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ORIGINAL PAPER

Extraction and Characterization of Microcrystalline Cellulose from Date Palm Fibers using Successive Chemical Treatments

Amina Hachaichi[1] · Benalia Kouini[2] · Lau Kia Kian[3] · Mohammad Asim[3] · Mohammad Jawaid[3]

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Abstract

The aim of present study is to extract microcrystalline cellulose (MCC) from fruit bunch branches fibers of Algerian date palm trees (phoenix dactylifera L) as biofiber for reinforcing green composite and thus replace synthetic fibers in various applications. The extraction of MCC from date palm fibers passed via serial chemical treatments, including alkali, bleaching and acid hydrolysis process. Subsequently, several analyses were implemented to determine the characteristics of each sample prepared at different stages of treatment. Fourier transform infrared spectroscopy (FTIR) analysis revealed the effectiveness in removing substantial amorphous components of lignin and hemicellulose from date palm fibers. Altered and irregular shaped morphology of microfibrils with slightly rougher surface was observed for microcrystalline date palm fibers (MCC-DP) through scanning electron microscope (SEM) examination. Furthermore, X-ray diffraction (XRD) presents the increasing of the crystallinity from 55% in raw date palm (R-DP) to 76.26% in MCC-DP. Also, the results of TGA and DSC indicate the MCC-DP has greater thermal stability than that of R-DP, A-DP and B-DP fibers. These results demonstrate the feasibility of using date palm waste (fruit bunch branches fibers) to extract a good reinforcing material (MCC) with high properties and low cost, which qualifies its use in composite materials. Also, it can be transformed into nano-scale for isolating nanocrystalline cellulose with the aim of using it, in the future to produce ecofriendly bionanocomposites in different fields of applications, biomedical, pharmaceutical and packaging.

Keywords Microcrystalline cellulose · Date palm · Chemical treatments · Morphology · Crystallinity

Introduction

Date palm (*Phoenix dactylifera L*) is a desert palm tree that categorized under the *Arecaceae* family. Its height is estimated at about 20–30 m, and this tree is widely cultivated in semi-arid and arid areas, such as United States, Middle East and North African countries. The life cycle of these trees can be more than 100 years. Algeria is the 5th country in the world ranking in terms of the number of palm trees (over 18.6 millions trees) [1, 2].

Each year, these trees can produce about 200,000 tonnes of lignocellulosic waste after planting or fruit harvesting (branches, stalks and trunks). Meanwhile, these agro-wastes can be used in the production of environmental friendly materials with low costs for many applications, such as in textile, baggage, sports item, and automotive parts [1, 3]. The extraction of date palm fibers from various wastes parts of date palm trees can be conducted by immersing these parts in a water [4]. Date palm fiber is multi cellular lignocellulosic fiber comprises 35–45.1 ± 3.4% cellulose, 9.75–27.7 ± 1.5% hemicellulose and 11.45–29.48% lignin [5]. It's is a more sustainable biofiber resource and possesses good mechanical strength which is better than other natural fibers [6]. Only a limited number of research in open literature related to the use of date palm fibers as a reinforcement for composite materials, Al-Othman et al. [3] made an attempt to develop a polypropylene composite by reinforcing with date palm fibers, while Abdal-Hay et al. [2] reported

✉ Mohammad Jawaid
jawaid@upm.edu.my

1 Research Unit Materials, Processes and Environment (URMPE), Faculty of Technology, M'hamed Bougara University, 35000 Boumerdes, Algeria

2 Laboratory of Coatings, Materials and Environment, Faculty of Technology, M'hamed Bougara University, 35000 Boumerdes, Algeria

3 Laboratory of Biocomposite Technology, Institute of Tropical Forestry and Forest Products (INTROP), Universiti Putra Malaysia, 43400 UPM, Serdang, Selangor, Malaysia

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having obtained an epoxy composite reinforced with date palm fiber.

Plant cell wall is primarily composed of the cellulose, which give support and rigidity to the structures [7]. Cellulose can be extracted from natural fibers such as date palm fiber and other fibers such as oil palm, coconut, cotton, wood, roselle, sisal, flax, ramie, and jute [8]. It is a semi-crystalline material represented by the chemical formula of $(C_6H_{10}O_5)n$ [9]. Cellulose is characterized by high mechanical properties, good thermal resistance, low cost, and low density, in addition to its remarkable reinforcing capability, renewable and biodegradable criteria [8, 10]. These advantages have drawn the attention of researchers to utilize cellulose as an excellent bio-filler to develop ecofriendly polymer composites [11].

