Recovery of Palm Oil and Valuable Material from Oil Palm Empty Fruit Bunch by Sub-critical Water

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Abstract: Oil palm empty fruit bunch (EFB) is one of the solid wastes produced in huge volume by palm oil mill. Whilst it still contains valuable oil, approximately 22.6 million tons is generated annually and treated as solid waste. In this work, sub-critical water (sub-cw) was used to extract oil, sugar and tar from spikelet of EFB. The spikelet was treated with sub-cw between 180-280°C and a reaction time of 2 and 5 minutes. The highest yield of oil was 0.075 g-oil/g-dry EFB, obtained at 240°C and reaction time of 5 minutes. Astonishingly, oil that was extracted through this method was 84.5% of that obtained through Soxhlet method using hexane. Yield of oil extracted was strongly affected by the reaction temperature and time. Higher reaction temperature induces the dielectric constant of water towards the non-polar properties of solvent; thus increases the oil extraction capability. Meanwhile, the highest yield of sugar was 0.20 g-sugar/g-dry EFB obtained at 220°C. At this temperature, the ion product of water is high enough to enable maximum sub-critical water hydrolysis reaction. This study showed that oil and other valuable material can be recovered using water at sub-critical condition, and most attractive without the use of harmful organic solvent.

Key words: oil recovery, sub-critical water, oil palm empty fruit bunch, Soxhlet

1 Introduction

Palm oil mill produces many solid wastes such as empty fruit bunch (EFB), palm pressed fiber, trunks and fronds. Approximately 22.6 million tons/year of EFB is generated and the amount is 23% from processed bunches. To utilize these wastes, EFB are usually incinerated to produce energy¹⁾, while some are used for mulching. However, a study shows that EFB contains large amount of residual oil and nutrients that is worth recovered. EFB is known as lignocellulosic waste produced during oil extraction in palm oil mill²⁾ and is available as substrates in cellulose production¹⁾. However, the problem with the process is the difficulty of getting high yield of reducing sugar as the end product due to the complex structure of lignocellulosic materials³⁾. Mainly, EFB consists of cellulose, hemicellulose and lignin which decompose and form valuable component under certain condition. Md Yunos *et al.*⁴⁾ reported that the composition of EFB is comprised of approximately 20% cellulose, 23.9-25.1% hemicellulose and 23.5% lignin and about 7.4% oil. Despite the small amount of oil, it can be very significant if the oil can be extracted out from EFB. Residual oil, sugar and alternative heavy oil could be ex-

tracted out using various methods like Soxhlet extraction⁵⁾, pyrolysis⁶⁻⁸⁾, hydrolysis^{3, 9-11)}, supercritical liquefaction¹²⁾ of sample and others. However, some of the methods require organic solvent. Organic solvent such as hexane is not suitable for food processing and furthermore handling organic waste is costly. Thus, search for alternative environmental friendly solvent and process is needed for processing EFB.

Sub-cw is defined as liquid water that lies at temperature between its atmospheric boiling point and less than its critical temperature ($T = 374^{\circ}\text{C}$)¹³⁾. The region for sub-cw includes any region above critical pressure but lies between boiling and critical temperature. For sub-cw treatment, it is known that pressure have not much effect on the reaction and usually high enough to keep water in liquid state¹³⁾. Sub-critical water is able to extract oil, hydrolyze cellulose and hemicellulose into sugar and pyrolyse lignin into tar¹⁴⁾. The altered physicochemical properties of water at certain temperature allow this process to occur.

Water at room temperature has a dielectric constant of 78.5¹⁵⁾. Interestingly, the dielectric constant of water becomes 29 at 250°C, close to dielectric constant of ethanol

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at room temperature ($\epsilon = 27$). Ethanol is a good solvent for extraction, thus by having dielectric constant similar to the ethanol, water will behave like ethanol. Secondly, as the temperature and pressure increases, the ion production of water also increases 1000 times more at 250°C^{14, 16, 17}) than at room temperature. Hydrolysis reaction is strong when ion production is at the highest. Owing to the excellent physicochemical characteristics of sub-cw, it is expected that residual oil can be extracted from EFB, thus increase the extraction efficiency in mill operation. At the same time, valuable substances such as sugar and nutrients¹⁷) can be obtained from hydrolysis of EFB.

