

Preparation and Characterization of Microcrystalline Cellulose from Cellulose Based-Agro Wastes

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Abstract: Two types of agricultural wastes, Bagasse (BG) and Banana Stem (BS) were extracted and hydrolyzed into Microcrystalline Cellulose (MCC) by acid hydrolysis using HCl and H₂SO₄. The suitable condition for preparing MCC from each agricultural waste was investigated. From the results of Thermogravimetric Analysis (TGA), the suitable NaOH concentrations for delignification of BG and BS fiber were 0.5 and 1 M, respectively. The most appropriate bleaching condition giving high whiteness and less degradation of both pulps was 6% H₂O₂ in alkali solution. For H₂SO₄ hydrolysis, BG and BS MCC achieved their unchanged average particle sizes (6-7 µm) and equilibrium Degree of Polymerization (DP) values (233-256) within 60 and 30 min, respectively. Whereas, the optimum conditions for BG and BS hydrolyzed with 2.5 N HCl to obtain their unchanged average particle sizes (6-7 µm) and equilibrium DP values (238-303) were at 120 and 60 min, respectively. In addition, particle size of all obtained BG and BS MCCs was smaller than Commercial Microcrystalline Cellulose (CM-MCC).

Key words: Microcrystalline cellulose, acid hydrolysis, degree of polymerization, biopolymer, bagasse, banana stem

INTRODUCTION

Microcrystalline Cellulose (MCC) which is extracted from natural fiber is usable for pharmaceutical, cosmetic, food and plastic composite fields owing to its compatibility, hydrophilicity, acid-insolubility and biodegradability (Thoorens *et al.*, 2014). Alpha cellulose which is mostly found in plant and natural fiber is the well-known raw material for microcrystalline cellulose preparation. Cellulose chain consists of a large number of glucose molecules linked together with β-1,4 glycosidic bond. Its molecular chain is arranged in order and disorder forms resulted in its crystalline and amorphous structures (Thoorens *et al.*, 2014). Generally, three steps which are delignification, bleaching and hydrolysis are necessary for microcrystalline cellulose preparation from natural fiber. For the first and second steps, natural fiber or native fiber are needed to be treated with alkaline solvent to remove impurity from cellulose fiber before hydrolysis, since cellulose is covered with impurities that are wax, hemicellulose, lignin and other compositions (Adel *et al.*, 2010). Next, the hydrolysis process is used to reduce the crystalline cellulose into small size. The dilute acid

hydrolysis (e.g., hydrochloric and sulfuric acids) is widely used in industrial scale owing to its reasonable price. During this process, acid solution can penetrate into voids of amorphous region prior to crystalline region and break down at glycosidic bond, leading to small size of crystalline structure in micro scale (Adel *et al.*, 2010). However, many factors such as cellulose source, acid type, reaction time and temperature have been found to affect on morphology such as size and shape and Degree of Polymerization (DP) of obtained microcrystalline cellulose (Sakhawiy and Hassan, 2007). Thailand is one of the famous agricultural countries; consequently, after cultivation many kinds of agricultural wastes have been found and needed to be discarded or utilized in proper and useful approaches. Sugarcane and banana can grow very well in Thailand, particularly approximately 50 million tones of sugarcane is yearly produced in this country. These agricultural wastes are cheap and abundant; moreover, they are rich in cellulose content. Approximately 32-48% (Reddy and Yang, 2005) and 60-65% (Deepa *et al.*, 2011) of cellulose can be found in bagasse and banana stem, respectively. Accordingly, these agricultural wastes are of interest to

be used as raw materials to prepare crystalline cellulose. Therefore, the objective of this work was to prepare microcrystalline cellulose from agricultural waste. Two types of agricultural wastes, Bagasse (BG) and Banana Stem (BS) were transformed into microcrystalline cellulose by acid hydrolysis using HCl and H₂SO₄. The suitable condition for delignification and bleaching was determined according to the thermal properties of delignified fiber and whiteness of bleached pulp. The optimum condition for hydrolysis was defined in accordance with physical appearance, i.e., average particle size and morphology and DP of MCC. The effect of cellulose source and acid type on average particle size, reaction time and DP of the obtained MCC was studied.