Nowadays, microcrystalline cellulose (MCC) is gaining more attraction, since it can be used as reinforcing agent in composite materials or it can be transformed into nanoscale to develop nanocrystalline cellulose (NCC) filler for polymer nanocomposites. MCC can be applied in various applications like aerospace, construction, pharmaceutical and pack-aging industries [8, 12]. The extraction of MCC from native cellulose can be gone through various process, including chemical treatment (acid hydrolysis) [12, 13], biological treatments (enzymatic hydrolysis) [14, 15], mechanical treatments (steam explosion) or combination of them [16, 17]. The type of raw fiber and the conditions applied during the extraction process (temperature, reaction time, type and acid concentration) are essential influences on the final properties of the MCC product [18, 19]. In addition, Abdullah et al. [20] have reported the successfully isolation of MCC from pineapple leaf fibers and coconut husk fiber using alkali treatment with potassium hydroxide (6 wt. %) and bleaching treatment with acidified sodium chlorite. Kian et al.[8] obtained MCC from roselle fibers, utilizing sodium hypochlorite (NaClO) solution with about 10% (w/v) for bleaching, while sodium hydroxide (NaOH) solution with around 8.0% (w/v) for alkali treatment, followed by hydrochloric acid (HCl) with 2.5 mol/L for acid hydrolysis reaction. Moreover, Tarchoun et al.[13] isolated microcrystalline cellulose from giant reed using alkali treatment of 1.25 M sodium hydroxide to solubilize lignin and hemicellulose, which then followed by two bleaching steps for further delignification. In the bleaching treatments, it involved the utilization of 2.6 M H_2O_2 and 4.4 M CH_3COOH solution in the first step, while the second step with 2.1 M H_2O_2 and 1 M NaOH solution for purifying the pulp. Ultimately, the purified giant reed cellulose was treated with hydrolysis by employing single-acids and blended-acids. Hence, an integrated process of alkaline, bleaching, and acid hydrolysis is considered as the most common method used for extraction process due to the appropriateness and effectiveness in preparing MCC particles [17].

From previous studies. This work is considered the first of its kind as there are no paper reports on the extraction of MCC from fruit bunch branches fibers of Algerian date palm trees. In this respect, the aim of this research is to extract MCC from a novel type of fibers which was derived from fruit bunch branches of Algerian date palm trees, with goals of using it as a reinforcing material of biocomposites for packaging applications, also, it can be used as a starting material for nanocellulose isolation to develop Bio-nanocomposite materials in future. The MCC obtained after serial chemical treatments, was analyzed by applying different characterization techniques such as FTIR, XRD, SEM, TGA, and DSC.

Material and Experimental Details

Materials and Chemicals

The fruit bunch branches of date palm tree were collected from M'sila-Algeria.The date palm fibers were extracted by using water retting technique as described by Amroune et al. [1]. Sodium hydroxide (97%), sodium chlorite (80%), acetic acid glacial (99.8%) and sulphuric acid (95–97%) were used as purchased from supplier.

Microcrystalline Cellulose Isolation

Washing Process

Following the date palm fibers were cut into small pieces, they were crashed mechanically, Fig. 1a, then immersed in tap water at 100 °C with stirring for 1 h to remove hydrosoluble impurities, finally they were dried at 50 °C in a vacuum oven for 15 h.

Alkali Treatment

To obtain pure cellulose, the date palm fibers were treated with 4 wt.% NaOH solution for 2 h at 80 °C. The mixture was then filtered, washed several times with distilled water and dried at 50 °C in a vacuum oven for 24 h. This treatment was repeated twice, Fig. 1b.

Bleaching Treatment

After alkali treatment, 1.7wt % of sodium chlorite solution ($NaClO_2$) solution was prepared and it was added with acetic acid reaching pH 4. After which the palm fibers were treated with this solution at 80 °C for 2 h, this mixture was filtered with distilled water to obtain white color fiber, and the obtained fiber was dried in vacuum oven at 50 °C for 24 h.

Fig. 1 Isolation of MCC-DP from R-DP

Table 1 Abbreviations of samples names

Names of samples	Abbreviations
Raw date palm fiber	R-DP
Alkali treated date palm fiber	A-DP
Bleaching treated date palm fiber	B-DP
Microcrystalline date palm fiber	MCC-DP

The bleaching treatment was performed twice to purify the cellulose component, Fig. 1c.

Acid Hydrolysis Treatment

Afterwards, the pulp fiber was treated by using 64 wt. % of sulphuric acid (H_2SO_4) solution at 45 °C for 30 min under constant stirring to extract MCC. The obtained mixture was filtered and washed thoroughly with distilled water for complete removal of acid, then it was dried at 50ºC in a vacuum oven for 24 h, Fig. 1d, The abbreviations of samples names after each chemical treatment are shown in Table 1.

Characterization

Fourier Transform Infrared Spectroscopy (FTIR)

FTIR spectroscopy is an analysis method for identifying the functional groups of fiber samples from their vibrational properties. Perkin-Elmer 1600, a spectrometer was used in this analysis by conducting 32 infrared scanning in the range of 650–4000 cm^{-1}. Also, a Nicolet software program was used to track the positions of significant transmittance peaks at a given wavenumber.

Morphological Analysis

To determine the changing on the morphology of the different samples after each stage of chemical treatment, a scanning electron microscopy (Hitachi ModelS-3400 N) was used for the examination under an accelerated voltage of 15 kV. Prior to viewing, the samples were sputtered with gold to avoid the effect of charging.

