GREEN SOLVENT ENHANCED MECHANICAL EXTRACTION OF PALM KERNEL OIL

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ABSTRACT
Even though the mechanical extraction process offers a simple and environmentally friendly process, the recovery of oil is relatively low. Thermal pretreating the oilseed increases the oil yield but produces unwanted oil color. A new method that combines grinding and extraction using green solvents was developed to extract palm kernel oil. The performance of six different green solvents such as water, ethanol, isopropyl alcohol, dimethyl carbonate, ethyl acetate, and d-limonene in the extraction of palm kernel oil was determined using a controllable blender extractor (CBE), new extraction equipment modified from a household blender appliance. Furthermore, ethyl acetate, which produced the maximum oil yield, was used to study the effect of the operating parameters of the CBE. The oil yield of 34.2% was obtained in the extraction condition of the ratio of palm kernel to ethyl acetate of 1:7, the rotational speed of 5000 rpm, and 10 minutes extraction time. Compared with other green extraction methods, the CBE-intensified palm kernel oil extraction could save >70% energy consumption. In terms of extraction time, the CBE-intensified could extract palm kernel oil faster than existing extraction methods.

Keywords: Green Solvents, Ethyl Acetate, Extraction, Palm Kernel.

INTRODUCTION
Vegetable oil is currently produced either by solvent extraction or mechanical press of oil-bearing seeds.1 Due to the simple process and high extraction yield with an acceptable scent, solvent extraction, particularly using hexane, is industrially preferable.2 However, hexane is toxic and flammable, which raises health and safety concerns.1,3 In addition, the solvent extraction method requires thermal conditions for evaporation and consumes an excessive volume of hexane.4 Therefore, green extraction processes have been developed to accommodate those issues. The supercritical fluid utilizes carbon dioxide or solvents, particularly in its critical condition to disrupt seed cell walls, releasing oil to the environment, and mechanical disruption, i.e., Screw press, which applies a non-inherent force to the seeds are the common green extraction process.5,6 Even though a high oil yield is extracted using the supercritical extraction method, a special reactor that can hold a high pressure is required for successful extraction, which, together with operating costs such as electricity, specialized operator, and periodic maintenance, limits its industrial application.3 Mechanical processes offer a simple and environmentally friendly extraction method. This extraction method is preferred in rural areas, as it requires low-cost investment and semi-trained persons to operate.7,8 In addition, the by-product contains no toxic organic residue and can be used directly for other purposes, particularly for animal feed uses.9 However, the oil yield obtained using this method is quite low and is preferably applied to oleaginous oilseed/ biomass.7,9,10 Many researchers observed that 10–20% of residual oil remains in various oleaginous seeds after a mechanical continuous extraction process.2,8,11 In addition, this method cannot be applied to microalgae biomass as the cell walls are too small and rigid.12 Modification of the mechanical extraction machine combined with heating has proved to increase the extraction efficiency.11 Subroto et al.13 observed an increase of 28.6% in oil recovery when increasing the temperature to 105°C. However, darker oil color is obtained due to the denaturation of protein in the Jatropha kernel, lowering the oil viscosity. It was found that preheating
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the oilseed can also decrease the residual oil.\textsuperscript{11,14} Pre-treating palm fruit with microwave irradiation for 3 min increased the oil yield from 7\% to 19\%. Even though oil recovery was increased with the increased irradiation time, black-colored oil was produced.\textsuperscript{15} Recently, as environmental and health risk concerns increase, the utilization of green solvents in vegetable oil extraction has been developed.\textsuperscript{3,16-19} Green solvent was used combined with other cell wall disruption methods such as supercritical, pressurized liquid extraction, ultrasound, microwave, or maceration.\textsuperscript{3,17} The combination of mechanical press followed by the supercritical carbon dioxide process to extract the residual oil showed increasing extraction efficiency.\textsuperscript{9,20} Furthermore, similar oil yields could be extracted from \textit{Echium plantagineum} L seeds using pressurized ethanol running at 150°C for 10 min and the Soxhlet-hexane extraction method conducted for 480 min.\textsuperscript{17} In addition, some bioactive compounds were extracted using green solvent-producing oil with good oxidation stability.\textsuperscript{16} However, seed grinding processes are required to reduce the seed size and to increase the contact surface area of the cell walls. Some oil detected sticks to the grinding machine, which reduces the recovery yield. Therefore, this study aims to simultaneously grind and extract palm kernel oil using green solvents as the extracting agent. The home appliance capsule blender design was adopted and equipped with a controlled motor. Furthermore, the best performance solvent was used to study the effect of the ratio of palm kernel to solvent, reaction time, and the CBE rotation speed. Finally, the energy consumption of the CBE was calculated and compared with other extraction methods.

