

Solely Cellulose-based Adsorbent Derived from Oil Palm Empty Fruit Bunches for Dye Removal

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Abstract

The purpose of this research is to determine the adsorption capability of micro and nano-cellulose derived from oil palm empty fruit bunch (EFB) as dye removal. Cellulose based adsorbents were successfully obtained from EFB in the form of microcellulose (eMC) via both delignification (D-eMC) and bleaching processes (B-eMC) as well as in nanocellulose (eNC) by using acid hydrolysis method and hence termed as delignified-eNC (D-eNC) and bleached-eNC (B-eNC) respectively. Dye adsorption test was carried out by UV-Spectrophotometer by comparing initial dye MO concentration (as a control solution) to the treated MO solution upon the addition of micro- and nano- cellulose based adsorbent. It was clear that the dye removal efficiency of micro-cellulose both D-eMC and B-eMC were lower than the nano-cellulose structures. The higher adsorptive capacity of nano-size cellulose compared to the micro-size cellulose was confirmed by the distinct presence of FTIR shifting peak of hydroxyl and sulfonyl groups. It was expected as the micro-size of cellulose would not be able to provide a good adsorptive capability of hydroxyl surface active agent to adsorb the dye. In the case of eNC, the result showed that D-eNC afforded a better dye adsorption than B-eNC. At the same concentration of eNC at 3 wt. %, D-eNC could adsorb at about 19.3% of MO while only 2.4% of MO could be removed by B-eNC. Finally, all of adsorbent could maintain pH and TDS within water quality specification.

Keywords: adsorption, dye removal, nano-cellulose, oil palm empty fruit bunches, water treatment

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INTRODUCTION

The massive development of the textile industry has significantly increased the production of dyes or synthetic pigments. Although this massive development offers the benefit to the economy, this industry has been

responsible for the serious water pollution as 80% of wastewater comes from the textile industry (Anbia and Salehi, 2012). This waste is difficult to degrade and easy to be accumulated in the environment lead to the

reduction of water quality and health problems for humans and the ecosystem.

Studies were made to remediate this water pollution via chemical like a photocatalytic system (Rusadi *et al.*, 2018), biological that use enzyme azoreductase as an azo dye degradation, and physical treatments (Shah *et al.* 2013). Among these methods, adsorption is considered a promising technology because of low-cost, easy and effective operation (Mahmoodi *et al.*, 2010). In particular, the use adsorbents derived from natural materials has been attracted academia to reduce dyes contaminant due to good mechanical strength, resistance to abrasion, as well as the stability of remediation after repeated regeneration and biodegradability (Laraous *et al.*, 2005).

Oil Palm Empty Fruit Bunch (EFB) is one of the solid wastes produced from the palm oil industry. It is reported that at about 7 million tons per year of EFB was generated. There is a limit utilization of EFB which is only 10% of EFB has been used for boiler and compost fuel, while most of them are discarded or burnt causing serious environmental problem and giving no benefits (Ngadi and Lani, 2014). EFB contains high cellulose about 43%, hemicellulose 24%, and lignin 21% (Septevani *et al.*, 2019; Septevani *et al.*, 2018). The high content of cellulose can be used as a potential advance material due to, low density, low abrasiveness, high biodegradable capability and recently reported as effective adsorption capacity (Septevani *et al.*, 2018; Septevani *et al.*, 2020).

At nanoscale, cellulose possesses a large surface area, exceptional mechanical properties, and low coefficient of thermal expansion (Septevani *et al.*, 2017; Batmaz *et al.*, 2014). In these particular studies, it is reported that nano-cellulose (NC) is a potential eco-friendly adsorbent for dye removal. In previous research showed, NC with sulfonyl group functionality (NCs) that prepared by acid hydrolysis with sulphuric acid displayed a high uptake adsorption capacity for methylene blue dyes with a maximum adsorption capacity was 118 mg/g at 25 °C and pH 9 (Batmaz *et al.*, 2014). Furthermore, tunability of the adsorption capacity by surface modification of NCs was shown by oxidizing the primary hydroxyl groups on the NCs surface with TEMPO reagent and the adsorption capacity increased from 118 to 769 mg /g (Batmaz *et al.*, 2014).