X-ray Diffraction (XRD)

The crystallinity of each sample was examined by using a diffractometer (SIEMENSD 5000 X-ray) under Ni-filtered Cu Kα radiation at 5° to 40° angular incidence of 2θ angle

range. The equation as below was used to calculate the crystallinity index (CrI):

$$CrI(\%) = \frac{(I_{002} - I_{am})}{I_{002}} \quad (1)$$

where, I_{am} represents the intensity of amorphous domain at $2\theta = 19.2°$, I_{002} represents the intensity of the crystal domain at $2\theta = 22.5°$.

Thermal Analysis

To determine the thermal stability, around 6 mg of each fiber was weighed and scanned using a thermal gravimetric analyzer (TA Instruments Q500) within a temperature profile of 30–900 °C at the heating rate of 20 °C/min under the condition of flowing nitrogen gas. Meanwhile. DSC thermograms have also been implemented by using TA Instruments Q20 in a temperature range of 30–500 °C at heating rate of 10 °C/min under atmosphere of nitrogen purge. DSC analysis enables us to study the thermal molecular change behaviour within the date palm fiber.

Results and Discussion

FTIR Spectroscopy

Figure 2, represents the infrared analysis of R-DP, A-DP, B-DP and MCC-DP samples. All samples show closely similar spectra, confirming that they have the same chemical composition. The obtained spectra also proved the insignificant effect of the various treatments on the lignocellulosic compounds due to the presence of identical functional groups. These spectra typically show two prominent absorption regions, where the first region noted at high wavenumbers range of 2700–3700 cm^{-1} and the second region observed at low wavenumbers range of 800–1800 cm^{-1}, which corresponded to the MCC extraction reports by Kian et al. [8], Tarchoun et al. [13], and Hermawan et al. [21]. A wide absorbance band appears from 3300 cm^{-1} to 3500 cm^{-1} range, are attributing to the presence of hydroxyl groups (OH) of cellulose for each fiber sample. However, the band intensity of MCC-DP appeared weaker when compared to others samples, due to the agglomerated MCC particles that had led to the less exposure of cellulose molecular chains [11, 13, 22]. The reduced band intensity was also noticed for MCC-DP sample from 2800 cm^{-1} to 2900 cm^{-1}, because of the lessened C-H groups vibration in its highly crystalline cellulose component [8, 13]. Furthermore, a hemicellulose characteristic peak at 1739 cm^{-1}, relating to the acetyl and

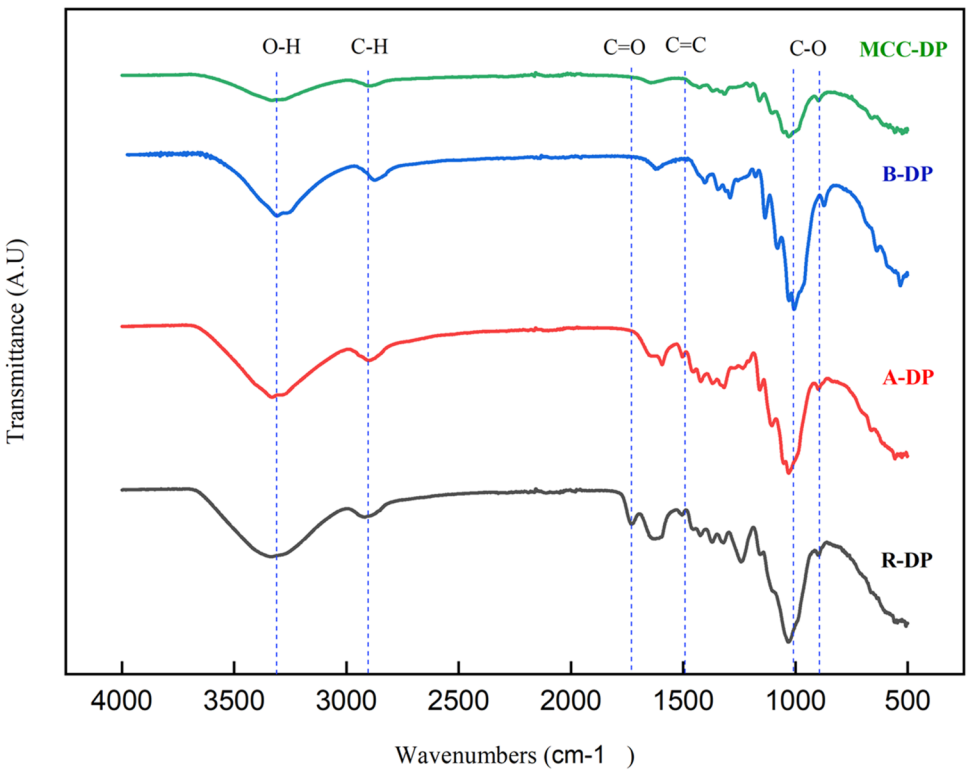

Fig. 2 FTIR spectra of R-DP, A-DP, B-DP and MCC-DP

uronic ester (C = O) groups had disappeared after successive treatment of date palm fiber, indicating the total removing of hemicellulose [23]. Also, a small peak observed at about 1492 cm^{-1} was associating to the C = C groups of lignin. This peak had disappeared in the spectrum of the MCC-DP sample, which indicate the completely removing of lignin compound through the disruption of cellulose-lignin linkage [9, 24]. The complete disappearing of amorphous parts (hemicellulose and lignin) is interesting point indicate the strong role of successive chemical treatments which was used to extract microcrystalline cellulose [10, 25]. These results are much better compared to some literature by [8, 12].