MATERIALS AND METHODS

Experimental: Bagasse (BG) and Banana stem (BG) were used as raw materials for microcrystalline preparation. Sodium hydroxide was purchased from Fluka Co., Ltd., Thailand. A technical grade of 35% hydrogen peroxide was provided by Peroxy Thai, Co., Ltd., Thailand. Sulfuric acid (95-98%) and hydrochloric acid (35-37%) were purchased from Sigma Aldrich Co., Ltd., Thailand. Cupriethylenediamine hydroxide solution supplied by Calo Elba Co., Ltd., Singapore, was used as a solvent for microcrystalline cellulose. A commercial grade of microcrystalline cellulose with an average particle size of 20 μm purchased from Fluka Co., Ltd., USA was used as a reference.

Microcrystalline cellulose preparation: The agricultural wastes, BG and BS were dried in an oven overnight and then cut into small pieces before delignification with NaOH solution. For delignification, the small pieces of sample were soaked in various NaOH solution concentrations (0.5, 1, 2 M) for 24 h and then heated and stirred at 80-90°C for 4 h to produce fibrils. The obtained fibrils were washed with distilled water prior to bleaching at different H₂O₂ concentrations (2, 4, 6, 8 and 10 v/v H₂O₂ in 15 w/v NaOH solution) at 100°C for 1 h. This bleaching step was treated twice. Subsequently, the bleached pulp was washed several times with distilled water until the pH of bleached pulp became neutral. After that the bleached pulp was hydrolyzed with two types of acid, 2.5 N of HCl and H₂SO₄ and stirred vigorously at 70-80°C for various times (30, 60, 120, 180 min). Then, the suspension was washed several times with distilled water until the pH became neutral and then it was filtered. After that, the microcrystalline was dried in an oven at 60-70°C overnight. The final microcrystalline was ground to decrease its agglomeration for 4 min and kept drying in a desiccator until being used.

Characterization: Thermal behavior of delignified and bleached samples was investigated using thermogravimetric analyzer (TGA; SDTA851e, Mettler-Teledo, Greifensee, Switzerland) under nitrogen atmosphere at a heating rate of 20°C/min from 50- 500°C. Colour Spectrophotometer (color-eye 7000A, Gretag Macbeth, NewYork) was used to study whiteness and yellowness of bleached pulp according to ASTM 1925 and 313, respectively. Bleached sample was cut into 5×5 cm. Each sample was measured three times and the average value was reported. Size and shape of MCC were investigated by Scanning Electron Microscope (SEM; JSM 6480, JOEL, Tokyo, Japan). The surface of sample was coated with a thin layer of gold before being scanned. The system was running at 15 kV. Average particle size of MCC powder was determined by the laser particle size analyzer (Matersizer 2000, Malvern, Worcestershire, UK). The MCC powder was suspended in 20 mL of distilled water before being tested. The study of crystallinity behaviors of the obtained MCC was performed by means of on X-ray diffractometer (XRD; D8-Advanced, Bruker, Germany) The scan scope was between 2 and 50° with a scanning rate of 2°C min⁻¹ with CuK α radiation operating at 45 KV and 40 mA.

Degree of polymerization: Degree of Polymerization (DP) of the MCCs (BG MCC, BS MCC and CM-MCC) was examined by intrinsic viscosity measurement according to the ASTM D1795-96 (Ogeda *et al.*, 2012) The MCC powder was dissolved in cupriethylene diamine hydroxide solution, then the intrinsic viscosity of cellulose solution was investigated by Ubbelohde capillary viscometer and finally this intrinsic viscosity of the cellulose solution was used to calculate the degree of polymerization. The details of measurement are as follows. Firstly, the stock solution of cellulose (2g⁻¹) was prepared by dissolving 0.1 g of dried MCC in 50 mL of 0.5 M cupriethylene diamine solution. The mixture was stirred vigorously until the MCC was completely dissolved under nitrogen atmosphere. After that, the stock solution of cellulose was diluted into five concentrations (1, 0.8, 0.6, 0.4, 0.2 g dL⁻¹) by adding distilled water and then stirred for 30 min under nitrogen atmosphere. Next, the viscosity of all dilute solutions and 0.5 M cupriethylene diamine was measured by using an Ubbelohde capillary viscometer at 25°C. This process was repeated three times and the flow time of each dilute cellulose solution was measured and averaged. The relative viscosity (η_{rel}) was calculated from the ratio of cellulose solution flow time (t_{r}) and solvent flow time (t_0) as displayed in this equation: $\eta_{\text{rel}} = t_{\text{r}}/t_0$. These data were plotted with relationship between $\log [(\eta_{\text{rel}}-1)/c]$ and c where c refers to concentration of the