This study is aimed on investigating the potential use of micro-cellulose and nano-cellulose derived from oil palm empty fruit bunch (EFB) as the adsorbent of dye contaminant for water remediation. The structure-property relationship of EFB-based adsorbent was investigated in terms of dye removal, pH and total dissolved solids (TDS) analysis.

RESEARCH METHODS

Materials

Oil palm empty fruit bunches (EFB) was obtained from PT. Perkebunan Nusantara I, Sumatera Utara, Indonesia. Technical grade sodium hydroxide and hydrogen peroxide were purchased for alkaline

pre-treatment and bleaching process respectively. Sulphuric acid 98% was purchased from Merck for acid hydrolysis. Methyl Orange (4-dimethylaminoazobenzene-4'-sulfonic acid) (MO) was purchased from Merck for dye solution.

Preparation Microcellulose (eMC) and Nanocellulose (eNC)

Both eMC and eNC can obtained from raw EFB through, delignification, bleaching, and acid hydrolysis as our previous reported study (Septevani *et al.*, 2018 and Septevani *et al.*, 2019). The delignification proses was performed in the stirred reactor by adding NaOH 10% wt. into EFB at 150 °C and 4 bar pressure for 30 minutes. The delignified EFB (D-eMC) was then washed with water to a neutral pH. The D-eMC then went to the bleaching process by using 1 liter H₂O₂ and MgO 2% wt. The obtained bleached cellulose, denoted as B-eMC, was then washed with water to a neutral pH and dried overnight at 50 °C. Both B-eMC and D-eMC was then further hydrolyzed using 36 % of H₂SO₄ at 50 °C for 3.5 hours under vigorous stirring process, followed by neutralization and freeze drier to obtain nanoscale cellulose, denoted as B-eNC and D-eNC, respectively.

Preparation Solution Methyl Orange

Methyl Orange was chosen as a control solution because MO has azo bonding (-N=N-) that normally found in textile dyes and a more difficult to adsorbed than simple dye molecule like methylene blue. Stock solution of 1000 ppm of MO was prepared by dissolving 0.1 gr MO with 100 ml demineralized aqua. The MO solution was prepared by diluting demineralized water of stock solution to gain concentration 10 ppm. For standard solution, the concentration is made from 10 ppm, 8 ppm, 6 ppm, 4 ppm, and 2 ppm.

Characterization

Determination adsorption efficiency by using eNC & eMC adsorption was performed in 50 mL beaker glass each containing 25 mL of 10 ppm MO. eNC & eMC were added to each backer glass with the concentration 0.1%; 0.5%; 1%; 2%; and 3% (w/v). These mixtures were stirred using a magnetic stirring bar at 400 rpm for 30 minutes. The treated MO solution was separated by using a filter paper under vacuum condition. The final concentration of MO after adsorption was measured using Spectrophotometer UV-Vis at λ_{max} 465 nm. The adsorbent before and after adsorption was then dried and characterized by IR Prestige 21, Shimadzu. For pH and TDS analysis, the MO solution before and after the adsorption test was measured using L-AQUA pH/ion/cond. meter F-74 BW, Horriba Scientific. Hitachi SU3500 with an acceleration voltage of 0.5 KV was performed in the Research Center for Physisc, LIPI to analysis the morphology of cellulose after treatments. Prior to SEM analysis, the samples were

coated with gold to provide about 200 Å gold layer thickness using a vacuum sputter coater. At the nanoscale, the morphology of NC was analyzed by Transmission Electron Microscopy (TEM) at Eijkman Research Institute. The NC suspension was spotted onto a gold glider grid G400G and then stained with 2% aqueous uranyl acetate. The prepared samples were analyzed in a JEOL 1010 (JEOL, Japan) at 80 kV. The average diameter and length of cellulose fibers were measured using digital image analysis (Image-J) software with a minimum of 10 measurement.

