

Synthesis and Characterization of Cellulose Ethers from Screw Pine (*Pandanus tectorius*) Leaves Cellulose as Food Additives

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Abstract. Isolation of cellulose from Screw pine (*Pandanus tectorius*) leaves and its transformation into cellulose ethers have been successfully conducted. Crystalline cellulose was obtained as a white powder in 40.24% yield from dried Screw pine leaf powder through alkalization with sodium hydroxide followed by bleaching with sodium hypochlorite. The crystalline cellulose was converted to methyl cellulose (MC), carboxymethyl cellulose (CMC), and hydroxypropyl cellulose (HPC) and obtained 94.6%, 71.47%, and 81.54% yields, respectively. The physicochemical properties of MC, CMC, and HPC, such as shape, color, pH, Degree of Substitution (DS), and viscosity meet the standards for application as a food additive. The pH and viscosity of the synthetic cellulose ethers were 7.53-7.60 and 61.98-62.41 cP, respectively. The DS value for MC, CMC, and HPC were 1.680, 0.996, and 0.696, respectively. The study revealed the potential of Screw pine cellulose to obtain food-grade cellulose ethers since the properties of MC, CMC, and HPC agree with food additive criteria.

Keywords: Cellulose; Cellulose ethers; Food additive; *Pandanus tectorius*; Screw pine

1. Introduction

Food additives are often added during food processing to improve the taste, flavor, color, texture, and nutritional value. Food additives are also used as preservatives for longer storage. The consumption of food additives, especially synthetic additives, can cause long-term negative health effects (Sahu, 2017). Therefore, there is a great interest in the use of food additives from natural sources to minimize the side effects. Demand for functional food has been growing globally (Suryanti *et al.*, 2021; 2020; 2016). Biopolymer-based materials from polysaccharide, especially cellulose has great potential to be used as food additives (Li *et al.*, 2018). In particular, cellulose derivatives are widely used as food since they have low viscosity which adds new properties to food products.

Cellulose can be obtained from natural resources by acid-alkali treatment. The production of cellulose and its derivatives from raw materials that are economically cheaper and locally available is desirable. Cellulose is commonly converted into useful derivatives by reactions typical of compounds containing hydroxyl groups. The hydroxyl groups (-OH) of cellulose can be partially or fully reacted with various reagents to obtain

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cellulose ethers with useful properties (Tosh, 2014). The isolation of cellulose ethers in living organisms, such as plants, animals, bacteria, fungi, and yeast, is not reported yet. Therefore, synthetic cellulose ether needs to be developed. Etherification of cellulose changes cellulose's solubility significantly, where cellulose ethers dissolve in water, dilute acids, dilute alkalis, or act as organic solvents. Cellulose ethers, such as methyl cellulose (MC), carboxymethyl cellulose (CMC) and hydroxypropyl cellulose (HPC), have demonstrated useful applications in various areas including food, personal care products, oil-field chemicals, construction materials, the paper industry, as well as adhesives and textile industries (Kimani *et al.*, 2016; Bhatt *et al.*, 2011).

In Indonesia, Screw pine (*Pandanus tectorius*) grows wild along the northern coast of Java, Kepulauan Seribu, and Sumatra. Screw pine leaves produce strong fibers that are often used to make ropes and also to weave hats and mats. Screw pine leaf contains cellulose (37.3%), hemicellulose (34.4%), pentose (15.7%), lignin (24.0%), and extractive (2.5%) (Sheltami *et al.*, 2012). Thus, Screw pine leaf is a promising source of cellulose. Cellulose has been isolated from Screw pine and used as a polymer composite (Afolabi *et al.*, 2019). Nevertheless, the synthesis of cellulose ethers such as MC, CMC, and HPC from the cellulose of Screw pine leaves has not been reported yet. Therefore, this study focuses on the synthesis and characterization of MC, CMC, and HPC from the cellulose of Screw pine leaves.