EXPERIMENTAL

Materials

Palm kernel was collected from a local palm oil industry in Medan, Sumatera Utara – Indonesia, and stored in a cold room before use. All green solvents used in this study were purchased from local chemical stores and were used without any treatment.

Conventional Extraction of Palm Kernel

The Soxhlet extraction method was used to extract palm kernel oil and the oil yield was used as a benchmark to calculate the extraction efficiency of the intensified CBE. The procedures for Soxhlet extraction followed the published method described in the previous studies.\textsuperscript{21-23} About 20 g of palm kernels were ground to a homogeneous powder using a coffee grinder (Klaz Cg9100) set to fine (particle size <200 μm) and were placed in a thimble which was connected to a Soxhlet apparatus and extracted using hexane under reflux conditions for 30 min. The oil yield was calculated after solvent evaporation and stored in a desiccator for analysis.

The CBE Intensified Palm Kernel Oil Extraction

The CBE has a maximum speed of 16,000 rpm and 2 blades to chop up the seed. The oil yield, free fatty acid (FFA) content, and lipid profile in the form of monoglyceride (MG), diglyceride (DG), and triglyceride (TG) were the outcomes of the CBE intensified palm kernel oil extraction operated at ambient temperature and pressure. The achievements as solvent extractors of green solvents such as water, ethanol, isopropyl alcohol, ethyl acetate, dimethyl carbonate, and d-limonene were evaluated. Six different green solvents were added to the palm kernels with a ratio of 1:3 (w/v) in the CBE (Fig.-1). The extraction was conducted for 10 min with a rotational speed of 5000 rpm at ambient temperature and pressure. The performance of hexane, a non-polar organic solvent, was also studied as a comparison. The oils and solvents were separated from the palm kernel cake using centrifugation working at 8000 rpm for 5 minutes. The green solvent was evaporated and the oils were stored in a desiccator until analysis. The acid value was determined using the standard titration method following the ASTM D664 procedure. The extraction efficiency was calculated based on the comparison of the CBE-intensified oil yield with Soxhlet and Screw press oil yield.

\[
\text{Yield}_{\text{CBE}} = \frac{W_{\text{CBE}}}{W_{\text{palm kernel}}} \times 100\% \quad (1)
\]

\[
\text{Extraction Efficiency (\%)} = \frac{\text{Yield}_{\text{Soxh}} \text{ or screw press}}{\text{Yield}_{\text{CBE}}} \times 100\% \quad (2)
\]
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Where, $W_{CBE}$ is the weight of CBE-intensified oil extracted and $W_{palm\ kernel}$ is the weight of palm kernel used.

**Fig.-1:** Photographic Image of CBE

**Fatty Acid Profile and Lipid Content Analysis**
The fatty acid profile and lipid content in the form of MG, DG, and TG were determined following previously published protocol\textsuperscript{24,25} using a gas chromatograph spectrometer (GC Shimadzu type 2010) equipped with a capillary column (DB 23 SN 9980937) with a length of 30 m, the film thickness of 0.25 $\mu$m and ID 0.25 mm. The column oven was set to an initial temperature of 90$^\circ$C with a hold time of 5.0 min and a rate of 7$^\circ$C/min. A 1.0$\mu$m of the sample was injected into the injection port in split injection mode with the injection port temperature was set to 260$^\circ$C using helium as carrier gas with a column and total flow of 1.04 and 54.1 ml/min, respectively. The flame ionization detector which is set to a temperature of 260$^\circ$C at a sampling rate of 40 msec and a stop time of 26.86 min was used. A lipid standard, mono-, di- and triglyceride mixture (Supelco) were used for identification and quantification.

**Statistical Analysis**
The data from all experiments are expressed as mean ±SD and were subjected to one-way ANOVA and Tukey’s post hoc test using Statistica 13.3 software. The significance level was set to $\alpha = 0.05$ and differences were considered significant at $P<0.05$.

**RESULTS AND DISCUSSION**
The amount of oil contained in palm kernel seeds was measured using the Soxhlet extraction method with hexane as a solvent. The result was used as a divisor to determine the extraction efficiency of the CBE intensified palm kernel extraction using a green solvent. The oil yield using the Soxhlet oil method was 39.53 ± 2.2% with an acid value of 1.06 ± 0.2%. This result is in agreement with previous studies.\textsuperscript{26} Lauric acid dominated the fatty acid profile at a concentration of 47% while oleic acid (15.6%) was the highest for unsaturated fatty acids. As mentioned, the residual oil remaining after the Screw press method is 10 – 20%; therefore, 15% as the middle number was used as a comparison to calculate the extraction efficiency based on the Screw press.