The aim of this work is to study the possibility of using sub-cw to recover oil and other valuable materials from EFB. The optimum temperature for the recovery was investigated and evaluated by comparison to other conventional methods.

2 Materials and Experimental Procedure

2.1 Preparation of raw material

Fresh wet EFB used in this research was taken from Sri Langat Mill in Dengkil. The samples were cut into pieces of 5-10 mm long. The heating bath contains molten salts from sodium nitrate (Industrial grade), and potassium nitrate (Industrial grade) with ratio of 1:1. The reagents involved in this treatment were sulphuric acid, hexane, phenol solution and acetone where all were analytical grade. Meanwhile, ultra-pure water was prepared in house by Milli-Q with purity of <0.1 µS.

2.2 Drying of sample

Wet EFB which have been cut 5-10 mm long were weighed (~ 5 g) and put into beaker and were then dried at 80°C and were weighed again after 24 h until the weight become constant. The moisture content (MC) was calculated using equation (1).

$$\text{MC (Dry basis)} = \frac{\text{Initial weight sample} - \text{final weight sample}}{\text{Initial weight sample}} \times 100 \quad (1)$$

2.3 Sub-cw reaction

A batch laboratory-scale sub-critical fluid extraction system was used for this study. The reactor was a stainless steel batch reactor (SUS316, i.d 7.5 mm × 150.4 mm) capped with Swagelok fittings. Molten salt bath (Celcius 600H, Tomasu Kagaku) was preheated up to a temperature between 180°C to 360°C for a few minutes. Total volume of the reactor was 7.3 cm³. 1 g of crushed EFB were inserted into the reactor and later filled with 5 mL Milli-Q water. Then air was drawn out by purging argon gas. Reactor was tighten and was weighed again to obtain the actual amount

of water inserted. The reactor was then placed into the preheated salt bath to initiate the reaction. The reaction time was either 2 or 5 minutes with continuous shaking. The pressure of the reaction was estimated using the steam table. By this method, the heating-up rate achieves a steady state condition in less than 25 sec¹⁴). After reaching the reaction time, the reactor was quickly quenched in flowing tap water at room temperature to terminate the reaction. The reactor content was transferred into test tube and photographs were taken before centrifuged at 4000 rpm for 5 min to obtain oil, aqueous and solid phases.

Hexane was added into the reactor to recover oil which sticks on the reactor wall and then poured into the test tube. The test tube was shaken gently and then the hexane-soluble component was removed into sample bottle. This was repeated until the color of hexane layer obtained was colorless. Then, hexane soluble product was filtered before dried in the fume hood for at least two days. The yield of oil extracted was calculated by equation (2).

$$\begin{aligned} &\text{Yield of oil extracted (g-oil/g-dry EFB)} \\ &= \frac{\text{Weight of oil extracted (g - oil)}}{\text{Weight of charged EFB (g - dry EFB)}} \quad (2) \end{aligned}$$

The water-soluble components were recovered using distilled water. The water and solid phase remained in the test tube was centrifuged and the aqueous phase was transferred to another sample bottle. Appropriate amounts of distilled water were added to the reactors and vigorously shaken to clean the inner wall of the reactor and then transferred into the test tube. These procedures were repeated until the aqueous phase becomes colorless. The content in the test tube was mixed thoroughly before centrifuged. After the reactor was clean, an appropriate amount of distilled water was added into the test tube in which only solid phase remains. The contents in the test tube were mixed thoroughly using a tube mixer, centrifuged and transferred to the same sample bottle. These steps were repeated until the aqueous phase become colorless. The final aqueous phase was filtered using membrane filter (Mixed Cellulose Ester 0.45 µm Hydrophilic, Toyo Roshi Kaisha Ltd, Japan). Lastly, the filtered aqueous phase was diluted to 50 ml by distilled water. The aqueous phase was then kept in a refrigerator before analyzed.