cellulose solution. The intrinsic viscosity $[\eta]$ was obtained from the interception of this plot. The DP of MCC was then calculated by multiplying the intrinsic viscosity by 190 as presented in this equation: $DP = [\eta] \times 190$.

RESULTS AND DISCUSSION

Delignification

Physical appearance: The photographs of BG and BS fibrils treated with various NaOH concentrations are shown in Fig. 1a-h, respectively. Bagasse fiber can be separated into fibrils at 0.5 M NaOH (Fig. 1b); whereas at higher NaOH condition the bagasse fiber cannot be changed into fibrils, probably due to the degradation of the fiber resulted from higher NaOH concentration. For BS as shown in Fig. 1 (e-h), it can be separated into fibrils at every NaOH concentrations. However, at 2 M NaOH solution a small amount of dark fibrils also occurred (Fig. 1h). The dark fibrils are probably resulting from the degradation of cellulose structure at high NaOH concentration. After alkaline treatment, fibrils still exhibited slightly yellowish and brownish colors. These colors may indicate that partial lignin and hemicellulose still remained in the fiber.

Thermal behavior: Figure 2 shows the TGA thermograms of delignified bagasse and banana stem fibrils at various NaOH concentrations comparing with the untreated fibers. As presented, all samples displayed a decomposition temperature nearly at 100°C due to moisture decomposition. As shown in Fig. 2a, the untreated bagasse fiber showed three main degradation steps. The first stage displayed the Td (onset) in the range of $200\text{--}230^\circ\text{C}$ is due to hemicellulose degradation; whereas the second one occurred in the range of $240\text{--}300^\circ\text{C}$ is attributed to lignin degradation. The last step appeared in the range of $340\text{--}360^\circ\text{C}$ is resulted from cellulose degradation (Chen *et al.*, 2005).

After delignification, Td (onset) of the delignified bagasse treated with 0.5 M NaOH solution was closely to that of the one treated with 1 M NaOH solution. The decomposition temperatures of these delignified bagasse fibers presented at 295 and 292°C , respectively, were due to cellulose degradation. While the Td (onset) of the delignified bagasse treated with 2 M NaOH solution occurred at lower temperature than those treated with 0.5 and 1 M NaOH concentrations, resulting from the degradation of cellulose structure occurred at higher NaOH concentration. Unlike bagasse fiber, Fig. 2b shows that untreated banana stem fibers had two main stages of degradation. The first stage of Td (onset) at 177.94°C

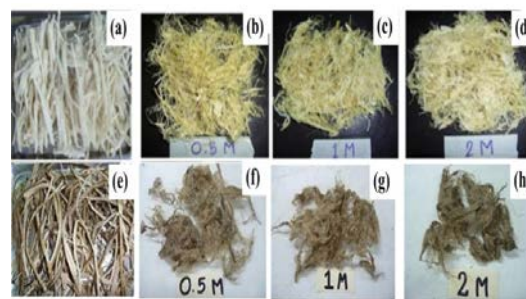


Fig. 1: Photographs of: a) untreated BG ; b-d) treated BG with 0.5, 1 and 2 M NaOH, respectively; e) untreated BS; f-h) treated BS with 0.5, 1 and 2 M NaOH, respectively

is due to hemicellulose degradation; whereas, the second step of Td (onset) at 263.39°C is probably due to the combination of cellulose and lignin degradation (Deepa *et al.*, 2011; Ogeda *et al.*, 2012; Chen *et al.*, 2005; Zeronian and Inglesby, 1995). After delignification, the delignified banana stem fibrils exhibited only one step of degradation ranging between $250\text{--}350^\circ\text{C}$ which is due to the cellulose degradation (Reddy and Yang, 2005; Deepa *et al.*, 2011; Ogeda *et al.*, 2012; Chen *et al.*, 2005). Clearly, the Td (onset) of banana stem treated with 1 M NaOH solution was higher than that of the one treated with 2 and 0.5 M, respectively.