RESULT AND DISCUSSION

Synthesis Microcellulose (eMC) and Nanocellulose (eNC) adsorbent

The delignification process was carried out by dissolving EFB in NaOH solution so that some components of lignin, hemicellulose, oil, and wax can be removed from cellulose fibers by increasing the surface accessibility of lignocelulose fibers towards chemicals (Burhani *et al.*, 2017) and thus enable a greater dissolvability of the lignin and hemicellulose, to produce higher content of cellulose at about 70.12% (Septevani *et al.*, 2019). The obtained delignified cellulose is thus denoted as D-eMC. The bleaching process was then performed to remove the remaining lignin and hemicellulose in D-eMC using H₂O₂ as an environmentally friendly bleaching agent because after the reaction, it decomposes into hydrogen and oxygen with almost no dangerous residues (Bensah and Mensah, 2013). H₂O₂ is also strong oxidizing agent so that white and high cellulose at 81.5% (denoted as B-eMC) was obtained (Septevani *et al.*, 2019; Septevani *et al.*, 2018). Both B-eMC and D-eMC were then used to produce nano-cellulose through the acid hydrolysis process, and thus denoted as B-eNC and D-eNC respectively.

Figure 1A and 1C show that microsize fibre was obtained after delignification and bleaching. Individual well-separated microsize fibre was observed in the B-eMC while some microfibre bundles were still observed in D-eMC. Further process by acid hydrolysis reduce the size into nanoscale for both D-eNC and B-eNC. The presence of acid will diffuse into the cellulose fibers, and selectively attack the amorphous cellulose by separating the glycosidic bonds leading the acid to reduce the size of the fiber and increase crystallinity (Batmaz *et al.*, 2014; Adiatama *et al.*, 2007). Figure 1B shows that D-eNC possessed a long entangled nanofiber structure with length in microns and diameter in about 10-20 nm. Meanwhile, rod-like nanocellulose was observed in the B-eMC (Figure 1D) with the low aspect ratio of 13 at 10.7 ± 1.8 nm in width and 128.4 ± 17.2 nm in length. High purity of cellulose after bleaching (B-eMC) is postulated to be easier deconstruction into a smaller size compared to D-eMC which still contained complex structure of lignin and hemicellulose (Septevani *et al.*, 2020).

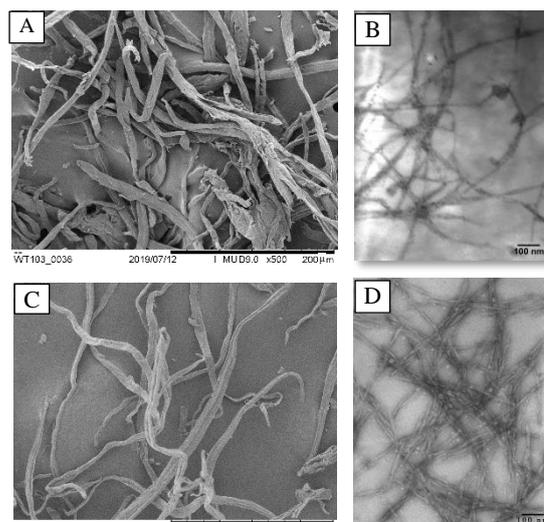


Figure 1. Microscopy of micro and nanocellulose from EFB: A) D-eMC; B) D-eNC; C) B-eMC, and D) B-eNC