**Effect of Different Extraction Solvents on Extraction Efficiency and Lipid Profile**
Six green solvents such as water, ethanol, d-limonene, dimethyl carbonate, isopropyl alcohol, and ethyl acetate were used as solvent extractors in CBE-intensified palm kernel oil extraction and the solvent with the highest extraction efficiency was selected to study the operational variable of the CBE. For comparison, hexane was also used either as a solvent for traditional Soxhlet extraction or CBE-intensified palm kernel oil extraction. Figure-2A shows that all green solvents could extract oil from the palm kernel with an average oil yield of 26% except water, which could not extract the lipid out of the cell walls. It is interesting to note that hexane, which has similar polarity to lipids, only extracts 24% of palm kernel oil in the CBE-intensified extraction method. This is probably because the extracted oil in hexane at that
volume ratio has reached its saturation point.\textsuperscript{27} Increasing the volume of hexane used showed significantly increased oil yield. Other works in the extraction of jatropha oil have also shown that increasing the volume of solvent could increase the oil yield as the concentration gradients of the solid and liquid phase has increased, providing better mass transfer between reactant.\textsuperscript{28} In addition, only ethanol oil yields less than hexane. A different result was reported by Rincón-Cervera et al.\textsuperscript{16} who obtained a similar oil yield extracted using hexane and ethanol as solvents from purple viper’s bugloss (Echium plantagineum). The highest oil yield was obtained using ethyl acetate (29.5 ± 1.0%). This result is in agreement with Cascant et al.\textsuperscript{29} who concluded that ethyl acetate has the potential to replace environmentally hazardous hexane as the extracting agent due to its ability to extract all classes of lipids. The average extraction efficiency based on the Soxhlet method was quite low (66%), while the Screw press method’s extraction efficiency was 84%. Ethyl acetate as the best extracting agent in this study showed extraction efficiencies of 74.6 ± 2.5% and 95.9 ± 3.2% based on Soxhlet and Screw press, respectively. These results showed that the CBE-intensified palm kernel oil extraction using green solvents has a lower performance than the Soxhlet and Screw press methods. However, the green solvent has the potential to improve the mechanical disruption method with further variations of the operational parameters of CBE using ethyl acetate. The average concentration of MG, DG, and TG extracted from CBE-intensified palm kernel oil was 4.0, 2.2, and 84.4 mol\%, respectively (Fig. 3A). The highest concentrations of MG and DG were extracted using \textit{d}-limonene of 9.9 ± 0.6 and 6.4 ± 1.0 mol\%, respectively, which are 2.5 and 7.4 times higher than the Soxhlet method as shown in figure 3A. Even though the oil yield extracted using ethanol was the lowest, the highest TG concentration of 87.9 ± 1.1 mol\% occurred using this green solvent. Overall, the TG extraction efficiency of 99% showed that all the green solvents used have similar performance to the Soxhlet-hexane method in the TG extraction process.

![Fig.-2: The effect of solvents type (A); rotational speed (B); extraction time (C) and the ratio of palm kernel to ethyl acetate (D) on extraction efficiency based on the Soxhlet and Screw press methods of CBE-intensified palm kernel oil extraction. Ethyl acetate was used as a solvent to determine the effect of B and C](image)

**Effect of Rotational Speed on Extraction Efficiency and Lipid Profile**

The rotational speed of CBE is one of the important factors that impact the oil yield. Normally in chemical reactors, increasing the agitation / rotational speed could increase the mixing intensity of reactants, yielding high mass transfer and enhancing reaction rates.\textsuperscript{30} Therefore, in this study, 6 different rotational speeds starting from 3000 to 8000 rpm with increments of 1000 rpm were investigated in extraction conditions of the ratio of palm kernel to ethyl acetate of 1:3 (w/v) and extraction time of 10 minutes. As shown in Figure -2B, the rotational speed of CBE did not affect the oil yield. This is due to the diffusion solvent to the solute controlling extraction process and the constant oil yield obtained when it reaches equilibrium.\textsuperscript{27,30}
Fig. 3: The effect of solvents type (A); rotational speed (B); extraction time (C) and the ratio of palm kernel to ethyl acetate (D) on the concentration of MG, DG, and TG extracted using CBE. Ethyl acetate was used as a solvent to determine the effect of B and C.

