52.18^{b}± 0.03

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

$$A = \frac{BC - DE}{F}$$

Where, A = milli-equivalent of consumed acid per gram of specimen, B = volume of Sodium hydroxide added, C = concentration in molarity of sodium hydroxide added, D = volume of consumed hydrochloric acid, E = concentration in molarity of hydrochloric acid used, F = weight of sample used (g),

Sodium chloride content

The sodium chloride content of the synthesized CMC was determined using the standard method of ASTM, 1995 and JECFA 1998 and the NaCl content was calculated as follows:

$$NaCl\ (\%) = \left[\frac{(a \times 0.001169 \times 5)}{b}\right] \times 100$$

Where: a = ml of the silver nitrate utilized, b = dry weight of the sample (g)

The actual NaCl content was then obtained by subtracting the blank value from the sample value.

Instrumental analysis

Fourier Transform Infrared (FTIR) Spectroscopy was conducted using ThermoNicolet Avatar 370 FT-IR Spectrometer operating in the attenuated total reflection (ATR) mode (SmartPerformer, ZnSe crystal)

Statistical analysis

Data obtained in triplicate were analyzed using Duncan's Multiple Range Test (DMRT) and Analysis of Variance (ANOVA)

RESULTS

Physicochemical properties of raw materials

The results of the physicochemical properties of the plant raw materials samples are shown in Table 1 and sample of raw materials is shown in Figure 1a to c.

Holocellulose and alpha cellulose

The holocellulose content of the sample materials as shown in Table 1 indicates that *T. diversifolia* (68.81±0.20) and *M. parasidiaca* stalk (64.13±0.03) had the highest holocelluose content while *M. parasidiaca* stem (54.04±0.13) had the lowest. The alpha cellulose content of the lignocellulosic raw materials also followed the same pattern with the holocellulose. *T. diversifolia* (52.18± 0.03) had the highest holocelluose content, *M. parasidiaca* stalk (46.03±0.20, while *M. parasidiaca* stem (29.17±.0.01) had the lowest.

Ash and silica content

The ash contents of *M. paradisiaca* (stalk), *M. paradisiaca* (unripe peel), and *T. diversifolia* (stalk) ranged from 6.71 - 11.19% while the silica content ranged from 2.13 -3.36%. *T. diversifolia* (stalk) had the lowest silica content whereas the silica contents of *M. paradisiaca* (stalk), *M. paradisiaca* (unripe peel) were higher.

Cold and hot water solubility

The cold water solubility ranged from 16.82 to 29.86%. *M. paradisiaca* (Stalk) has the highest solubility when compared to *M. paradisiaca* and *T. diversifolia*. However, the hot water solubility of *M. paradisiaca* (Stalk), *M. paradisiaca* (unripe peel) and *T. diversifolia* (stalk) was 37.70, 30.76 and 19.24 % respectively.

Alkali solubility

The 1% NaOH solubility of *M. paradisiaca* (stalk), *M.*

Figure 1. Plant raw materials sample of, a) *M. paradisiaca* stem; b) *T. diversifolia*; and c) Unripe *M. paradisiaca* fruit peel.

paradisiaca (unripe peel), and *T. diversifolia* (stalk) are 43.61, 59.42 and 36.93%, respectively. *M. paradisiaca* (unripe peel) has the highest solubility when compared to the *M. paradisiaca* (stalk) and *T. diversifolia* (stalk).

Ethanol-Benzene solubility

The result indicates that *M. paradisiaca* (unripe peel) had the highest content of 6.22% followed by *T. diversifolia* (stalk) 2.56% and *M. paradisiaca* (stalk) having the lowest value of 2.33%.

Physicochemical properties of pulp cellulose samples

The results of the physicochemical properties of the pulp samples are shown in Table 2 and pulp samples are shown in Figure 2a to c.

Pulp yield

The percentage pulp yield ranged from 58.20 - 30.43%. *Tithonia diversifolia* had the highest yield of 58.20% which was followed by *Musa paradisiaca* (stalk), 37.03%, and *Musa paradisiaca* (peel), 30.43%.

Ash and silica contents

The ash and silica contents of the cellulose pulp samples ranged from 6.71 to 11.19%, with *Musa paradisiaca* (stem) the lowest at 6.71% which was followed by *T. diversifolia* with 8.67% and *M. paradisiaca* (peel) the highest with 11.19%. The silica content ranged from 2.13 to 3.36% with *T. diversifolia* recording the lowest at

Table 2. Physicochemical properties of pulp samples.

Parameter (%)	*M. paradisiaca* (stalk)	*M. paradisiaca* (unripe peel)	*T. diversifolia* (stalk)
Yield	37.03	30.43	58.20
Moisture	8.29^{c}±0.20	7.65^{b}±0.17	7.41^{a}±0.39
Ash	2.54^{a}±0.10	2.71^{b}±0.05	3.70^{c}±0.13
Silica	1.60^{b}±0.02	1.02^{a}±0.07	1.67^{c}±0.02
Kappa no	24.61^{a}±0.03	40.21^{c}±0.01	29.30^{b}±0.15

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

Figure 2. Cellulose pulp sample of, (a) *Musa paradisiaca* stem; (b) *Tithonia diversifolia*; and (c) Unripe *Musa paradisiaca* fruit peel.

2.13%, *M. paradisiaca* (stalk) 3.02% and *M. paradisiaca* (peel) 3.36%.

Table 3. Properties of bleached pulp samples.

Parameter (%)	*M. paradisiaca* (stalk)	*M. paradisiaca* (unripe peel)	*T. diversifolia* (stalk)
Yield	35.03	27.11	52.01
Moisture	7.12^{a}±0.20	6.42^{c}±0.13	6.23^{b}±0.02
Ash	1.82^{c}±0.14	2.07^{a}±0.02	3.21^{b}±0.07
Silica	1.14^{b}±0.03	0.97^{c}±0.23	1.31^{a}±0.01
Kappa no	9.46^{c}±0.01	11.06^{a}±0.11	7.15^{b}±0.06
Bulk density	0.43^{b}±0.10	0.38^{a}±0.01	0.58^{c}±0.03
Tap density	0.54c±0.10	0.47a±0.02	0.69b±0.10

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

Kappa number

The kappa number is an index of lignin content (Solange et al*.,* 2008). The result of the kappa number indicates that *M. paradisiaca* (peel) had the highest (40.21%) followed by *T. diversifolia* (29.30%) and *M. paradisiaca* (stalk) the lowest (24.61%).

