

Extraction of methylene blue using palm kernel oil as a green organic solvent: A parametric study

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ARTICLE INFO

Article history:

Received 27 January 2026

Revised 20 August 2026

Accepted 1 September 2026

Online first 30 September 2026

Published 30 September 2026

Keywords:

Palm Kernel Oil

Solvent Extraction

Methylene Blue

Green Organic Solvent

Free Fatty Acid

DOI:

10.24191/esteem.v22iSeptember.10400

ABSTRACT

Methylene blue (MB), a synthetic dye widely used in industries such as textiles, pharmaceuticals, and food processing, has become a major environmental pollutant due to its toxicity and persistence in aquatic environments. Conventional solvent extraction methods often rely on hazardous organic solvents, posing risks to both human health and the environment. This study investigates a sustainable alternative by using palm kernel oil (PKO), a green organic solvent, for MB extraction. A parametric study was conducted to evaluate the effects of pH, organic-to-aqueous volume ratio, and initial MB concentration on extraction efficiency. Batch extractions were performed using the shake-out method. The results showed a maximum extraction efficiency of 99% at pH 4.6 and a 1:1 volume ratio. Extraction efficiency was also influenced by the initial dye concentration. Additionally, a stripping process using hydrochloric acid (HCl) achieved 99% recovery of MB from the PKO phase, enabling solvent reuse. The presence of MB in PKO was confirmed using FTIR analysis. This study highlights PKO's potential as a greener and effective alternative to conventional solvents in dye removal, offering a safer and more sustainable approach.

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1. INTRODUCTION

The textile industry extensively uses synthetic and reactive dyes for their favourable colour fastness and cost-effectiveness, with classification based on their chemical properties [1]. Research shows that over 10,000 tons of dyes are used globally each year in the production of 40 million tons of textiles, resulting in the discharge of 3,600 tons of concentrated dye waste into natural water sources [2]. According to the United Nations Industrial Development Organization (UNIDO), dye pollution is the second-largest global water pollutant, following agriculture [3]. This dye-loaded wastewater not only disrupts ecological stability but also poses significant risks to human and animal health. For instance, methylene blue (MB) is a cationic dye widely used in the textile and tanning industries. Excessive exposure to MB has been linked to health issues such as skin sensitivities, cardiovascular problems, and nausea in both humans and aquatic organisms [4]. Despite its relatively low toxicity, MB's slow degradation and high visibility even at low concentrations can damage ecosystems by reducing photosynthetic activity and chemical oxygen demand (COD) [4].

Hence, effective treatment of dye-loaded wastewater is essential for protecting the environment and living organisms. Several methods, including chemical precipitation, electrolysis, membrane, oxidation, biological treatments, and adsorption, have shown promising results in removing MB from wastewater [4], [5], [6], [7], [8]. However, all these methods have their own advantages and disadvantages. The electrolysis process involves high electricity costs, while adsorption requires efficient and cost-effective adsorbents, which may need to be replaced or regenerated frequently. Additionally, chemical precipitation may produce hazardous sludge as a by-product, and biological treatments can be time-consuming and may not be effective for all types of dyes.

Given these limitations, alternative separation techniques have been explored to enhance the efficiency and sustainability of dye removal. Solvent extraction is one such method and is widely used in chemical separation processes because it is simple, cost-effective and efficient at extracting pollutant solutes from aqueous solutions. The solute is transferred from the aqueous phase to the organic phase through facilitate transport involves an extractant dissolved within the organic phase, interacting with the solute to form solute-extractant complexes [9]. The organic phase generally consists of an extractant as the active compound, a modifier to prevent emulsions between the two immiscible solvents, and a diluent to control the overall conditions. Commonly used diluents include harmful chemicals such as kerosene, hexane and chloroform, while extractants and modifiers are also often petroleum-based solvents [10], [11], [12].

