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Solvent selection for efficient phenolic extraction from palm kernel cake using microwave-assisted techniques

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ABSTRACT

This study investigated the recovery of phenolic compounds from palm kernel cake (PKC) using microwave-assisted extraction (MAE), focusing on the effects of solvent type and liquid-to-solid ratio on extraction efficiency. MAE was performed under constant microwave power (300 W) and extraction time (25 minutes), while varying the solvent types (ethanol, methanol, and water) and liquid-to-solid ratios (20:1, 30:1, and 40:1). Among the solvents tested, ethanol achieved the highest extraction yield (45%) and total phenolic content (TPC) of 0.086 mg GAE/g dry oil, while water produced the lowest yield (10%) and TPC (0.052 mg GAE/g dry oil). Regarding the liquid-to-solid ratio, the highest extraction yield (48%) was observed at a 40:1 ratio. However, the highest TPC was obtained at a 30:1 ratio, emphasising the dilution effect at higher solvent volumes. These findings demonstrate that optimising both solvent selection and liquid-to-solid ratio is critical for maximising phenolic recovery from PKC. Moreover, the use of ethanol, a food-grade and biodegradable solvent, coupled with MAE technology, provides a sustainable and cost-effective approach for the valorisation of agricultural by-products. The optimised process offers potential applications in the food, nutraceutical, and pharmaceutical industries.

1.0 INTRODUCTION

The most common species in the genus *Elaeis*, a member of the Palmae family, is the oil palm (*Elaeis guineensis*), native to West Africa. Its cultivation has spread worldwide over time, and Malaysia is now one of the top producers The oil palm sector is an important factor in Malaysia's agricultural economy and a major driver of its economic expansion. It is estimated that around 115.86 million tons of oil palm Fresh

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fruit bunches (FFB) are processed yearly in Malaysia (Awoh et al., 2023). There are multiple steps in the production process as shown in Fig. 1. Sterilisation of fresh fruit bunches is carried out to inhibit enzymatic activity and facilitate fruitlet separation (Mahlia et al., 2019). The stripped fruitlets are then mechanically pressed to extract crude palm oil (CPO), while the resulting press cake contains residual kernels and fibres. The CPO undergoes clarification and filtration to ensure its quality for further processing. Kernels are subsequently processed through winnowing, nut cracking, and depericarping to produce palm kernel oil. Despite its economic importance, palm oil production faces challenges related to waste generation. Various solid and liquid by-products, such as empty fruit bunches (EFB), palm kernel shells, and palm oil mill effluent (POME) are generated at different stages of processing (Awoh et al., 2023). It is estimated that for every ton of crude palm oil produced, approximately 0.70 tons of palm fibre, 0.35 tons of palm kernel shells, and 1.1 tons of empty fruit bunches are generated (Ni'Mah et al., 2019). Although these wastes pose environmental challenges, they also present opportunities for valorisation into value-added products and bioenergy. The palm oil milling process is thus a complex operation that must balance environmental management, industrial efficiency, and agricultural productivity. Achieving sustainable growth in the sector requires careful integration of these factors, especially in countries like Malaysia where palm oil production plays a critical role in the economy.