Therefore, 0.5 M NaOH solution is the suitable concentration for bagasse delignification because at this concentration fiber can be separated into many fibrils than other NaOH solution concentrations. Similarly, the suitable NaOH concentration for delignification of banana stem fiber was at 1 M. Since the bagasse fiber treated with 1M NaOH solution had greater Td (onset) than the ones treated with other NaOH concentrations. However, as mentioned earlier all of the delignified fibrils presented yellowish and brownish colors as a result of the partly remaining lignin on the fibrils. The observation of thermal degradation of lignin and celluloses are overlapped because thermal behavior of lignin and cellulose are closely, attributing to one step of the degradation of the delignified fibrils.

Bleaching

Whiteness: The remaining lignin not only has influenced on the color or whiteness of pulp but also affected on the hydrolysis process. The outer layer of lignin can prevent an acid from penetrating into an amorphous region and breaking down the glycosidic bond of cellulose. Figure 3 a, b show that whiteness of bleached bagasse

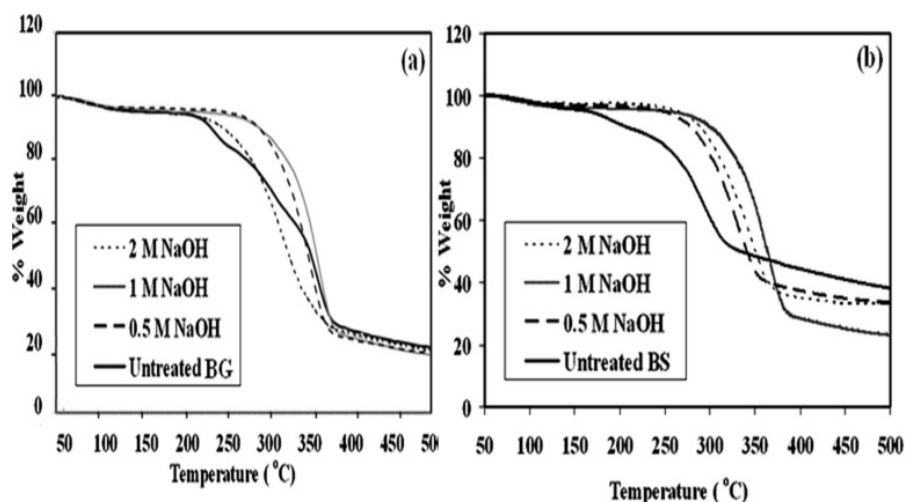


Fig. 2: TGA curves of untreated and delignified: a) bagasse fibrils and b) banana stem fibrils at different NaOH solution concentrations

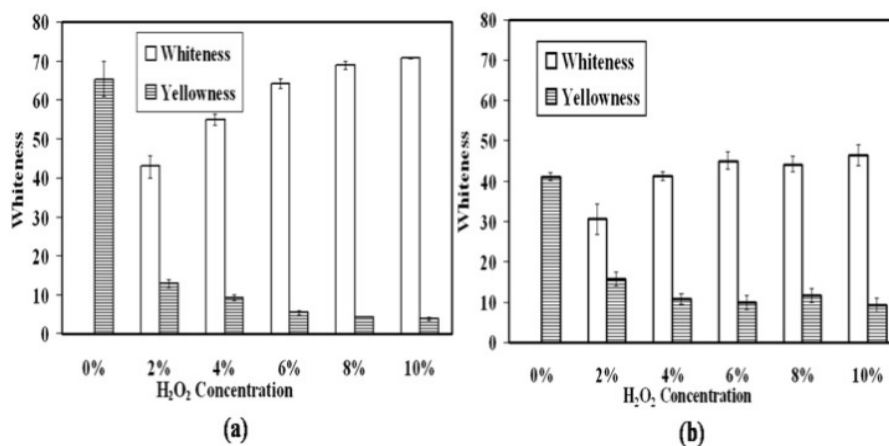


Fig. 3: Whiteness and Yellowness of: a) bleached bagasse pulp and b) bleached banana stem pulp at various H_2O_2 concentrations

