

Effect of microcrystalline cellulose on the strength of oil palm empty fruit bunch paper

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Abstract. Oil palm empty fruit bunch (EFB) is one of the most copious residues which are left behind after the oil refining process. It is categorized as short fiber which can be used in the production of pulp and paper. In most cases, EFB fibers need other supporting agent to enhance their wet and dry strength of paper properties. Therefore, the aim of this study is to investigate the effect of paper strength after incorporating different percentage of microcrystalline cellulose (MCC) of 0, 3, 6 and 9% with EFB fibers. The standard method in producing the hand-sheet was applied. Comparison is carried among all samples to determine the optimum percentage of MCC which is suitable to strengthen the paper. Results showed that physical properties of paper in terms of porosity do not have significant effect while the opacity was slightly decreased. The mechanical properties such as tensile strength and tear resistance for MCC papers are better especially paper tensile strength. The scanning electron microscopy (SEM) image showed the morphological structure of paper surface that contains without and with MCC. In conclusion mechanical properties of the hand-sheets are improved with the incorporation of less than 10% MCC into the pulp fibers.

1. Introduction

One of the most modern industries and the oldest crafts is papermaking. Paper is an aqueous deposit of any plant fiber in the sheet form in its technical form. The word paper originated from the Latin word "Papyrus" (Cyperus papyrus), a native plant to the Nile valley, which was made into sheets that could be used to write on by the Egyptians [1]. Extensive use of wood as a raw material for pulp and paper production are affected by the increasing demand of paper in the world due to several reasons; population growth, better literacy, development of communication, and industrialization in developing countries causing global deforestation which gave harmful effect to the environment [2-3]. Tewari [4] estimated that there will be over million tons of paper demand in 2020. Due to the necessity to supply the paper industry with raw materials, new sources of primary pulp fibers had to be identified [5]. There are ranges of non-woody and cellulosic plant materials from which pulp fibers can be extracted. Common species used include rice straw, wheat straw, bamboo, bagasse, kenaf, flax, cotton, sisal, jute, and hemp, to name a few [6]. They can also be divided into three groups based on their availability which are agricultural residues, natural growing plants and non-wood crops grown primarily for their fibers [7]. This has caused a significant growth in the production of pulp for papermaking from the non-wood fibrous materials such as oil palm empty fruit bunch (EFB) [6], [8], straws [9], grass [10] and kenaf [11] and due to their cost-effectiveness and abundance [7].



EFB is one of the most abundant lignocellulose materials available which are not optimally used and have been left until rotten in the plantation by the miller [8]. Waste generated from oil palm plantation includes empty fruit bunches (EFB), oil palm trunks (OPT), oil palm fronds (OPS), palm shells and palm oil mill effluent palm (POME). The vast quantity of biomass available in the industry can also be transformed into significant additional products, which may be a possible revenue generator for the oil palm-producing countries [12]. EFB is one of the promising potential raw materials to be used for the pulp and paper manufacturing [13]. Lignocellulose material such as EFB is primarily composed of cellulose, hemicellulose, and lignin. Studies done by Yunos [14] have reported that the amount of hemicellulose in EFB was estimated to be around 15–35 % while cellulose and lignin contents were around 30–50 %, and 20–30 %, respectively. Rosli [6] reported that EFB are weaker than the trunk in tensile and burst indices but despite the relatively short fiber (<1mm), they give exceptional tear index.

Chemical pulping involves cooking of raw materials using aqueous chemical solutions at elevated temperature and pressure to extract pulp fibers. Example of chemical pulping include kraft, soda, and sulphite [15]. Kraft pulping involves the fiber cooking in a solution of sodium hydroxide (NaOH) and sodium sulfide (NaS₂). According to Tanaka [16], active alkali (AA) concentration is the most effective factor on the yield and properties, compared to liquor/EFB ratio, cooking temperature and cooking time for kraft pulping.

Addition of additive and filler can improve the physical properties of paper in term of wet and dry strength. Uniformity of the surface, dimension stability and unchanging quality in usage were affected by the dispersion of the additive and filler [17]. Filler addition can give benefits such as cost and energy saving [18], raised in furnish drainage rate, enhancement in optical properties, printability, and appearance of paper produced [19].