Water-insoluble tar was recovered by acetone. After the reactor and test tube were dried thoroughly in dryer at 60°C, a suitable amount of acetone was added into the reactor and was shaken vigorously. The content was transferred into the test tube. This was repeated until acetone become colorless. The test tube was well mixed before centrifuged. The liquid phase recovered was dried in air. The acetone extracts was obtained after the acetone was totally vaporized.

Free fatty acids in the oil phase were measured using

GC-FID (Shimadzu GC14B) equipped with ZB-1 column (30 m length, 0.25 mm diameter and 0.25 μ m thickness) after methyl esterification. pH of the water phase was measured using pH meter (Horiba Ltd). Total sugar was measured using the phenol-sulphuric acid method. 0.60 mL for each standard solution or aqueous phase of sample was put into different test tube. Then, 0.60 mL of 5 wt% phenol solution was added into the test tube and well mixed. 3 mL of concentrated sulphuric acid was added into the test tube and was then left to cool for 20-30 min. The absorbance for each standard solution was measured using ultraviolet-visible absorption spectrometer (Shimadzu, UV-1600) at wavelength 490 nm. The absorbance values were recorded and calibration curve were constructed. Aqueous phase was also tested using TOC Analyzer. Potassium hydrogen phthalate and a mixture of sodium hydrogen carbonate, NaHCO_3 with sodium carbonate were used as standards for total carbon and inorganic carbon respectively.

Yield of oil obtained using Soxhlet extraction method was set as benchmark for comparison with extraction by sub-cw. 8 g of EFB was inserted into the thimble and put into the distillation path equipped with a condenser. The oil extracted by hexane in the round-bottomed flask was dried until completely dry. Then, the round-bottomed flask was then weighed again to obtain the weight of oil extract-

ed.

3 Results and Discussion

3.1 Moisture Content

The average moisture content of the EFB obtained was 47.4%. It was lower than that recorded by common palm oil processing company (70%). The duration of fresh fruit bunch (FFB) exposed to air and steam in the sterilizer and also the handling method of FFB might have contributed to the difference in moisture content. The average moisture content was used to calculate the actual dry weight of sample charged during reaction. Thus, all results were based on the same weight basis.

3.2 Sample after sub-critical water treatment

Figure 1 shows the photos of centrifuged sample products after sub-critical water treatment for 5 minutes carried out at various temperatures. Three phases consisting of oil layer, water and solid phase were observed. The water layer was a clear yellowish-orange color at all temperature except at 220°C. The clear brown color was observed when treatment was performed at 220°C, probably caused by the degradation of hemicellulose and cellulose

Fig. 1 Photos of centrifuged sample after sub-critical water treatment for 5 minutes.

Table 1 Average and standard deviation of all products at various reaction temperatures and yield of oil obtained from various methods.

Method	Yield of oil		Yield of sugar		Yield of tar	
	%	σ	%	σ	%	σ
Soxhlet*	8.9					
Treatment and pressing ²¹⁾	1.5					
Sub-cw (180°C)*	1.7	0.001	1.6	0.050	5.4	0.008
Sub-cw (200°C)*	5.4	0.026	8.2	0.033	3.4	0.003
Sub-cw (220°C)*	5.6	0.022	19.6	0.040	2.0	0.028
Sub-cw (240°C)*	7.5	0.015	9.6	0.033	12.9	0.005
Sub-cw (260°C)*	3.0	0.002	5.3	0.008	17.3	0.018
Sub-cw (280°C)*	2.4	0.003	4.0	0.004	18.5	0.047

* This study

into simple sugar and furfural. Meanwhile, solid residue decreased as reaction temperature increases. It changed to powder form when reaction temperature was higher than 250°C.

3.3 Yield of product (solid residue, oil, total organic carbon, tar and sugar)

Table 1 shows the average and standard deviation of all products at selected reaction temperatures. All product have standard deviation of less than 0.1, thus the results obtained were consistent and valid. Figure 2 depicts the yield of product formed after sub-critical water treatment. The solid residue shows a decreasing trend as reaction temperature increases. Yield of remaining solid residue was 0.74 g-solid residue/g-dry EFB at 180°C. Only 26% of EFB decomposed to water soluble compound during the sub-critical water treatment. Meanwhile, the lowest yield of solid residue was 0.33 g-solid residue/g-dry EFB that was obtained at 260°C. This is caused by the degradation of EFB especially when the temperature was above 250°C¹⁴⁾. Sukiran *et al.* reported that pyrolysis reaction occurs at this temperature⁸⁾, which might significantly cause the degradation of the hemicellulose, cellulose, and lignin in the EFB to smaller compounds. Thus, from the present study, it is shown that waste EFB could be reduced to more than 60% by treatment using sub-critical water.

