

DELIGNIFICATION OF PALM OIL EMPTY FRUIT BUNCH BY HYDROXYL RADICAL OF MICROWAVE RADIATION UNDER ALKALINE CONDITIONJONI PRASETYO^{1,2}¹NATIONAL RESEARCH AND INNOVATION AGENCY (BRIN)
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ABSTRACT

This work assessed how to remove lignin of palm oil empty fruit bunch (EFB) in environmentally friendly method and less energy. Moreover, delignification was strengthened with additional H₂O₂ to perform hydroxyl radical by microwave radiation in low NaOH solution of concentration up to 0.8 M, 10%. Setting on microwave, solution was heating up and cause delignification work more effective. Observation at black liquor showed that temperature was influence by microwave radiation duration while NaOH concentration affected total suspended solid increasing. Optimally treated EFB at 0,2 M NaOH with effective lignin removal and holocellulose increasing reached 27% and 26%, respectively. Unfortunately, at higher NaOH concentration, the decomposition penetrated EFB interior, holocellulose and made lignin percentage high again. Therefore, EFB yield got less at higher NaOH concentration because more EFB was sludgy and lost during washing. XRD analysis showed the increasing crystallinity by 2.2%, closer to α -cellulose. SEM showed successful impurities removal from EFB surface and Ca, Nb, Si, and Mg detected by EDS.

KEYWORDS: Palm oil empty fruit bunch, hydroxyl radical, delignification, microwave, NaOH solution.

INTRODUCTION

Advanced oxidation process (AOP) offered applicable technology for delignification in term of strong oxidation ability of lignin degradation efficiency. The work could occur due to the existence of oxidative agent, hydroxyl radicals (OH°) that formed by hydrogen peroxide (H_2O_2) and microwaves irradiation. AOP has been applied successfully in waste water treatment process. OH° has an important role in the degradation of many organic compounds (Deng 2015, Pera-Titus 2004). AOP treatments for organic pollutant in water had been reported in some literatures like polychlorinated dibenzo-p-dioxins and dibenzofurans (Vallejo 2015). In addition, AOP was applicable for high temperature treatment such as supercritical water oxidation (Zhao 2014). In case of palm oil empty fruit bunch (EFB) pretreatment, innovation is still needed to develop further especially for efficiency energy consumption, economic feasibility approach (Gogate 2004). Therefore, AOP was applied to improve the delignification of EFB.

Indonesia is the largest palm oil producer in the world. Crude palm oil (CPO) production reached 27 million tons in 2017 (Hasanudin 2022). Palm oil plantation was spread wide through some big islands, mainly Sumatera and Borneo, stretch across 14.03 million hectares by 2017 (Sari 2019). As the consequence, the more CPO production generated the more solid waste as well. Especially EFB from Sulawesi Island was considered low quality because of high lignin content. So far, EFB utilization was limited, only used to substitute boiler fuel, road pavers in oil palm plantations and composting.

EFB is a lignocellulosic biomass with generally comprise of 66.97% holocellulose, composites of cellulose and hemicellulose, and the rest is 24.45 % of lignin (Sugiwati 2021). On a more elaborate analysis, EFB consist of components that described on Tab. 1. However, the quality of EFB depends on plantation management besides plantation environment.

Tab. 1: Composition of EFB (wt%) (Abdullah 2011, Chang 2014).

Components	Composition (wt %)
Cellulose	24 - 65
Hemicellulose	21 - 34
Lignin	14 - 31
Carbon	48.9
Hydrogen	6.3
Nitrogen	0.7
Sulphur	0.2
Oxygen	36.7
Potassium	2.24
K_2O	3.08-3.65

Fresh fruit bunch processing at palm oil mill would generate EFB between 25-34% of fresh bunches. EFB consisted of three main components, cellulose, hemicellulose and lignin (Abdullah 2011, Rahayu 2019). Delignification is a process for lignin removal whereas lignin encircled cellulose and hemicellulose (Mohtar 2015). Delignification is a very important stage and must be carried out in order to improve accessible any reagents including cellulase to access cellulose and hemicellulose (Kusmardini 2018). Some researchers conducted in pilot scale using base or acid method as well as steam explosion method (Akhtar 2015). Chemical delignification either with

strong acid or base would produce chemical waste. Bio-delignification with *Trichoderma reesei* and *Aspergillus niger* conducted in 6 days. Bio delignification was time consuming and produced only 1.251 g^l⁻¹ glucose. Previous research for bio-delignification conducted in 2016 using local isolate and identified as *A. fumigatus*, lignin removal was low but saccharification increased significantly rather than untreated EFB (Kusmardini 2016).