Nanocellulosic materials that have been explored in recent decades for using in pulp and paper is microfibrillated cellulose (MFC), which is prepared by mechanical processes that involve very high shear forces to defibrillate cellulose fibers. Cellulose nanocrystals or microcrystalline cellulose (MCC) are the second material, which are prepared by the acid hydrolysis of cellulose fibers, followed by mechanical action, and the third materials are nanocomposites, which may be the combination of first and second types of nanocellulosic materials, along with polymers or other matrices [20]. Incorporating cellulose with the pulp fiber also can help to improve the wet and dry strength of the paper. Many published studies have reported the properties of sheets made by incorporating microfibrillated cellulose (MFC) such as [21-24], and nano-fibrillated cellulose (NFC) [25-29] as papermaking additive. Fewer studies have been published on the effect of incorporating microcrystalline cellulose (MCC) with paper pulp fibers on the properties of paper sheets prepared by the standard method for paper sheet making.

The microcrystalline cellulose (MCC) is formed through strong hydrogen bonding among individual cellulose crystals/whiskers and is a type of cellulose that has micro-scaled fibers liberated from larger plant based cellulose fibers and has a broad range of potential applications on tensile strength (wet and dry), tear resistance, burst strength, opacity, and porosity of paper sheets [20, 30]. Ioe Zou [31] reported that addition of 5% MC as additive in the pulp sheet increased the physical properties of pulp sheet. Elongation and TEA increased respectively with concentration of MC at the beginning and reached the highest value on a dose of 8% concentration of MC [17]. Thus, the aim of this work was to study the effect of adding certain amounts of microcrystalline cellulose in kraft paper made from oil palm empty fruit bunches.

2. Materials and Methods

2.1 Raw materials

Oil palm empty fruit bunch fibers were received in bale from a local mill at Pahang. Toluene, acetic acid and acetone were purchased by Bendosen Laboratory Chemical. Ethanol, sodium hydroxide (NaOH) and sulphuric acid were acquired from R&M Chemicals. The MCC was also obtained from R&M Chemicals with degree of polymerization, 210, pH at 5.5, conductivity of more than 75.0 $\mu\text{s}/\text{cm}$ and water soluble substances, 0.25.

2.2 Chemical composition of EFB

The chemical composition of EFB was determined according to the following standard methods: ethanol–benzene extractives (TAPPI T204 cm-97), lignin (TAPPI T222 cm-02), holocellulose (Wise et al., 1946) and α -cellulose (TAPPI T 203 cm-99).

2.3 Preparation of pulp and papermaking

The kraft pulping was carried out using Twin Digester (MK Twin Tub Digester). Pulping condition was selected according to Tanaka [16] by adjusting the level of active alkali (AA). The parameters for kraft pulping were fixed at 18% active alkali and 25% sulphidity. The ratio of 1:10 liquor to EFB was selected. The pulping was done for 120 min at 170°C. After completing pulping, the pulps were washed and screened in a Somerville Fractionator. Prior to screening, these pulps were dispersed in a Hydropulper. Finally, unbleached pulps were obtained and applied for papermaking by using Handsheet Machine. Papermaking was carried out based on TAPPI Standard T 205 sp-02. On the other hand, MCC was sonicated using ultrasonic processor (Sonics Vibra-Cell VCX 400) for 5 mins and 10 pulses for 5 secs before adding it into the pulp slurry for papermaking.

In papermaking, there are four samples prepared which contained EFB pulp and EFB pulp added with MCC at 3%, 6% and 9%, w/w MCC over pulp. Firstly, the pulp slurry was disintegrated using Pulp Disintegrator (Regmed DSG 200) at 25,000 rpm to ensure uniform distribution of MCC and EFB fibers. Prior to papermaking, the papers were conditioned at 50% relative humidity and 23°C temperature for 24 hr before testing the paper physically, mechanically and optically. The samples were coded as MP0, MP3, MP6 and MP9.

2.4 Morphological observation of MCC powder and paper

The morphological properties of MCC powder and papers were observed via Scanning Electron Microscopy (SEM) (Hitachi S-3400N and Jeol JXA 840A) under an accelerating voltage of 15 kV. Before scanning, samples were coated with gold using a sputter coater system (Edwards Sputter Coater; BOC Edwards, Sussex, United Kingdom) to obtain excellent image by avoiding any charging effect.

