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EXTRACTION AND CHARACTERIZATION OF CELLULOSE FROM JACKFRUIT (*Artocarpus integer*) PEEL

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Artocarpus, cellulose

Jackfruit peel

Extraction of cellulose

Characterization

ABSTRACT

In the present study, cellulose was extracted from de-pectinated peel of Jackfruit. The peel was treated with alkali followed by a chemical process treatment. In present study, yield of jackfruit cellulose was 27g / 100g. The average particle size and zeta potential value of extracted cellulose is 0.730nm and -15.7mV respectively. Further, morphological characterization was carried out by using scanning microscopy. FTIR spectroscopy analysis revealed the presence of major absorptions indicative of cellulose and hemicellulose. The thermal profiles of the residual waste occurred in a minimum three steps, which representing the degradation profiles of hemicellulose, cellulose, and lignin

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1 Introduction

Cellulose is the foremost constituent of all plant materials. Cellulose is a polysaccharide consisting of a linear chain of several thousands of β(1→4) linked D-glucose units. Cellulose is the main component of the plant cell. Although some animals (such as tunicates) and some bacteria contain cellulose, the content of cellulose in these species is negligible when compared with plants. Cellulose is probably the least soluble of all fiber components, being insoluble not only in cold or hot water but also in hot dilute acids and alkalis also (Singanusong et al., 2014).

From the economic point of view it is preferable to produce fibres from locally available biomass waste in order to avoid high transport costs of the raw materials. In recent years, the interest in cellulose based materials has been increasing due to the demand for renewable resources and growing environmental awareness (Mohanty et al., 2005). Agro-wastes that have been explored for cellulose include materials such as coconut husk fibres, mulberry bark, wheat, straw, (Johar et al., 2012), pea hull, corn bran, dried beet pulp, oat hull (Vail, 1991), orange peel (Arslan et al., 2007); banana plant wastes (Elanthikkal et al., 2010); banana peel (Singanusong et al., 2014), durian rind (Penjumras et al., 2014); pomelo (*Citrus grandis*) albedo peel (Zain et al., 2014); tomato peel (Jiang & Hseih, 2015); *Platycodon Grandiflorum* peel (Zhou et al., 2015); rambutan peel (Oliveira et al., 2016), brewer's spent grain (Mishra et al., 2017). Though a range of natural fibers are investigated intimately, the utilization of jackfruit (A*rtocarpus integer*) peel as a natural supply for the assembly of cellulose has not been explored nevertheless.

Jackfruit (*Artocarpus integer*) can be a genus of roughly sixty species of Southeast Asia and Pacific origin, belonging to the mulberry family, *Moraceae* (Zerega & Motley, 2001). Jackfruit is indigenous to India and unfolded out into tropic regions, including Republic of Indonesia (Ismadji et al., 2008; Sundarraj et al., 2018). A. *integer* plant has been used as ancient medication to treat protozoal infection and symptom (Syah et al., 2006). The cellulose fibres obtained from waste materials can be used for the preparation of micro and nano cellulose fibres, micro and nanoparticles and nanowhiskers. The total annual amount of cellulose is several billion tons, revealing the huge economic value of it.

Despite the various medicinal and economic importances, approximately 2714 - 11,800 kgs per tree jackfruit peel production reported annually (Sulochana & Inbaraj, 2004). In India, 75% of jackfruits are wasted and Kerala alone accounts for about 35 crore jackfruits annually. "Assuming that one jackfruit costs only Rs. 3 and the national average waste is only 50%, India is losing Rs 214 crore worth of food every year", (Sundarraj et al., 2018). Appropriate methods to convert these wastes into value-added products will serve the dual purpose of environmental protection and value addition (Sundarraj & Ranganathan, 2017). Cellulose could be a valuable by-product that can be obtained from these wastes which might be extensively employed in the food trade (Raj & Ranganathan, 2018). The objective of this study was to extract cellulose from jackfruit peels and analysis of its physicochemical properties.

2 Materials and Methods

2.1 Materials

Mature jackfruits were collected from the local market of Pudukkottai (district), Tamil Nadu, India. Plant species authentication was done by Botanical Survey of India (BSI), Coimbatore, India (Ref no. BSI/SRC/5/23/2013-14/Tech/1714). For comparison study, commercial cellulose powder was procured from M/s. Loba Chemie Pvt. Ltd., Mumbai, Maharashtra, INDIA.