Besides this, the absorption peak at 1624 cm^{-1} had reflected the occurrence of water absorption (H–O-H), which attributing to the hydrophilic nature of cellulosic material [8, 25]. Moreover, the noted peaks at 1031 cm^{-1} and 893 cm^{-1} were attributed to the vibration of C–O–C skeletal pyranose ring and β-1,4-glycosidic bonding, revealing the typical structural chemistry of cellulose molecules [26].

Morphological Analysis

The Fig. 1e, represents the experimental samples appeared in different colors after each chemical treatment, indicating the change of their morphology. Significantly, it was observed that the fine powder feature of R-DP fiber particles had changed to larger globular shape particles for A-DP fiber, showcasing the self-assembly behaviour of fibrils structure after reaction with NaOH. With bleaching treatment, the brown color of A-DP had turned into a white color powder for B-DP sample, revealing the typical criterion of pure cellulose pulp. The color change also indicated the effective dissolution of amorphous components for date palm fiber by successive chemical processes. In final acid treatment, the irregular shape of B-DP pulp fiber was hydrolysed into the extremely fine particles for MCC-DP sample, evidencing the successful disintegration of large fibrous structure into small particles.

Apart from that, the topographic surface of these samples was further examined by scanning electron microscope (SEM) as seen in Fig. 3a–d. From Fig. 3a, the R-DP is shown as a compact bundle of fibrous feature that generally bound with 'cementing materials, such as lignin, hemicellulose and pectin amorphous compounds [24]. Meanwhile, some small particulates were observed on the surface, revealing the presence of residual cellulosic substances in these fibers [1]. After alkaline treatment, a longitudinal soft structure with the exposure of parallel aligned fiber surface morphology was presented by A-DP in Fig. 3b. It was contributed by the separation of bundles into individual fibers through the solubilization of non-cellulosic materials [24, 25]. The surface became clearer and smoother for B-DP fiber following the bleaching treatment, implying the complete removal of amorphous components, Fig. 3c [18, 27]. With further acid hydrolysis treatment, an intertwine and entangled microfibrils structure was exhibited by MCC-DP sample, Fig. 3d [28]. During hydrolysis, the hydronium ions generated by the H_2SO_4 had penetrated the internal fiber structure, and subsequently disintegrated the fibers into micro-size fibrous feature [21, 29]. The sulfuric acid contributed to obtaining this result, which is considered better than that obtained when using hydrochloric acid (HCl) as proven by other reported studies [8, 12, 30]. The small particle size of MCC-DP could be a favourable starting material for isolating nanocellulose product [8, 31].

X-ray Diffraction

To assess the crystallinity property of the R-DP, A-DP, B-DP and MCC-DP samples, XRD analysis is carried out for these samples as shown in Fig. 4, and the results are summarized in Table 2. Two main peaks were appeared in all spectra of samples at $2\theta = 16.1°$ and $2\theta = 22.5°$, reflecting that all date palm fibers contained crystal region for cellulose compartment. The presence of additional peak at $2\theta = 34.9°$ was noted for B-DP and MCC-DP samples, revealing both fibers were with native cellulose I_β type structure [11, 26]. However, the unobservable of this extra peak for R-DP and A-DP, possibly embedded by the large proportion of binding components that somehow reduced the detection of cellulose crystalline structure. In among the fibers, the crystallinity index for R-DP was estimated the lowest of 55%, which in line with its low intense crystallinity peak at 22.5°. After successive chemical treatments, the crystallinity index was increased with the sharpness of 22.5° peak for A-DP, B-DP, and MCC-DP samples, Table 2. The crystallinity index obtained in this study (CrI = 76.26%) was higher than the crystallinity index obtained by several reported works, Fruit Bunch Stalk of date palm (CrI = 70%) by Alotabi et al. [22] Olive MCC (CrI = 74.2%) by Kian et al. [30] and Jute MCC (CrI = 71%) by Jahan et al. [32]. The increment of crystallinity index of date palm fiber after each chemical treatment was related to the removal of lignin and hemicelluloses constituents [10, 26]. The high crystallinity of MCC-DP implied the high rigidity of its pure cellulose structure, and that qualifies it to be used as biofiller for composite materials in order to provide them with high mechanical properties [14, 26].