2.5 Determination of paper properties

The paper physical properties comprised grammage, thickness, and bulk. The mechanical properties involved tensile, tear and folding endurance while optical properties consisted of brightness and opacity. The Standard Methods referred are listed as in Table 1.

Table 1. TAPPI Standard used in this study.

Properties	Method
Grammage	TAPPI T 220 sp-01
Thickness	TAPPI T 411 om-97
Bulk Thickness	MS ISO 534:2007
Tensile strength	TAPPI T494 om-01
Tear strength	TAPPI T414 om-98
Brightness	MS ISO 2471:2001
Opacity	MS ISO 2471:2010
Folding endurance	MS ISO 5629:1999

3. Results and Discussions

3.1 Chemical composition of EFB

In order to gain clearer picture on chemical composition of EFB fiber, comparison was done among findings by Sharma [32] and Onuorah [33]. The EFB did not go through any process such as pulping prior to chemical analysis. The content of extractive from this study was found slightly higher than the

other two findings which may be due to the dissimilarities in soil characteristics, climatic surroundings or agricultural arrangement [31]. Onuorah [33] concluded that water and alcohol-benzene solubility can affect the total pulp yield, drainage, paper quality and high alcohol benzene solubility (ABS) can be signed of potential problems which could influence paper formation while Rosli [6] implied that the strands of stem and EFB showed slightly lower solubility in alcohol-benzene as their parenchyma cells had been, more or less, removed.

The lignin content also was considered as high than Onuorah [33] but not much difference than other findings. This shows that EFB is suitable to be pulped using kraft/soda pulping due to percentage amount of lignin as EFB utilize fewer chemicals. Bleaching can be done more easily as lignin content is low, which indicates that this material can undergo bleaching with the minimal usage of chemical [34].

The holocellulose content obtained in this study is lower than Onuorah [33] that can be significantly related to different plant varieties used. However, the chemical composition was at par with findings by Ferrer [35] and α -cellulose content was also almost similar with Onuorah [33]. The cellulose is very important in papermaking process because it affects the strength of paper. Cellulose also will affect the property and economic production of fibers for various uses [34].

Table 2. Chemical composition of EFB fiber.

Analysis	Content, %			
	This study	Ferrer [34]	Sharma [32]	Onuorah [33]
Alcohol benzene extractive	4.80 ^a	1.17	4.10	1.6 ± 0.01 ^b
Acid insoluble lignin	25.74 ^a	24.45	30.99	17.80 ± 0.28 ^b
Holocellulose	66.99 ^a	66.97	72.00	77.35 ± 0.16 ^b
α -cellulose	43.66 ^a	47.91	-	43.51 ± 0.30 ^b

^aAverage values of duplicate experiments

^bAverage values ± standard deviations of triplicate experiments

3.2 Effect of MCC incorporation on oil palm EFB pulp property

Table 3 displayed the effect of adding MCC with the EFB kraft pulp in the papermaking process. Canadian Standard Freeness (CSF) test showed that there was a decrease in the freeness of the pulp with the increase of MCC percentage. Addition of 3% of MCC resulted in higher freeness compared to the control sample while sample MP9 showed the same amount of freeness with the control. Increased in freeness may be the result of MCC loss with the water during the test. As the percentage of the MCC increased, the bonding between the fibers resulted in decline trend in freeness.

Table 3. Freeness of kraft EFB pulp.

Sample	CSF (ml)
MP0	465
MP3	525
MP6	490
MP9	465

^aAverage values ± standard deviations of triplicate experiments

3.3 Effect of MCC incorporation on oil palm EFB paper: morphological observation

Morphological properties of MCC are shown in Fig 1 which exhibited a disordered, ruptured and aggregated microstructure. The particle size distribution of the commercial MCC indicated that the volume weighted mean diameter is 277.27 μ m. The evaluation of hand-sheet structure was determined using SEM. Fig 2 showed the morphology of the reference sample which has not been incorporated with any additive. Addition of MCC to the hand-sheet have a tendency to smooth the surface of the paper while as the concentration of the MCC increases the SEM assessment showed an increase in the bonding of the fiber which tends to cover the pores (Figure 3). Structural properties in addition with MC has been reported previously by Bahar [17] which conclude that the presence of MC, develop

greater surface area and binds cellulose fibers with another because of their small size fiber. Increment of surface area will lead to escalation of hydrogen bonding. Study on retention of nanocellulose on paper sheet has not been extensively done as they were chemically alike to the pulp and hard to detect in paper. Fibrils cannot be seen with microscopic methods after drying process of the handsheet [26]. The same goes to the MCC as their properties were similar to the pulp.