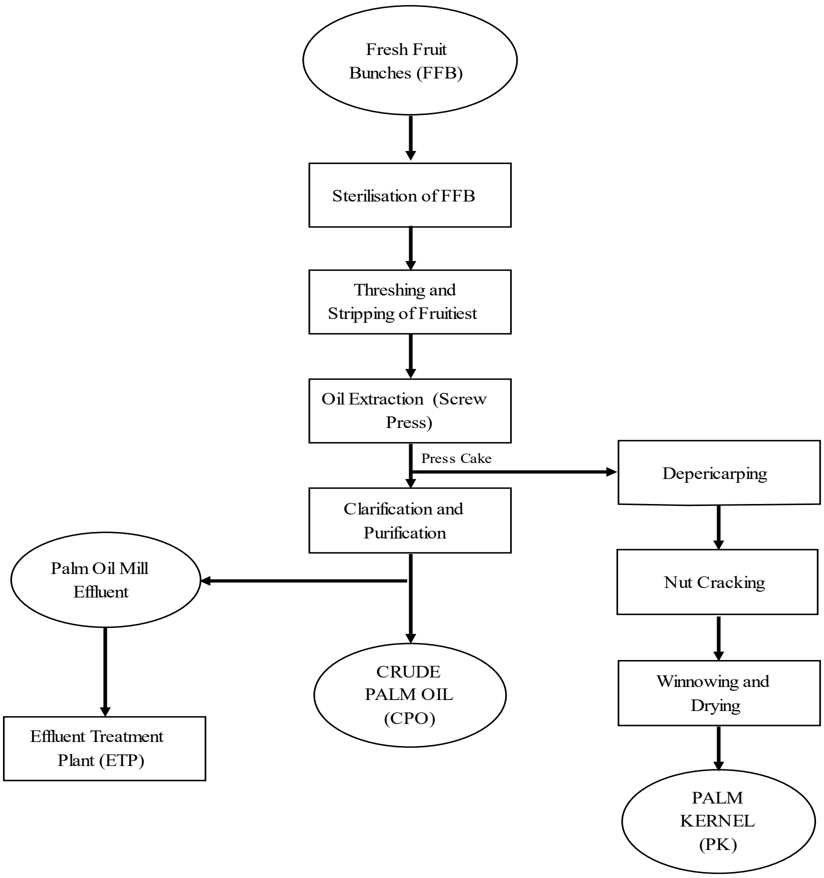

Fig. 1. Process of the waste generation in palm oil mill

Source: Mahlia et al. (2019)

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Phenolic compounds are the primary substances generated during the secondary metabolic processes in plants. The compounds exhibit unique chemical structures and are classified into five categories: phenolic acids, flavonoids, tannins, stilbenes, and lignans (Albuquerque et al., 2021). The oil palm fruits contain a significantly elevated phenolic content. Each year, 21.63 tons of by-products per hectare of oil palm agriculture produce 225 kg of palm phytochemicals (Ofori-Boateng & Lee, 2013). Kua et al. (2015) studied PKC protein hydrolysate and discovered its antioxidant properties. The phenolic concentration of PKC extract was 18.2%, including 12.3% hydroxybenzoic acid and 5.9% 4H-pyran-4-one. P-hydroxybenzoic acid was the most common phenolic compound identified, followed by protocatechuic acid as the second most frequently detected phenolic. The PKC methanol extract included a small quantity of d-glucuronic acid and quinic acid. Salicylic acid and sinapic acid were only found in the PKC cell wall extract, in addition to d-glucuronic acid and quinic acid (Kua et al., 2015).

The phenolic compounds can be extracted both conventionally and unconventionally. Traditional phenolic extraction methods such as maceration, Soxhlet extraction, and hot reflux extraction. The phenolic extraction rates from these simple methods are sufficient and they use low-cost equipment. The intended components are destroyed by long extraction durations, significant amounts of environmentally harmful chemical solvents that are also harmful to human health, as well as by light, air, high temperatures, and enzyme interactions. Ultrasound-assisted extraction (UAE), microwave-assisted extraction (MAE), supercritical fluid extraction (SFE), high-pressure homogenisation (HPH), and pulsed electric fields (PEF) are some of the improved phenolic compound extraction techniques that are desired due to issues with the current extraction methods (Carpentieri et al., 2021). By using fewer organic solvents, these simple solutions reduce pollution emissions. MAE is one of the more recent technologies for extracting phenolic compounds (Gallo et al., 2010). Using 50% aqueous ethanol with MAE produced the best phytocomponent extraction from *Melilotus officinalis* flowering tops, according to Martino (2006). The advantages of the MAE method include reduced time, reduced solvent usage, higher extraction rates, and lower costs for superior goods (Gallo et al., 2010). Temperature, liquid-to-solid ratio, extraction time, microwave power, and solvent selection are the parameters of the optimal yield of the phenolic compound (Osorio-Tobón, 2020).

Among the critical factors in MAE, the selection of solvent type is particularly important. Water, methanol, ethanol, and combinations of water-ethanol are commonly used solvents for phenolic extraction (Widyawati, 2014). Water, a highly polar solvent, is effective for extracting highly polar phenolics such as hydroxybenzoic acids and glycosylated flavonoids. Methanol with slightly lower polarity can extract both highly polar and moderately polar phenolics (Jasiukaitytė-Grojzdek et al., 2020). Ethanol, possessing intermediate polarity, is often used for less polar phenolics such as aglycones and lipophilic compounds. Additionally, mixtures of ethanol and water offer a balance of polarity that enables the extraction of a broader range of phenolics. Ethanol stands out not only due to its solvent properties but also because of its environmental and regulatory advantages. It is food-grade, biodegradable, renewable, and safer for human health compared to other organic solvents like methanol. Consequently, ethanol is highly suitable for extracting phenolic compounds intended for applications in food, nutraceutical, and pharmaceutical industries. Its use aligns with the principles of green chemistry and sustainability, contributing to the development of eco-friendly extraction processes. Due to its chemical compatibility, extraction effectiveness, and sustainability, ethanol is an excellent solvent for the extraction of phenolic compounds from PKC and other biomass sources (Djenar et al., 2020). Fig. 2 schematically illustrates the interaction mechanism between ethanol and PKC during MAE.