The main goal of this work is to assess environmentally friendly delignification in alkaline solution using microwave irradiation for short period with H₂O₂ additional. Microwave irradiation would lead to OH[•] formation (Dewi 2018, Chavoshani 2018). Moreover, microwave irradiation gave advantage like heating up slowly in evenly and saving energy (Mikulski and Kłosowski 2022). This method is expected able to improve the efficiency and effectiveness of delignification.

MATERIAL AND METHODS

Materials

Palm oil empty fruit bunches (EFB) were provided from Sulawesi Island. The EFB was considered as low quality in terms of the lignin content. Undesirable lignin was significantly higher than that of Sumatera Island. Based on 5 years research, lignin mostly reached 40%. α -cellulose for the reference was provided from Sigma-Aldrich, Life Science. Technical grade sodium hydroxide (NaOH) and H₂O₂ were used for delignification.

Methods

Delignification was conducted at 10 g EFB in 200 ml NaOH at various concentration 0.2, 0.4, 0.6, and 0.8 M. Innovation this work was the addition of 500 ppm H₂O₂ to the NaOH solution before microwave irradiation. Power setting for microwave irradiation was at lowest level, 100 W for 15 min. The effect of microwave irradiation was carried out by measuring the temperature directly after the irradiation was completed. The solution was stirred up for an hour while cooling down. Black liquor and treated EFB were separated. Black liquor was analyzed for oxidation-reduction potential (ORP), total dissolved solid (TDS) and total suspended solid (TSS). Treated EFB was washed with tap water and dried at 60°C in oven for overnight. The treated EFB was analyzed for its component.

Analysis

Lignin was analyzed according National Renewable Energy Laboratory (NREL) procedure (Maryana 2022). Acid insoluble lignin (AIL) was treated using cellulose acetate filter paper 0,45 μ m with diameter 47 mm. Acid soluble lignin (ASL) was analyzed using UV spectrophotometry with coarse cuvette at wavelength 205 nm. Ash content was analyzed using crucible ceramic and put into the muffle furnace at 575°C for 3 hours.

Based on NREL guidance some monomer sugars, glucose and xylose, were analyzed through reducing sugar (RS) approach DNS method. Xylose was a representative for sugar C5 and glucose a representative sugar C6. Source of xylose was hemicellulose while source of glucose was cellulose. Both cellulose and hemicellulose was called holocellulose (Javier-Astete

2021). The color was measured against spectrophotometry at wavelength 540 nm (Prasetyo 2010).

TSS was measured by the gravimetric method. The black liquor sample, 25 ml, was filtered using membrane filter 0.45 μm . The filtration system was equipped by a centralized vacuum system. The filtrate was dried to remove the moisture. Based on the result, the concentration of TSS was calculated (Kamarudin 2018).

TDS of solution was analyzed using TDS meter, Thermo Scientific, Eutech Con 150.

X-Ray diffraction (XRD) analysis. Sample was dried at dryer chamber at 60°C for overnight. XRD was conducted to observed the crystallinity and compare to α -cellulose. XRD analyzer was PANalytical type Emoyrean equipped by furnace TCU 2000 N.

Scanning electron microscope (SEM) analysis. Morphology of untreated and treated was scanned with SEM apparatus JEOL model JSM-6390 and equipped by Energy Dispersive X-Ray spectrometer (EDS) to analyze elements distribution.

RESULTS AND DISCUSSION

Raw material assessment

Survey on the site was done in order to assess its availability and to determine which the most suitable part. Tab. 2 informs components capacity and availability. EFB was considered as the most potential biomass since already collected at mills.

Tab. 2: Biomass potential of palm oil tree parts (Sudiyani 2019).

Part	Capacity (million tons)	Availability
Trunk	34	scattered on the plantation
Fronde	124	scattered on the plantation
Shell	8	scattered on the plantation
EFB	30	collected at mills

In case of EFB from Sulawesi Island had low quality because of high lignin content. This work was such focused-on delignification of the EFB. EFB and other parts of palm tree: frond, shell, and trunk, were analyzed for its component (Fig. 1a). Measurement of holocellulose was carried out by measuring cellulose as a source of glucose and hemicellulose as a source of xylose. By measuring total sugar with strong acid, 72% H₂SO₄, glucose and xylose can be analyzed quantitatively using DNS method. Lignin and holocellulose were listed among various parts of palm tree. In worst case, the biggest content of lignin was confirmed in EFB from Sulawesi Island reached 51.229%, excluding ash content.