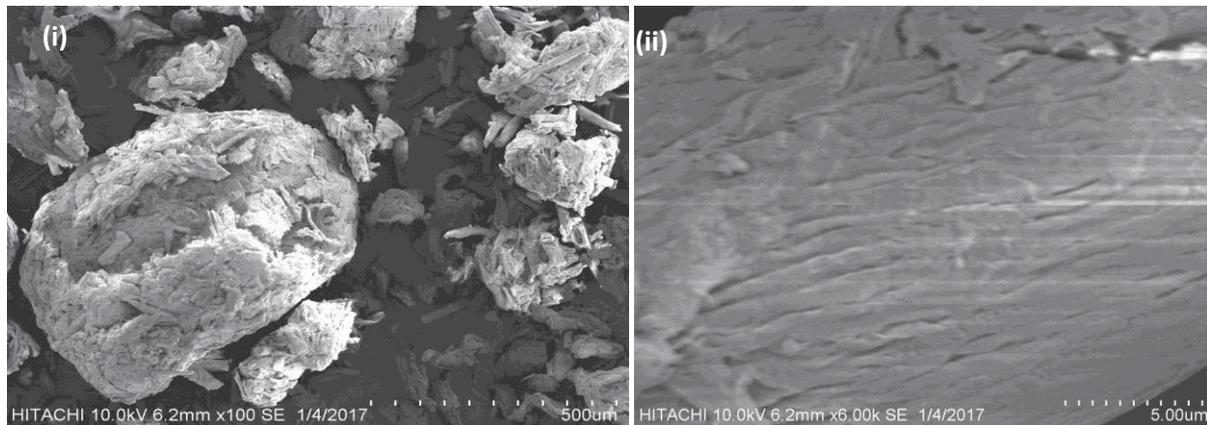


Figure 1. SEM analysis of the commercial MCC at (i) 100x and (ii) 6,000x magnifications