Thermal Properties

The thermal stability of MCC is an important parameter for the use of these fibers in the production of biocomposites at high temperature [32, 33]. Figure 5 and Fig. 6 represents the TGA and DTG curves respectively, for R-DP, A-DP, B-DP

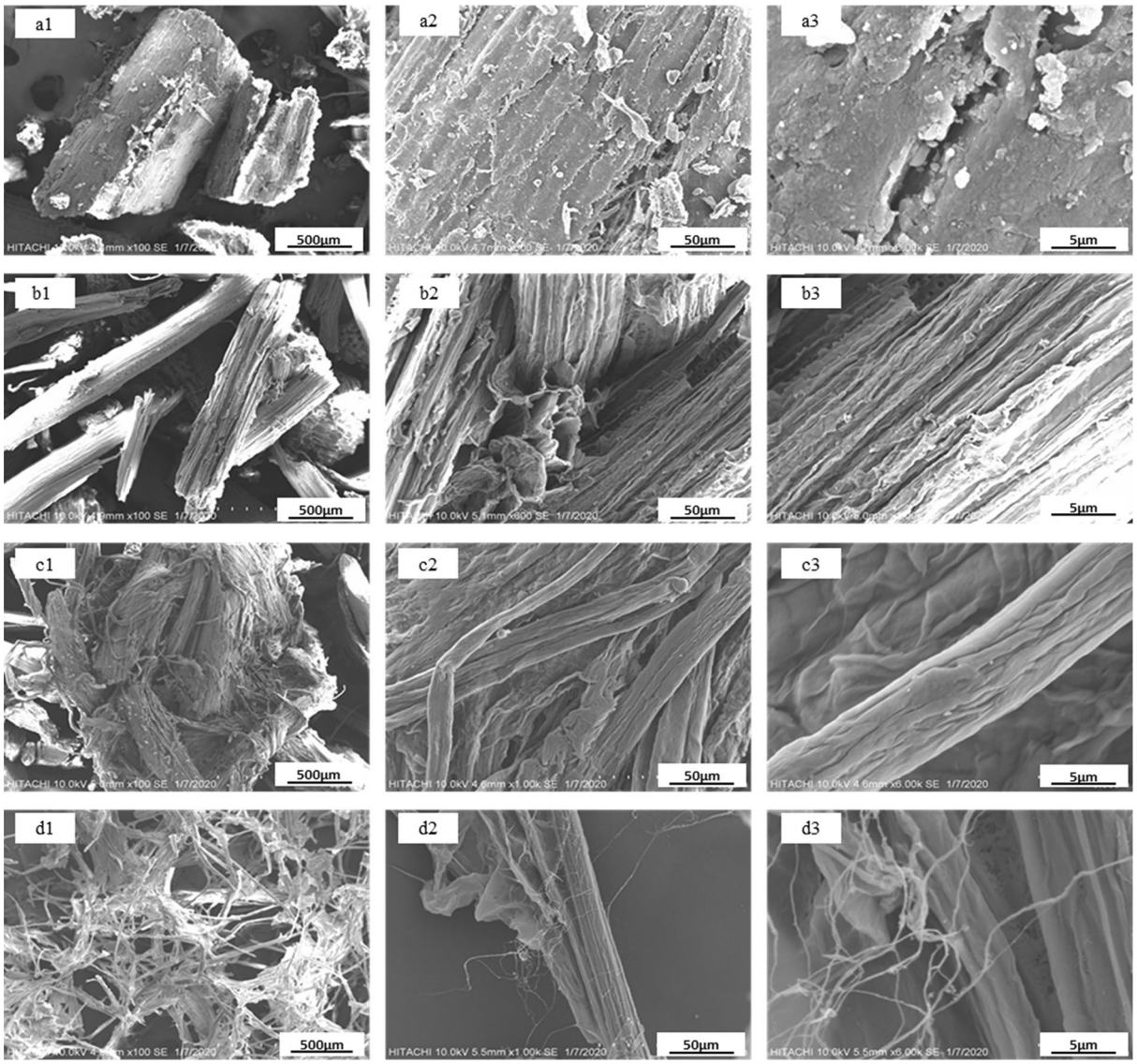

Fig. 3 SEM of R-DP (**a**), A-DP (**b**), B-DP (**c**) and MCC-DP (**d**)

and MCC-DP, the Table 3 show the thermal analysis data for these samples.