Adsorption MO onto EFB-NC

The adsorption efficiency was determined by using UV spectrophotometry by comparing the concentration of MO before and after treatment of EFB adsorbent. Figure 2 shows the adsorption efficiency of EFB-based adsorbents towards MO with varied concentration of adsorbent. Figure 2 clearly showed that the MO adsorption efficiency of micro-cellulose was lower than that of nano-cellulose adsorbent. The maximum adsorption efficiency by using 2% wt. of B-eMC was 1.4 % while 1 % adsorption efficiency was obtained by using D-eMC. Nano scale adsorbent of D-eNC was the most effective adsorbent compared to other adsorbents

The increasing concentration of D-eNC was proportional to increasing adsorption efficiency. The addition from 0.1 up to 3 % gradually increased the adsorption efficiency. At the same concentration, 3% of D-eNC could adsorb up to 19.3% of MO which was

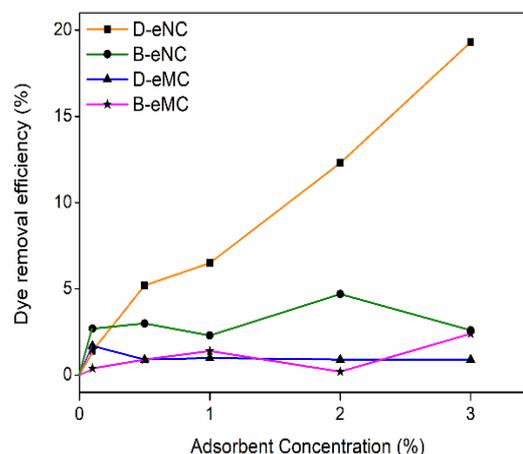


Figure 2. Efficiency adsorption of eNC and eMC

greater than that of B-eNC at only 2.6% adsorption efficiency of MO. The greater adsorption ability of D-eNC compared to B-eMC, might be attributed to the presence of complex aromatic lignin (at about 12% in D-eNC, which was much higher than that of B-eNC at only 0.8% wt.) to offer greater ability of dyes removal. Previous research has been reported that lignin from sugarcane bagasse can adsorb Methylene Blue with an excellent adsorption capacity (Filho *et al.*, 2007). The mechanism of adsorption process of dye removal for methyl orange by the presence of lignin is also reported by Budnyak *et al.*, 2018 due to simultaneously mechanism of electrostatic interaction between positively charge of nitrogen (N) in dye components and dissociated carboxyl and oxygen in the lignin sorbents as well as the hydrogen bounds and π -interactions during the process. The reported mechanism is illustrated in Scheme 1.

Figure 3 shows the reduction of colour intensity as reflected by reduction absorbance intensity by using the optimum condition of nanocellulose obtain from both bleaching (B-eNC) and delignified (D-eNC) process. It is



Scheme 1. Reported mechanism of dye adsorption due to the presence of lignin in the adsorbent, adapted from Budnyak *et al.*, 2018

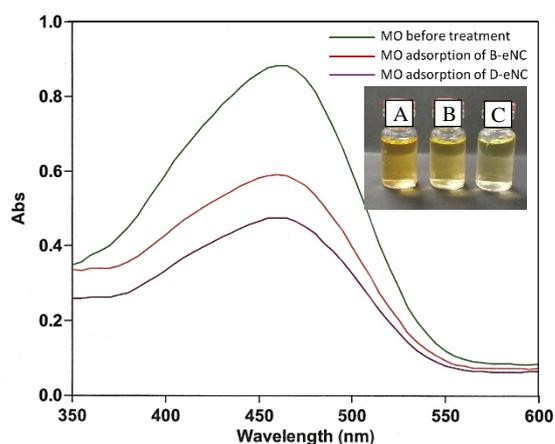


Figure 3. The UV-VIS Absorbance and physical photographs of MO before (A) and after adsorption of B-eNC (B) and D-eNC (C).

clear that the reduction absorbance intensity by using D-eNC are higher than B-eNC. The physical color change of control MO (inset Figure 3A) compared to treated MO after adsorption by using B-eNC (inset Figure 3B) and D-eNC (inset Figure 3C) is also in agreement with the reduction absorbance intensity and illustrated in Figure 3. The solution after D-eNC treatment shows a greater change in color towards control MO compared to the solution after B-eNC treatment showing the physical evidence of a greater efficiency as a dye removal for D-eNC.