and banana stem pulp increased when the H_2O_2 concentration increased; whereas, the yellowness was drastically decreased. This is because hydrogen peroxide is used with the alkaline solution sodium hydroxide or to produce high pH solution that is necessary for generating the active perhydroxyl ion, HO_2^- . This ion is a mild oxidant resulting in pulp whiteness (Zeronian and Inglesby, 1995). Thus, the active perhydroxyl ion increased when the concentration of H_2O_2 was increased, resulting in an increase in whiteness of all the pulps. However, when the H_2O_2 concentration was increased up to 8-10%, it seemed to cause some degradation of pulp as evidenced by the occurrence of many short fibrils. Comparatively, bleached bagasse pulp seemed to have greater whiteness than bleached banana stem pulp in

every condition. Generally, the suitable condition for bleaching should be the one that can remove higher amount of lignin from the fiber and yield high whiteness value. Although, in this research, the high concentration of H_2O_2 (i.e., 8 and 10%) contributed to the high whiteness value but it led to some degradation of bagasse and banana stem pulp. Thus, the suitable condition to bleach both pulps should be at 6% H_2O_2 solution since this concentration attributed to high whiteness and low degradation of pulp.

Hydrolysis

Average particle size and particle size distribution

Effect of hydrolysis time: The results of particle size analysis of MCC samples are summarized in Table 1.

Table 1: Average particle size and degree of polymerization (DP) of BG and BS MCC at various hydrolysis reaction times

| MCC | Time (min) | HCL | | | H ₂ SO ₄ | | |
|-----|------------|----------------------------|---------------------|-------|--------------------------------|--------------|-------|
| | | Average particle size (µm) | Particle range (µm) | DP | Average particle size (µm) | Range (µm) | DP |
| BG | 0 | 69.19 | 17.96-261.10 | 11685 | 69.19 | 17.96-261.10 | 11685 |
| | 30 | 13.62 | 3.37-30.590 | 344 | 12.12 | 3.30-41.630 | 341 |
| | 60 | 12.41 | 3.81-26.390 | 340 | 6.11 | 2.09-20.710 | 233 |
| | 120 | 6.77 | 2.41-19.410 | 238 | 6.46 | 2.37-15.810 | 232 |
| | 180 | 6.83 | 2.21-19.450 | 237 | 6.81 | 2.39-18.030 | 231 |
| BS | 0 | 38.98 | 8.48-201.57 | 12024 | 38.98 | 8.48-201.57 | 12024 |
| | 30 | 11.75 | 4.05-21.860 | 308 | 7.29 | 3.40-17.940 | 256 |
| | 60 | 8.59 | 3.23-17.200 | 303 | 6.09 | 3.02-19.160 | 234 |
| | 120 | 7.27 | 2.80-12.820 | 235 | 9.12 | 2.97-18.070 | 232 |
| | 180 | 8.81 | 3.92-14.900 | 234 | 9.10 | 3.83-18.030 | 231 |

These results showed that an average particle size and particle size range of hydrolyzed pulps can be changed into the micro scale because the amorphous regions or disorder phase can be removed by hydrolysis at glycosidic linkages (Adel *et al.*, 2010). Considering the bagasse pulp, an average particle size and particle size range of BG MCC decreased with an increase of the reaction time. Especially, these values were decreased rapidly after hydrolysis with acids for 30-60 min. However, upon further increased the reaction time, i.e., from 120-180 min, the average particle size of BG MCC seemed to be unchanged, having its values within the range of 6-7 µm.

In case of banana stem pulp, similarly, an average particle size of BS MCC was decreased when the reaction time increased from 0-30 and then 60 min. However, when the reaction time was increased from 60-180 min, the particle size of BS MCC treated with HCl seemed to be unchanged. Its particle size treated with HCl was in the range of 7-8 µm. On the other hand, in case of H₂SO₄-BG MCC, when the reaction time was increased from 60-180 min, an average particle size was somewhat increased. This increasing of BS MCC particle size might be contributed to an agglomeration.