2. Methods

2.1. Materials and Methods

The Screw pine leaves were obtained from Gunung Kidul coastal area, Yogyakarta, Indonesia. All chemicals were purchased from Sigma-Aldrich (Germany) and used without any further purification. Fourier Transform Infrared (FT-IR) spectrum was obtained by SHIMADZU FT-IR-prestige 21 using a KBr pellet in the range of 4000–400 cm^{-1} . The powder was characterized by X-Ray diffraction XPert MPD using Cu-K α radiation ($\lambda = 1.5406 \text{ \AA}$; $2\theta = 5\text{--}60^\circ$) at the voltage of 40 kV and current at 30 mA. Scanning Electron Microscope (SEM) data were collected by SEM JSM-6510 with voltage at 10 kV and pressure at 70 Pa. Thermogravimetric Analysis (TGA) of STA Linseis pt 1600 was used where the sample was heated to 600°C at a heating rate of 10°C/min under atmospheric pressure.

2.2. Isolation of Cellulose from Screw Pine Leaves

Cellulose was isolated from Screw pine leaves according to the previous method with some modifications (Afolabi *et al.*, 2019). Screw pine leaves were cut into 10-12 cm in length, washed with water, and then sun-dried. The dried leaves were then cut into 1 cm lengths and soaked with water for 72 h. The water was changed once every 24 h. The leave pieces were then sun-dried and ground to powder. The leave powder (100 g) was mixed with 10% (w/v) NaOH solution at a ratio of 1:20 (w:v). The mixture was heated to 100 °C and stirred for 2 h. The mixture slurry was then filtered. The solid phase was neutralized by washing with distilled water and then dried. The dried pulp was subjected to bleach with 4% NaOCl (pH = 4.5) at a ratio of 1:20 (w:v) at 100 °C for 2 h. After cooling, the mixture was filtered and the solid phase was neutralized by rinsing with distilled water and then dried at 60 °C for 1 h. The percentage yield of cellulose isolated was calculated using equation (1).

$$\text{Yield of cellulose (\%)} = \frac{\text{Mass of cellulose (g)}}{\text{Mass of dried leaf powder (g)}} \times 100 \quad (1)$$

The isolated cellulose was then analyzed by Fourier Transform Infra-Red (FTIR) spectroscopy, Scanning Electron Microscopy (SEM), Thermogravimetric Analysis (TGA), and X-ray Diffraction (XRD).

2.3. Synthesis of Methyl Cellulose (MC)

MC was synthesized following [Viera *et al.* \(2007\)](#) method with some modifications. Screw pine leaf cellulose (1 g) was added to a mixture of 50% NaOH (5 mL) and isopropanol (30 mL). The mixture was stirred for 1 h at room temperature. The mixture was then filtered and the residue was mixed with acetone in a ratio of 1:20. The mixture was refluxed for 6 h at 50°C with dichloromethane being added dropwise in a ratio of 1:10. The mixture was neutralized with 10% acetic acid and then washed with 95% ethanol. The residue was dried at 50°C to obtain pure MC.

2.4. Synthesis of Methyl Cellulose (CMC)

CMC was synthesized from Screw pine leaves cellulose according to [Parid *et al.* \(2018\)](#) method with some modifications. The CMC synthesis was performed in two steps: alkalization and etherification of cellulose. In the alkalization reaction, 1 g of cellulose was stirred in a mixture of 3 mL of 20% NaOH and 20 mL isopropanol for 1 h at room temperature. For the etherification reaction, the mixture was heated to 50°C and gradually added with a solution of trichloroacetic acid/isopropanol (1 g: 4 mL) and further stirred for 3 h. The resulting slurry was filtered and neutralized with 10% acetic acid. Then, the residue was rinsed with 95% ethanol to remove undesired by products. The residue was dried at 60°C for 6 h to obtain pure CMC.

2.5. Synthesis of Hydroxypropyl cellulose (HPC)

HPC was synthesized according to [Marseno *et al.* \(2014\)](#) method with modifications. Screw pine cellulose (1 g) was added and mixed with 10% NaOH (4 mL) and isopropanol (25 mL). The mixture was stirred for 1 h at room temperature. The mixture was then refluxed for 3 h at 55°C where propylene oxide (1.4 mL) was added in a dropwise. The resulting mixture was filtered and the residue was neutralized with 10% acetic acid and then washed with 95% ethanol. The residue was dried at 60°C for 6 h to obtain pure HPC.