Physicochemical properties of bleached pulp cellulose samples

The physicochemical properties of the bleached pulp cellulose samples are indicated in Table 3.

Yield

There was a general decrease in the yield of the bleached cellulose pulp compared to the unbleached pulp. The bleached pulp yield ranged from 27.11 - 52.01% with *T. diversifolia* recording the highest and *M. paradisiaca* (peel) the lowest.

Ash and silica contents

The ash content of the bleached cellulose pulp ranged from 1.82 to 3.21% with *M. paradisiaca* (stalk) the lowest at 1.82%, followed by *M. paradisiaca* (peel) at 2.07% and *T. diversifolia* the highest at 3.21%. The result for the silica content showed *M. paradisiaca* (peel) having the lowest at 0.97%, followed by *M. paradisiaca* (stalk) at 1.14% and *T. diversifolia* the highest at 1.31%.

Kappa number

The kappa number ranged from 7.15 to 11.06% with *T. diversifolia* the lowest at 7.15%, followed by *M. paradisiaca* (stalk) while *M. paradisiaca* (peel) recorded the highest at 11.06%.

Bulk and tap densities

The bulk and tap densities of the bleached pulp samples followed the same order in their decrease from *M. paradisiaca* (peel) to *T. diversifolia* stem to peel. The bulk densities of the bleached pulp samples were 0.38, 0.43 and 0.58% for *M. paradisiaca* (peel), *M. paradisiaca (*stalk) and *T. diversifolia* respectively. While the tap densities were 0.47, 0.54 and 0.69% for *M. paradisiaca* (peel), *M. paradisiaca* (stalk) and *T. diversifolia* respectively.

Physicochemical properties of synthesized carboxymethyl cellulose

The physicochemical properties of synthesized carboxymethyl cellulose are presented in Table 4 and samples of synthesized CMC are presented in Figure 3.

Yield

Yield is a function of the amount of materials lost during dialysis step. The yield of the synthesized CMC ranged from 33.21-62.57%, with *T. diversifolia* recording the highest, closely followed by *M. paradisiaca* stem, and *M. paradisiaca* peel recording the lowest.

pH

The pH which is a measure of the acidity or alkalinity of the CMC ranged from 6.51 - 6.74. *M. paradisiaca* had the highest pH of 6.74, closely followed by *M. paradisiaca* peel with 6.61, and *T. diversifolia* the lowest having 6.51.

Degree of substitution (DS)

The DS is one of the most important properties of CMC. It does not only influence the solubility of the CMC

Table 4. Properties of synthesized carboxymethyl cellulose.

Parameter (%)	*M. paradisiaca* (stalk)	*M. paradisiaca* (unripe peel)	*T. diversifolia* (stalk)
Yield	41.37	33.21	62.57
Moisture	9.87^{b}±0.01	11.96^{c}±0.10	7.45^{a}±0.03
Ash	1.78^{c}±0.14	1.93^{a}±0.02	2.81^{b}±0.07
pH	6.74^{b}±0.04	6.61^{c}±0.02	6.51^{a}±0.11
Bulk density	0.76^{b} ±0.01	0.65^{a} ±0.01	0.82^{c} ±0.02
Tap density	0.80^{b} ±0.02	0.73^{a} ±0.13	0.89^{c} ±0.12
True density	0.85±0.11	0.92±0.01	0.97±0.03
Swelling capacity	553.37^{c}±0.11	350.14^{b}±0.13	687.01^{a}±0.17
Sodium Chloride (%)	0.15^{b}±0.02	0.16^{c}±0.11	0.13^{a}±0.23
Viscosity (Cp)	30.47±0.51	26.25±0.31	32.17±0.07
DS	0.28^{b} ±0.14	0.17^{c} ±0.06	0.33^{a} ±0.11

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

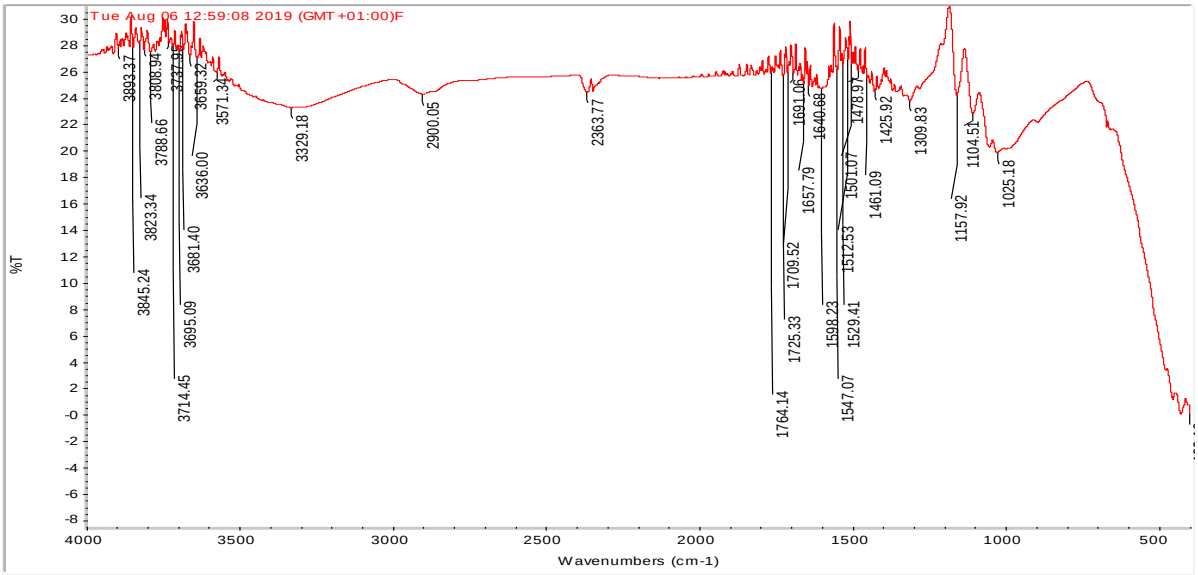

Figure 3. FT-IR of commercial carboxymethylcellulose.

molecules but also affects the solution characteristics (Barba et al., 2002). The DS of the CMC samples ranged from 0.17 to 0.33. *T. diversifolia* recorded the highest (0.33), followed by *M. paradisiaca* stem (0.28), and the least was *M. paradisiaca* unripe peel (0.17).