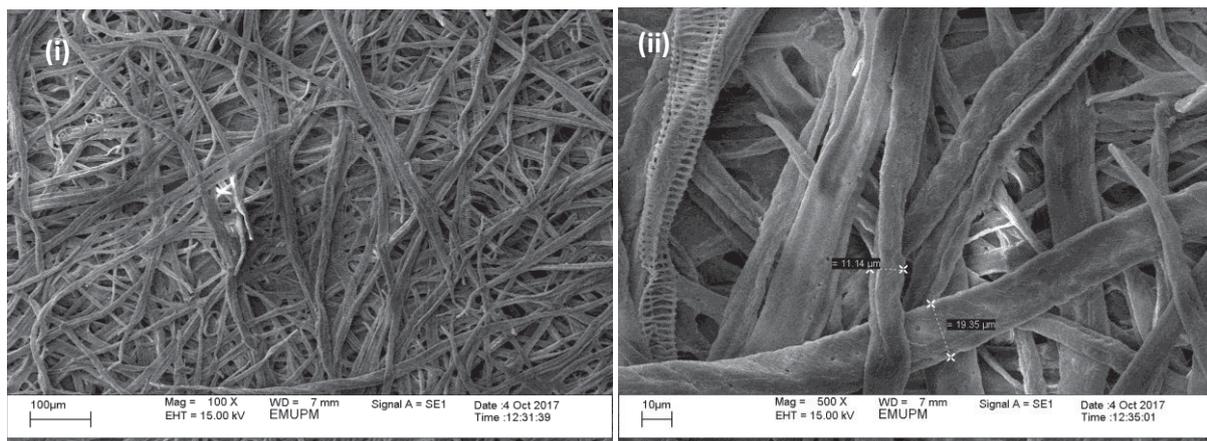
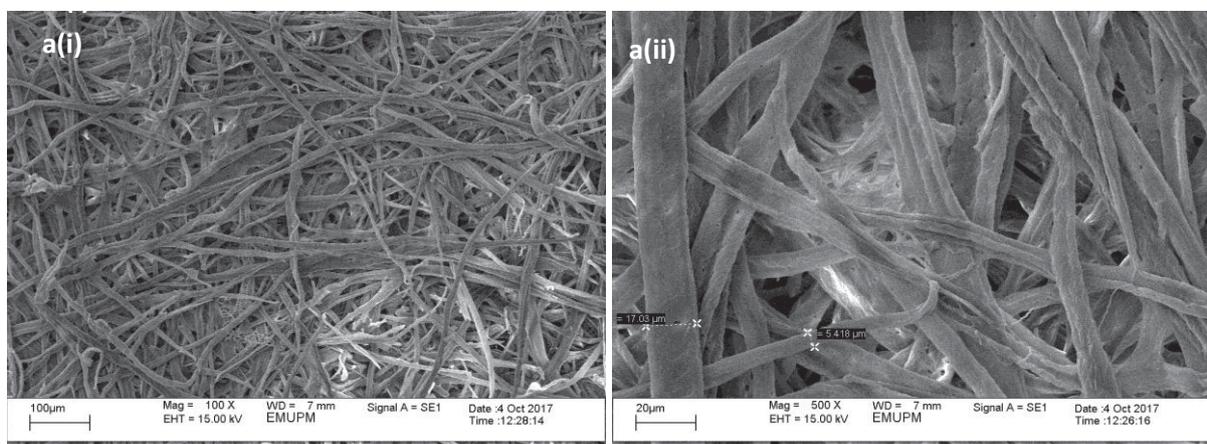


Figure 2. SEM analysis of the sheet structure composed of kraft EFB paper, MP0 at (i) 100x and (ii) 500x magnifications