2.2 Preparation of Jackfruit peel powder

Jackfruit was peeled manually to discard the edible part together with the seeds. The peels were cut into smaller pieces of 1 cm size and treated with sodium metabisulphite (Mohamed & Hasan 1995). The treated jackfruit peels were then washed with boiling water and pressed to remove an excess quantity of water. Subsequently, the jackfruit peels were dried in a cross flow dryer at 65°C for eight hours. The dried peels were ground and packed in polyethylene bags and keep at room temperature for further analysis.

2.3 Extraction of Cellulose from jackfruit peel

The extraction of cellulose procedure was supported methodology given by Arslan et al. (2007) with slight modification. The method followed is given in (figure 1) and the weight of jackfruit cellulose obtained was recorded.

Cellulose was kept in sealed container at temperature and prepared for analysis. The proportion of cellulose was calculated by method given by Penjumras et al. (2014)

$$\mathbf{Cellulose}(\%) = (\mathbf{W2/W1})\,\mathbf{x100}$$

Whereas, W1 = weight of jackfruit peel powder (g) and W2 = weight of jackfruit cellulose (g)

2.4 Characterization

Moisture content of the jackfruit peel was found to be 90% after carrying out the necessary experiments. Thus 100 g of dried peel was obtained on drying 1 kg of jackfruit peel.