Viscosity

The DS also influences the viscosity, a higher DS results in better viscosity and cation exchange ability. Additional carboxyl groups provide more sites for cross-linking by multivalent cations. The viscosity of CMC samples ranged from 26 - 32%. *T. diversifolia* had the highest viscosity (32%), followed by *M. paradisiaca* stem (30%), and *M. paradisiaca* unripe peel the lowest (26%).

Swelling capacity

The swelling capacity of prepared CMC in this work ranged from 350.14 - 687.01 with *T. diversifolia* having the highest (687.01) which was followed by *M. paradisiaca* stem (553.37), and the unripe peel recording the lowest (350.14).

Sodium chloride content

The CMC produced contained 0.13, 0.15, and 0.16% for

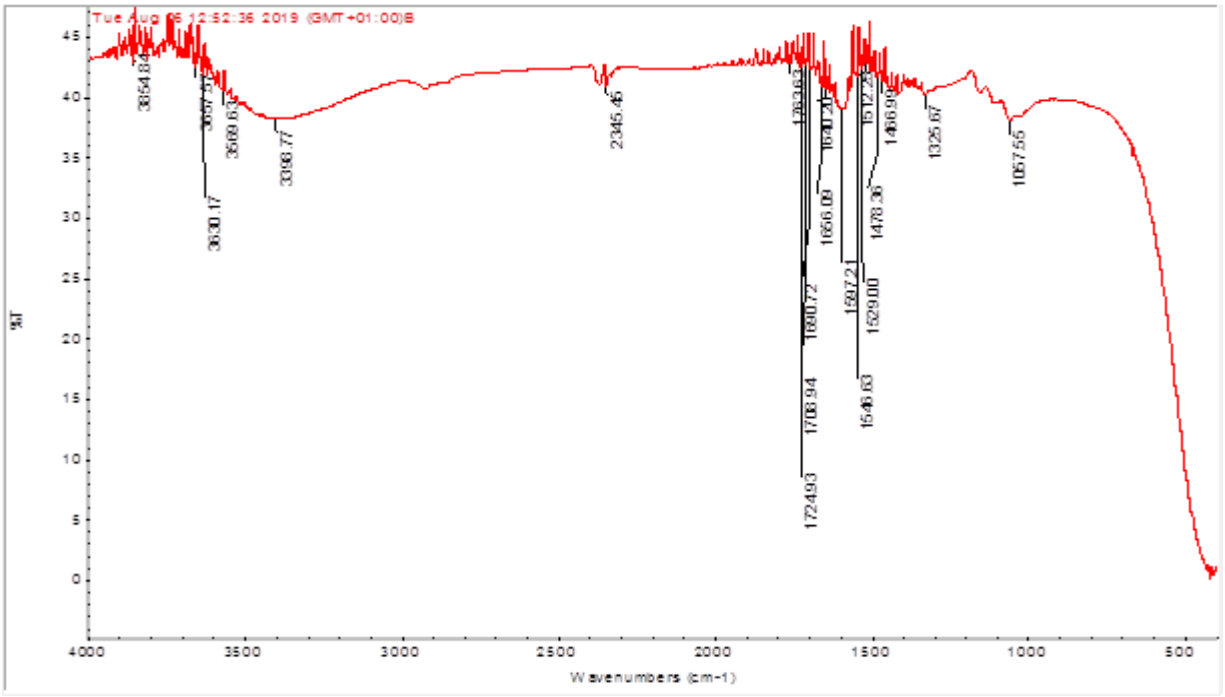

Figure 4. FTIR spectra of carboxymethyl cellulose derived from *T. diversifolia.*

T. diversifolia, M. paradisiaca stem, and unripe peel, respectively.

Bulk and tap densities

There is no much significant difference in the bulk and tapped densities as the bulk densities ranged from 0.65 - 0.82% and the tap densities ranged from 0.73 - 0.89% for CMC derived from *M. paradisiaca* peel, stem, and *T. diversifolia*, respectively. This means that *M. paradisiaca* stem and *T. diversifolia* bleached cellulose powders have better flow than *M. paradisiaca* peel.

Fourier transform infrared spectroscopy (FTIR)

Fourier transform infrared (FT-IR) spectroscopy was used to verify the successful etherification of cellulose. The FT-IR spectral of synthesized and commercial CMC are shown in Figures 3 to 6 and the spectral data analysis in Table 5.

DISCUSSION

Physicochemical properties of raw materials

Holocellulose and alpha cellulose

The percentage of holocellulose in the plant materials is in the mid-range holocellulose content. The quality of end product depends on the content of holocellulose; high content increases pulp and quality of end product (Zawawi et al*.,* 2013). The holocelluose content obtained in this research work compared well with those obtained by other researchers. Oluwasina et al. (2014) had reported 71.60% for *T. diversifolia,* 72.60 and 73.40% for *M. parasidiaca* and *M. sapentium,* respectively. Ibrahim et al*.* (2010) reported 67.80, 57.46, and 53.89% for corn cob, banana plant and cotton gin waste, respectively. The alpha cellulose content of raw materials gives an indication of pulp yield (Sezgin and Serhat 2018). Studies by other researchers have reported comparable results on non-woody lignocellulosic materials. Oluwasina et al. (2014) reported 54.00% for *T. diversifolia,* 55.00 and 55.33% for *M. parasidiaca* and *M. sapentium* respectively. Saelee et al*.* (2014) reported 44.5% for Sugarcane baggase.