This research emphasises the utilization of PKC as a sustainable source of phenolic compounds with significant antioxidant potential. By employing microwave-assisted extraction (MAE), this study aims to optimise key process parameters, particularly solvent type and liquid-to-solid ratio, to maximise the recovery of phenolic compounds. The outcomes are expected to support the sustainable utilization of oil palm by-products and contribute insights into enhancing the cost-effectiveness and environmental performance of phenolic extraction processes.

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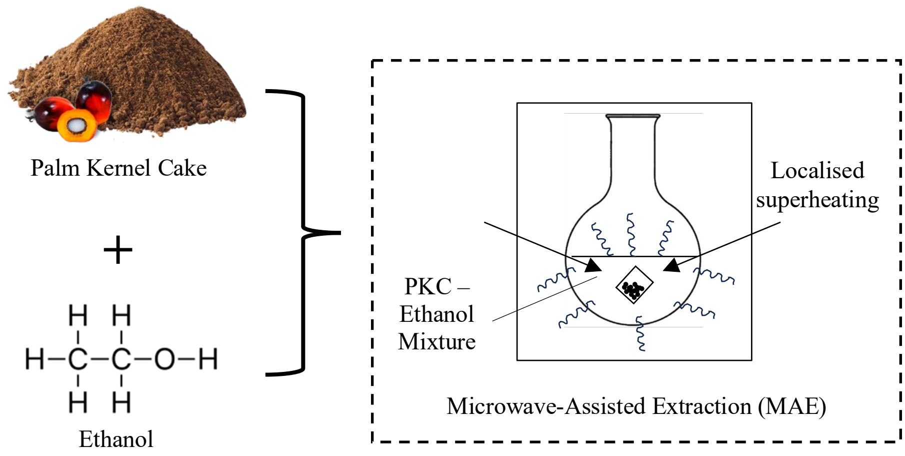

Fig. 2. Interaction mechanism between ethanol and PKC in MAE

Source : Author's illustration

2.0 MATERIALS AND METHOD

2.1 Materials

The chemical and reagent used in this study was palm kernel cake (PKC) purchased from a local source in Terengganu, located in Malaysia. Folin-Ciocalteu reagent and sodium carbonate (Na_2CO_3) were purchased from Sigma-Aldrich. Gallic acid was supplied by HmbG Chemicals. Ethanol (96%) and methanol (100%) were purchased from Emsure Merck.

2.2 Microwave-assisted extraction of PKC

The palm kernel cake (PKC) was first rinsed with deionised water to remove any remaining fine particles like debris that may be present on the surface of the PKC that could interfere with extraction efficiency (Hebbani et al., 2021). Then, the PKC would have been slow dried in an oven set to 40 °C for 48 hours (Yi et al., 2011), until a constant weight was achieved indicating that the moisture content had been sufficiently reduced for extraction process. This slow dry method offers several benefits for extraction, including the preservation of heat-sensitive phenolic compounds, minimization of bioactive degradation, and improved extraction efficiency (ElGamal et al., 2023). Following drying, the PKC were ground into a powder using a grinder until the samples were powdery. After processing, the PKC were sieved to produce particles ranging from 0.5 to 1 mm, ensuring a larger surface area to enhance solvent penetration during extraction (Senawong et al., 2023). A total of 10 g of ground PKC was placed into an empty tea bag. The extraction process was then performed using the MAE method. Approximately 10 g PKC sample and 300 mL of ethanol as a co-solvent were added into the round bottom flask. In this study, the MAE process parameters were fixed at a microwave power of 300 W and an extraction time of 25 minutes, while the varied parameters included the type of co-solvent (water, ethanol, methanol, and water-ethanol mixtures at ratios of 20:80, 60:40, and 40:60) (Rezaei et al., 2013) and the liquid-solid ratio of ethanol to palm kernel cake (PKC) at 20:1, 30:1, and 40:1 (Tungchaisin et al., 2022).