Hence, replacing harmful chemical solvents is essential for transitioning to green separation processes. Palm kernel oil (PKO), which contains natural fatty acids and ester groups, has shown promise as an environmentally friendly alternative for dye removal due to its biodegradability and ability to effectively adsorb dye molecules. The free fatty acids (FFA) in PKO act as a natural acidic extractant, capable of interacting with cationic dyes such as MB through electrostatic attraction, thus facilitating their removal from wastewater [13]. Hence no addition of extractant or diluent is needed and PKO was used as the sole organic phase. Additionally, PKO is abundantly available, particularly in Malaysia, one of the largest global producers of palm oil, making it a cost-effective and sustainable option. The widespread availability of PKO further enhances its potential as a green alternative for wastewater treatment, offering a renewable and environmentally friendly solution. In this study, we explore the potential of PKO as the organic solvent for MB removal from aqueous solution. The effects of aqueous-phase pH, the organic-to-aqueous volume

ratio and the initial MB concentration were investigated. Stripping MB from palm kernel oil was also investigated to assess its potential for reuse.

2. METHODOLOGY

2.1 Material and Equipment

Palm Kernel Oil (PKO) was obtained from a local supplier and used as the organic solvent in this study. The chemicals employed included methylene blue (>75% purity), hydrochloric acid (HCl) ($\geq 37\%$ purity), sodium hydroxide (NaOH) ($\geq 99\%$ purity), diethyl ether (>99% purity), anhydrous ethanol (99.85% purity), potassium hydroxide (KOH) ($\geq 99\%$ purity), and phenolphthalein (Merck, 1% in ethanol). All chemicals were used as received without further purification.

The two immiscible solvents of PKO and aqueous solution were mixed in an orbital shaker (Lab Companion, SK-300). The equilibrium pH of the solution was measured using a pH meter (Hanna Instruments, HI11310). The concentration of the MB in the aqueous phase was measured using a spectrometer (HACH, DR3900). A calibration curve was established to correlate adsorptivity with dye concentration. Absorptivity values were obtained at the specific wavelengths corresponding to the maximum absorption of the MB at maximum wavelength of (λ_{max}) 665 nm [14].

2.2 Free Fatty Acid Analysis

The free fatty acid (FFA) content in PKO was determined using a titration method [15]. A 3 g sample of PKO was weighed and diluted in 30 mL of a 1:1 (v/v) solution of diethyl ether and ethanol. Two drops of phenolphthalein were added as an indicator. The mixture was then titrated with a 0.1 M KOH solution until a subtle colour change was observed. The FFA concentration was subsequently calculated using Eq. (1):

$$C_{FFA}(\text{wt.}\%) = \frac{(V_{KOH})(M_{KOH})(MW_{FFA})}{10 \times S_{PKO}} \quad (1)$$

where V_{KOH} is the volume of KOH solution (mL) used in the titration, M_{KOH} is the molarity of KOH solution (mol/L), MW_{FFA} is the average molar mass of FFAs, and S_{PKO} refer to the mass of PKO (g) used.

2.3 Preparation of Organic and Aqueous Solution

PKO was used as the organic phase without any dilution with other chemicals, such as diluents, or the addition of extractants and modifiers. Pure PKO was applied as the organic phase during the extraction process, while in the stripping process, the MB-loaded PKO served as the organic phase. The aqueous phase for the extraction process was prepared by dissolving an appropriate amount of MB in distilled water, while in the stripping process, 1 M HCl was used as the aqueous phase.

2.4 Extraction and Stripping Process

The extraction experiments were performed under batch conditions by contacting equal volumes (15 mL) of the PKO and MB- loaded solution in a conical flask. The mixture was agitated using an orbital shaker for 8 minutes to allow the system to reach equilibrium. The mixture was then left for a few minutes to allow complete phase separation into two immiscible layers. The aqueous phase (bottom layer) was withdrawn to measure the pH of the solution. The aqueous phase then was returned to the flask if the pH solution is not obtained, where the pH was adjusted using 1 M HCl or 1 M NaOH.