Ash and silica content

Ash content represents different metal salts such as carbonates, silicates, oxalates and potassium phosphates, magnesium, calcium, iron and manganese as well as silicon. From the results in Table 1, *M. paradisiaca* (unripe peel) has the highest ash and silica content when compared with the other two lignocellulosic materials. Jaramogi et al*.* (2016) reported higher ash content (9.1%) for *M. paradisiaca* (stalk) and *T. diversifolia* (stalk), but lower than the ash content of *M.*

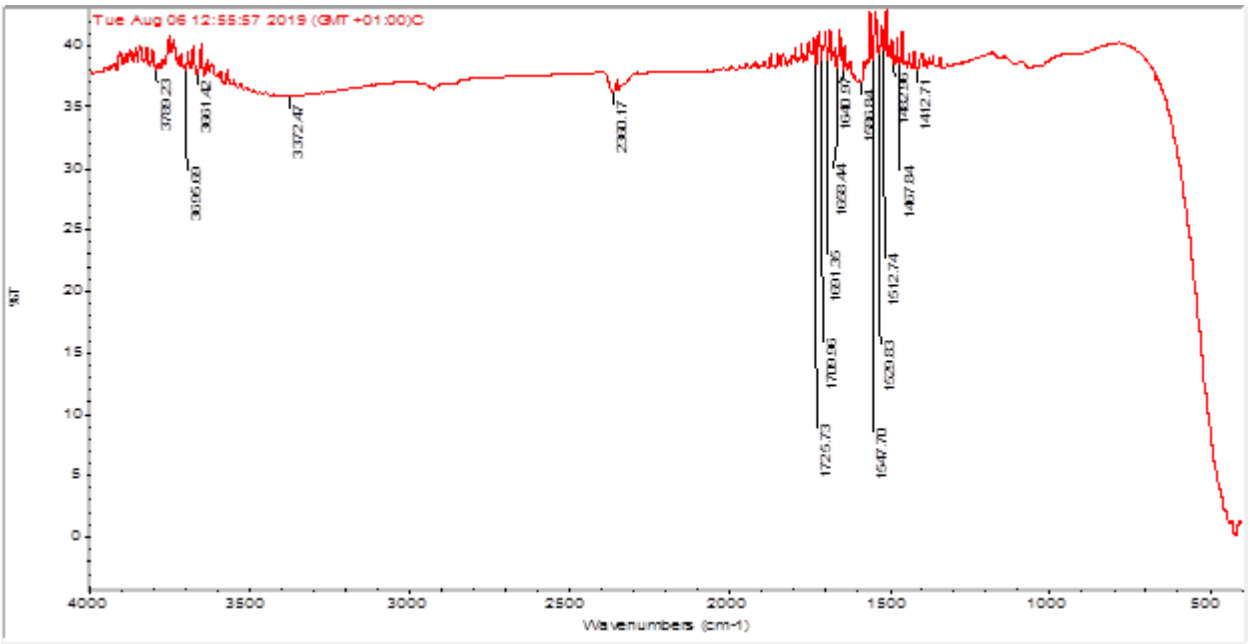

Figure 5. FTIR spectra of carboxymethyl cellulose derived from *M. paradisiaca* stem.

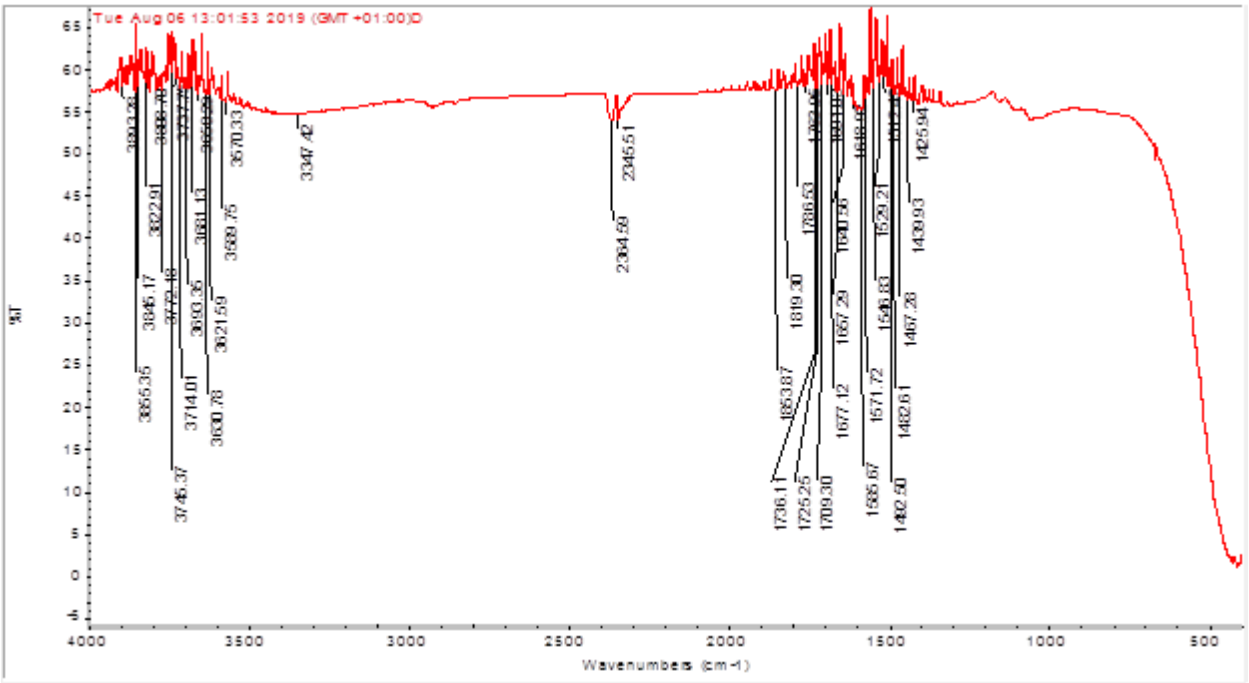

Figure 6. FTIR spectra of carboxymethyl cellulose derived from *M. paradisiaca* peel.

paradisiaca (unripe peel). The values of the ash were indicative of the presence of high mineral (especially the macrominerals) content in the lignocellulosic materials. The higher the ash content, the higher the mineral composition.

Cold and hot water solubility

From the results in Table 1, *M. paradisiaca* (Stalk) has the highest solubility when compared to *M. paradisiaca* and *T. diversifolia*. Water solubility removes a part of

Table 5. Characterization of CMC using FTIR spectral data analysis.

Commercial CMC	Major absorption band (cm^{-1})			Functional group
	T. diversifolia	*M. paradisiaca* stem	*M. paradisiaca* peel	
3329.18	3398.77	3372.47	3347.42	OH Hydroxyl group
2900.05	2345.45	2360.17	2364.59	-CH stretching of methyl and methylene
1588.23	1597.21	1588.81	1585.67	-C=O carbonyl group
1425.92	1466.99	1467.84	1467.28	$-CH_2$ bending
1309.83	1325.67	-	-	C-O ether group
1025.18	1051.56	-	-	O-C-O stretching of ether

extraneous components, such as inorganic compounds, tannins, gums, sugars and colouring matter present in the lignocellulosic plant and hot water removes, in addition, starches (Shakhes et al*.,* 2011). It can therefore be inferred that *M. paradisiaca* (Stalk) was more prone to the removal of extraneous components.