Effect of acid use: From Table 1, similar trend was observed for both types of acid but with different magnitude, that the equilibrium values of average particle size were attained at different reaction time. As presented, the optimum reaction time for bagasse pulp hydrolyzed with HCl and H₂SO₄ was at 120 and 60 min, respectively. Whereas, the optimum reaction time for hydrolysis of banana stem pulp with HCl and H₂SO₄ was at 60 and 30 min, respectively. These results indicated that MCC prepared using H₂SO₄ required shorter reaction time to reach its constant value or equilibrium value than HCl. This result can be described by the fact that H₂SO₄ is a stronger acid than HCl since H₂SO₄ can give higher proton into water than HCl (Adel *et al.*, 2010; Sakhawy and Hassan, 2007; Reddy and Yang; Deepa *et al.*, 2011; Ogeda *et al.*, 2012; Chen *et al.*, 2005; Zeronian and Inglesby, 1995; Battista *et al.*, 1956).

Degree of Polymerization (DP): The degree of polymerization or DP refers to the number of repeating units in polymer molecule. The degradation of cellulose by acid hydrolysis to produce microcrystalline cellulose is known to occur through the degradation of glycosidic bonds of cellulose chain in amorphous regions which is effected on the DP of cellulose.

Effect of hydrolysis time: Table 1 shows that the degree of polymerization of BG and BS MCC was decreased when the reaction time increased. Considering BG MCC when the hydrolyzed time was increased from 0 to 30 min the DP of HCl-BG MCC was decreased rapidly. However, after 60 min, the DP of HCl-BG MCC slightly decreased and seemed to reach its equilibrium DP value within the range of 237-238 after hydrolysis for 120 min. Similar trend was noticed in case of H₂SO₄-BG MCC. However, its equilibrium DP value, (i.e., 231-233) was achieved within shorter reaction time (i.e., 60 min).

Similar results were also observed for BS MCC. The equilibrium DP value of HCl-BS MCC was in the range of 234-235 whereas that of H₂SO₄-BS MCC was in the range of 231-234. These results indicated that the optimum reaction time to hydrolyze both types of MCC using HCl and H₂SO₄ was 120 and 60 min, respectively. At these conditions, the DP value of MCC reached its equilibrium or constant value.

These results are similar to El-sakhawy and Coworker (Sakhawy and Hassan, 2007) who prepared MCC from bagasse, rice straw and cotton stalk bleached pulps by using two different kinds of acid hydrolysis (HCl and H₂SO₄). They reported that these pulps reached Level-off Degree of Polymerization (LODP) after their reflux with acids for 30-45 min.

Effect of acid use: As shown in Table 1, regardless of MCC type, the MCC prepared using H₂SO₄ required shorter reaction time to reach its equilibrium DP value than the MCC prepared using HCl. In addition, the DP value and equilibrium DP value of MCC prepared using HCL were higher than those of MCC prepared using