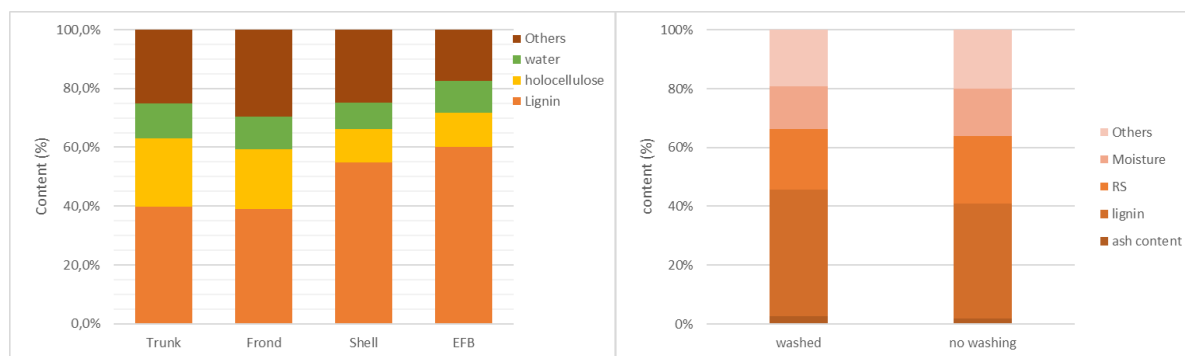


Fig. 1: Component analysis in palm oil tree: (a) comparison component in palm oil tree part, (b) component analysis washed EFB with original mechanical treatment of EFB.

Mechanical treatment EFB made some parts of the EFB are very fine as dust. Therefore, it was washed with tap water. EFB was not sieved since in the form of fiber. Both lignin in EFBs, directly after mechanical treatment and after washing, were rechecked for the component (Fig. 1b). Both EFB were almost the same, no lignin dissolved in water. This experiment was using EFB with length no more than 3 mm. Based on most component measurements of EFB, lignin content that used for base calculation in delignification efficiency was 43.089%.

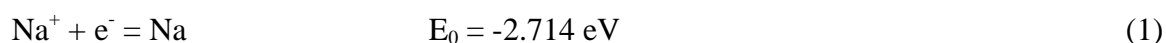
Delignification

Delignification in alkaline is the most common method. Previous research already established delignification method for EFB in 2,5 M NaOH and temperature up to 150°C (Sudiyani 2019). Unfortunately, this method used high concentration NaOH so that potentially caused environmental damage. In addition, the process in high temperature was consuming high energy. This work proposes environmentally friendly delignification that conducted at low concentration NaOH and irradiation microwave with H₂O₂ additional. H₂O₂ with microwave irradiation could generate effectively in OH[•] production, an oxidative agent (Chavoshani 2018, Miller 2013, Kim 2011). OH[•] has potential strength 2.86 eV (Muruganandham 2014).

EFB is categorized dielectric compounds that able to absorb microwave irradiation and heating up the solution (Hollertz 2013). Instead of fast heating, microwave irradiation, lignocellulose was also quickly cooled on removing heat effect caused by microwave irradiations.

Another preparation, power setting on microwave was tested between 100 and 300 W in water. The EFBs were irradiated and analyzed. Both samples had almost the same remain lignin 37.0 and 37.7, resp. Therefore, this work set the microwave power at minimum level 100 W. After microwave irradiation, temperature was measured directly. Each sample had temperature increment. This temperature increase was influenced by microwave duration. NaOH concentration did not affect the temperature rise (Fig. 2a).

Fig. 2b showed ORP that expressed in mV. This quantitative showed the tendency any chemical that acquire or lose electrons in black liquor. Existence of NaOH caused negative potential, according equation 1. Na⁺ ion needs an electron so that the potential was negative.



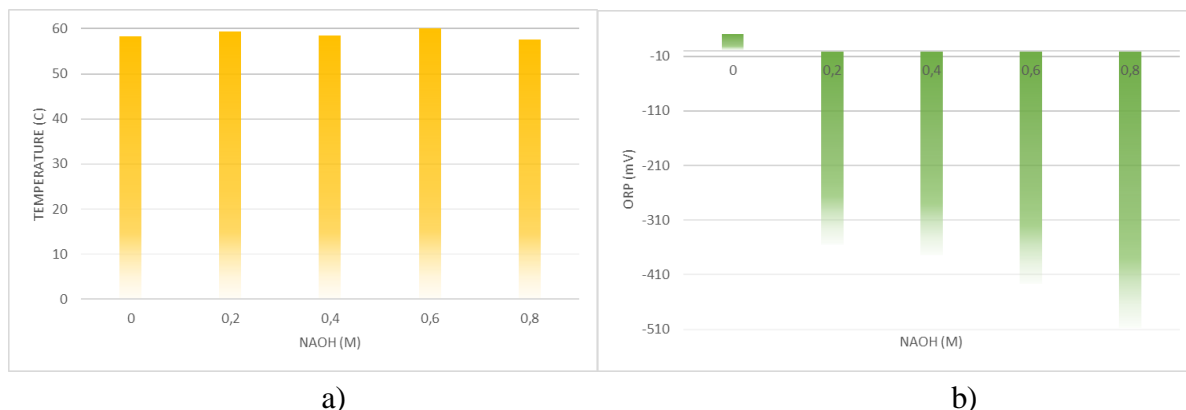


Fig. 2: Black liquor observation for: (a) temperature and (b) ORP.