Alkali solubility

The alkali solubility of sample indicates an extent of cellulose degradation during processes and has been related to strength and other properties of the further pulp of the sample. *M. paradisiaca* (unripe peel) has the highest solubility when compared to *M. paradisiaca* (stalk) and *T. diversifolia* (stalk). This indicates that *M. paradisiaca* (unripe peel) has higher cellulose degradation than the other two lignocellulosic materials.

Ethanol-Benzene solubility

The ethanol-benzene extractive consists of soluble materials not generally considered part of the plants substance and is primarily the waxes, fats, resins and some gums as well as some water soluble substances. The result in Table 1 indicates that *M. paradisiaca* (unripe peel) had the highest ethanol-benzene solubility content. These results obtained in this study were comparable with that obtained for some non-woody plants, corn stalk, 3.5% (Barbash et al*.,* 2012), canola stalk, 2.5% (Enyati et al*.*, 2009) and cotton stalk, 2.93-3.03% (Ali et al*.,* 2001)

Physicochemical properties of cellulose

Pulp yield

The results of the yield as presented in Table 2 for *T. diversifolia*, *M. parasidiaca* stalk and unripe peel compared favourably with that reported for extracted banana waste (EBW) and waste banana fibre (WBF) using soda pulping method (Arafat et al*.,* 2018); the results ranged from 46.7 to 66.8% for EBF and 29.3 - 38.8% for WBF. The result also compared favourably with report by Ogunsile et al*.* (2006) for *M. paradisiaca* Mid-Rib using soda pulping and ranged from 25.80 - 49.13%.

Ash and Silica contents

The results of the ash and silica content of the pulp showed a reduction in their content compared to the plant raw materials. This trend correlates with that of other reports on the effect of pretreatment and pulping on the reduction of ash and silica content of lignocellulosic materials (Ainun et al*.,* 2017; Serzgin and Serhat, 2018). The higher silica and ash content of *T. diversifolia* has been attributed to its grass nature (Jones and Handrick, 1967).

Kappa number

From the results in Table 2, *M. paradisiaca* peel recorded the highest kappa number and this could be attributed to its fruit covering duty which might have built in much lignin as plant glue which in turn assists in fruit covering.

Physicochemical properties of bleached pulp

Yield

From the results indicated in Table 3, there was a reduction in the yield of the bleached cellulose pulp. Higher and lower yield values on non-woody biomass have been reported by other authors (Shirkolaee, 2009; Mohsen et al*.,* 2015). Reduction in yield of bleach compared to non-bleached pulp could be attributed to the sequencing, type and concentration of bleaching agents used. However, Oluwasina et al*.* (2014) attributed the reduction as a result of removal of some residual lignin and other oxidizable compounds.

Ash and silica contents

There was also a reduction in the ash and silica content

of the bleached cellulose pulp as indicated in Table 3. Lower values of 0.06, 0.57 and 0.77% have been reported for *Musa sapentium, T. diversifolia* and *M. paradisiaca* (Oluwasina et al*.,* 2014) while higher value of 50.6% for rice straw using atmospheric acetic acid pulping and bleaching has also been reported (Xuejun et al*.,* 1999). Reduction in ash and silica content has been attributed to the removal of lignin and other oxidizable compounds which might have contained both ash and silica (Oluwasina et al*.,* 2014).

Kappa number

Kappa number of the bleached cellulose pulp also recorded a reduction compared to the unbleached pulp as depicted in Table 3. The reduction could be as a result of the washing and squeezing action during bleaching which might have caused removal of more lignin.

Physicochemical properties of synthesized CMC

Yield

Yield is a function of the amount of materials lost during dialysis step. Higher and lower yields have been reported. The yield of the synthesized CMC in this research work from Table 4 compared favourably with the work of Bono et al. (2009) (33.15%) on CMC from Palm Kernel cake, and Huang et al*.* (2017) (64.40%) on spent tea leaf. Higher yield, 121 to 128% from water hyacinth and 141% from Pod husk of Cacao have been reported (Saputra et al*.,* 2014; Hutomo et al*.,* 2012). The difference in yield could be attributed to temperature of the reaction and concentration of NaOH and MonoChloroacetic acid (MCA) applied during synthesis.

pH

The pH of the synthesized CMC in this work as depicted in Table 4 indicates that the samples are in a very weak acidic medium. Lower CMC pH values could indicate a lower purity of the product with non-reacted reagents such as monochloroacetic acid and reaction by-products. Saputra et al*.* (2014) reported pH range of 7 - 14. The pH of this research compared with the report of Bono et al*.* (2009) (6.5) for CMC from Palm kernel cake. The variation in properties of the different CMC could be as a result of the source of cellulose used, plant species, age and source which affect the cellulose content compositions (Chandra et al*.,* 2007; Carere et al*.,* 2008).

Degree of substitution (DS)

Since degree of substitution (DS) is the average number of hydroxyl groups replaced by the substituent in every anhydroglucose unit in the chain; therefore the result in Table 4 suggests that the various prepared CMC reacted differently. This results in different DS. The result compared favourably with report of Huang et al*.* (2017) on palm kernel cake (0.31) and oil palm fibre (0.29). Higher DS values of 0.35, 0.80, and 0.76 have been reported for *M. sinensis, E. crassipes* and *C. papyrus,* respectively (Kimani et al*,* 2016). Adinugraha et al*.* (2005) reported DS range of 0.26 - 0.76 for banana pseudo stem. The normal DS range for commercially available CMC is approx. 0.5 - 1.5 (Karatas and Arslan, 2016). When the DS is below 0.4, the CMC is swellable but insoluble, while above this value, then, CMC is fully soluble with its hydro-affinity increasing with increasing DS value (Arshney et al*.,* 2006). Since the DS of the prepared CMC are below 0.4, they are insoluble in water but swellable and therefore a good material for superabsorbent biopolymers.

Viscosity

Viscosity of CMC greatly influences the DS, a higher DS results in better viscosity and cation exchange ability. Additional carboxyl groups provide more sites for cross-linking by multivalent cations. From the results in Table 4, *T. diversifolia* possesses higher swelling capacity. Higher and lower viscosities for non-woody biomass have been reported by several authors. Higher viscosity (66.6 cP) was reported for CMC from PKC (Bono et al*.,* 2009) while lower viscosity (14.0 cP) was reported for CMC from Orange mesocarp.