Figure 2 also shows the yield of oil obtained at various reaction temperatures. Yield of oil increased from 180°C until it reached the highest at 240°C (0.075 g-oil/g-dry EFB). However, it decreased to 0.024 g-oil/g-dry EFB at 280°C. Yield of oil obtained at this temperature was one-third of the highest oil yield. Highest yield of oil was ob-

Fig. 2 Effect of temperature on the yield of oil, total organic carbon, alternative heavy oil, and solid residue (reaction time: 5 minutes). (□) Oil, (*) Toc, (▲) Tar, and (▽) Solid residue.

tained at 240°C probably because of the low dielectric constant of water at this temperature to allow maximum oil extraction to occur. However, decreasing trend was observed when temperature was higher than 250°C probably due to further degradation of the extracted product as reported by Kronholm *et al.*¹³⁾.

From Fig. 2 also, the yield of water insoluble tar maintained between 0.021 to 0.054 g-tar/g-dry EFB from 180°C to 220°C. Then it increased a little from 0.13 to 0.14 g-tar/g-dry EFB with increasing in temperature up to 250°C. Yield of tar increased to 0.19 g-tar/g-dry EFB when temperature reached 280°C. The increment was probably caused by the decomposition of lignin by pyrolysis, especially at highest temperature where lignin degraded into acetone soluble product. Research carried out by Salema and Ani¹⁸⁾ found out that tar from EFB contain 2-methoxy-phenol, 2,6-dimethoxy-phenol, 2-methoxy-5-methyl-phenol, 4-ethyl-2-methoxy-phenol and *p*-cresol. Meanwhile, yield of TOC increased with temperature and became constant between 0.090-0.11 g-TOC/g-dry EFB at temperature above 220°C. TOC detected was from the glycerol, sugar and probably some organic acid contained in the water phase after treatment with sub-cw. This is because hydrolysis of hemicellulose and cellulose may produce simple sugar^{12, 19)}, furfural¹⁹⁾ and organic acid such as formic acid²⁰⁾.

Analysis by GC-FID demonstrated that oil produced from sub-cw treatment contained FFA between 3-12%. Increased in temperature might have contributed to the increment in the FFA formation due to hydrolysis of triglyceride. Thus, the best quality oil obtained with lowest FFA content was one treated at 220°C, 0.056 g-oil/g-dry EFB.

Figure 3 shows the relationship between dielectric constant of water at various temperatures with the yield of oil. The yield of oil obtained was inversely proportional to the dielectric constant of water below 250°C. Correlation coef-

Fig. 3 Effect of dielectric constant of water (---) on the yield of oil (■).

ficient calculated from this relationship between oil yield and dielectric constant was 94%. This shows that below 250°C, there is somehow a relation between dielectric constant and yield of oil. However, this relationship is not valid at higher temperatures probably because EFB may be physically altered and thus reducing the extraction recovery¹³⁾. Kronholm *et al.*¹³⁾ also reported that many compounds from natural origin like plant, leaves, and root, can easily decompose at low temperature. This might explain the reason why the trend only partially showed the relation between yield of oil extracted and dielectric constant of water at temperatures below 250°C.

pH of water phase was in between 3 and 4 for all temperature. The low pH is probably caused by the effect of temperature on the increasing formation of hydrogen ion of water¹⁴⁾. Besides that, there is probability that it was also affected by the formation of organic acid due to further degradation of simple sugar and furfural²⁰⁾.