2.6. Characterization of MC, CMC, and HPC

The percent yields of the MC, CMC, and HPC were calculated using Equation (2), where W_{CE} is the weight of synthesized cellulose ether; W_c is the weight of cellulose.

$$\text{The yield of cellulose ether (\%)} = \frac{W_{CE}}{W_c} \times 100 \quad (2)$$

MC, CMC, and HPC were also measured for pH, degree of substitution (DS), and viscosity. The cellulose ethers were analyzed by Fourier Transform Infra-Red (FTIR) Spectroscopy and Scanning Electron Microscopy (SEM).

The pH of 1% aqueous solution of MC, CMC, or HPC was measured using a pH meter. The viscosity of 1% aqueous solution of MC, CMC, or HPC was measured using an Ostwald viscometer. The viscosity of cellulose ether was calculated according to Equation (3). Determination of cellulose ether viscosity coefficient (η_1) with a known density (ρ_1) is done by measuring the time required for the liquid to flow (t_1) under the influence of gravity on the viscometer relative to the time required for the reference fluid (t_2) with known coefficient of viscosity (η_2) and density (ρ_2) to flow between the same two lines at the same temperature.

$$\eta_1 = \eta_2 \frac{\rho_1 t_1}{\rho_2 t_2} \quad (3)$$

The determination of the degree of substitution (DS) of cellulose ether was carried out based on the FTIR spectral analysis using Shimadzu FTIR Prestige 21-8201 PC brand with a sample ratio: KBr of 1:100. The intensity value of % T in the C-O cellulose ether number

was compared with the C-O cellulose wave number according to Equation (4) (Li, Cai, and Zhang, 2019).

$$DS = \frac{\text{Absorbance of CO CE}}{\text{Absorbance of CO Cellulose}} \quad (4)$$

2.7. Instruments

The functional groups of the cellulose and cellulose ethers were determined using a Shimadzu FTIR Prestige 21 model 8201 PC. The surface morphologies of samples were examined by SEM (JEOL JSM- 6510LA). The thermal stability of cellulose was studied by thermogravimetric analysis using an STA Linseis pt 1600 TGA analyzer. The degree of crystallinity of cellulose was determined using an X-Ray diffractometer (XRD XPert MPD diffractometer).

3. Results and Discussion

3.1. Isolation of Cellulose

Cellulose was isolated from Screw pine leaves by alkalization and bleaching processes. The white cellulose was obtained in a 40.24% (w/w) yield. FTIR spectrum of isolated cellulose is consistent with the results reported by Sheltami *et al.* (2012). Obtained SEM image showed a typical shape of cellulose. Cellulose fibers were individually dispersed which were separated and elongated like ribbons with approximate diameters of 5.97 μm in the range of 1.5–17 μm . The cellulose fibers' size is longer than that of Screw pine leaf fibers. These results are consistent with research conducted by Sheltami *et al.* (2012). Thermogravimetric analysis of purified cellulose showed that thermal degradation of cellulose occurred in the range 205–407°C with a significant weight loss of 86.52%. The X-Ray diffractogram revealed that cellulose structure was categorized as a crystal I type (Sheltami *et al.*, 2012). The crystallinity index of the isolated cellulose was 83.03% which is higher than the value reported for the cellulose of rice straw at 69.5% (Sheltami *et al.*, 2012) and bagasse cellulose at 58.77% (Nu *et al.*, 2019). The data obtained from FT-IR, SEM, TGA, and X-Ray Crystallography analyses confirmed that cellulose has been obtained from Screw pine leaves.