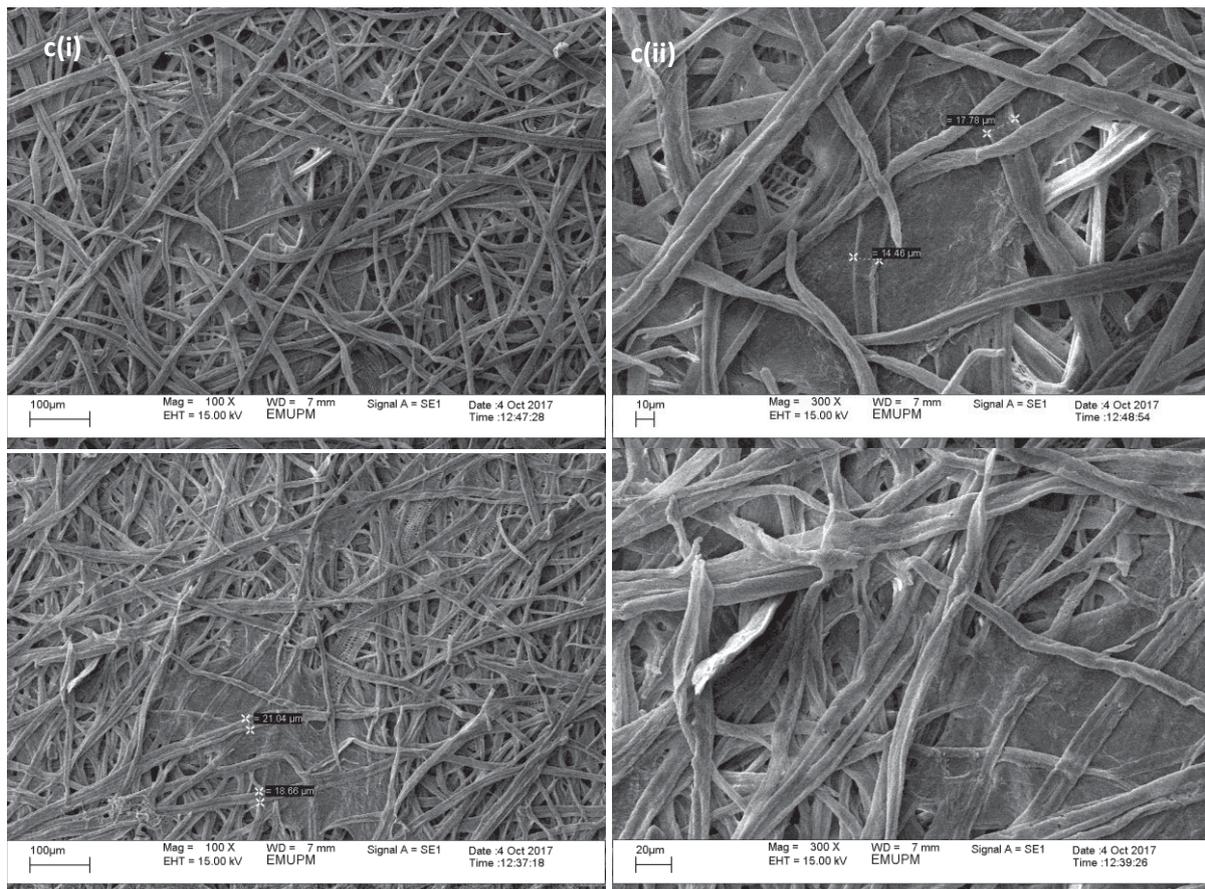


Figure 3. SEM analysis of the sheet structure composed kraft EFB and EFB-MCC papers (3%, 6%, and 9%), (a) MP3, (b) MP6, (c) MP9 .The corresponding image acquired at 100x and 300x for (a), (b), and (c).

3.4 Effect of MCC incorporation on oil palm EFB paper: physical properties

An EFB paper is produced as reference to compare the potential benefits of incorporating MCC with the kraft EFB pulp. This is presented in Table 4. The thickness of the paper produced after incorporating with MCC is lower than the reference sheet. Petroudy [28] also reported that decrement in thickness happened with addition of 5% MFC caused by combination of the pulp with MFC which both are anionic in neutral water thus they repel each other [22]. In fact, thickness, grammage and bulk thickness of the paper decreased with the increased of percentage of the MCC. Addition of 3% MCC give about 5% reduction in thickness. There is no significant difference between the grammage of the reference hand-sheet compared to the hand-sheet that was added with MCC.

Table 4. Physical properties of EFB paper (MP0) and EFB-MCC paper (MP3, MP6 and MP9).

Sample	MP0	MP3	MP6	MP9
Grammage (g m^{-2})	32.68	33.08	32.03	33.38
Thickness (μm)	81.65 ± 4.4^a	77.61 ± 4.0^a	71.4 ± 2.9^a	63.23 ± 2.5^a
Bulk Thickness ($\mu\text{m/g m}^{-2}$)	2.50	2.35	2.23	1.89

^aAverage values \pm standard deviations of 10 replications

3.5 Effect of MCC incorporation on oil palm EFB paper: mechanical properties

The mechanical properties of paper are controlled by fiber strength, the bonding degree of fiber network and strength of the bonds [22]. Fiber-to-fiber joint strength as well as on bonded area highly affect the bonding strength. The plateau in tensile strength may represent the amount of bonded area when the sheet is so strongly bonded that bonds are not broken when the paper is subjected to tensile forces but the fibers themselves are broken [27]. As expected, the result indicated that incorporation of MCC help to increase the tensile strength of the paper (Figure 4). As tensile strength of paper depends on the fiber strength and the bonding strength, this showed that MCC promoted higher amount of bonded areas (Figure 4) as they have larger surface areas. The positive effect of MCC addition in the papermaking was also reported by Ioe Zou and Hsieh, (2007).