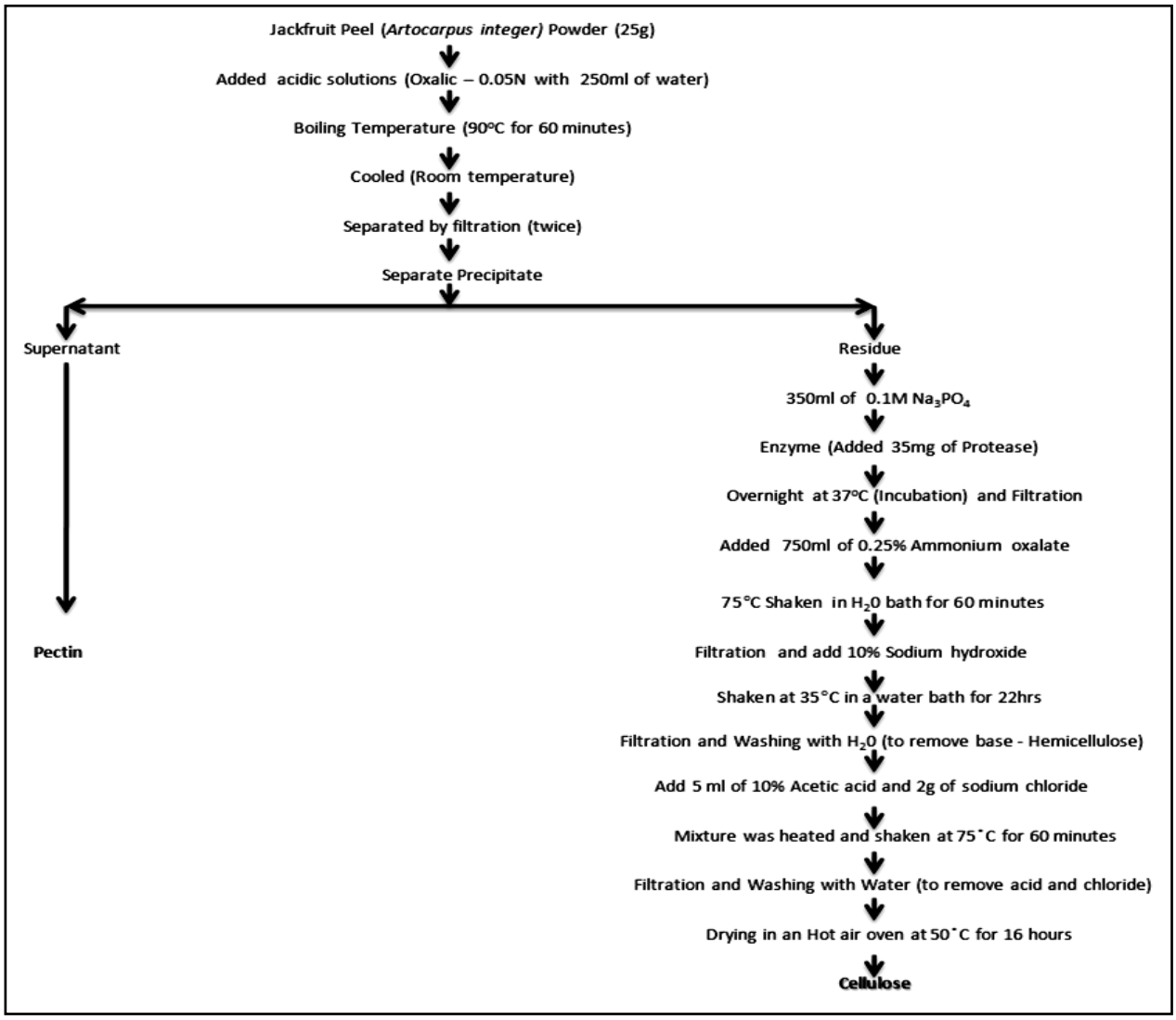


Figure 1 Extraction of cellulose from jackfruit Peel

2.5 Particle Size Distribution

Particle size distribution parameters was expressed as Sauter mean diameter (Mu & Ma, 2016), [$D_{3,2}$ (µm)] using Optical Diffraction in a Malvern particle size instrument.

2.6 Zeta potential Distribution (ζ)

Zeta potential for 0.1 wt (%) aqueous suspensions of jackfruit and industrial cellulose was measured using a Zetasizer Nano S90 - Malvern Instrument. The zeta potential was calculated from the natural process quality using Huckel approximation (Jiang & Hseih, 2015).

2.7 Scanning Electron Microscopy (SEM)

Scanning microscope JEOL JSM 6390, SEM, Japan was used to investigate the microstructure of jackfruit and commercial cellulose. The micrographs were determined at a magnification of X 500 at an accelerated voltage of twenty kV (Vaidya et al., 2016).

2.8 Fourier Transform Infrared Spectroscopy (FTIR)

Functional groups of jackfruit and commercial cellulose was analyzed by FTIR transmission spectra of powder samples by KBr technique. The mixed powder was mounted on the instrument to

create a straight forward measure of the transmission % T mode (Ismadji et al., 2008). The analysis was carried out in a Shimadzu IR PRESTIGE 21 FTIR instrument with frequency variation from 4000^{-1} to $500cm^{-1}$ with a resolution of $4cm^{-1}$ and total of 10 scans (Deka & Khawas, 2016).

2.9 X - ray Diffraction (XRD)

XRD patterns were measured employing a SHIMADZU - XRD 6000 instrument at an ambient temperature using Cu kα radiation supply of the wavelength of λ = 0.154nm at 45kV and an incident current of 30 mA. The angular region ranged from 0° to 80° with a scanning speed of 0.01°/min (Mu & Ma, 2016). The method of Segal et al. (1959) was considered to evaluate the crystallinity of the different samples. The crystallinity index Crl was determined (Oliveira et al., 2016) based on the reflected intensity data following;

$$Crystallinityindex\ (CrI)\ = 100x\frac{(I_{002} - Iam)}{I_{002}}$$

where: I_{002} is that the maximum intensity of the (002) lattice diffraction peak and (I_{am}) amorphous - intensity scattered by the amorphous part of the sample.

The diffraction peak of the plane (002) is located at a diffraction angle and the lowest intensity scattered by the amorphous part is measured as the lowest intensity at a diffraction angle.

2.10 Thermogravimetric Analysis (TGA)

Thermal degradation of cellulose was carried out in a TGA, Model SDT Q600 from TA instruments. Thermogravimetric analyses of the samples was conducted from 25°C to 700°C, below the nitrogen atmosphere at a flow rate of 50 ml/min, (Boumediene et al., 2015). A specimen was first placed into an aluminum pan on the platinum basket in the chamber. The heating rate of 10°C/ min was maintained and 10mg of sample mass was utilized in each experiment trials.

2.11 Color

The color values of the cellulose were measured with a Hunter Calorimeter. Cellulose Specimens were placed on a white standard plate (L = 96.86; a = - 0.02; b = 1.99) and the L, a and b color values were measured. All measurements were performed in triplicates.

3 Results and Discussion

3.1 Yield of Cellulose (%)

Cellulose was obtained from dried jackfruit peel (moisture content of 12.98% ± 0.42) by depectination followed by mercerization. The amount of cellulose obtained was 21.10 ± 1.06 g for 100 g of the peel taken. This amounts to 77.77% yield, considering that the cellulose content of the peel was determined to be 27g / 100 g of dry matter. The cellulose content and yield from jackfruit peel is found to be higher than other peels including albedo peel (21.29±1.90 %, Zain et al., 2014), orange (24.06% Bicu & Mustata, 2013) and rambutan (24.28%, Oliveira et al., 2016). This indicates that jackfruit peel can be used as a potential source of cellulose.