3.2. Synthesis of Cellulose Ethers from Cellulose of Screw pine (*Pandanus tectorius*) Leaf

Williamson's etherification reaction was used to synthesize the MC, CMC, and HPC. Williamson ether synthesis utilizes deprotonated alcohol and an organohalide to form an ether. The alkoxide ion is a good nucleophile and displaces halide ions from the alkyl halide to give a new carbon oxygen bond. An alkoxide is produced by the treatment of alcohol with either a base or an alkali metal. In this reaction, sodium hydroxide is reacted with cellulose to obtain Na-cellulose. The initial step of cellulose ether synthesis was performed by stirring screw pine leaf cellulose and sodium hydroxide, forming a yellow-brown mass.

In the synthesis of MC, the etherification step was carried out by the addition of dichloromethane to Na-cellulose. The MC was obtained as white powders in 94.61% yield. The synthesis of CMC was accomplished by reacting Na-cellulose and trichloroacetic acid (TCA). CMC is commonly prepared by the reaction of cellulose with monochloroacetic acid (MCA). In this work, TCA was used instead of MCA. The CMC was obtained as a white powder with a 71.47% yield. This yield is double compared to that reported by Bono *et al.* (2009) (33.15%) for CMC from palm kernel cake; and Huang *et al.* (2017) (64.40%) for CMC from spent tea leaf. The difference in yield could be attributed to the temperature of the reaction, and the concentration of NaOH and carboxymethylating agent. The synthesis of HPC was conducted by mixing Na-cellulose and propylene oxide at 55°C. The HPC was obtained as white powders in 81.54% yield. The highest yield was obtained for the

synthesis of MC may be due to the steric reason where the methyl group caused the least steric bulk.

The general mechanism for Williamson ether synthesis of MC, CMC, and HPC is shown in Figure 1. NaOH withdraws the proton of the OH of the cellulose to form the Na-cellulose and H₂O as a by product. The alkoxide then reacts with esterification agents. The nucleophile oxygen of Na-cellulose attacks the electrophilic carbon of dichloromethane, trichloroacetic acid, and propylene oxide to form MC, CMC, and HPC, respectively.

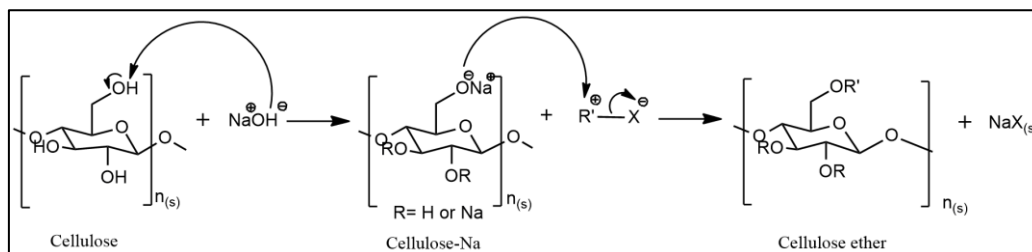


Figure 1 Proposed reaction mechanism for the synthesis of cellulose ethers

3.3. FT-IR Spectral Analysis of Cellulose Ethers

FT-IR spectra of the cellulose ethers confirm the successful synthesis of MC, CMC, and HPC from Screw pine cellulose (Figure 2 and Table 1). There are typical absorption peaks of MC at 1500-800 cm⁻¹ (Ibrahim *et al.*, 2015; Viera *et al.*, 2007). An absorption of -CH alkane appears at 1414 cm⁻¹ indicating the presence of methyl compounds of MC. Additionally, the presence of a peak at 1158 cm⁻¹ for C-O ether and a peak at 1574 cm⁻¹ peak for C-C confirmed that MC was obtained. The typical cellulose peaks are also found at 3389-3445 cm⁻¹ for -OH groups, at 2902 cm⁻¹ for -CH (stretching) groups, at 1339 cm⁻¹ for -CH (bending) groups, at 1022 and 1062 cm⁻¹ for carbonyl (C=O) group (stretching) and at 895-927 cm⁻¹ for -CH uptake (rocking) deformation of the 1,4-glycosidic bond.