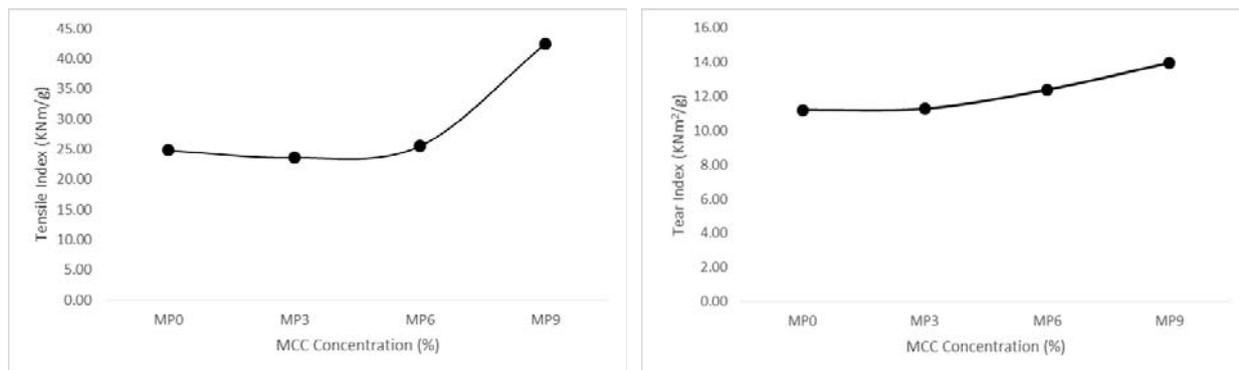


Figure 4. The effect of different percentage of MCC on a) tensile index and b) tear index of EFB paper (MP0) and EFB-MCC paper (MP3, MP6 and MP9).

Both tensile index and tear index showed linear increment with respect the MCC concentration (Figure 4). Addition of 9% MCC during the papermaking process increased both tensile index and tear index by 71.4% and 24.5% respectively. Increasing the number of bonds or the mechanical entanglement resulted in the enhancement of strength of the fiber network. The increased bonding hold the fiber and contribute to a greater network strength [22]. Petroudy [27] reported that tear resistance of the sheet negatively affected by addition of MFC but result obtained shows positive effect on the tear index of the paper sheet which supported Hassan [21] discovery as tear resistance did not deteriorated the strength of paper sheet with addition of 30% MFC to bagasse pulp.

Elongation and toughness energy absorption (TEA) show negative effect at the beginning for 3% addition of MCC but gradually increase for 6% and reaching highest value on dose of 9% concentration of MCC which is higher than the reference pulp (Figure 5). Positive result for the TEA and elongation by adding cellulose was previously reported by several studies. Bahar [17] reported that addition of 2% nanocellulose (70 nm) raised the TEA by 50% and elongation by 24%. An increase occurred by as much as 3 times for the hand-sheet which was added with 10% of nanofibrillated cellulose (NFC) to the unbeaten pulp [27]. By increasing the dose of MCC concentration improved fiber-fiber adhesion which is probably contribute to this effect.

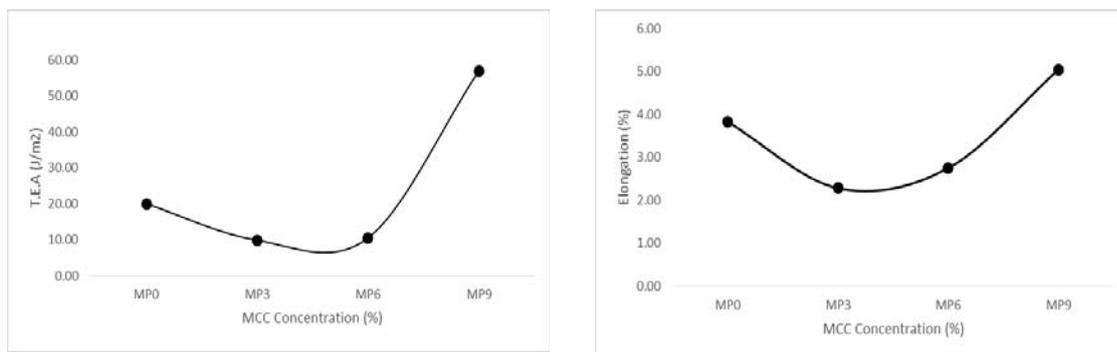


Figure 5. Effect of different percentage of MCC addition on a) T.E.A and b) elongation of EFB paper (MP0) and EFB-MCC paper (MP3, MP6 and MP9).

Increase of bonding can support folding endurance paper property. Fig. 6 exhibited that increase of folding endurance are linear with the increase of MCC concentration. Reference pulp showed lower folding endurance than Tanaka [16]. As EFB are short fiber, they tend to produce close sheet due to lower air permeability and bulk [35]. Folding endurance reached the highest with 9% addition of MCC which is almost 3 times compared to the reference paper.