Swelling capacity

The swelling capacity of the synthesized CMC in this study as shown in Table 4 followed the same trend as the DS. This suggests that swelling capacity is a function of the degree of substitution since *T. diversifolia* with the highest swelling ability still has the highest DS value of 0.33. Higher and lower swelling capacity of synthesized CMC on non-woody plants have been reported by several authors. Kimani et al*.* (2016) reported 488.59, 205.55, and 419.66 for *M. sinensis, E. crassipes* and *C. papyrus,* respectively. Higher values of 748.17 and 801.73% were reported for *C. gigantea* CMC and *G. sepium* CMC, respectively (Abe et al*.,* 2018)

Sodium chloride content

The sodium chloride level in CMC is an important parameter; it is a reaction minor-product, considered a contaminant. From the results in Table 4 *M. paradisiaca* (peel) had more minor bye-products than *M. paradisiaca* (stalk) and *T. diversifolia*. The sodium chloride values

obtained in this work compared favourably with sodium chloride values of 0.15 and 0.19 % reported for cotton linters (Latif et al*.,* 2007)

Bulk and tap densities

Bulk and tap densities provide an estimate in the ability of a material to flow and be packed into a confined space. Generally, the higher the bulk and tap densities, the better the potential for a material to flow and to rearrange under compression (Azubuike et al*.,* 2012). From the results in Table 4, there is no much significant difference in the bulk and tapped densities. This means that *M. paradisiaca* stem and *T. diversifolia* bleached cellulose powder have better flow than *M. paradisiaca* peel.

Fourier transform infrared spectroscopy

The spectral data analysis is shown in Table 5. The introduction of strong peaks at 1597, 1588 and 1585 cm^{-1} could be attributed to the presence of carbonyl group (C=O) stretching, confirming the presence of the –COO group and the successful etherification. This suggests that the cellulose from *T. diversifolia, M. paradisiaca* stem, and unripe peel were successfully modified into CMC (Huang et al*.,* 2017; Asl et al*.,* 2017; Bono et al*.,* 2009). Strong absorption band at 3398, 3372 and 3347 cm^{-1} was due to the stretching frequency of the -OH group and another band at 2345, 2360 and 2364 cm^{-1} was due to C-H stretching.

Conclusion

The result of the present work provided an insight into the physicochemical properties of synthesized cellulose and carboxymethylcellulose from *M. paradisiaca* stem and unripe peel and *T. diversifolia*. The high alpha cellulose content of *T. diversifolia* makes it a potential source of sustainable industrial grade cellulose production. The yield from *M. paradisiaca* peel was significantly low compared to the fruit stem and *T. diversifolia.* Major functional groups present in the commercial (Fidelo) CMC were also identified in *T. diversifolia* CMC whereas functional groups such as C-O of the ether and O-C-O stretching of ether were not identified in *M. paradisiaca* stem and unripe peel CMC. Furthermore, higher DS and viscosity of *T. diversifolia* CMC when compared to *M. paradisiaca* stem and unripe peel CMC makes it superior as a useful bio-polymer for industrial applications.

CONFLICT OF INTERESTS

The authors have not declared any conflict of interests.

ACKNOWLEDGEMENT

The authors appreciate the Raw Materials Research and Development Council and the Sheda Science and Technology Complex for their support towards the conduct of this research work.

REFERENCES

Abbas K, Rizwani GH, Zahid H, Asif A (2015). Pharmacognostic Evaluation of *Musa Paradisiaca* L. Bract, Flower, Trachea and Tracheal Fluid. World Journal of Pharmacy and Pharmaceutical Sciences 4(4):1461-1475.

Abe TO, Lajide L, Owolabi BJ, Adebayo AO, Ogunjobi JK, and Oluwasina OO (2018). Synthesis and application of carboxymethylcellulose from *Gliricidia sepium* and *Cola gigantean*. BioResources 13(3):6077-6097.

Adinugraha MP, Marseno DW, Haryadi (2005). Synthesis and characterization of sodium carboxymethyl cellulose from cavendish banana pseudo stem (Musa cavendishii LAMBERT). Carbohydrate Polymers 62(2):164-169.

Ainun ZMA, Muhammad KI, Rasmina H, Hazwani HA, Sharmiza A, Ntaziratulasikin AK, Latifah J (2017). Effect of Chemically Pretreatment on Pulp and Paper Characteristics of Bamboo *gigantochlou Scorthechinu* Kraft Fibers. Materials Science and Engineering 368(1):012044.

Akanbi WA, Olaniran OA, Adebayo TA (2007). Control of insect pests of cowpea in the field with allelochems from Tephrosia vegell and Petiveria allecea in Southern Guinea Savanah of Nigeria. Journal of Agriculture 2(3):365-369.

Akinsanmi AO, Oboh G, Akinyemi JA, Adefegha AS (2015). Assessment of the Nutritional, Anti nutritional and Antioxidant capacity of Uripe, ripe, and over ripe Plantain (*Musa paradisiaca*) Peels. International Journal of Advanced Research 3(2):63-72.

Akinyele BJ, Agbro O (2007). Increasing the nutritional value of plantain waste by the activities of fungi using solid state fermentation technique. Research Journal of Microbiology 2(2):117-124.

Akubor PI, Ishiwu C (2013). Chemical composition, Physical and Sensory Properties of Cakes supplemented with Plantain peel flour. International Journal of Agricultural Policy and Research 1(4):087-092.

Ali M, Byrd M, Jameel H (2001) Soda-AQ pulping of cotton stalks. Presented at 2001 TAPPI Fall Technical Conference (Atlanta) 1-9

Ambjornsson HA, Schenzel K, Germgard U (2013). Carboxymethyl Cellulose Produced at Different Mercerization Conditions and Characterized by NIR FT Raman Spectroscopy in combination with Multivariate Analytical Methods. BioResources 8(2):1918-1932.

Arafat KMY, Nayeem J, Quadery AH, Quaiyyum MA and Jahan MS (2018) Handmade paper from waste banana fibre. Bangladesh Journal of Science and Industrial Research 53(2):83-88.

Arisa NU, Adelekan AO, Alamu AE, Ogunfowora EJ (2013). The Effect of Pretreatment of Plantain (Musa Parasidiaca) Flour on the Pasting and Sensory Characteristics of Biscuit. International Journal of Food and Nutrition Science 2(1):10-24.