3.2 Particle Size Distribution

Dynamic light scattering is used for the determination of the dimensions of particles of suspension, emulsions, colloids, chemical compound etc. (Chandra et al., 2016). Particle size plays a very important role in physiochemical and functional properties (Wuttipalakorn et al., 2009; Peerajit et al., 2012). The water suspended cellulose samples was diluted and analyzed by the Malvern particle size analyser. The particle size of cellulose samples is given in the figure 2a. The result of study revealed that cellulose particle are in the nano region, with the jack fruit cellulose having a larger particle size of 730 nm as compared to that of commercial cellulose (312.3 nm), Figure 2a (i) and (ii).

3.3 Zeta Potential Distribution (ζ)

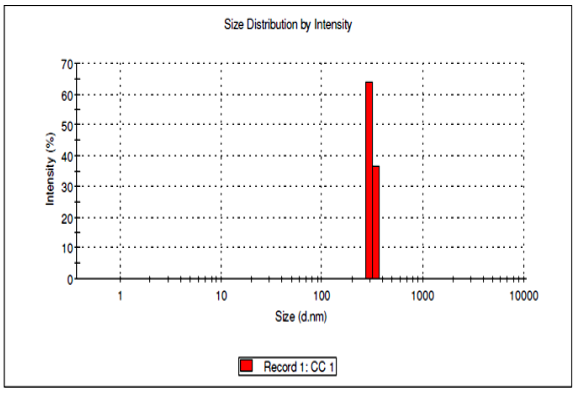
The zeta potential is an important parameter for examining the dispersion stability of colloids, in this study, the zeta potential was negative for both commercial (- 17.7 mV) and jackfruit cellulose (- 15.7 mV), indicating incipient instability (Figure 2b). Similar zeta potential have also been observed from banana cellulose (- 16.1 mV, Pelissari et al., 2014).

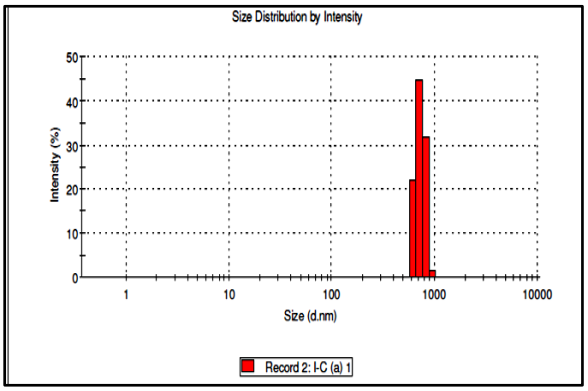
3.4. Scanning Electron Microscopy (SEM) Analysis

Changes in the surface morphology of celluloses (commercial and jackfruit peel) were determined by SEM. The SEM pictures of each cellulose with a magnification of five hundred times are shown in figure 2c. In the case of jackfruit cellulose, figure 2c (i) the diameter was less than 100µm, showing roughness image due to aggregation; similar report has also been reported from cassava cellulose (Sukyai et al., 2015). In figure 2c (ii) shows the structure of commercial cellulose particles, confirming the reports by other authors (Singanusong et al., 2014).

3.5. Fourier Transform Infrared Spectroscopy (FTIR)

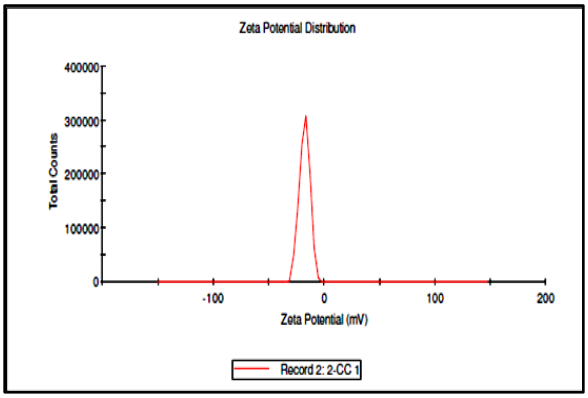
The FTIR has the flexibility to predict structural variations not seen by the other physiochemical analyses (Azubuike & Okhamafe, 2012) and therefore the vibrations or stretch on the chemical structure of the commercial cellulose is shown in figure 3(a) and jackfruit cellulose is shown in figure 3(b). The spectra present the characteristic bands which will be found on IR