The result of ORP showed potential redox of black liquor. NaOH solution had redox properties whereas Na^+ from NaOH would bind electron, act as an oxidator. However, during delignification, some alkaline element like Na and K from EFB dissolved in black liquor so that ORP measurement gave higher oxidation, more negative (Fig. 2b).

Additional H_2O_2 that converted to OH^\ominus caused some compound of EFB was dissolved and suspended more. The amount of suspended solid significantly affected by alkaline concentration (Fig. 3a) (Rossi 2005). TDS analysis increased according to NaOH concentration. However, TDS value was dominated with TDS original solution. As shown on Fig. 3b, the dissolved solid of EFB reached only 12%. Therefore, delignification was effective to suspend solid of EFB rather than the dissolved solid. TSS in NaOH solution usually consisted of organic and inorganic compounds. While the K, Na, and Ca were recognized as dissolved solid that detected as TDS. Regarding monomer of cellulose and hemicellulose could be estimated by glucose and xylose analysis, respectively. Both monomers were analyzed through RS measurement, DNS methods. Therefore, this work estimated holocellulose by RS measurement. RS analysis in black liquor was less than 0.8% that indicated holocellulose almost did not degraded during process of delignification. At low NaOH concentration, holocellulose that degraded less than 1% was negligible. However, the RS reached 1.2% at 0.8M NaOH because the chemical including oxidator could penetrated holocellulose and break it apart to glucose and xylose. Fig. 4 shows RS at 0,8 M NaOH was considered as high concentration.

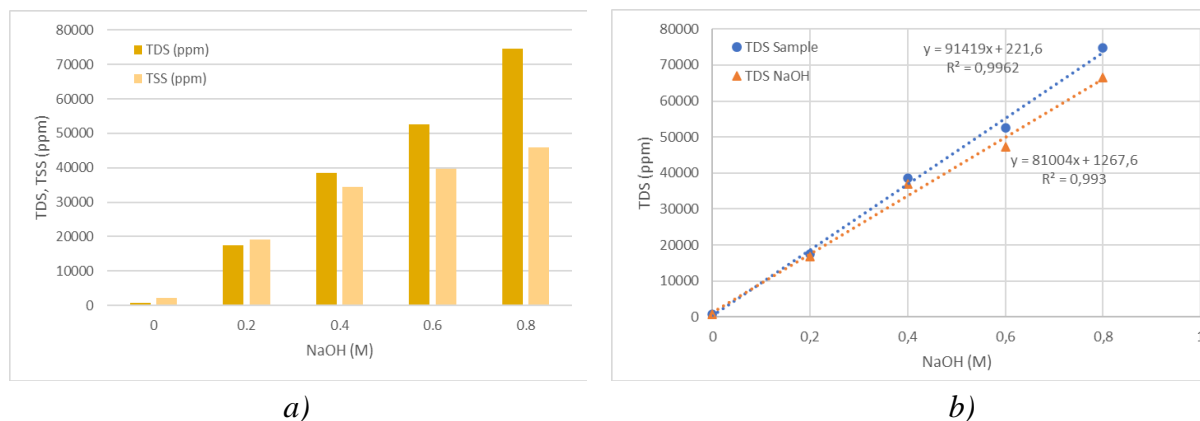


Fig. 3: TDS and TSS in NaOH solution: (a) after microwave processing, (b) TDS comparison with NaOH solution.

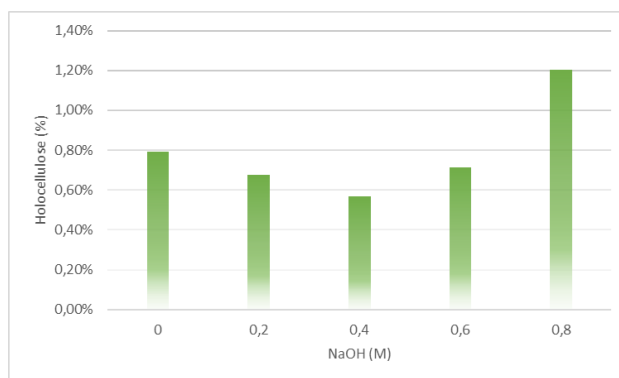


Fig. 4: Decomposition of holocellulose in black liquor.