Arshney VK, Gupta PK, Naithani S, Khullar R, Bhatt A, Soni PL (2006). Carboxymethylation of α-cellulose isolated from *Lantana camara* with respect to degree of substitution and rheological behavior. Carbohydrate Polymers 63(1):40-45.

Arun KB, Persia F, Aswathy PS, Chandran J, Sajeev MS, Jayamarthy, Nisha P (2015). Plantain Peel- A Potential source of antioxidant dietary fibre for developing functional cookies. Journal of Science and Technology 52(10):6355-6364.

Asl SA, Mousavi M, Labbafi M (2017).Synthesis and Characterization of Carboxymethyl Cellulose from Sugarcane Bagasse. Journal of Food Processing and Technology 8(8):1-6

ASTM D 1439-94 (1995). Standard test method for sodium carboxymethylcellulose, annual book of American society for Testing and Materials (ASTM) Standards, part 21, ASTM, Philadephia. https://www.document-center.com/standards/show/ASTM-D1439.

ASTM D1104-56 (1985). American Society for Testing and Materials. Method of Test for Holocellulose in Wood (https://www.document-center.com/standards/show/ASTM-D1104)

ASTM D1104-56 (1985). American Society for Testing and Materials. Method of Test for Alpha-cellulose in Wood. https://www.document-center.com/standards/show/ASTM-D1103

ASTM D-1439-03 (2005). Analytical method for determining degree of substitution in the product, Document CK-G06 Edition, 05: D-1439-03 https://www.astm.org/DATABASE.CART/HISTORICAL/D1439-03.htm

Azubuike CP, Oduluja JO, Okhamafe AO (2012). Physico-technical, spectroscopic and thermogravimetric properties of powdered cellulose and microcrystalline cellulose derived from groundnut shells. International Journal of Excipients and Food Chemistry 3(3):106-115.

Barba C, Montané D, Farriol X, Desbrières J, Rinaudo M (2002). Synthesis and characterization of carboxymethyl celluloses from non-wood pulps II. Rheological behavior of CMC in aqueous solution. Cellulose 9(3-4):327-335.

Barbash V, Trembus I, Nagorna J (2012). Pulp Obtaining From Corn Stalks. Chemistry and Chemical Technology 6(1):83-87.

Bisht BS, Joshi RK (2017). Comparative terpenoid composition of the leaf and root essential oil of *Tithonia diversifolia* (Hemsl.) A. Gray. American Journal of Essential Oils and Natural Products 5(3):21-24.

Bono A, Ying PH, Yan FY, Muei CL, Sarbatly R, Krishnaiah D (2009). Synthesis and Characterization of Carboxymethyl Cellulose from Palm Kernel Cake: Advances in Natural and Applied Sciences 3(1):5-11.

Buragohain R (2016). Growth performance, nutrient utilization, and feed efficiency in broilers fed *Tithonia diversifolia* leaf meal as substitute of conventional feed ingredients in Mizoram. Veterinary World 9(5):444-449.

Carere CR, Sparling R, Cicek N, Levin DB (2008). Third generation biofuels via direct cellulose fermentation. International Journal of Molecular Sciences 9(7):1342-1360.

Chandra RP, Bura R, Mabee WE, Berlin A, Pan X, Saddler JN (2007). Substrate pretreatment: The key to effective enzymatic hydrolysis of lignocellulosic. Advance Biochemical Engineering Biotechnology 108:67-93.

Chukwuka KS, Ojo OM (2014). Extraction and Characterization of Essential Oils from *Tithonia diversifolia* (Hemsl.) A. Gray. American Journal of Essential Oils and Natural Products 1(4):1-5.

Drechsel P, Reck B (1998). Composted Shrub-prunnings and other manures for small holders' farmer system in Southern Rwanda. Agroforestry Systems 39:1-12.

Enyati AA, Hamzeh Y, Mirshokraie SA, Molaii M (2009). Paper from Canola Stalks. BioResources 4(1):245-256.

Fang J, Liu H, Zhang G (2016) Preparation of carboxymethyl cellulose from corncob. Procedia Environmental Sciences 31(1):98-102.

Friday ON, Muhammad MM (2015). Isolation and Physicochemical Characterization of Lignin from *Chromolaena Odorata* and *Tithonia Diversifolia*. Journal of Applied Sciences and Environmental Management 19(4):787-792.

Huang CMY, Chia P, Lim SS, Nai JQ, Ding DY, Seow PB, Wong CW, Chan WC (2017). Synthesis and Characterisation of Carboxymethyl Cellulose from various Agricultural Wastes, Cellulose Chemistry and Technology 51(7-8):665-672.

Hutomo GS, Marseno DW, Anggrahini S, Supriyanto A (2012). Synthesis and Characterization of Sodium Carboxymethylcellulose from Pod Husk of Cacao (*Theobroma Cacao* L.). African Journal of Food Science 6(6):180-185.

Ibrahim MM, Agblevor FA, El-Zawawy (2010). Isolation and characterization of Cellulose and Lignin from steam-exploded lignocellulosic Biomass. BioResources 5(1):397-418.

Itiola OA (1991). Compressional characteristic of three starches and the mechanical properties of their tablets. Pharmacy World Journal 8:91-94.

Iwuagwu MA, Okoli PC (1992). The disintegrant properties of pregelatinized cassava and white yam starches. Pharmacy World Journal 8(3):91-94.

Jama B, Palm CA, Buresh RJ, Niang A, Gachengo C, Nziguheba G, Amadalo B (2000). *Tithonia diversifolia* as a green manure for soil improvement in Western Kenya: A review. Agroforestry Systems 49(2):201-221.

Jaramogi GH, Sally J, Michieka M, Biot G, Josephat BO (2016). Evaluation of proximate and mineral composition of (*Musa paradisiaca)* wastes as livestock feeds. International Journal of Animal Breeding and Genetics 3(6):138-142.

JECFA (1998). "Sodium Carboxymethylcellulose, Enzymatically hydrolysis," Superseding tentative specifications prepared at the 49th JECFA. Available at http://www.inchem.org/documents/jecfa/jeceval/jec_2138.htm. Retrieved on 12/06/2017

Jones LHP, Handrick KA (1967). Silica in soils, plants and animal. Advances in Agronomy 19(2):107-149.

Karatas M, Arslan N (2016). Flow behaviours of cellulose and carboxymethyl cellulose from grapefruit peel. Food Hydrocolloids 58:235-245.