The main parameters affecting the %E of MB, i.e. aqueous pH, oil-to-aqueous volume ratio (O: A), and initial MB concentration, were investigated. The experiment was conducted using a one-factor-at-a-time (OFAT) approach to determine the optimum conditions for MB extraction. The pH was varied from 4.6 to 8.5, while the O: A phase ratio was varied from 0.2 to 5. The initial MB concentration was investigated at 100, 200, and 300 mg/L. These parameters were selected based on their potential influence on the distribution of MB between the aqueous and organic phases. The selected ranges were used to systematically evaluate the effects of these operating parameters on the %E of MB.

Following extraction, the organic phase, which contained MB, was subjected to a stripping process to remove the MB, allowing the PKO to be recycled. An illustration of the extraction and stripping process is presented in Fig. 1. In the extraction process, PKO served as the organic phase, while the aqueous phase consisted of the MB solution. The MB molecules were transferred from the aqueous phase into the organic phase. The MB-loaded organic phase was then subjected to a stripping process using HCl as the stripping agent. The effect of the A:O phase ratio, ranging from 1 to 5, on %S of MB was investigated. The treated MB solution and concentrated MB were subsequently collected at the respective outlets of the aqueous extraction and stripping processes.

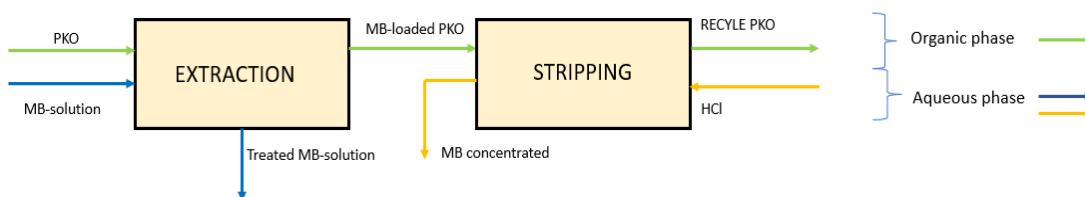


Fig. 1. Extraction and stripping process

All the samples for extraction and stripping were run in duplicate. The standard deviation was calculated to assess the variability of the experimental results.

The percentage extraction (%E) of MB was determined using Eq. (2):

$$\%E = \frac{MB_{ext.1} - MB_{ext.2}}{MB_{ext.1}} \quad (2)$$

Where $MB_{ext.1}$ is the initial MB concentration in the aqueous phase while $MB_{ext.2}$ is the final MB concentration in the aqueous phase from the extraction process. The percentage stripping (%S) was determined using Eq. (3).

$$\%S = \frac{MB_{strip.1} - MB_{strip.2}}{MB_{strip.1}} \quad (3)$$

where $MB_{strip.1}$ is the initial MB concentration in the aqueous phase while $MB_{strip.2}$ is the final MB concentration in the aqueous phase from the stripping process.

2.5 FTIR Analysis

Fourier-transform infrared (FTIR) analysis was performed to determine the functional groups of PKO before and after MB extraction. The PKO samples were analysed using an attenuated total reflectance (ATR) technique. FTIR spectra were measured over the wavenumber range of 4000–400 cm^{-1} at a resolution of 4 cm^{-1} , with [16] scans performed at room temperature. The characteristic absorption bands of PKO, including those corresponding to aliphatic C–H stretching, C=O stretching, and C–O stretching, were examined. Particular attention was given to the 1650–1790 cm^{-1} region, which corresponds to the C=O stretching vibrations of carboxylic acid groups associated with the free fatty acid (FFA) content of PKO. This region was selected for detailed comparison of the spectra before and after methylene blue extraction to evaluate changes in the functional groups associated with the active component involved in the extraction process.