EFB solid analysis

Treated EFB was analyzed for AIL, ASL, ash content and RS as presented in Fig. 5. Delignification effectiveness was influenced by temperature (Håkansdotter 2001). Treatment with microwave irradiation caused the solution including EFB heating up (Hollertz 2013) so that delignification should work more effective. Delignification with this treatment worked effective for both AIL and ASL (Fig. 5a). AIL removal for 0.2 and 0.4 M NaOH was almost the same, 25.767 and 25.132% respectively. While ASL removal reached 5.646 and 5.353%, respectively. This work reached 27% lignin removal. Unfortunately, if the NaOH was made more concentrated, the reactant could damage EFB interior as well. With the reduction of holocellulose, the percentage of lignin increased again. Any minerals or elements in EFB that measured as ash content. The ash content was not significantly reduced when microwave irradiation for EFB delignification in water, detected 2.33%. In NaOH solution, the ash content for most samples were less than 1%, a relatively small amount (Fig. 5b) (Shariff 2014). Metals or minerals formed in the delignification process are dissolved in black liquor and affect TDS measurements. When TDS got higher, this indicated the reduction of ash content in the EFB.

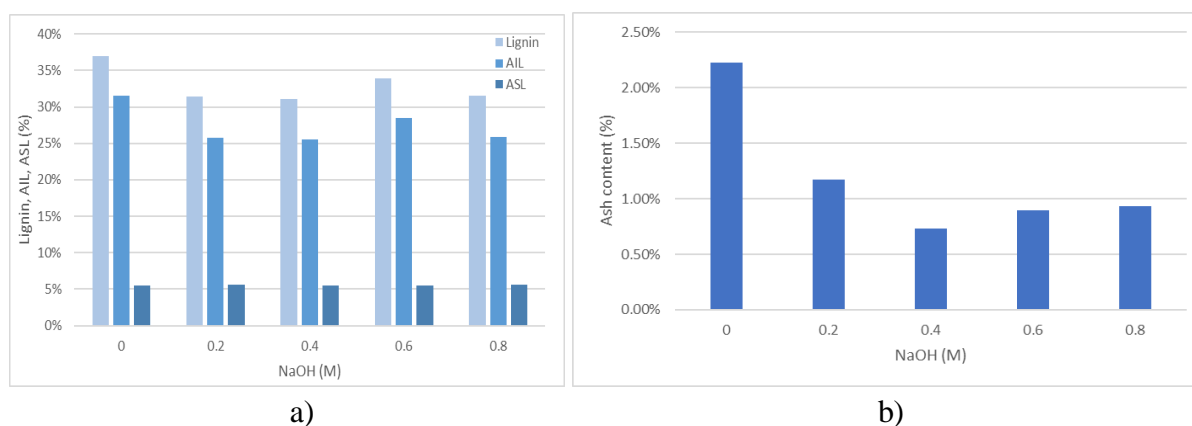


Fig. 5: Treated EFB analysis (a) lignin, (b) ash content.

In term of holocellulose in treated EFB, treatment in 0.2 M NaOH with OH⁰ using microwave irradiation increased holocellulose percentage to 26%, 6% higher than raw material (Fig. 6). Therefore, 0.2 M NaOH considered as optimal conditions as well as lignin removal that was also in 0.2 M NaOH.

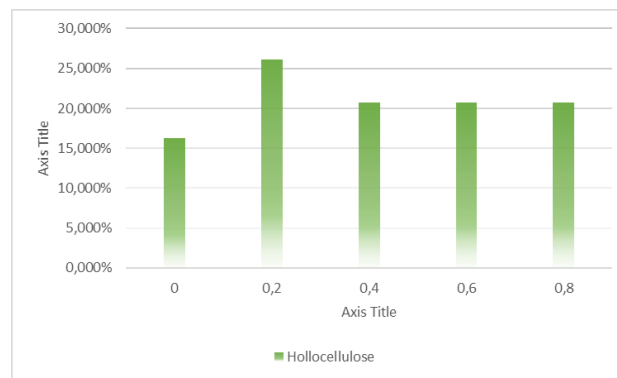


Fig. 6: Holocellulose content in EFB based on reducing sugar.

XRD Analysis

XRD analysis treated EFB was acquired for crystalline cellulose. Crystallographic plane of raw material, treated EFB and α -cellulose were calculated (Fig. 7). Based on the control, α -cellulose, the peak in some 2θ s was $14-16^\circ$, 22° and $34-35^\circ$ assigned (Park 2010). A broad peak in those places were observed and diffractogram was significantly different especially for raw material and treated EFB 21.1% and 23.3%, resp.