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2.3 The determination of extraction yield

After extraction, the mixture was filtered to remove solid residues. The filtrate was then concentrated using rotary evaporation (Rotavapor R-215) under reduced pressure to remove the solvent. The remaining extract was dried completely and weighed to ensure no residual solvent remained. The extraction yield was calculated using the following Eq. (1) (Onoji et al., 2019):

$$\text{Yield (\%)} = \left(\frac{\text{Mass of extracted oil (g)}}{\text{Mass of raw material used (g)}}\right) \times 100 \tag{1}$$

2.4 The determination of total phenolic content (TPC)

The TPC of the PKC extracts was determined using the Folin-Ciocalteu method, with gallic acid as the standard reference (Nomanbhay et al., 2018). A stock solution of gallic acid was prepared by dissolving 0.5 g of gallic acid in 10 mL of ethanol and diluting it to 100 mL with distilled water. Standard solutions with concentrations of 50, 100, 250, and 500 mg/L were prepared. For the assay, 40 µL of sample or standard solution was mixed with 0.2 mL of 0.2 N Folin-Ciocalteu reagent and 3.16 mL of distilled water, followed by the addition of 0.6 mL of 7.5% (w/v) sodium carbonate (Na_2CO_3) solution. The mixture was vortexed and incubated in the dark at room temperature for 2 hours. Ethanol was used as the blank. The absorbance was measured at 765 nm using a double-beam UV-Vis spectrophotometer. TPC was calculated using the gallic acid calibration curve and expressed as milligrams of gallic acid equivalents per gram of dry oil (mg GAE/g dry oil), with all measurements conducted in triplicate (Tsouko et al., 2019).

3.0 RESULTS AND DISCUSSION

3.1 Effect of different types of solvent in MAE of PKC

The selection of an appropriate solvent plays a critical role in the efficiency of phenolic compound extraction during microwave-assisted extraction (MAE). Solvents with different polarities influence the solubility of phenolic compounds, thereby affecting the overall extraction yield and total phenolic content (TPC) (Vieito et al., 2018). Polar solvents such as ethanol, methanol, and water are commonly used due to their compatibility with phenolic compounds and ability to extract a wide range of bioactive components (Osorio-Tobón, 2020). Fig. 3 shows the result of extraction yield and total phenolic content (TPC) obtained using various solvent types which are ethanol, methanol, and water.

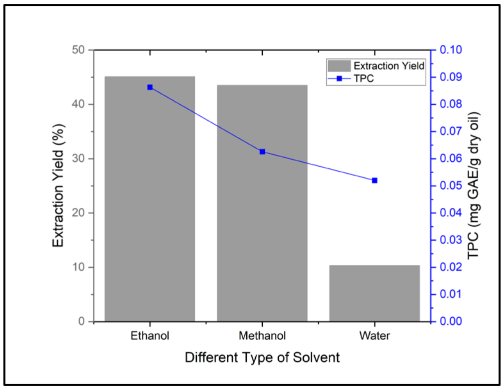

Fig. 3. Extraction yield and total phenolic content at different types of solvent

Source : Author's own data

Ethanol and methanol show far greater extraction yields, at around 45% and 43%, respectively compared to water, which produces 10% as shown in Fig. 3. Ethanol has the greatest TPC value which was 0.086 mg GAE/g dry oil, followed by methanol 0.063 mg GAE/g dry oil and water 0.052 mg GAE/g dry oil. The polarity of ethanol and methanol allows for improved solubilization and extraction efficiency, making them more compatible with phenolic compounds and contributing to their higher performance. In contrast, water is less effective than organic solvents because it has a reduced capacity to solubilise phenolic chemicals. These results are consistent with earlier research that shows that ethanol and methanol are excellent solvents for extracting phenolic compounds because of their mild polarity and capacity to create hydrogen bonds with phenolics. The results correspond with other research highlighting the essential influence of solvent type on the extraction of phenolic compounds. Hashim et al. (2020) indicated that ethanol and methanol serve as very efficient solvents for the extraction of phenolic compounds, attributed to their intermediate polarity that enhances interaction with phenolic structures. These solvents achieve equilibrium between hydrophilic and lipophilic characteristics, enabling them to dissolve a diverse array of phenolics. Djenar et al. (2020) emphasised that ethanol is preferred for its effective extraction of phenolics, as well as its environmental safety and suitability for culinary and medicinal uses. This study's observation of water's inferior performance as a solvent aligns with Fabian (2012) results, which indicated that water's strong polarity restricts its capacity to solubilise less-polar phenolic compounds, resulting in diminished extraction yields and total phenolic content (TPC) values. This constraint renders water less efficacious in comparison to organic solvents like ethanol and methanol.