The thermal degradation of all samples presents two stages. The first drying stage from 30 to 140 °C represented all fibers have lost considerable weight due to the evaporation of water and volatile compounds on theirs surface [8, 11, 34]. In the second stage beyond 200 °C, the thermal decomposition of R-DP sample began at 223.84 °C indicates its low thermal stability compared to the other samples. Also, the MCC-DP sample shown higher final decomposition temperature at 357.08 °C due to its high molecular arrangement, which makes its decomposition requires high thermal energy [8, 12]. The degradation peak temperature of MCC-DP occurred at 380◦C, which was higher than R-DP, A-DP and B-DP. R-DP sample lost a weight of 61.71%, which is the lowest weight compared to the other samples. In addition, the residual weight of this sample is the highest value of 23.78% which is due to the presence of non-decomposing components that contributed to the char formation [35]. Also, the residual weight is the highest of 89.10% in MCC-DP sample because the cellulose is highly pure [36]. At 150 °C, the components of cellulose are degraded and their decomposition continue up to 385 °C, this is when decarboxylation, depolymerization and decomposition occur in the cellulose and hemicellulose fragments. Biomass is expose to aromatization, decomposition, combustion, lignin pyrolysis and char residue formation beyond the temperature of 385 °C [8, 12].

DTG peak temperature of MCC-DP at 380 °C was greater than those of MCC extracted from: Roselle fibers at

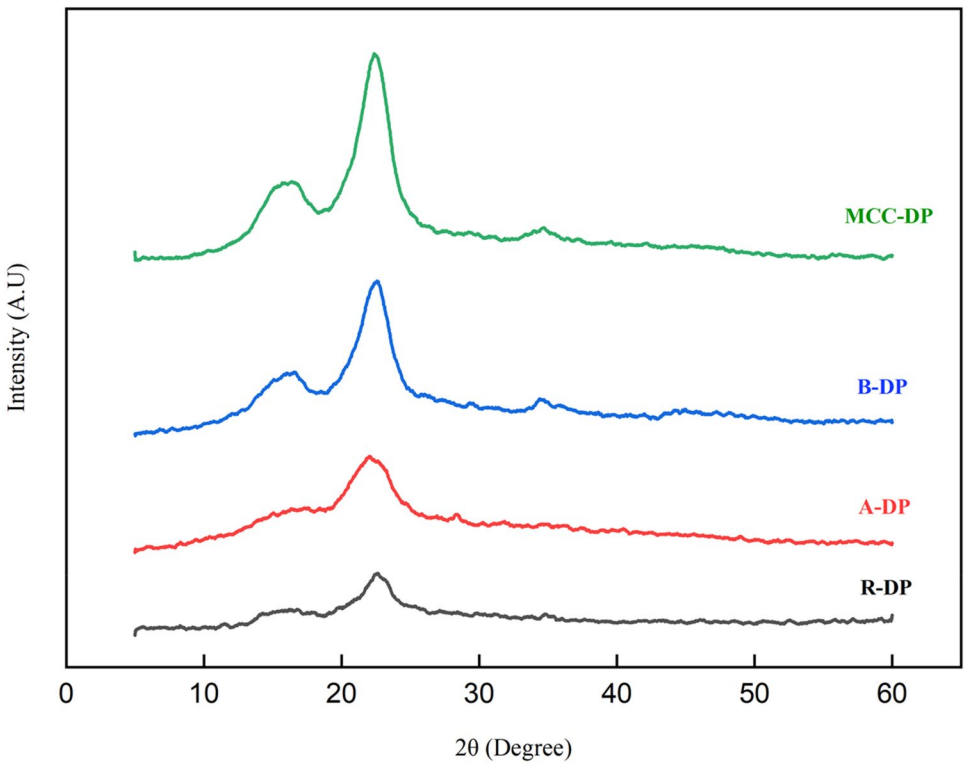

Fig. 4 X-ray diffractograms of R-DP, A-DP, B-DP, and MCC-DP samples

Table 2 Crystallinity index of date palm fibers at different stages of chemical treatment

Samples	CrI (%)
R-DP	55
A-DP	68.48
B-DP	72
MCC-DP	76.26

340.12 °C, as reported by Kian et al. [8], Bamboo fibers at 353 °C,as reported by Rasheed et al. [12], and Fruit Bunch Stalk of Date Palm at 364.2 °C by Alotabi et al. [22], from these results, it can be concluded that the MCC-DP has a hight thermal stability which allow its use as reinforcing material for composite.

The DSC curves are illustrated in Fig. 7 and their data is presented in Table 3. The endothermic peaks shown from 40 °C to 150 °C were related to the process of water evaporation [22]. Small endotherms were differently observed at 143.59 °C, 144.94 °C, 141.66 °C, and 140.20 °C for R-DP, A-DP, B-DP and MCC-DP, respectively. According to reported study, the thermal energy required by fibers to evaporate their water content was different due to the varying non-substituted hydroxyl groups existed on their surface. For fibers had high affinity for water molecules would require more thermal energy to evaporate their water content. In this work, the decrement of hydroxyls groups was reflected with the reduced temperature of endotherms from A-DP to MCC-DP samples, evidencing the necessitated thermal energy became smaller for the chemically-treated fibers following the successive treatment processes [8].

From 160 °C onwards, the occurrence of large endothermic peaks is significantly noticed at 177.17 °C,182.23 °C,182.85 °C and 185.73 °C,respectively for R-DP, A-DP, B-DP and MCC-DP, these endothermic peaks present the depolymerization and decarboxylation of cellulose [30, 37]. This is due to the predominant amorphous regions in R-DP, which made it more susceptible to initial thermal degradation, when compared to MCC-DP, which is considered more thermally stable, so it can be concluded that, the chemical treatment has an effect on the thermal stability of the fibers [38].