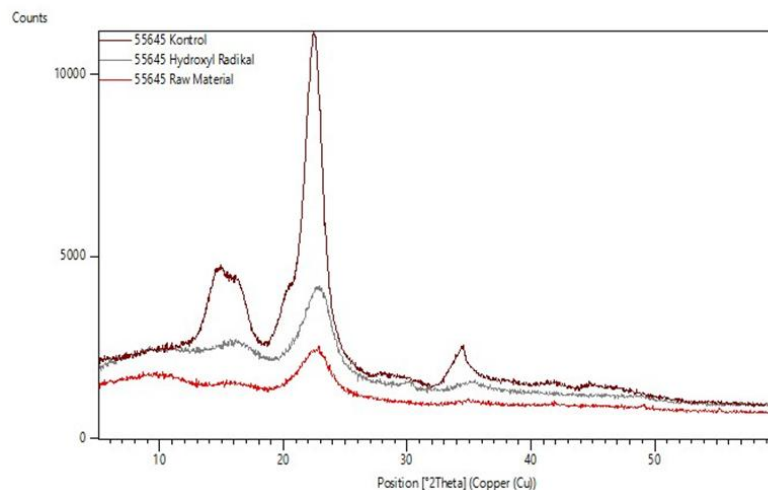


Fig. 7: XRD diffractogram raw material, treated EFB and α -cellulose.

Visual observation with SEM