The FTIR spectra show typical peaks of CMC (Alabi *et al.*, 2020; Huang *et al.*, 2017; Haleem *et al.*, 2014; Sun *et al.*, 2004). The presence of methylene group was confirmed by a peak at 1416 cm⁻¹ for -CH alkane (bending). Peaks at 1570-1638 cm⁻¹ for carboxyl (C=O) group stretching confirm the etherification reaction successfully occurred. In addition, there are peaks at 1066 and 1159 cm⁻¹ for C-O ether (stretching). Typical cellulose peaks were still found at 3445 cm⁻¹ for stretching vibration of the -OH group and at 2899 for -CH stretching, 1330 and 1024 cm⁻¹ for -CH bending. This suggests that cellulose was successfully modified into CMC.

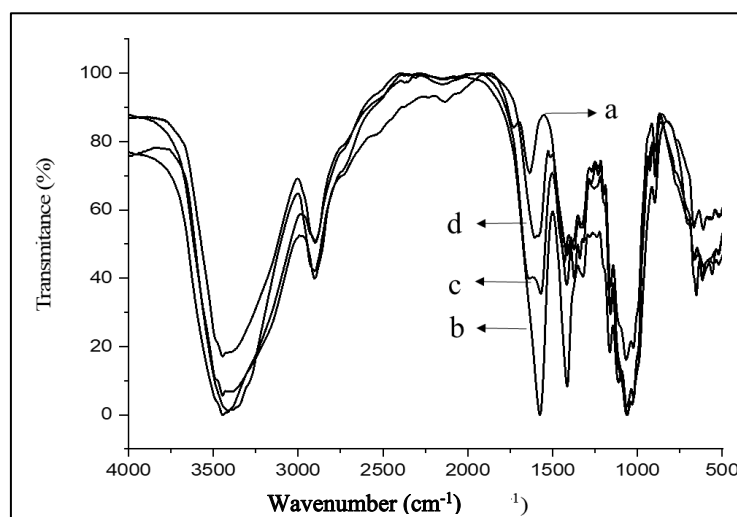


Figure 2 FTIR spectra of cellulose (a), MC (b), CMC (c), and HPC (d)

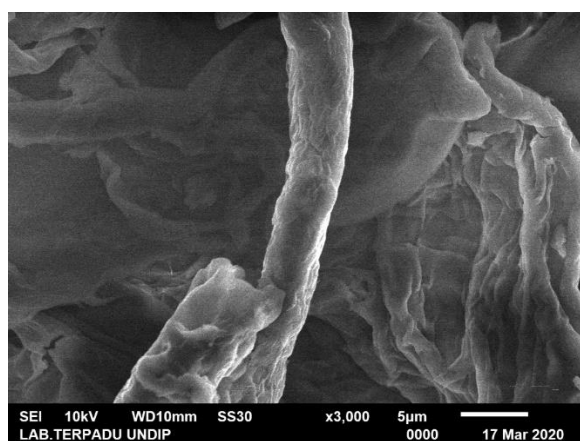
Table 1 FTIR spectra analysis of cellulose, MC, CMC, and HPC

Cellulose	Absorption band (cm ⁻¹)			Functional Groups
	MC	CMC	HPC	
3384	3389-3445	3445	3445	-OH stretching
2902	2902	2899	2899	-CH stretching of methyl and methylene
-	-	-	1650	OH bending (water)
-	-	1570-1638	-	-C=O stretching
1431	-	1416	1416	-CH ₂ and -CH ₃ bending
-	1317-1414	-	-	-CH ₂ , -CH ₃ scissoring
1321	-	1330	-	C-O stretching ether group
1112-1163	1158	1159	1159	C-O-C stretching asymmetric
1031-1058	1022-1062	1024-1066	1063	O-C-O stretching of ether
898	896	895	896	C-H bending (β -1,4 glycosidic)