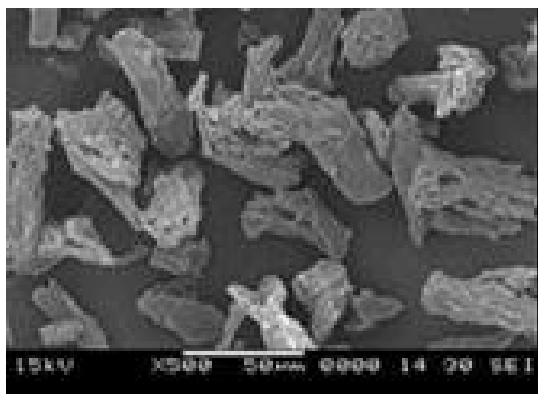


Fig. 4: SEM micrograph of CM MCC

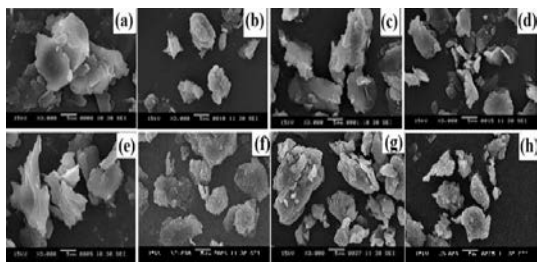


Fig. 5: SEM micrographs of BG MCC hydrolyzed at 2.5 N HCl for: a) 30 min; b) 180 min and 2.5 N H_2SO_4 for: c) 30 min; d) 180 min and SEM micrographs of BS MCC hydrolyzed at 2.5 N HCl for: e) 30 min; f) 180 min and 2.5 N H_2SO_4 for: g) 30 min; h) 180 min

H_2SO_4 . For example, at 60 min, the DP values of HCl treated BG MCC and BS MCC were 340 and 303 whereas those values of H_2SO_4 -BG MCC and H_2SO_4 -BS MCC were 233 and 234.

Effect of cellulose source: Comparing between these two pulps, it seemed that similar trend was observed. The DP values obtained from each type of MCC were comparable when treated with the same acid. Additionally, the DP value of Commercial Microcrystalline Cellulose (CM-MCC) was higher than that of both BG and BS MCC. For example, at 60 min, the DP value of H_2SO_4 -BG and H_2SO_4 -BS MCC was 233 and 234, respectively; whereas, the DP value of CM MCC was 345 (Rojas *et al.*, 2011).

Morphology of microcrystalline cellulose: Figure 4 shows SEM micrographs of CM-MCC while SEM micrographs of BG MCC and BS MCC are shown in Fig. 5. As seen, all prepared MCC showed similar shape which was a mixture between flake and pellet shape. Obviously, the particle size of both BG MCC and BS MCC

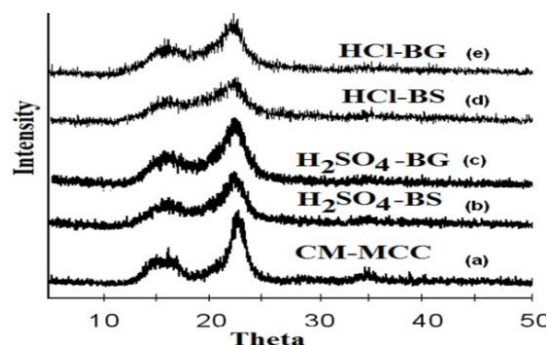


Fig. 6: The XRD patterns of BG and BS MCC hydrolyzed at 2.5 N of HCl

was decreased when the hydrolysis time or reaction time increased. When comparing between MCC treated with HCl and H_2SO_4 , it was found that the particle size of the MCC treated with H_2SO_4 seemed to be smaller than that of the one treated with HCl as confirmed by the particle size analysis. Considering the cellulose source, the average particle size of BG MCC treated with both of HCl and H_2SO_4 acid Fig. 5a, d seemed to be larger than that of BS MCC (Fig. 5e-h). However, average particle size of H_2SO_4 -BS MCC which was obtained from long reaction time, i.e., 120-180 min was slightly larger than BS MCC- H_2SO_4 . It is probably due to the agglomeration. Comparing with the commercial MCC while the prepared MCC showed a mixture between flake and pellet form with smooth surface; the CM MCC exhibited shortening fiber and rod shape with rough surface (Fig. 5).

Crystallinity of microcrystalline cellulose: Figure 6 shows the X-ray diffraction patterns of BG and BS-MCC samples compared with that of the commercial sample (CM-MCC). As shown, all the samples showed crystalline peak at $2\theta = 14, 16$ and 22° . Similar to work of Ogeda and co-workers (2012), this pattern belongs to α -cellulose in cellulose I structure (Sakhawy and Hassan 2007; Ogeda *et al.*, 2012). From this figure, the crystalline peak of MCC prepared using H_2SO_4 (curves (b) and (c)) had higher intensity than those prepared using HCl. This result may be implied that the H_2SO_4 -MCC had higher degree of crystallinity than those prepared using HCl. Unlike the work reported by Sakhawy and Hassan (2007) which was found that the kind of acid used had no effect on the crystallinity of the prepared MCC. When comparing with the CM-MCC, it seemed that the intensity of crystalline peak ($2\theta = 22^\circ$) of CM-MCC was slightly higher than those of the prepared MCC, in particular for those hydrolyzed with HCl which exhibited relatively

broadener crystalline peak (curve (d) and (e)). Therefore, CM MCC had higher degree of crystallinity than the prepared microcrystalline cellulose.