Conclusion

The major finding in this work is the extraction process was successfully done, through three chemical analysis steps of alkali, bleaching, and acid hydrolysis, which was confirmed by different analyses. FTIR analysis confirmed the crystalline structure of MCC-DP which also shown by XRD analysis with crystallinity index of CrI = 76.26% endowing it with rigidity for reinforcing composite material. Moreover, the SEM results demonstrated that the MCC-DP has a rough and irregular shape which is appropriate morphology to isolate nanocrystalline cellulose in future, based on the TGA and DSC analysis, MCC-DP represent greater thermal stability,

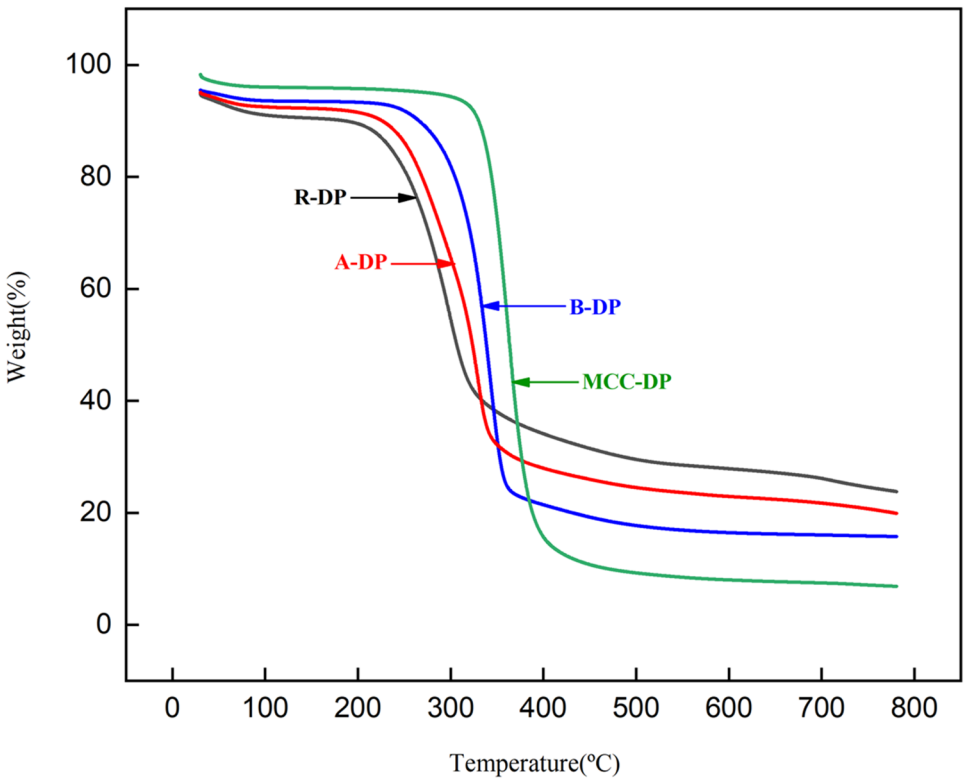

Fig. 5 TGA curves of R-DP, A-DP, B-DP, and MCC-DP samples

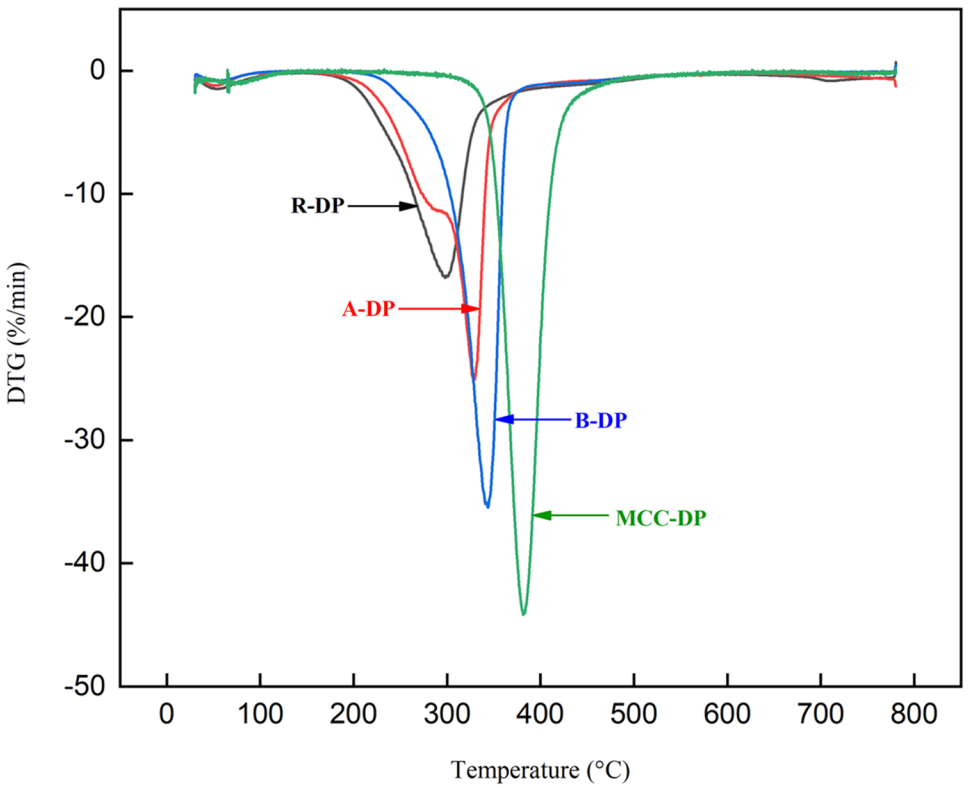

Fig. 6 DTG curves of R-DP, A-DP, B-DP, and MCC-DP samples

Table 3 Thermal data of R-DP, A-DP, B-DP and MCC-DP

Samples	TGA analysis					DSC analysis		
	$T_{initial}$ (°C)[a]	T_{final} (°C)[b]	T_{peak} (°C)[c]	W_{loss} (%)[d]	$W_{residue}$ (%)[e]	$T_{initial}$ (°C)[f]	T_{peak} (°C)[g]	ΔH (J/g)[h]