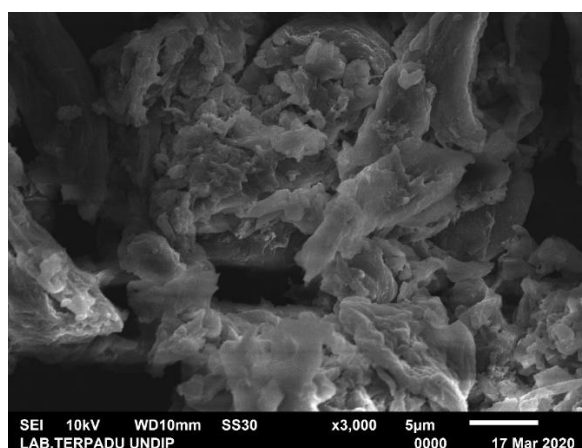
The FTIR spectra of HPC reveal the presence of functional groups of HPC (Joshi *et al.*, 2019; Marseno *et al.*, 2014; Bhatt *et al.*, 2011; Sharma *et al.*, 2009). Absorption at 2899 cm⁻¹ and 1416 cm⁻¹ shows C-H and methylene groups of hydroxypropyl cellulose, respectively. In addition, there is also absorption at 1063 and 1159 cm⁻¹ for C-O ether (stretching). The typical absorption of cellulose was found at 3445 cm⁻¹ for absorption of the -OH group (stretching), 1323 cm⁻¹ for the -CH group (bending), and 896 cm⁻¹ for absorption of -CH (stretching) deformation of the 1,4-glycosidic bond. This indicates that cellulose was successfully modified into HPC.

3.4. SEM Analysis of MC, CMC, and HPC

The success of MC, CMC, and HPC synthesis was also evidenced by the SEM data where the morphology of the etherification products changed in comparison to cellulose (Figure 3). Figure 3 shows that cellulose was completely elongated with fibrils of various lengths and thicknesses at higher magnification. The SEM micrograph of cellulose revealed the packed fibril structures were separated from each other. The MC product shows twisted and blunt flakes which are identical to the commercial MC (Nasatto *et al.*, 2015). CMC showed a rough surface with a partial round fibril which is not elongated like cellulose structures and is closely packed together to form clusters (Suriyatem, 2020; Haleem *et al.*, 2014). Substitution of the OH groups in cellulose with CH₃COO⁻ groups resulting amorphous phase of CMC. The HPC product has a wavy structure that is coarser than cellulose. This HPC structure is identical to HPC from *Lantana camara* (Bhatt *et al.*, 2011), bamboo waste (Sharma *et al.*, 2009), and paper waste (Joshi *et al.*, 2019).



(a)



(b)

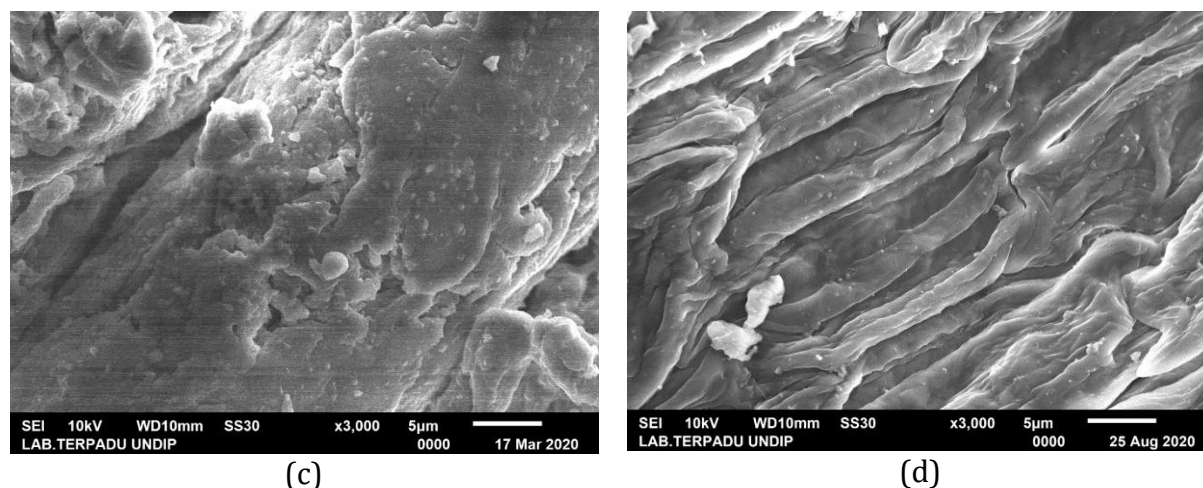


Figure 3 SEM micrograph of cellulose (a), MC (b), CMC (c), and HPC (d) at magnification 3000X