3. RESULTS AND DISCUSSION

3.1 Effect of pH on %E of MB

The pH of the aqueous solution is one of the main factors that significantly influence the %E of MB. The pH was varied from 4.6 to 8.5 and the results were presented in Fig. 2. The results show that %E increased from 97% to 99% as pH rose from 4.6 to 4.7. However, %E declined to 46% at pH 8 and 36% at pH 8.5. This observation indicated that MB extraction was more effective under acidic conditions, whereas alkaline conditions (pH 8.0–8.5) hindered its transfer into the organic phase. This result may be explained by the high FFA content of PKO (61 wt.%), as determined by the titration method described in Section 2.2. The high FFA content could enable PKO to act as an acidic extractant.

Generally, at pH levels above 3.5, FFAs undergo deprotonation, forming negatively charged carboxylate ions (COO^-) [17]. These COO^- ions electrostatically interact with the positively charged MB cations, facilitating their removal from the aqueous phase and subsequent transfer into the organic phase. However, at higher pH levels (8.0–8.5), %E decreases primarily due to soap formation resulting from the addition of NaOH for pH adjustment, as shown in Fig. 2. The presence of soap increases resistance to mass transfer, thereby reducing the efficiency of MB extraction from the aqueous to the organic phase [18].

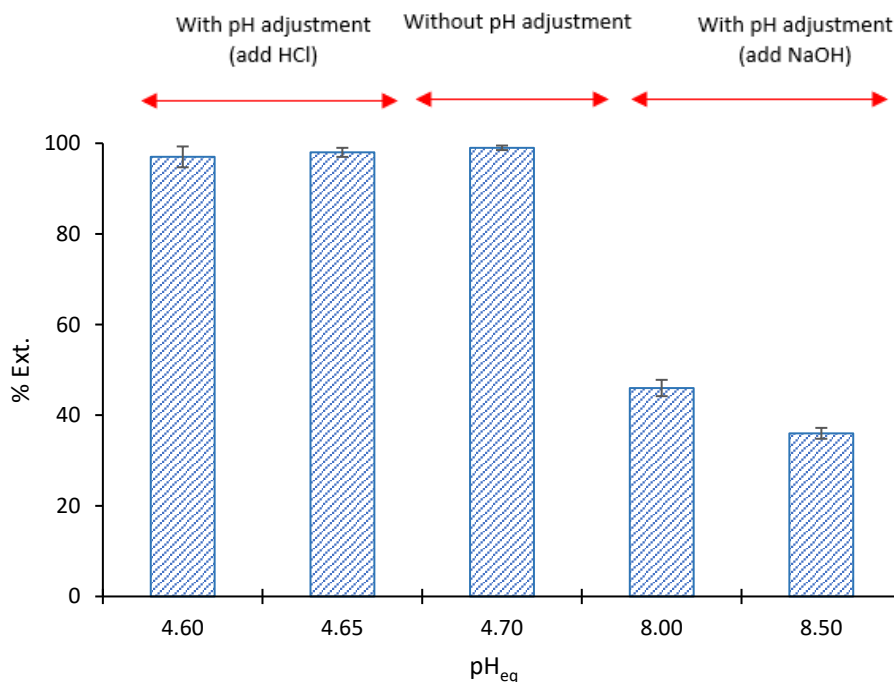


Fig. 2. Effect of pH on %E of MB

3.2 Effect of Organic to Aqueous Phase Ratio on %E of MB

The organic-to-aqueous (O: A) volume ratio is a significant parameter in solvent extraction, as it directly influences the mass transfer efficiency and phase equilibrium between the aqueous and organic phases. Investigating the O:A ratio can help identify the optimum solvent volume for maximum %E, which is important for cost-effectiveness and sustainability in large-scale applications. The O:A ratio was varied from 0.2 to 5, as shown in Fig. 3. At a low O:A ratio of 0.2, %E is relatively low at 40%, suggesting insufficient organic phase volume to facilitate the extraction of MB. As the O:A ratio increases to 0.5, %E improves to 60%, indicating enhanced mass transfer due to a greater availability of organic solvent for MB extraction. A further increase in O:A to 1.0 results in a substantial increase in %E to 99%. This improvement may be attributed to the greater availability of FFAs from PKO, which enhance MB extraction. Meanwhile,

at lower O:A ratios, the %E of MB was reduced due to the limited availability of FFA present in the solution. This finding emphasises the importance of optimising the O: A to balance the solvent consumption and efficiency of the MB removal. An O:A ratio of 1.0 was selected as the optimal condition because the maximum %E was achieved at this ratio while avoiding unnecessary solvent consumption. Similar results were also reported previously by other researchers [19].