The findings of the current study are consistent with those of Mueanmas et al.\textsuperscript{30} which reported no significant increase in oil yield with increasing mixing speed in the extraction of waste coffee grounds oil using hexane as the solvent. The average oil yield of 27.8\% was obtained, which is less than the Soxhlet and Screw press method. The analysis of the variance test indicated a significant effect of changing the rotational speed on the CBE-intensified extraction efficiency of both the Soxhlet and the Screw press method. The significant effect was mainly driven by the rotational speed of 6000 and 8000 rpm, which yielded the lowest extraction efficiencies of 66.5 ± 2.4\% and 67.1 ± 0.5\%, respectively in comparison with the Soxhlet method and 85.5 ± 3.1\% and 86.3 ± 0.6\% for extraction efficiency based on the Screw press method. For all variables tested, the highest extraction efficiency was obtained under the extraction condition of 5000 rpm rotational speed.

The average concentrations of MG, DG, and TG (3.9, 0.8, and 84.7 mol\%) extracted using the CBE-intensified method were similar to the concentrations extracted using the Soxhlet method (Fig. 3B). However, for some rotational speeds, the extraction efficiency of MG, DG, and TG exceeded the concentration extracted using the Soxhlet method. The highest extraction efficiencies of MG, DG, and TG were 138.0, 125.0, and 103.0\%, respectively, observed at rotational speeds of 6000, 3000, and 4000 rpm, respectively. An ANOVA one-way test detected a significant effect either for MG, DG, or TG. A Tukey test post hoc analysis established that the significant effect was driven by a high extraction of MG at the rotational speed of 6000 rpm (5.5 ± 0.6 mol\%) while, in contrast, the low extraction of DG at a rotational speed of 6000 – 8000 rpm (0.4 ± 0.1 – 0.5 ± 0.1 mol\% respectively) causes the significant effect.

Effect of Extraction Time on Extraction Efficiency and Lipid Profile

It is noted that extraction time has a significant effect on oil yield.\textsuperscript{4} Furthermore, regarding the production cost, electrical consumption and extraction/reaction time have the biggest impact, as prolonged extraction/reaction time could increase the electricity consumption of the reactor.\textsuperscript{31} Therefore, finding the specific extraction time is necessary. In this present study, the extraction time was determined as ranging from 5 to 30 minutes in the extraction condition of the ratio of palm kernel to ethyl acetate of 1:3 at a rotational speed of 5000 rpm (Figure-2C). The average oil yield of 28.3\% obtained from this parameter ranged from 25.2 ± 0.4\% to 29.5 ± 0.2\%. The average extraction efficiency of the Soxhlet method was 71\% and 92\% of the Screw press method. The highest extraction efficiencies were observed after 10 minutes of extraction time with values of 74.6 ± 2.5\% and 95.9 ± 3.2\% for the Soxhlet and Screw press methods, respectively. Significant effects were observed for this parameter both for the extraction efficiencies based on Soxhlet and Screw press methods. The further Tukey post hoc test did not detect the
main effect. Extraction time had a significant effect on the extraction efficiency of MG, DG, and TG (Figure-3C). The extraction of MG was significantly highest at 10 minutes extraction time, achieving an extraction efficiency of 99.5%, while a 122.0% extraction efficiency of DG was achieved in the same extraction time. In contrast, the TG was completely extracted in 5 minutes extraction time compared to TG concentration using Soxhlet-hexane.

Effect of Ratio Palm Kernel to Ethyl Acetate on Extraction Efficiency and Lipid Profile