Morphology SEM at Fig. 8 showed that treated EFB looked cleaner than raw material.

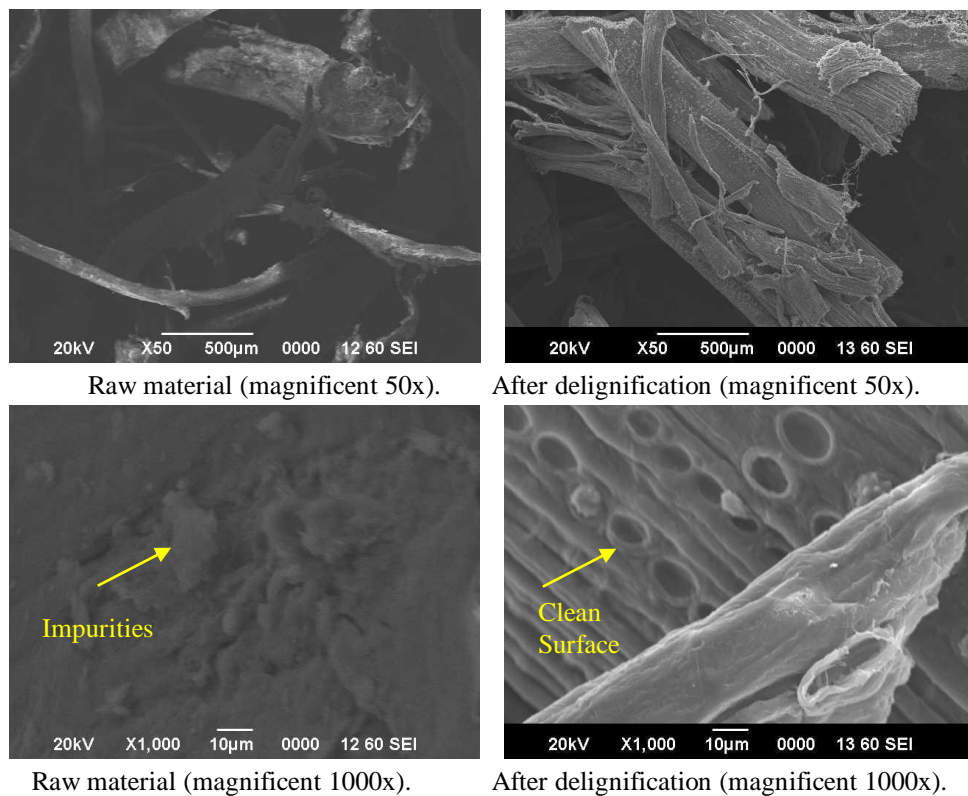
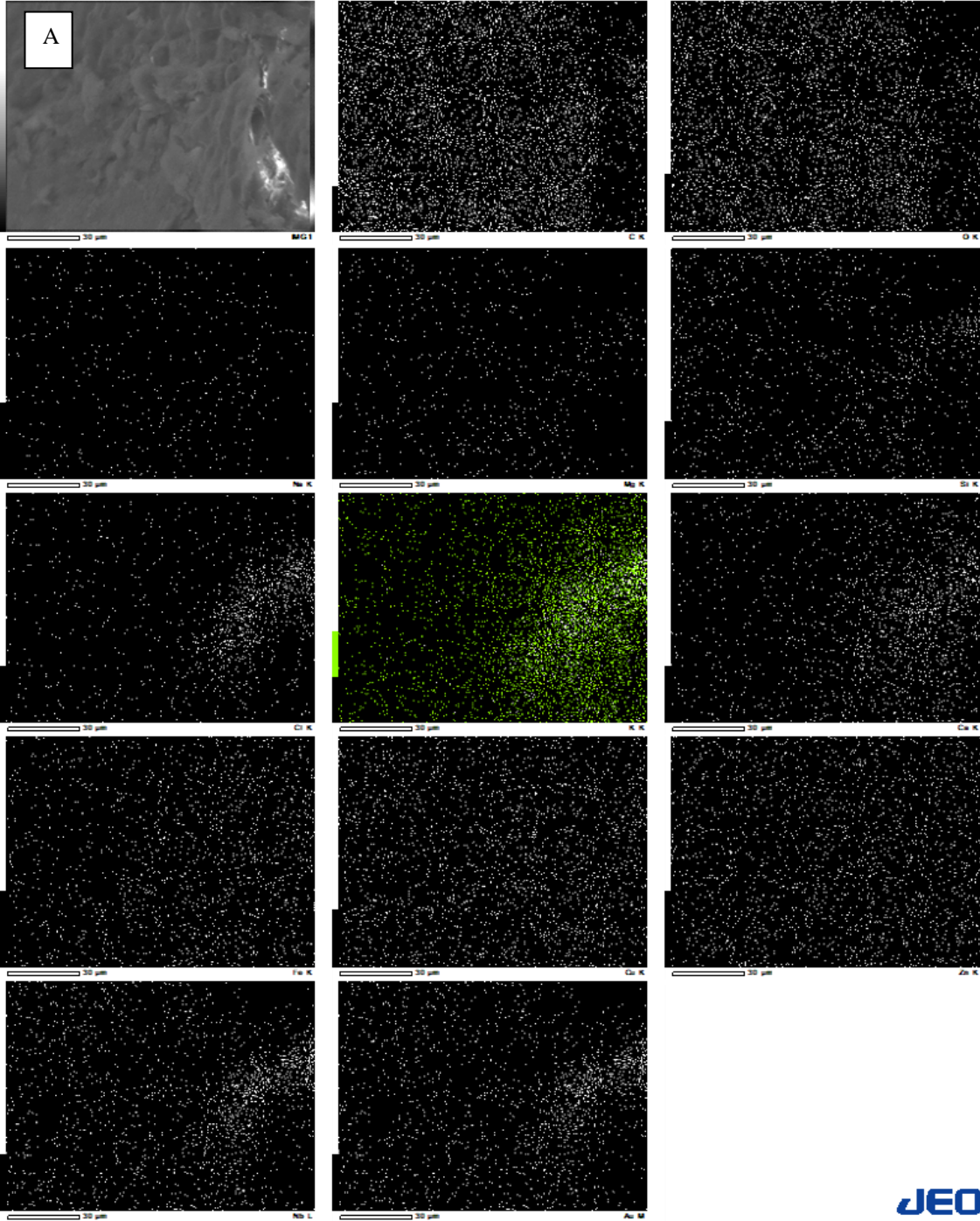