In this study, an extraction yield of 45% was achieved using MAE with ethanol as the solvent. Compared to previous studies, this yield is notably higher than that reported for *Pandanus amaryllifolius* (23.53%) (Djenar et al., 2020) and *fiddlehead ferns* (18.18%) (Fabian, 2012), both extracted under MAE conditions. Although slightly lower than the 83.33% yield obtained from *Peperomia pellucida* (Hashim et al., 2020), the differences can be attributed to the tougher, fibrous structure of PKC compared to softer plant materials. Thus, a 45% yield can be considered high for PKC, supporting the effectiveness of the optimised MAE conditions employed in this study.

Based on these findings, ethanol was selected for further optimization studies. Although water alone exhibited lower extraction efficiency, ethanol-water mixtures were investigated to determine whether combining solvents of differing polarity could enhance overall phenolic recovery, as reported by Gallo et al. (2010). Fig. 4 illustrates the effects of different ethanol-water ratios of 100% ethanol, 20:80, 40:60, and 60:40 on extraction yield and TPC.

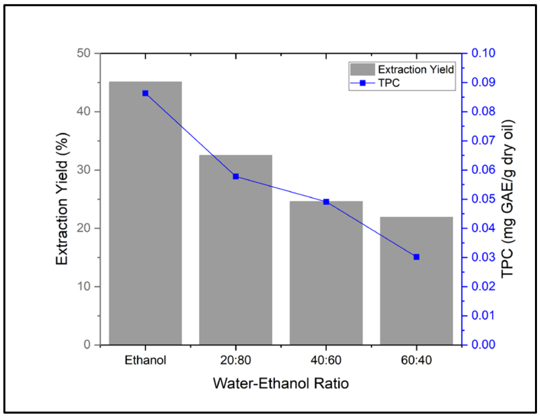

Fig. 4. Extraction yield and total phenolic content at different ratios of water-ethanol solvent

Source: Author's own data

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Pure ethanol has the greatest extraction yield which is 45% and TPC 0.086 mg GAE/g dry oil as shown in Fig. 4. As the quantity of water in the ethanol-water combination increases, extraction yield and TPC gradually decreases. The 60:40 water-ethanol ratio results in the lowest performance, with an extraction yield of 21% and a TPC of 0.030 mg GAE/g dry oil. As the water content increases, the solvent mixture's polarity compatibility decreases, explaining the downward trend. Phenolic compounds are more successfully extracted using solvents of intermediate polarities, such as pure ethanol, which can solubilise a broader spectrum of phenolics. As the water increases, the solvent becomes more polar, lowering its attraction for less polar phenolic chemicals and decreasing extraction efficiency. This finding is consistent with prior research, such as Hashim et al. (2020), which found that ethanol is a suitable solvent for phenolic extraction since it can dissolve polar and non-polar chemicals well. In contrast, combinations with a higher water content were demonstrated to restrict solubility and diminish interaction with phenolic structures (Fabian, 2012). These results support the idea that pure ethanol or ethanol-dominant combinations are best for optimising extraction yield and TPC.

Interestingly, in this study, pure water extracted a slightly higher TPC (0.052 mg GAE/g) compared to the 60:40 ethanol-water mixture (0.030 mg GAE/g). This phenomenon may be attributed to the specific phenolic profile of PKC, which likely contains highly polar phenolic compounds better solubilised by pure water than by hydroalcoholic mixtures. Similar findings were reported by Widyawati et al. (2014), where pure water extract of *Pluchea indica* leaves exhibited a higher total phenolic content than ethanol extract, supporting the idea that certain highly polar phenolics are more effectively extracted by water. These observations highlight the importance of matching solvent polarity carefully to the polarity of the target compounds during extraction.