FTIR Analysis

FTIR spectra for both fresh D-eNC and D-eMC was illustrated in Figure 4. The aliphatic C-H group is indicated by a peak of 2895 cm^{-1} at D-eNC and 2854 cm^{-1} on D-eMC. Lignin is indicated by a peak between $1200 - 1300\text{ cm}^{-1}$ with aromatic skeletal vibrations (Reddy and Yang, 2005). Both D-eNC and D-eMC indicate the

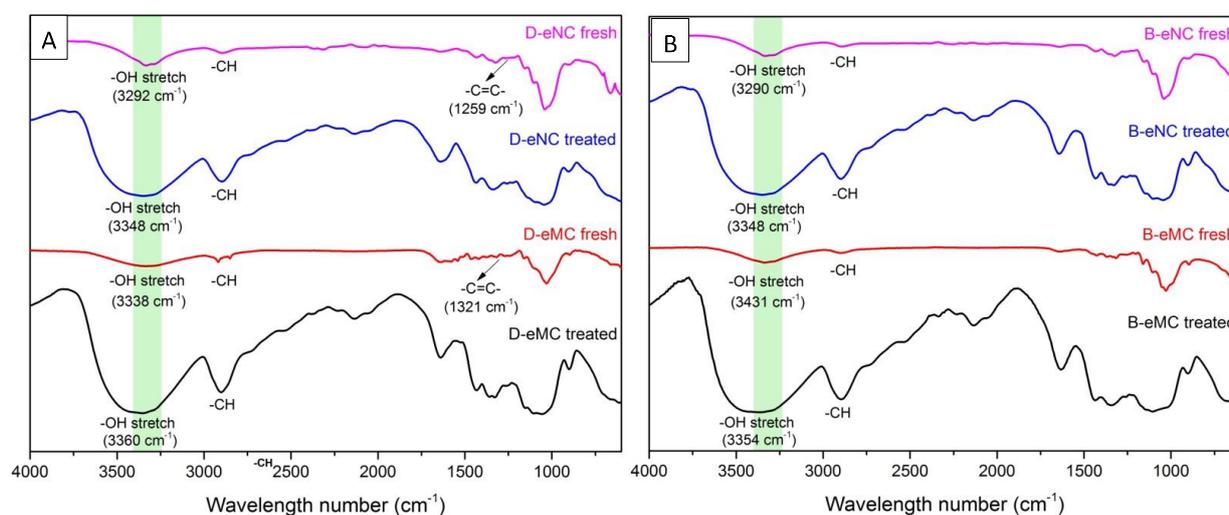


Figure 4. FTIR spectrum of micro and nano-adsorbent from (A) delignification and (B) bleaching process before and after adsorption

presence of lignin content which is shown at the absorption peak of 1259 cm^{-1} and 1321 cm^{-1} respectively. Meanwhile, the aromatic skeletal vibration does not present in B-eMC and B-eNC, which mean that lignin was substantially removed in the bleaching process.

There was a difference in intensity of -OH stretching. It is seen that the absorption of -OH stretching at D-eNC (3332 cm^{-1}) was sharper than D-eMC. This sharper absorption of -OH stretching indicates the presence of intermolecular hydrogen bonds. The breakdown of intermolecular hydrogen from both cellulose and lignin leads to the formation of greater presence of free -OH groups on the surface of D-eNC as an effective functional group to adsorb MO molecules. In previous research, it has been reported that the mechanism adsorption of the Methylene Blue (MB) can be proposed when MB diffuse through the boundary layer onto the surface of the adsorbent (micro-cellulose). The MB molecules adsorbed on the active site (hydroxyl groups) of the surface and into the interior pores of the micro-cellulose particles (Tan *et al.*, 2018).