R-DP	223.84	323.855	299.04	61.71	23.78	163.59	177.17	125.81
A-DP	240.77	340.27	330.02	68.90	19.88	180.20	182.23	103.30
B-DP	279.81	340.40	343.66	76.63	15.76	181.66	182.85	100.50
MCC-DP	288.60	357.08	380	89.10	6.807	184.94	185.73	85.50

[a]TGA initial decomposition temperature

[b]TGA final decomposition temperature

[c]DTG peak temperature

[d]TGA maximum weight loss

[e]TGA char residue weight

[f]DSC Initial decomposition temperature

[g]DSC Peak temperature

[h]DSC Heat of decomposition

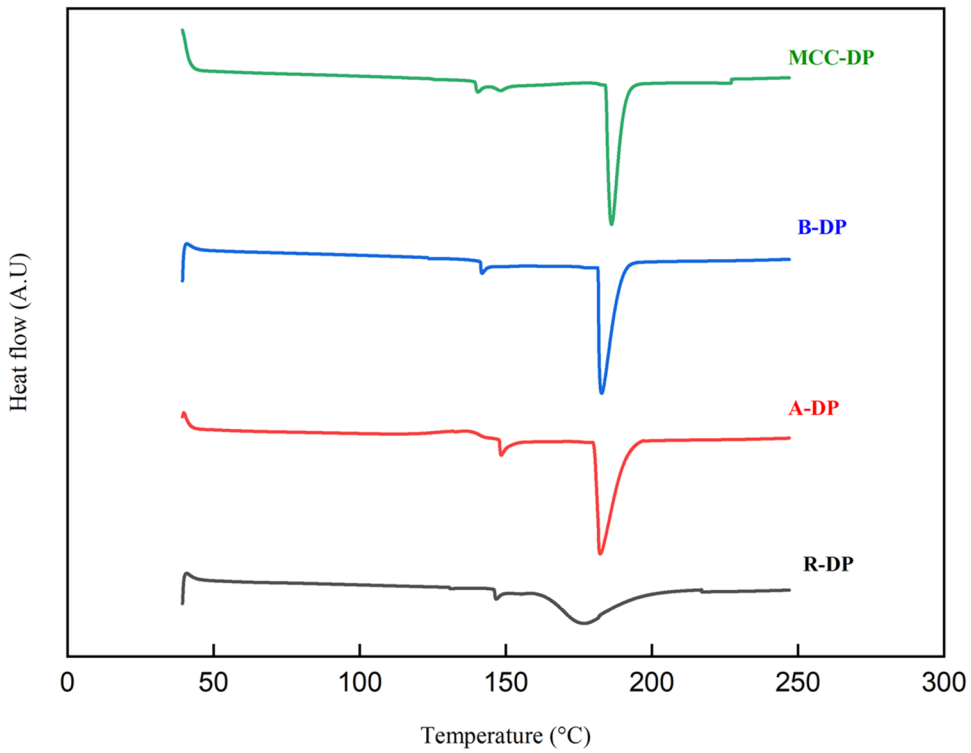

Fig. 7 DSC curves of R-DP, A-DP, B-DP, and MCC-DP samples

that is suitable as a good reinforcing element for application in polymer composites of higher processing temperature. The MCC-DP extracted in this study can be utilized for isolation of nanocrystals Cellulose (NCC), which also can be used to prepare cellulosic aerogels, therefore to use it as reinforcing materials, for future bionanocomposite fabrication process in packaging applications.

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