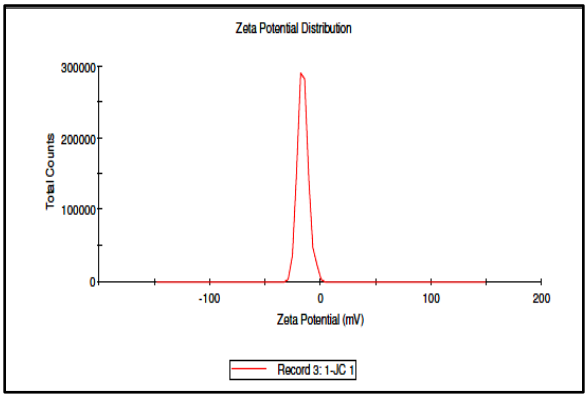




(i) Commercial Cellulose (CC) (ii) Jackfruit Cellulose (JC)

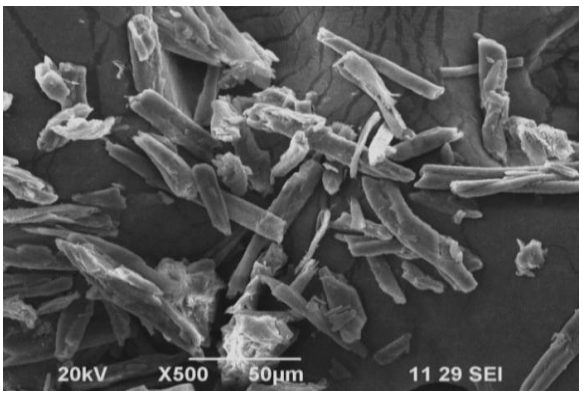
Figure 2a: Particle size distribution curve for cellulose (i) and (ii)

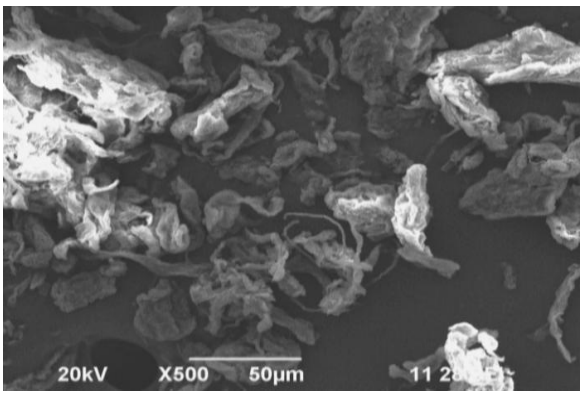




(i) Commercial Cellulose (CC) (ii) Jackfruit Cellulose (JC)

Figure 2b: Zeta potential distribution of cellulose (i) and (ii)



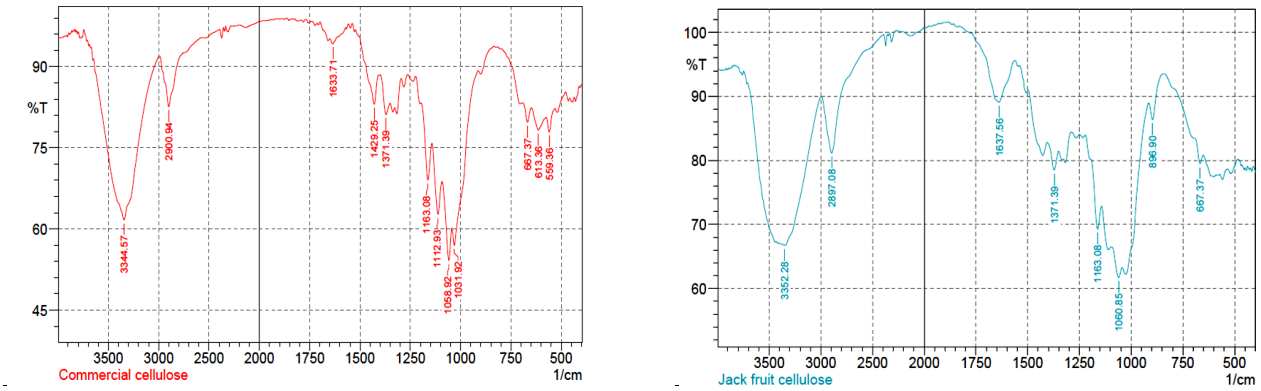


(i) Commercial Cellulose (CC) (ii) Jackfruit Cellulose (JC)

Figure 2c: Micro-structural characteristics of cellulose (i) and (ii)

spectra of cellulose and hemicellulose and are in sensible agreement with the literature (Li et al., 2008; Zapata et al., 2009).