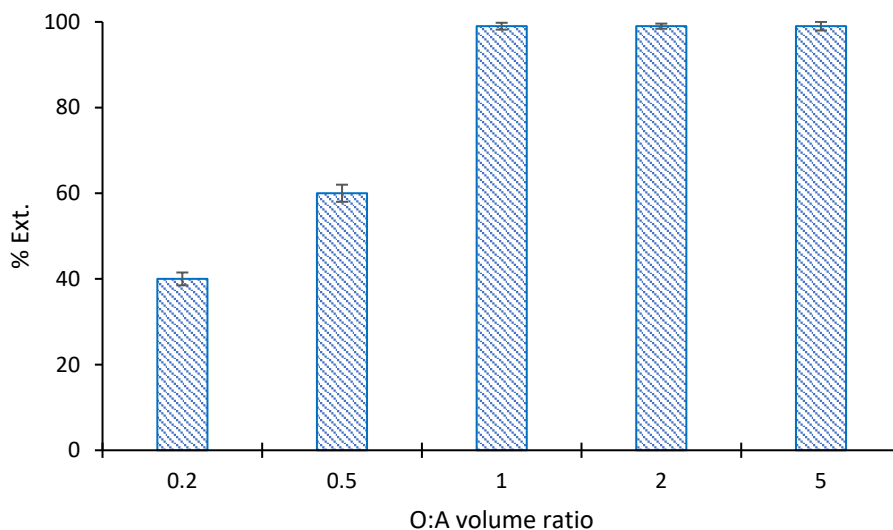


Fig. 3. Effect of O:A ratio on %E of MB

3.3 Effect of Initial MB Concentration on %E of MB

The concentration of MB in wastewater may vary depending on the source of pollution. Therefore, investigating the effect of the initial MB concentration is important for optimising MB removal efficiency, as variations in solute concentration can significantly influence the mass transfer driving force. Fig. 4 presents the effect of the initial MB concentration on the extraction efficiency (%E) of MB at various pH levels. Overall, the %E decreased with increasing pH. The highest %E, ranging from 63% to 78%, was obtained at pH 4.6–4.8. The %E gradually decreased as the pH increased, with a more pronounced reduction to below 30% at pH values above 8.5. The %E also increased with increasing initial MB concentration from 100 to 200 mg/L. This increase may be attributed to the greater concentration gradient between the aqueous and organic phases, which provides a higher driving force for mass transfer. However, at 300 mg/L, a slight reduction in %E was observed, suggesting a possible saturation effect, whereby the extraction capacity of the organic phase approaches its limit and consequently restricts further mass transfer of MB. These findings are consistent with the discussion in Section 3.1, which indicates that MB extraction is more favourable under acidic conditions and decreases under alkaline conditions. A similar trend was reported by Sipi and Chang [19], who found that %E decreased with an increase in the initial solute concentration

during the extraction of MB from an aqueous solution using waste cooking oil and tributyl phosphate as the organic solvent.