Fig. 8: Comparison of visual untreated EFB and treated EFB by SEM.

Clearer surface EFB fiber was got by 1000x magnificant. The images clarified the difference with some spheres appeared on the surface of treated EFB. Spheres on EFB surface indicated eroded during the delignification.

Supporting the visualization of SEM, the elements distribution of untreated EFB was only potassium (K) detected. Most surface of untreated EFB covered by impurities. Element K was predicted from fertilizer. While on the surface of treated EFB showed some elements detected Ca, Nb, Si, and Mg (Fig. 9) (Madhiyanon 2013). Sequence elements indicated the number of elements. Ag indicated as the coating agent. Na was the remaining solvent.



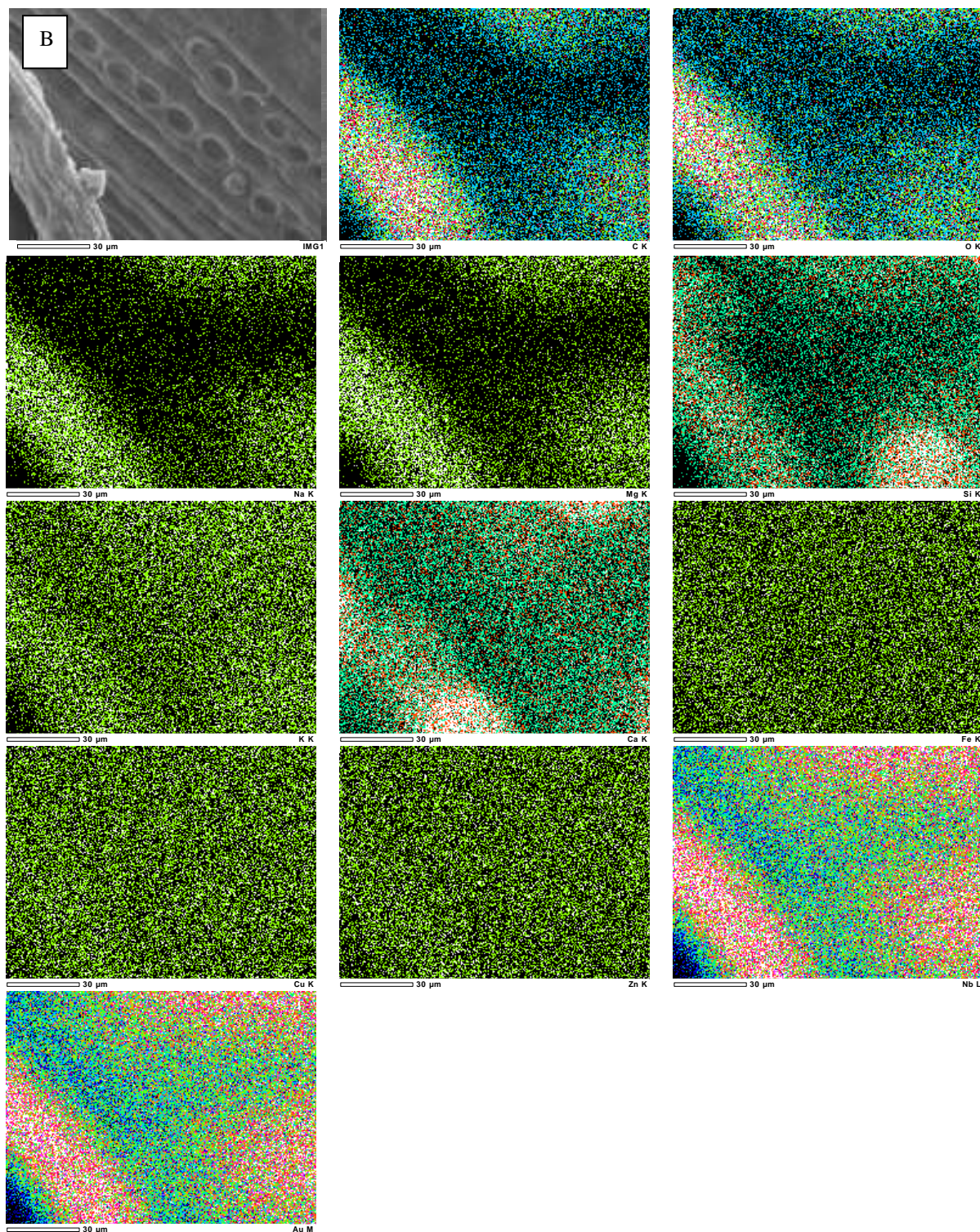


Fig. 9: Elements mapping on the surface of (a) raw material EFB, (b) treated EFB.

Yield calculation

The yield was calculated based on treated EFB that already dried in an oven at 60°C for overnight:

$$\text{Yield (\%)} = \text{Yield (\%)} = \frac{m_{\text{treated EFB}}}{m_{\text{EFB}}} \cdot 100\% \quad (2)$$

As shown on Fig. 10, some part of treated EFB was degraded and suspended in black liquor, and other parts were sludgy. Unfortunately, those parts were lost during washing.

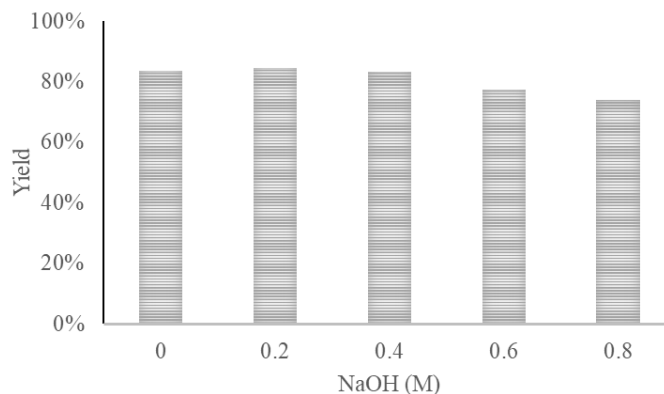


Fig. 10: Yield of treated EFB.

Therefore, the amount EFB got less. In overall, EFB parts were lost more at higher NaOH concentration so that the yield decreased. Satisfyingly, the overall yield was still more than 74% and considered as tolerable lost.

CONCLUSIONS

EFB is classified as dielectric compound so that microwave irradiation treatment was considered a suitable method for delignification because EFB would absorb the heat of microwave irradiation. Delignification effectiveness could be seen from temperature increment and suspended solid. Lignin was the target to be removed while keeping the holocellulose from breaking apart. Both requirements were achieved with involving OH^\ominus , NaOH solution and microwave irradiation. 0.2 M NaOH considered as an effective lignin removal with high holocellulose achieved at 27% and 26%, respectively. Unfortunately, higher NaOH concentration caused the decomposition was not only attack lignin but also holocellulose. Therefore, EFB yield would decrease by increasing NaOH concentration. Morphology EFB with SEM visualized the surface treated EFB as effect of delignification treatment. Analysis by XRD was also confirmed the morphology that this delignification improved crystallinity by 17.7% closer to the control, α -cellulose. Elements Distribution was also detected on the surface of treated EFB that dominated by was Ca, Nb, Si, and Mg.

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