The FTIR spectrum of commercial and jackfruit cellulose displayed broadband at 3500cm^{-1} - 3000cm^{-1}, that corresponds to the hydrogen secure O-H Stretching vibration of radical teams originating primarily from cellulose and hemicelluloses (Ismadji et al., 2008; Suresh et al., 2011; Ramli et al., 2014). The peaks at 2897.08cm^{-1} of jackfruit cellulose and 2900.94cm-1 of commercial cellulose correspond to the C-H asymmetrical stretching vibration of acyclic structures, whereas alternative bands of the fingerprint region (1800cm^{-1} to 900cm^{-1} of jackfruit



(a) Commercial Cellulose (b) Jackfruit Cellulose

Figure 3: Infrared Spectroscopy spectra of cellulose (a) and (b)

and commercial cellulose) are complex; this is often a results of various vibration modes in carbohydrates and lignin (Bodirlau et al., 2008; Sanchez et al., 2014). The peak at 1637.56cm^{-1} of jackfruit cellulose and 1633.71cm^{-1} of commercial cellulose, which indicates the presence of O-H bending in the absorbed water (Zain et al., 2014) respectively. It is determined that the absorption most of water peak is slightly shifted to higher oftenness as wet will increase (Velazquez et al., 2003).

A robust/strong band seems at 1429.39cm^{-1} of jackfruit and commercial celluloses indicates the aliphatic compound deformations with reference to CH and CH_2 (Mammino & Kabanda, 2009). Moreover, the intensity peak refers the cellulose structure involves the dominant peak at 1371.39cm^{-1} of jackfruit and commercial celluloses to be attributed to the O-H bending vibration (Sukyai et al., 2015). The peak showing in the 1330cm^{-1} - 1369cm^{-1} of jackfruit and commercial cellulose regions of the spectra could be attributed to the in-plane bending vibration of the C-H and C-O teams of the monosaccharose ring in the cellulose (Muniyandy et al., 2016). Similar results have been reported on cellulose isolated from sugarcane waste (Schulz & Baranska, 2007). The peak at 1163.08cm^{-1} of both celluloses (jackfruit and commercial) confirmed the presence of cellulose (Mulinari et al., 2009; Anna et al., 2017).

The FTIR spectrum of jackfruit cellulose (peak at 1060.85cm^{-1}) and commercial cellulose (peak at 1058.92cm^{-1}) are because of the C-O-C stretching vibrations of the β-1,4-glycosidic ring linkages between the D-glucose units in cellulose (Marchessault, 1962). Additionally, the foremost important intense peak at 1037.92cm^{-1} of commercial cellulose and a shoulder peak of the region 896.90cm^{-1} corresponded to the -C-H glycosidic deformation of cellulose part (Chandra et al., 2016). Moreover, 2 sharp signals at 896.90cm^{-1} of jackfruit cellulose and at 1429.25cm^{-1} of commercial cellulose reflect the crystalline band of cellulose (Fan et al., 2012).

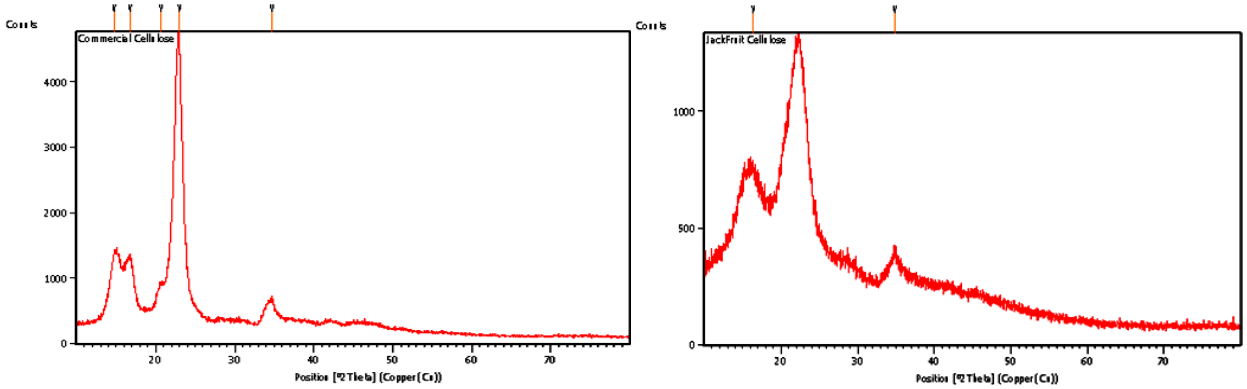
3.6 X- ray Diffraction (XRD)

In the present study, the XRD pattern of commercial and jackfruit cellulose obtained by Cu kα with the wavelength of (λ= 0.154nm), based on the report of Ismadji et al. (2008) was investigated. XRD analysis was conducted to probe the crystallinity of the cellulosic particles. Commercial cellulose has sturdy crystalline peaks at 2θ values of 14.7°, 16.7° and 23° cherish to (110), (110) and (002) planes of crystals main characteristic peaks of cellulose I and weak crystalline peak at 34.7° to the (004) plane is shown in figure 4 (a) .