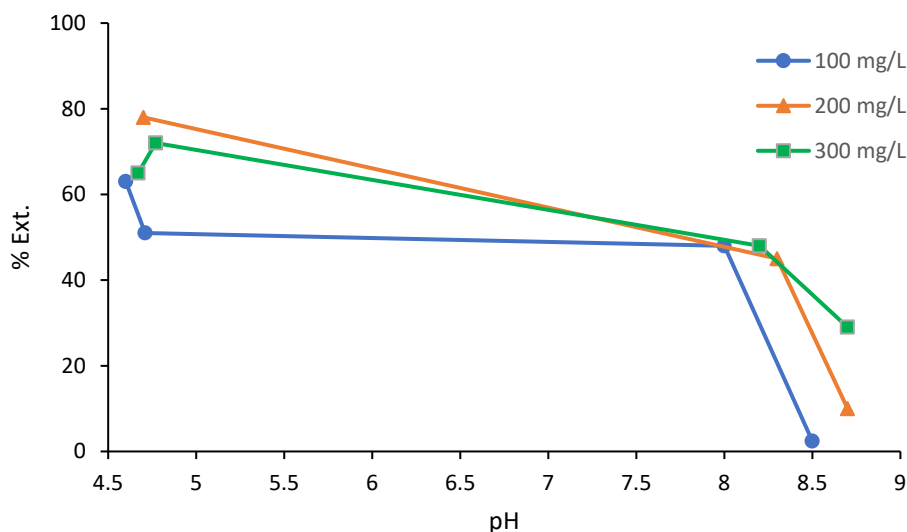


Fig. 4. Effect of initial MB concentration on %E of MB at various pH values

3.4 Stripping of MB-loaded PKO

Stripping, also known as back-extraction, is an important step in solvent extraction for solute recovery and enables the reuse of the organic solvent. Therefore, investigating the effect of the A:O phase ratio on the %S is important to determine the optimum amount of hydrochloric acid (HCl) required for effective MB recovery. Fig. 5 shows the effect of the A:O phase ratio on %S. The results showed that %S increased as the A:O ratio increased. Partial recovery of MB from the PKO phase was obtained at an A:O ratio of 1. Increasing the A:O ratio to 2 resulted in a substantial increase in %S to 84%, indicating improved transfer of MB from the organic phase into the aqueous phase. Further increasing the A:O ratio to 5 resulted in almost complete stripping, with a %S of 99%. This result indicates that a higher volume of HCl enhances the driving force for MB transfer into the aqueous phase. However, excessive use of the stripping agent may increase chemical consumption and adversely affect the economic and environmental performance of the process. Therefore, an A:O ratio of 2 was selected as the optimum condition, which considers both stripping efficiency and operational practicality.

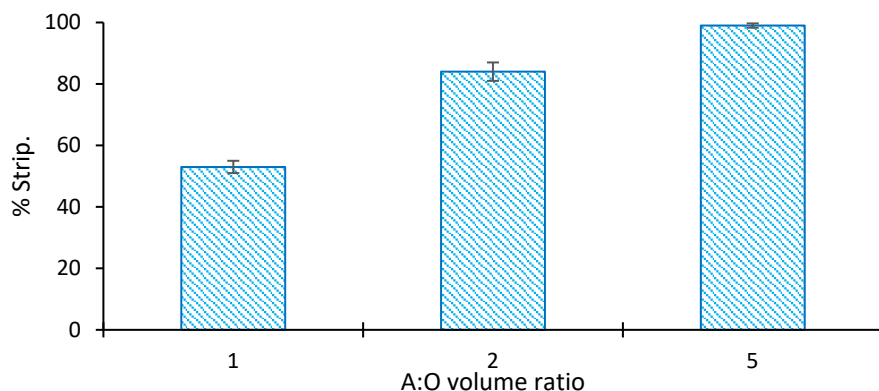


Fig. 5. %S of MB from MB-loaded PKO at various A:O ratios

3.5 Spectroscopic Evidence of MB Extraction into PKO

The presence of MB in the MB-loaded PKO was confirmed through Fourier-transform infrared (FTIR) spectroscopy. Two samples, pure PKO (before extraction) and MB-loaded PKO (after extraction), were analysed, and their respective transmittance spectra are presented in Fig. 6. A broad absorption band in the range of 1650 to 1790 cm^{-1} , attributed to the C=O stretching vibrations of carboxylic acid group (FFA) [20] was observed. This band exhibited greater intensity in the pure PKO sample, indicating a higher concentration of free carboxylic acid groups. In contrast, the reduced intensity in the MB-loaded PKO sample suggests interaction between the dye molecules and the FFA components. This interaction is likely facilitated by electrostatic attraction between the negatively charged carboxylate groups (FFA) in PKO and the cationic MB species. These spectral changes provide evidence for the presence of MB in the PKO phase.