Kimani PK, Kareru PG, Madivoli SE, Kairigo PK, Maina EG, Rechab OS (2016). Comparative Study of Carboxymethyl Cellulose Synthesis from Selected Kenyan Biomass Chemical Science International Journal 17(4):1-8.

Kumar V, Kothari SH (1999). Effect of compressional force on the crystallinity of directly compressible cellulose excipients. International Journal of Pharmacy 177(2):173-182.

Latif A, Anwar T, Noor S (2007). Two step synthesis and characterization of Carboxymethylcellulose from Rayon grade wood pulp and cotton linter. Journal of Chemical Society, Pakistan 29(2):143-150.

Mohammed ZI, Saleha A (2011). Musa paradisiaca L. and Musa sapientum L: A Phytochemical Pharmacological Review. Journal of Applied Pharmaceutical Science 1(5):14-20.

Mohsen M, Ghasemian A, Resalati H, Zeinaly F (2015). Total Chlorine-Free Bleaching of Populus deltoides Kraft Pulp by Oxone. International Journal of Carbohydrate Chemistry doi.org/10.1155/2015/381242.

Ogunsile BO, Omotoso MA, Onilude MA (2006). Comparative Soda pulps from the mid-rib, pseudo stem and stalk of *Musa paradisiaca*. Journal of Biological Sciences 6(6):1047-1052.

Olayinka BU, Alex D, Obukohwo EE (2015) Phytochemical And Proximate Composition Of *Tithonia Diversifolia* (Hemsl.) A. Gray Annals. Food Science and Technology 16(1):195-200.

Oluwasina O, Lajide L, Owolabi B (2014). Performance of bonded boards using lignin-based resins. Wood Material Science and Engineering 10(2):168-177

Oluwasina O, Lajide L, Owolabi B (2014). Microcrystalline Cellulose from Plant Wastes through Sodium Hydroxide-Anthraquinone-Ethanol Pulping. BioResources 9(4):6166-6192.

Otoide JE, Jayeola AA, Akanni BA (2018). Pulping and Paper Making Potentials of Stem of *Tithonia Diversifolia*. European Journal of Botany, Plant Sciences and Phytology 4(1):1-5.

Padam BS, Tin HS, Chye FY, abdullahi MI (2014). Banana by-products: an underutilized renewable biomass with great potential. Journal of food science and Technology 51(12):3527-3545

Rana GK, Singh Y, Mishra SP, Rahangdale HK (2018). Potential of Banana and its by-products: A review. International Journal of current Microbiology and Applied sciences 7(6):1827-1832.

Saelee K, Yingkamhaeng N, Nimchua T, Sukyai P (2014). Extraction and characterization of cellulose from sugarcane bagasse by using environmental friendly method. Proceedings of the 26th Annual Meeting of the Thai Society for Biotechnology and International Conference. Available at http://tsb2014.mfu.ac.th/proceeding/03_PDF/(BN)%20Biomaterials%20_%20Nano/TSB2014%20Proceeding%20PR-BN-09.pdf. Retrieved on 05/10/2016

Saputra AH, Qadhayna L, Pitaloka AB (2014). Synthesis and Characterization of CarboxymethylCellulose (CMC) from Water Hyacinth Using Ethanol-Isobutyl Alcohol Mixture as the Solvents. International Journal of Chemical Engineering and Applications 5(1):36-40.

Sezgin KG, Serhat S (2018). Chemical Composition, Fiber Morphology, and Kraft pulping of Bracken Stalks (*Pteridium aquilinum* (L) *Kuhn)*. Drvna Industrija 69(1):23-33.

Shakhes J, Marandi MAB, Zeinaly F, Saraian A, Saghafi T (2011).

Tobacco residuals as promising Lignocellulosic materials for pulping and paper Industry. BioResources 6(4):4481-4493**.**

Shirkolaee YZ (2009). Comparative study on hydrogen peroxide bleaching of soda- organosolv and kraft rice straw pulps. India Journal of Chemical Technology 16 (2):231-239**.**

T204om-97 (2007). Technical Association of the Pulp and Paper Industry (TAPPI) Standard for Solvent Extractives of Wood and Pulp. (https://www.tappi.org/content/sarg/t204.pdf)

T207om-81 (2008). Technical Association of the Pulp and Paper Industry (TAPPI). Standard for Water solubility of Wood. (https://imisrise.tappi.org/TAPPI/Products/01/T/0104T207.aspx)

T211om-02 (2002). Technical Association of the Pulp and Paper Industry (TAPPI). Standard for Ash in Wood, Pulp, Paper and Paperboard.

T212om-02 (2002). Technical Association of the Pulp and Paper Industry (TAPPI). Standard for One Percent Sodium Hydroxide solubility of Wood and Pulp. (https://research.cnr.ncsu.edu/wpsanalytical/documents/T212.PDF)

T236om-99 (1999). Technical Association of the Pulp and Paper Industry (TAPPI). Standard for Kappa Number of Pulp (https://research.cnr.ncsu.edu/wpsanalytical/documents/T236.PDF)

T244om-93 (1999). Technical Association of the Pulp and Paper Industry (TAPPI). Standard for Silica in Wood, Pulp, Paper and Paperboard. (https://webstore.ansi.org/standards/tappi/244)

T550om-03 (2008). Technical Association of the Pulp and Paper Industry (TAPPI). Standard for Moisture in Pulp, Paper and Paperboard. (https://www.complianceonline.com/images/supportpages/501052/sample_T550.pdf)

Tona L, Kambu K, Ngimbi, N, Cimanga K, Vlietinck AJ (1998). Antiamoebic and phytochemical screening of some Congolese medicinal plants. Journal of Ethnopharmacology 61(1):57-65.

Uhuegbu CC, Onuorah LO (2014). Production of Biogas from Plantain Peels. Research Journal in Engineering and Applied Sciences 3(2):145-150.

Xuejun P, Toshiaki T, Yoshihiro S (1999). Atmospheric acetic acid pulping of ricehusk II: Behaviour of ash and silica in rice straw during Atmospheric acetic acid pulping and bleaching. Holzforschung 53(1):49-55.

Zawawi D, Mohd ZMH, Angzzas SMK, Halizah A, Ashuvila MA (2013). Analysis of the Chemical composition and Fiber Morphology Structure of Corn Stalk. Australian Journal of Basic and Applied Sciences 7(9):401-405.