The shifting peak at the functional group -OH stretch on D-eNC spectra was possible because the functional group -OH after hydrolysis with acid (H_2SO_4) was protonated to H_3O^+ (Hardjono, 1991). Protonated conditions indicate that the -OH bonding character increases in length resulting in a decreased vibration or a shifting in the D-eNC spectra.

The presence of the -OH bond indicates that the eMC and eNC tend to be more polar indicating the potential to be used as an adsorbent for substances that tend to be polar such as for solution and gas (Wardono *et al.*, 2019). A shifting in the peak absorption of -OH stretching on the fresh D-eNC (before adsorption) at 3332 cm^{-1} compared to the -OH stretching on the D-eNC after adsorption, (denoted as treated D-eNC) at 3348 cm^{-1} was observed. This is presumably because on the surface of treated D-eNC in the -OH group originating from lignin and cellulose interacting with adsorbate molecules (dyes) through hydrogen bonds which is detected by shifting the peak of the absorption -OH stretching. The resulting absorption at 3348 cm^{-1} on treated D-eNC was broader compared to fresh D-eNC indicating strong intermolecular hydrogen bonding (Silverstein *et al.*, 2005) between cellulose and/or lignin with MO molecule. It is like fresh D-eMC, B-eMC, and B-eNC where the -OH stretch region were very broad after adsorption, indicating there was hydrogen bonding between adsorbent and adsorbate.

The protonation of eNC after hydrolysis provides a significantly strong electrostatic attraction between the fibers surface and the dye molecules leading to maximum adsorption (Vinoth and Lim, 2005). Hence, the adsorption of eNC for the dye molecule (MO) was possibility attributed to two mechanisms, firstly, an electrostatic attraction between the protonated hydroxyl groups of adsorbents and dyes; and secondly, the interaction between MO and adsorbents via hydrogen bonding (Ho and Mc Kay, 1999).

pH and TDS properties

Analysis of pH level before and after adsorption is illustrated in Figure 5. The pH level after adsorption by D-eMC and B-eMC slightly increased from 6.04 to 6.14 and 6.34, respectively. Meanwhile, there was a significant decreasing in pH level on the treated MO solution for the adsorption by D-eNC and B-eNC at 5.87 and 4.85, respectively. The decreasing of pH level possibly was attributed to the release of sulphonyl groups on eNC from adsorbent back to MO solution. However, our previous studies showed that the amount of sulphonyl groups release back to the treated water was insignificantly changed (Septevani *et al.*, 2020).

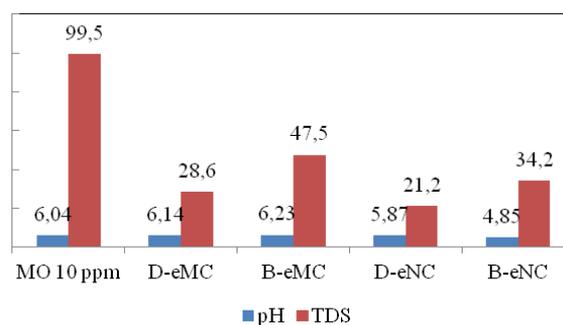


Figure 5. pH and TDS properties

The results for TDS analysis both eNC and eMC showed that there was a significant decrease in the amount of TDS after adsorption for all the adsorbents which achieved up to 78% TDS reduction. This indicates the potential application on the use of EFB as an effective adsorbent for organic contaminants. Finally, the results showed that all the adsorbent resulted in the changes of pH and TDS within the World Health Organization (WHO) specification which are 1000 mg/l and 6-9, respectively.