Figure 4 shows the effect of temperature on the yield of sugar and tar at reaction time 5 minutes. Yield of sugar increased drastically from 0.016 to 0.20 g-sugar/g-dry EFB and dropped above 220°C. The drastic increase below 220°C is because hydrolysis is favored at low temperature due to increases in ionic product value by three magnitudes higher than at room temperature. The acidic state of water at high temperature¹⁴⁾ might also contributed to the hydrolysis power of water. Besides that, Mazaheri *et al.*¹²⁾ also stated that the glycosidic bond of cellulose and hemicellulose is polar and therefore hydrolyzed very fast in water at subcritical condition. Through hydrolysis, hemicellulose and cellulose were probably broken down into cellobiose and it further hydrolyzed into xylose and glucose. Other than sugar, furfural and its derivative might also be produced¹⁹⁾. On the other hand, yield of tar showed no increment at low reaction temperature. At 220°C, yield

Fig. 4 Effect of temperature on the yield of (◆) sugar and (▲) tar (Reaction time: 5 minutes).

of tar increased rapidly indicating the high activity of pyrolysis. Highest yield of tar was at 280°C (0.19 g-tar/g-dry EFB). This could be because lignin is the most resistant towards hydrolysis followed by cellulose and hemicellulose³⁾, therefore more energy is required to break lignin. This explains the sequence of hydrolysis and pyrolysis reactions when treatment with sub-critical water evidenced from the opposite trend for yield of sugar and tar.

Figure 5 shows the effect of ion production constant on the yield of sugar at various temperatures. The yield of sugar increased as the ion product constant increases. An outstanding 92% correlation coefficient was obtained, which showed an excellent relationship between the yield of sugar and ionic product. However, when treatment was carried out above 240°C, correlation obtained was very low, probably because hydrolysis was no longer favored.

Figure 6 shows the effect of reaction time on the yield of oil extracted. The reactions were carried out for 2 and 5 min. Maximum yield of oil obtained was at 240°C for reaction time 2 min (0.032 g-oil/g-dry EFB) and 5 min (0.075 g-oil/g-dry EFB). Yield of oil extracted at reaction time 5 min were twice higher than in 2 min at all temperature except 260°C. The differences in oil extracted due to reaction time were possibly caused by the different amount of disrupted cell containing oil in the fiber. Longer reaction time allows more cells to be disrupted. Thus, more oil is easily extracted by the water at sub-critical condition.

3.4 Soxhlet extraction

Table 1 shows the yield of oil obtained from various methods. The highest yield of oil obtained was 0.089 g-oil/g-dry EFB using Soxhlet method from this work. On the other hand, oil obtained from treatment and pressing was only 1.5% as reported by Jorgensen²¹⁾. Meanwhile from Fig. 6, the highest yield of oil obtained using sub-cw was at reaction temperature and time of 240°C and 5 min which

Fig. 5 Effect of ion production constant on the yield of sugar. (◆) Yield of sugar and (×) ion production of water.

Fig. 6 Effect of reaction time on the yield of oil extracted.

Fig. 7 Efficiency of sub-critical water at various temperatures (5 min) when compared to soxhlet extraction.

was 0.075 g-oil/g-dry EFB. Surprisingly, this result is very close to that obtained by Soxhlet method. There is possibility that the slight difference were caused by the quality of the EFB used. Studies have shown that the oil extraction rate (OER) from the fresh fruit bunch is affected by several factors such as ripeness, age of palm tree, type of soil and others²²⁾.

By using the yield of oil obtained from Soxhlet extraction method as reference, the efficiency of sub-critical water extraction was measured as shown in Fig. 7. The efficiency of sub-critical water achieved in this experiment was 84.5% of that from Soxhlet method. Consequently, this study showed that sub-critical water can be used to extract oil from EFB and can compete with other standard method using organic solvent.

4 Conclusions

Sub-cw at certain temperature allowed extraction, hydrolysis and pyrolysis reaction to occur. Highest residual oil can be extracted at temperature 240°C, while highest yield of sugar and tar were at 220°C and 280°C respectively. In addition, the solid residue was reduced to 67% just by using sub-critical water. This showed that EFB still contain a lot of valuable material which are worth recovered and achievable using only water at its sub-critical state. On top of that, environmental problem related to palm waste could also be reduced.

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