Jackfruit cellulose has sturdy crystalline peaks at 16° and 23° cherish to (110) and (002) planes of crystals is shown in figure 4 (b), and weak crystalline peak at 34.84° to the (004) is shown in figure 4 (b). The XRD pattern confirms the presence of the most characteristic peaks of cellulose I at 2θ values of 16° - 23°, 2θ region indicating the preservation of native crystalline structure even in the presence of sodium hydroxide treatment.

The crystallinity index improved because of the partial removal of amorphous cellulose, hemicelluloses, and lignin, confirming similar reports on cellulose from various fruit peels like, banana peel (Elanthikkal et al., 2010; Deka & Khawas, 2016) and tomato peel (Jiang & Hseih, 2015). As observed in figure 4, the crystalline structure of the celluloses considerably differed. The CrI values of both cellulose samples are shown in table 1 (a) and (b), respectively. The standard cellulose I structure was confirmed by the diffraction pattern.

In jackfruit cellulose, 2θ angle at 34° obtained 100% crystallinity index; similar crystallinity index have also been reported from



(a) Commercial Cellulose (b) Jackfruit Cellulose

Figure 4: X - ray Diffraction Spectra of various cellulose (a) and (b)

Table 1a Crystallinity index (CrI) of commercial cellulose

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
14.7540	878.43	0.5353	6.00429	23.56
16.7394	813.17	0.4684	5.29636	21.81
23.0361	3728.01	0.9368	3.86093	100.00
34.7319	309.86	0.2676	2.58293	8.31

Table 1b Crystallinity index (CrI) of jackfruit Cellulose

Pos. [°2Th.]	Height [cts]	FWHMLeft [°2Th.]	d-spacing [Å]	Rel. Int. [%]
16.4102	99.06	0.9368	5.40185	96.90
34.8484	102.24	0.5353	2.57456	100.00

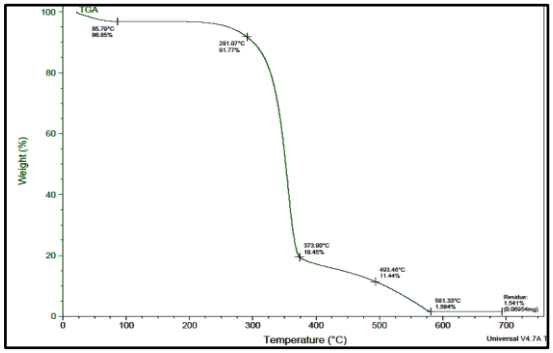
various celluloses (different fruit peels) like, tomato cellulose (78% to 85%, Jiang & Hseih, 2015) and banana cellulose (100% crystallinity index, Pelissari et al., 2014). Crystallinity index for both celluloses is found to be 100%. Similar studies were observed from cellulose of pomelo albedo peel by Zain et al. (2014). The enlarged crystallinity of each celluloses were additionally expected to justify their stiffness, rigidity and strength (Alemdar & Sain 2008; Azubuike & Okhamafe 2012).

3.7 Thermogravimetric analysis (TGA)

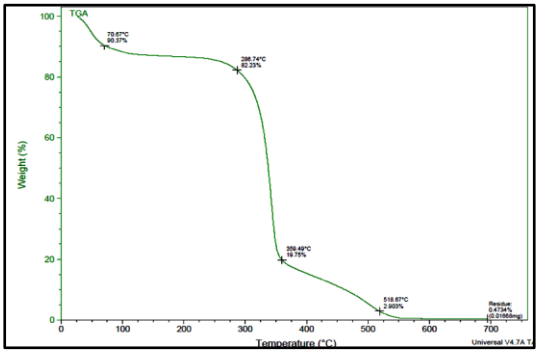
Thermal treatment produces a spread of chemical and physical changes (Yang et al., 2007). Thermogravimetric analysis is shown in figure 5.