Thermogravimetric Analysis

Thermal stability of cellulose-based adsorbents was investigated by Thermogravimetry Analysis. The initial thermal degradation temperature of eNC based adsorbent was lower than that of eMC based adsorbents. The onset degradation temperature (T_{onset}) of eMC (D-eMC and B-eMC) was begun at approximately $285\text{ }^{\circ}\text{C}$ while for nanocellulose (D-eNC and B-eNC), T_{onset} was at about $215\text{ }^{\circ}\text{C}$. It was expected that the presence of a sulfonyl group upon acid hydrolysis can accelerate the dehydration process leading to a lower initial decomposition temperature (Septevani *et al.*, 2017). Nevertheless, this eNC based-adsorbent is still considered to possess a higher thermal stability at above $200\text{ }^{\circ}\text{C}$ indicating potential results for the possible treatment at high-temperature contaminant water.

Raw e-EFB possesses a higher thermal stability as a note by lower percentage of weight losses, simply because it still contains many impurities including Silica to give a higher resistance at high temperature (Septevani *et al.*, 2020).

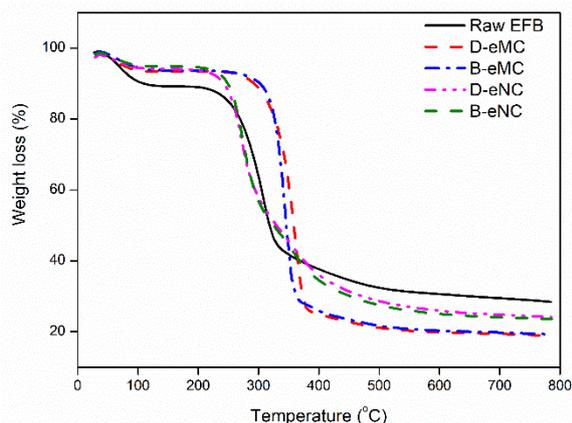


Figure 6. Analysis TGA of adsorbent

Further, eMC based adsorbent (D-eMC and B-eMC) exhibited a single-step and sharp decomposition with a greater weight loss compared to eNC samples (D-eNC and B-eNC). Observed final weight losses for various samples at 700 °C follow the order: eMC (D-eMC and B-eMC) at $\pm 81.5\%$ > eNC (D-eNC and B-eNC) at $\pm 75.2\%$ > raw EFB (70.6%). A greater thermal stability at high temperature of eNC compared to eMC was possible due to the higher crystallinity of eNC compared to eMC. As earlier discussion, the presence of strong acid upon nanocellulose preparation selectively attack the amorphous cellulose by separating the glycosidic bonds leading the acid to reduce the size of the fiber and increase crystallinity (Batmaz *et al.*, 2014; Adiatama *et al.*, 2007). Our previous reported studies showed that crystallinity of eMC derived from EFB was at about 70-80% while eNC exhibited 86-96%. It is known that ordered domain found in crystal structure would provide unique and outstanding properties include mechanical, optical and thermal properties, which expected to give a greater thermal degradation at higher temperature in this study.

CONCLUSION

The adsorption capability of adsorbent based on empty fruit bunches towards dye contaminant of methyl orange was thoroughly studied at room temperature. The effect of the adsorbent concentration for both eNC and eMC was investigated. Nanosize cellulose provide a greater adsorption capability compared to micro-size cellulose. Based on the UV-VIS Spectra, at the same adsorbent concentration, D-eNC which could adsorb a greater MO compared to B-eNC. The lignin and cellulose structure from D-eNC plays an important role in the mechanism in the adsorption of dye, which was postulated due to electrostatic mechanism as supported by FTIR analysis. Further, the eMC adsorbent showed an insignificant increase in pH level, while a decrease in pH level was observed by eNC which was attributed to the sulfonyl release. In terms of TDS analysis, both eMC and eNC resulted in a decreasing TDS value after adsorption indicating a potential use of EFB as eco-friendly based adsorbent for organic contaminant remediations.

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