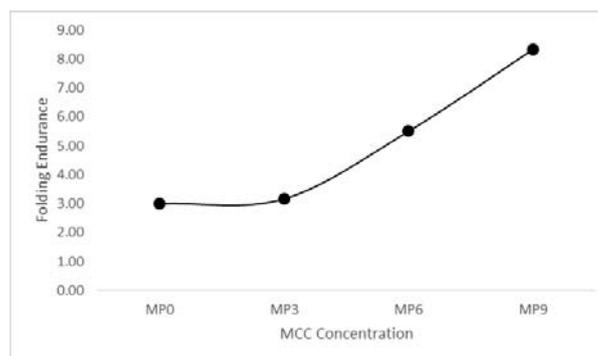


Figure 6. Effect of different percentage of MCC addition on folding endurance of EFB paper (MP0) and EFB-MCC paper (MP3, MP6 and MP9).

3.6 Effect of MCC incorporation on oil palm EFB paper optical properties

Both results indicated that opacity and brightness decreased linearly (Figure 7) with respect to MCC concentration. Brightness showed significant difference between the EFB paper and the EFB-MCC paper while opacity showed a little difference as the opacity ranging from between 99.99 to 99.93. The concentration of 9 % MCC concentration gave 0.06 % and 29.00 % decrement in terms of opacity and brightness respectively. Study done by Petroudy [27] reported that replacing 5 % soda bagasse pulp with MFC resulted in significant decreased of opacity. Kajanto [26] also reported that optical properties of paper worsened with addition of nanocellulose as 2-3 % points lower opacity is measured at constant weight basis. These slight decreases of brightness and opacity resulted from improvement of interfiber bonding with the MCC causing reduction on the light-scattering surface. The produced sheet was more compact which consequently reduce the brightness and opacity.

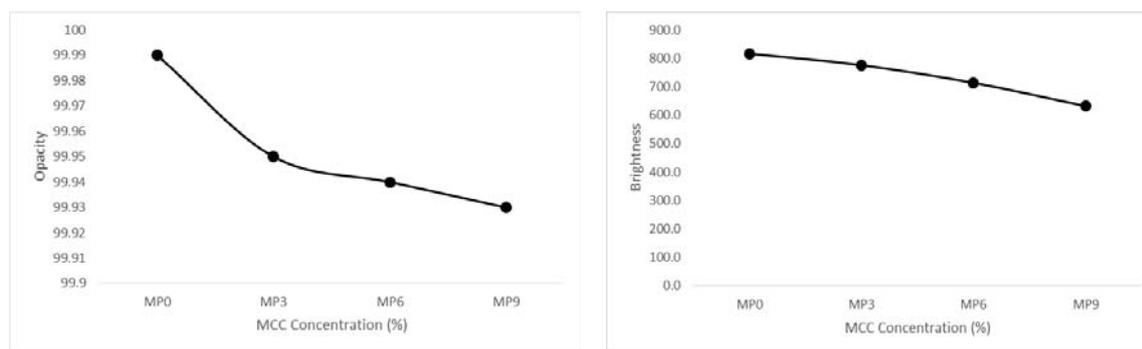


Figure 7. The effect of different percentage of MCC on a) opacity and b) brightness of EFB paper (MP0) and EFB-MCC paper (MP3, MP6 and MP9).

4. Conclusions

In conclusion, all results demonstrated excellent properties especially paper mechanical properties due to the incorporation of MCC. The results indicated that this type of MCC has great potential as an additive and reinforcing agent in papermaking by increasing the strength of paper even at low concentration. Looking at beneficial effect on tensile and tear indices allows high potential for high grade and specialty usages of writing and printing. Combination of low porosity but high strength is crucial characteristics required for such niche application of specialty paper. The MCC also has extra advantage as they are derived from pulp itself which make them highly compatible with the fiber and are more environmentally friendly. Their larger surface area helps to increase the binding and help to reduce the frequency of paper to rupture. Further study can be carried out such as drainage time and beating of pulp prior to incorporation with MCC.

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