For commercial cellulose, the primary step was observed between 25° - 85.79°C (figure 5a) with an initial weight loss. The second step in the temperature range between 85.79°C - 291.07°C could be attributed to decomposition of hemicelluloses and organic extractives (Boumediene et al., 2015). Hemicellulose has a random amorphous structure and it is easily hydrolyzed (John & Thomas, 2008; Poletto et al., 2014). The third step decomposition in the temperature range between 291.07°C - 373.90°C, was associated to the thermal degradation of cellulose. The last step overlaps between 373.90°C - 493.46°C, could be attributed to lignin degradation. Similar results have also been reported for orange peel (Orozco et al., 2012) and orange skin (Boumediene et al., 2015).

For jackfruit cellulose, the primary step was observed between 25° - 70.67°C (figure 5b) corresponds to an initial weight loss due to loss of moisture. A second step in the temperature range between 72°C and 286.74°C could be attributed to decomposition of hemicelluloses (Boumediene et al., 2015). The higher activity



(a) Commercial Cellulose



(b) Jackfruit Cellulose

Figure 5 Thermogravimetric analysis of both celluloses (a) and (b)

Table 2 Pyrolysis characteristic parameters of jackfruit and commercial cellulose

SI.NO:	Sample	T_1 (°C)	T_2 (°C)	T_3 (°C)	$T_{maximum}$ (°C)	Residue (%)
1.	Jackfruit cellulose	25 - 70.67	70.67 - 286.74	286.74 - 359.49	359.49 - 516.67	0.4734
2.	Commercial cellulose	25 - 85.79	85.79 - 291.07	291.07 - 373.90	373.90 - 493.46	1.541

*for $T_{1, 2 \& 3}$ - Temperature

Table 3 The Hunter color values of L, a, b and total color difference (ΔE) of jackfruit and commercial cellulose

Sample	L	a	b	ΔE^*_{ab}
Jackfruit Cellulose (JC)	84.64 ± 0.011	1.18	13.72 ± 0.005	13.25 ± 0.005
Commercial Cellulose (CC)	112.72 ± 0.01	1.99 ± 0.01	6.74 ± 0.01	19.76 ± 0.01

of hemicellulose in thermal decomposition might be attributed to its chemical structure (John & Thomas, 2008; Poletto et al., 2014). The third step in the temperature range between 286.74°C - 359.49°C, could be correlated with the random cleavage of the glycosidic linkage of cellulose (Severiano et al., 2010). The temperature range between 359.49°C - 516.67°C, could be attributed to the weight loss due to lignin degradation (Vaidya et al., 2016). The results are in good agreement with those reported for cellulose from apple pulp (Garcia et al., 2002), orange peel (Orozco et al., 2012), orange skin and almond peel (Boumediene et al., 2015).

Thermal stability of commercial cellulose was found to be higher than the jackfruit cellulose. The residue of jackfruit cellulose (0.4734%) was found to be less as compared to commercial cellulose (1.541%) is shown in table 2. Among the cellulose samples, commercial cellulose had higher residual char value of 690°C indicating higher nonvolatilisable carbonous material generated during thermal decomposition of organic material (Sain & Panthapulakkal, 2006).

3.8 Color

The appearance or color of cellulose was a crucial factor in terms of consumer acceptance. The lightness, *L**, represents the darkest black at *L** = 0, and the brightest white at *L** = 100. The red/green opponent colors were represented along the *a** axis, with green at negative *a** values and red at positive *a** values. The yellow/blue opponent colors were represented along the *b** axis, with blue at negative *b** values and yellow at positive *b** values. The L, a and b Hunter Lab color values, and total color distinction (ΔE), of celluloses, are shown in table 3.

The major variation in colour values of commercial cellulose was a high L* values (brightest white) and lower b* values. Jackfruit cellulose showed lower L* value, higher b* value. This indicated that commercial cellulose was clearer than the jackfruit cellulose. The colour change might be due to alkali treatments of jackfruit cellulose (Bolin & Huxsoll, 1991).

Conclusion

Cellulose was extracted from de-pectinated peel. The peel was treated with alkali followed by a chemical process treatment. The outer skins of jackfruit (A*rtocarpus integer*) could be used as a source of cellulosic material and its cellulose derivatives. The yield of jackfruit cellulose was 27g / 100g of dry matter. SEM images showed that jackfruit cellulose displayed roughness image due to aggregation. XRD results revealed that chemical/alkali treatment improved crystallinity. Results shows that jackfruit peel as a potential source of natural cellulose has been comparing favourably with commercial grade cellulose used for food and pharmaceutical applications.

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Conflict of Interest

Authors would hereby like to declare that there is no conflict of interests that could possibly arise

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