The effects of the ratio of palm kernel to ethyl acetate on CBE-intensified oil yield and MG, DG, and TG extraction were investigated at seven different ratios in extraction conditions of 5000 rpm rotational speed and 10 minutes extraction time. The oil yield was increased from 21.9 ± 0.4% when using a ratio of 1:2 to 34.2 ± 1.4% at the ratio of 1:7 and was decreased to 27.3 ± 0.6% with increasing solvent volume. The present findings seem to be consistent with other research which observed increasing lipid extraction efficiency of black soldier fly larvae with increasing ratio of biomass to solvent and remains plateau after reaching the peak.\textsuperscript{32} The average extraction efficiency based on the Soxhlet method was 74% with the highest (86.5 ± 3.6%) achieved at a ratio of 1:7. In contrast, the extraction efficiency based on the Screw press method exceeded 100% at a ratio of 1:6 and 1:7 and achieved 108.7 ± 1.3% and 111.2 ± 4.6%, respectively (Figure-2D). The univariate test of significance for both the extraction efficiency based on Soxhlet and Screw press methods showed significant effects. The post hoc Tukey test further detected that the significant effect was mainly driven by the parameters tested. The results are similar to previous results in studies in which solvent to seed ratio, time, and temperature have a significant effect on oil yield in the extraction of Crambe seed oil assisted by ultrasound.\textsuperscript{33} Significant effects were determined for MG, DG, and TG in this study. However, the Tukey test post hoc did not detect the main effect. The average concentrations of MG and DG obtained were quite similar to the concentrations extracted using the Soxhlet-hexane method, While the average TG concentration exceeded the standard. The highest concentrations of MG and DG of 3.9 ± 0.4 and 1.1 ± 0.0 mol% were observed at a ratio of 1:3, respectively, and a concentration of 90.5 ± 0.4 mol% was obtained for TG at a ratio of 1:4 (Figure-3D).

Energy Consumption Comparison of Palm Kernel Oil Extraction Methods

Energy consumption of CBE-intensified palm kernel oil extraction was determined as kilowatt-hour electricity consumption per kilogram palm kernel (Table-1).

| Extraction Method | Seed | Extraction Condition | Energy Consumption (kWh Kg\(^{-1}\)) | Ref. |
|-------------------|------|----------------------|--------------------------------------|-----|
| CBE green solvent | Palm kernel | Palm kernel 20 gram, ratio palm kernel: ethyl acetate (1:7), rotational speed 5000 rpm, extraction time 10 minutes, oil yield = 34.2% | 12.53 | This study |
| Screw press | Palm kernel | Fabricated palm kernel screw press, capacity 101.7 kg h\(^{-1}\), speed 56 rpm, oil yield = 39% | 45 | \textsuperscript{34} |
| Subcritical fluid | Jatropha curcas | Jatropha seed = 2.4 gram, ratio jatropha : solvent (1:20), temperature 90°C, pressure 0.5 MPa, 15 minutes extraction time, oil yield = 40.3% | 57.3 | \textsuperscript{35} |
| Soxhlet-hexane | Palm kernel | Palm kernel 20 gram, 150 ml Hexane, 30 minutes extraction time, oil yield = 39.5% | 15 | This study |

In comparison, the traditional Soxhlet-hexane extraction method was conducted using a hot plate magnetic stirrer as the heating source. The data on palm kernel oil extraction using the Screw press method was obtained from published research.\textsuperscript{34} Due to insufficient data on the extraction of palm kernel oil using a subcritical fluid, the published result on the extraction of Jatropha oil was used, as the oil content in \textit{Jatropha curcas} is in a range similar to palm kernel.\textsuperscript{35} As shown in Table-1, the energy consumption of CBE-intensified palm kernel oil using a green solvent was determined based on the
maximum oil yield obtained in the extraction condition. The CBE consumed 1.5 kW to operate at room temperature. Therefore, the total energy required for processing 20 g of palm kernel at a rotational speed of 5000 rpm for 10 minutes was 12.5 kWh kg\(^{-1}\) which is equal to 45.1 MJ kg\(^{-1}\)seed. The energy consumption of CBE-intensified palm kernel oil extraction was lower than for other extraction methods (Table-1). The CBE-intensified method provides an energy saving of 72, 78, and 16% compared to the Screw press, subcritical fluid, and Soxhlet-hexane processes, respectively. In addition, the CBE-intensified process could be conducted in only 10 minutes, which is 33 and 67% faster than subcritical fluid and Soxhlet-hexane processes, respectively.

**CONCLUSION**

The CBE-intensified palm kernel oil extraction combines grinding and extraction processes into a single step with a green solvent acting as the extracting agent to collect the oil at ambient temperature and pressure. The CBE-intensified process combined with ethyl acetate could improve traditional mechanical disruption palm kernel oil extraction. The result shows that the CBE-intensified is superior in palm kernel oil extraction to the Screw press method and this was done in only 10 minutes of extraction time, which is faster than the traditional Soxhlet and mechanical press methods. The CBE-intensified is energy efficient and could save >70% energy compared to other green extraction processes and 16% compared to the conventional Soxhlet method. In terms of time-saving, the CBE-intensified process is faster than Screw press, subcritical fluid, and Soxhlet-hexane extraction methods. Furthermore, the rapid high extraction efficiency of CBE-intensified palm kernel oil using ethyl acetate as the extracting solvent opens the possibility of economically feasible palm kernel oil production.

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