3.2 Effect of liquid-to-solid ratio in MAE of PKC

The liquid-to-solid ratio is a crucial parameter in microwave-assisted extraction (MAE) as it directly impacts the efficiency of phenolic compound recovery. This ratio determines the amount of solvent available for dissolving and extracting phenolic compounds from the biomass. An optimal liquid-to-solid ratio ensures sufficient solvent interaction with the sample while avoiding excessive dilution, which could reduce the concentration of phenolics in the extract. In this study, the effects of three liquid-to-solid ratios (20:1, 30:1, and 40:1) were investigated to evaluate their influence on extraction yield and total phenolic content (TPC) from palm kernel cake (PKC). Understanding the impact of this parameter is essential for optimising the MAE process and maximising phenolic compound recovery. Figure 5 shows the effect of different liquid (ethanol)-to-solid (PKC) ratios of 20:1, 30:1, and 40:1 on the extraction yield and TPC, mg GAE/g dry oil).

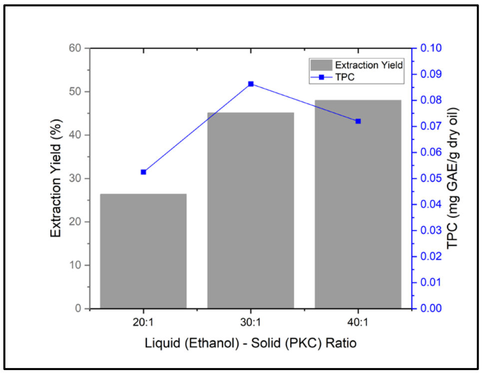

Fig. 5. Extraction yield and total phenolic content for liquid-solid ratio

Source: Author's own data

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According to Fig. 5, the extraction yield consistently increased with the liquid-to-solid ratio, reaching the highest value of 48% at 40:1. However, the TPC exhibited a different trend, increasing from 20:1 to 30:1 and peaking at approximately 0.086 mg GAE/g dry oil, before decreasing to 0.072 mg GAE/g dry oil at the 40:1 ratio. Despite the higher extraction yield at 40:1, the decline in TPC may be attributed to the dilution effect caused by the increased solvent volume. According to Wong et al. (2013), when the liquid-to-solid ratio surpasses the optimal level, the concentration gradient that facilitates the extraction of phenolic compounds decreases, leading to a reduction in phenolic recovery. As a result, the increased solvent volume may raise the total extract volume but dilute the phenolic concentration, reducing the solvent's capacity to effectively solubilise phenolic compounds. This finding aligns with the observed decrease in TPC at higher liquid-to-solid ratios, particularly at 40:1, where the dilution effect likely outweighed the benefits of the increased extraction yield.

In addition, the decrease in TPC may also be influenced by the polarity dilution effect, where the addition of water into ethanol alters the solvent polarity and reduces the solvent’s capacity to solubilise moderately polar phenolic compounds (Galanakis et al., 2013). This highlights that both solvent volume and solvent composition must be carefully optimised to maximise phenolic extraction efficiency. Therefore, the 30:1 liquid-to-solid ratio is considered optimal for maximising phenolic compound recovery, as it provides a balance between sufficient solvent availability and efficient extraction. Increasing the solvent volume beyond this point did not further improve phenolic recovery, as reflected in the lower TPC observed at the 40:1 ratio.

4.0 CONCLUSIONS

This study successfully optimised the microwave-assisted extraction (MAE) parameters for phenolic recovery from palm kernel cake (PKC). Among the solvents tested, ethanol exhibited the highest extraction efficiency, achieving an extraction yield of 45% and a total phenolic content (TPC) of 0.086 mg GAE/g dry oil. The optimal liquid-to-solid ratio was identified as 30:1, where the highest TPC was obtained without significant dilution effects, fulfilling the objective of maximising phenolic recovery. Furthermore, the results demonstrate that the use of ethanol, a food-grade, biodegradable, and renewable solvent, combined with MAE technology, enhances the sustainability and cost-effectiveness of the extraction process. MAE reduced extraction time and solvent usage compared to the conventional method while valorising an agricultural by-product (PKC) supports waste minimisation efforts. Overall, the findings directly address the study objectives by optimising extraction parameters for high phenolic yield while promoting an environmentally friendly and economically viable extraction strategy. These insights contribute to advancing sustainable resource utilisation and support the development of green technologies within the palm oil industry.

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CONFLICT OF INTEREST STATEMENT

The authors affirm that there are no conflicts of interest related to this research, including financial, personal, or professional relationships that could have influenced the outcomes of this study. The work was carried out independently and is free from any competing interests.

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AUTHORS' CONTRIBUTIONS

Ellisa Natasya Rosli: Experimental design, methodology, investigation and writing original draft; **Norhusna Mohamad Nor**: Supervision, writing-review and editing; **Siti Hawa Mat Yaman:** Experimental design, methodology, and investigation.

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