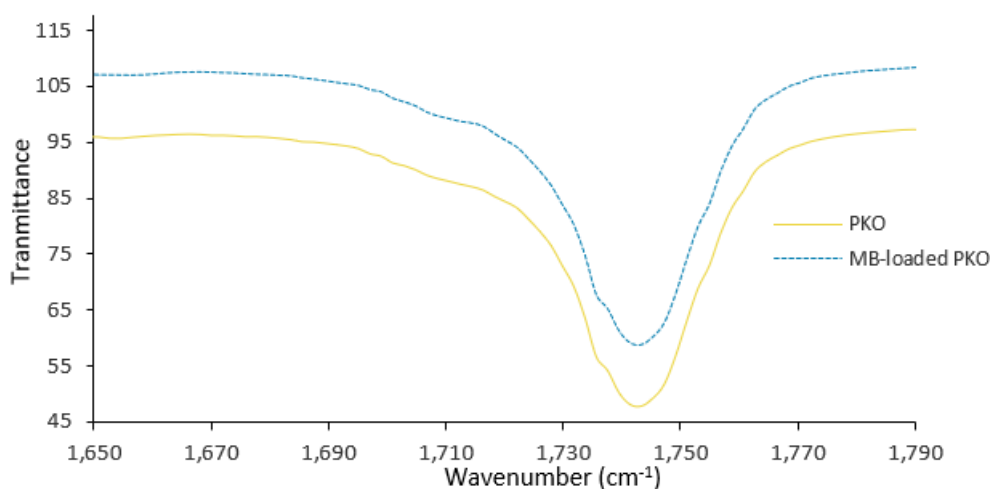


Fig. 6. FTIR Spectra of Pure PKO and MB-Loaded PKO

4. CONCLUSION

This study demonstrates the potential of PKO as a green organic solvent for the extraction of MB from aqueous solutions. The parametric study revealed that the highest %E of 99% was achieved at a pH of 4.6 with an organic-to-aqueous phase ratio of 1:1. The %E was also influenced by the initial MB concentration. In addition, the stripping process using HCl was successfully demonstrated, indicating the potential for PKO reuse in the extraction process. The significance of this study lies in demonstrating the feasibility of using PKO, a renewable plant-derived oil, as an alternative organic phase for MB extraction and in establishing the influence of the operating parameters on extraction performance. These findings provide a basis for further investigation of PKO-based solvent extraction systems for dyes and other solutes, including metal ions, under a wider range of operating conditions.

5. ACKNOWLEDGEMENTS AND FUNDING

This research was financially supported by Universiti Teknologi MARA, Malaysia, through the MyRA LPHD Research Grant (600-RMC/GPM LPHD 5/3 (074/2023)).

6. CONFLICT OF INTEREST STATEMENT

The authors declare that they have no personal, commercial or financial conflicts of interest related to this research or its funders.

7. AUTHORS' CONTRIBUTIONS

Muhammad Zahin Ikhwan Ariza Fattah: laboratory work and data collection; **Siti Fatimah Abdul Halim:** Conceptualisation, investigation, methodology, data collection, data analysis, original draft preparation, editing, and supervision; **Siu Hua Chang:** supervision, writing; **Nurulhuda Amri:** writing

8. DECLARATION OF GENERATIVE AI USE IN THE WRITING PROCESS

During the preparation of this work, the authors used ChatGPT to improve the language, grammar, and readability of the manuscript. After using this tool, the authors reviewed and edited the content as needed and take full responsibility for the content of the publication.

9. DATA AVAILABILITY AND SUPPLEMENTARY MATERIALS

The datasets used and/or analysed during the current study are available from the corresponding author on reasonable request.

10. ETHICS STATEMENT

This study did not involve the use of animals or animal-derived experimental subjects. Therefore, ethical approval for animal experimentation was not required.

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