CONCLUSION

Two types of agricultural wastes, i.e., bagasse and banana stem, were delignified, bleached and then hydrolyzed with 2.5 N of HCl or H₂SO₄ to obtain microcrystalline cellulose. The conditions for preparing crystalline cellulose from each agricultural waste were investigated. The suitable NaOH concentrations for delignification of bagasse and banana stem fiber were 0.5 and 1 M, respectively. For bleaching step, the suitable condition which gave high whiteness without degradation for both pulps was to use 6% H₂O₂ in alkali solution. For hydrolysis step, either cellulose source or type of acid used had influenced on the average particle size, reaction time and DP of MCC. The results indicated that H₂SO₄ is more efficient than HCl for hydrolysis. The proper reaction time to hydrolyze BG and BS with 2.5 N H₂SO₄ to achieve their unchanged average particle size and equilibrium DP value were at 60 and 30 min, respectively; whereas, the optimum conditions for BG and BS hydrolyzed with 2.5 HCl were at 120 and 60 min, respectively. Considering the cellulose source, banana stem pulp seem to be hydrolyzed easier than bagasse pulp since banana stem required shorter reaction time to reach its unchanged average particle size and equilibrium DP value. However, the selection of the suitable condition for MCC preparation may depend on its applications; for example, the size and shape of MCC are significant properties for reinforcement in polymer application.

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REFERENCES

- Adel, A.M., Z.H.A. El-Wahab, A.A. Ibrahim and M.T. Al-Shemy, 2010. Characterization of microcrystalline cellulose prepared from lignocellulosic materials. Part I. Acid catalyzed hydrolysis. *Bioresource Technol.*, 101: 4446-4455.
- Battista, O.A., S. Coppick, J.A. Howsmon, F.F. Morehead and W.A. Sisson, 1956. Level-off degree of polymerization. *Ind. Eng. Chem.*, 48: 333-335.
- Chen, Y., L. Sun, I.I. Negulescu, M.A. Moore and B.J. Collier, 2005. Evaluating efficiency of alkaline treatment for waste bagasse. *J. Macromol. Sci. Part B. Phys.*, 44: 397-411.
- Deepa, B., E. Abraham, B.M. Cherian, A. Bismarck and J.J. Blaker *et al.*, 2011. Structure, morphology and thermal characteristics of banana nano fibers obtained by steam explosion. *Bioresour. Technol.*, 102: 1988-1997.
- Ogeda, T.L., I.B. Silva, L.C. Fidale, O.A.E. Seoud and D.F. Petri, 2012. Effect of cellulose physical characteristics, especially the water sorption value, on the efficiency of its hydrolysis catalyzed by free or immobilized cellulase. *J. Biotechnol.*, 157: 246-252.
- Reddy, N. and Y. Yang, 2005. Biofibers from agricultural byproducts for industrial applications. *Trends Biotechnol.*, 23: 22-27.
- Rojas, J., A. Lopez, S. Guisao and C. Ortiz, 2011. Evaluation of several microcrystalline celluloses obtained from agricultural by-products. *J. Adv. Pharm. Technol. Res.*, 2: 144-150.
- Sakhawy, M.E. and M.L. Hassan, 2007. Physical and mechanical properties of microcrystalline cellulose prepared from agricultural residues. *Carbohydr. Polym.*, 67: 1-10.
- Thoorens, G., F. Krier, B. Leclercq, B. Carlin and B. Evrard, 2014. Microcrystalline cellulose, a direct compression binder in a quality by design environment-A review. *Intl. J. Pharmaceutics*, 473: 64-72.
- Zeronian, S.H. and M.K. Inglesby, 1995. Bleaching of cellulose by hydrogen peroxide. *Cellul.*, 2: 265-272.