3.5. Physical Properties of Cellulose Ethers

The physical properties of cellulose ethers are presented in Table 2. DS is one of the important parameters in determining the quality of cellulose ethers. DS is the average number of hydroxyl groups substituted in every anhydroglucose unit in the chain. The higher the DS value of the product obtained, the better the cellulose ether quality. DS of cellulose ethers was found to be dependent upon the source of cellulose, therefore cellulose ethers prepared from various cellulose sources may have various DS values.

The DS of the MC product was 1.68 which is in the range of 1.3-2.6 for the DS value of MC as a food additive. The commercial DS of CMC is 0.5-1.5 (Karatas and Arslan, 2016). CMC having DS below 0.4 is swellable but insoluble in water. CMC having DS above 0.4 is soluble in water, because the hydro-affinity of CMC is enhanced with increasing DS value (Varshney *et al.*, 2006). The DS value of synthesized CMC was 0.996, indicating it is soluble in water. The result is higher than the DS of CMC from palm kernel cake (0.31) and oil palm fiber (0.29) (Huang *et al.*, 2017). DS values of CMC from *Miscanthus sinensis*, *Eichhornia crassipes*, and *Cyperus papyrus* are 0.35, 0.80, and 0.76, respectively (Kimani *et al.*, 2016). The DS of synthesized HPC was 0.696. This result is in accordance with the characteristics of HPC as a food additive where the DS value of HPC should be above 0.12.

The pH of the 1% solution of MC, CMC, and HPC in distilled water were 7.53, 7.60, and 7.56, respectively. The pH of food grade MC, CMC, and HPC according to FAO standards are 5-8, 6-8.5, and 5-8.5, respectively. These results suggest that the pH of the synthesized MC, CMC, and HPC meet the standards as food additives.

The viscosity value of 1% MC, CMC, and HPC solution in distilled water was 62.39; 62.41, and 61.98 cP, respectively. These results are in accordance with the MC, CMC, and HPC standards as food additives. The viscosity of CMC is correlated with DS value: a higher DS results in higher cation exchangeability and viscosity. The low DS value indicates fewer hydrophilic groups, which implies the reduction of the bonding of polymer with water molecules. Higher viscosity (66.6 cP) was reported for CMC from Palm Kernel Cake (PKC) (Bono *et al.*, 2009) while lower viscosity (14.0 cP) was reported for CMC from orange mesocarp. The variation in properties of the different CMC could be a result of the source of cellulose used, plant species, age, and source which affect the cellulose content compositions (Carere *et al.*, 2008).

Table 2 Physicochemical properties of MC, CMC, and HPC

Properties	Cellulose Ethers		
	MC	CMC	HPC
Physical state and Appearance	white powder	white powder	white powder
pH	7.53	7.60	7.56
Viscosity	62.39 cP	62.41 cP	61.98 cP
DS	1.680	0.996	0.696

4. Conclusions

Cellulose was isolated from Screw pine (*Pandanus tectorius*) leaves in 40.24 % yield employing alkali treatment followed by bleaching with sodium hypochlorite. The structure of the obtained cellulose was confirmed by FTIR, SEM, and TGA analysis. Cellulose ethers have been successfully synthesized from Screw pine leaves cellulose through Williamson ether synthesis. Cellulose ethers MC, CMC, and HPC were obtained in 94.6%, 71.47%, and 81.54% yields, respectively. The products have been confirmed by FTIR spectra and SEM images. The physicochemical properties, such as shape, color, pH, viscosity, and DS suggest that synthesized MC, CMC, and HPC meet the criteria as food additives.

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