Research Paper

Transport of Malachite Green Using the Polyeugenol-Based Polymer Inclusion Membrane (PIM) Method

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Article info

Keywords

Highlights

- Making PIM membrane
- MG transport studies: Variations of pH MG, HNO_3 concentration, membrane thickness, carrier concentration, and transport time
- Analytical characterization
- Results of Characterization (FTIR and SEM Characterization)

Graphical abstract
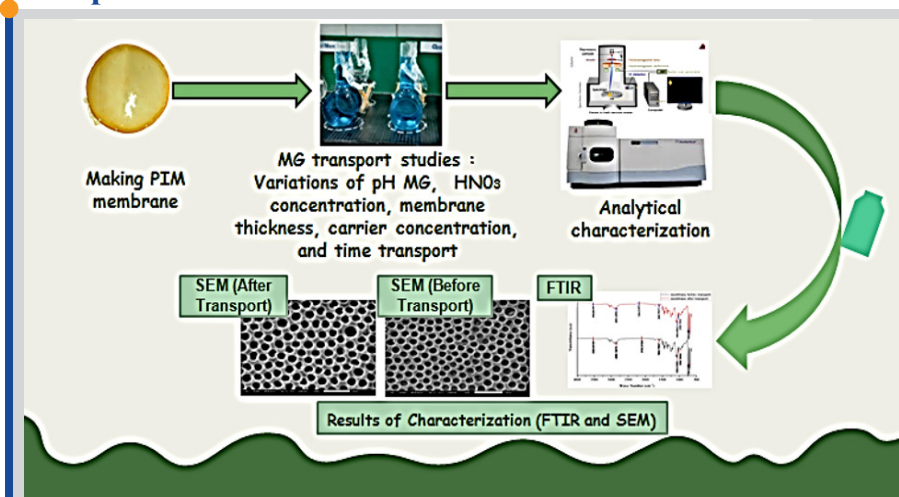
Abstract

This research aimed to optimize the reduction of Malachite Green (MG) dye waste using the Polyeugenol-based Polymer Inclusion Membrane (PIM) Method as a carrier. The carrier used polyeugenol. Mixing polyeugenol, dibenzyl ether (DBE), and polyvinyl chloride (PVC), and membrane preparation was carried out. The MG concentration after the transport process was measured using a UV-Vis spectrophotometer with 613 nm wavelength. The PIM before and after transport were characterized by SEM and FTIR. The research results showed that the optimum MG transport yield was 89.09% at a source phase MG pH of 7, HNO_3 concentration of 0.75M, concentration of carrier of polyeugenol 0.04 M, and transport time was 24 hours. The characterization SEM and FTIR assumed that components were lost during the transport process. Membrane loss results obtained at the optimum MG transport time for 24 hours were a 5% loss of the membrane component during the transport of the MG process.

1. Introduction

The dye industries sector has become the source of environmental pollution due to various industrial activities which have an impact on increasing industrial waste produced. Apart from polluting the environment, dyes can harm the environment, and biodiversity, and harm human health. Many industries use synthetic dyes because of the ease and cost-effectiveness of synthesis, color boldness, and light stability [1]. The textile industry is a source of waste in large quantities. Removing color from wastewater is a very urgent problem. Dyes are used in textiles and many other sectors of factories. Dyes are chemical compounds that are used to absorb or settle on the surface of a solution which can provide color. This molecule has the ability to stick to the surface of the fabric to provide a color effect. There are around 100,000 synthetic dyes, with annual production reaching more than 7×10^5 tons per year [2]. Dyes are the main component that can produce large amounts of wastewater which can have serious negative impacts on the environment and

human health. The textile industry is a large producer of wastewater containing organic and inorganic compounds. During the dyeing process in the textile industry, some of these dyes remain in high concentration. So it must be cleaned or changed color before the water is removed or recycled [3].

Malachite Green (MG) has become a topic of discussion because it can potentially harm the immune system and reproductive system and has genotoxic and carcinogenic properties. The presence of MG in water can cause serious health impacts such as mutagenesis, teratogenicity, chromosomal abnormalities, respiratory toxicity, and carcinogenesis, which are highly dependent on exposure time, temperature, and dye concentration [1]. MG is used in the textile industry as a dye and in the food industry as a dye. MG is a triphenylmethane dye, very difficult to decompose because it is synthetic in origin and cannot be biodegraded [3]. Moreover, MG exhibits high levels of persistence and contributes to atmospheric pollution [4].

Therefore, special treatment of this dye is very important to overcome the environmental problems it causes because the MG content is very dangerous. The presence of MG dye can be removed from waters using various techniques, such as precipitation, flocculation, adsorption, ion exchange, and the use of liquid membranes [5]. One of the effective methods for dealing with MG waste is using liquid membrane-based separation technology. There are several main types of liquid membranes, including bulk liquid membranes (BLM), emulsion liquid membranes (ELM), supported liquid membranes (SLM), and polymer inclusion membranes (PIM) [1]. A liquid membrane technique known as the Polymer Inclusion Membrane (PIM) method has emerged as a more stable option compared to other liquid membrane approaches to separate and purifying phenol compounds from wastewater with different carriers based on Kiswandono et al. 2019 [6]; Saka et al. 2020 [7]; Kiswandono et al. 2019 [8]; Benosmane et al. 2018 [9]; Kiswandono et al. 2013 [10]. The enhanced stability of the PIM method can be attributed instead to two main factors. Firstly, it involves the incorporation of a fundamental polymer (polyvinyl chloride-PVC), which is anticipated to mitigate carrier loss [11-14]. The PIM method in using liquid membranes is widely chosen because it has broad and selective separation capabilities, is simple in application, requires little energy, can extract metals and non-metals selectively [15], reduces the use of chemicals, and has a membrane composition flexible [16].

A study conducted by Sun et al., (2017) [17] showed that PIMs are an environmentally friendly method because they do not use a lot of solvents and use relatively few carriers. Polymer requirements must have a large molecular weight, lipophilic properties, and structures that allow the structure of bonds, interactions, and complexes for compounds being transported. Eugenol is a natural material that has the potential can be used as a polymer. Eugenol is a chemical found in clove leaf oil. This substance can undergo a polymerization process to become polyeugenol and various derivative compounds. The result of polyeugenol has properties for the requirements of a membrane because it has a high molecular weight, contains active -OH groups, and has a benzene ring structure.

2. Materials and Methods

2.1. Tools and Materials

Tools used in this research consisted of a magnetic stirrer and bar, analytical digital (Galaxy HR-150A), drop pipette, 3.5 cm diameter chamber with a capacity of 50 mL, pH meter (Water Taster EZ-9901), measuring cup, spatula, beaker, aluminum foil, tissue, stirring rod thickness gauge (Mitutoyo 7301 Dial Thickness Gauge), measuring flask, membrane mold, tissue, Fourier Transform Infrared (FTIR) (JSM 6360LA), and Scanning Electron Microscopy (SEM) (ZEISS EVO MA 10).

Materials used in this research consisted of malachite green (329.5 g/mol), polyeugenol (164.20 g/mol), distilled water, aquabides, polyvinyl chloride (PVC) (1.1-1.35 g/cm^3), dibenzylether (DBE) (198.28 g/mol), tetrahydrofuran (THF) (72.12 g/mol), HNO$_3$ 63 g/mol, PbCO$_3$ (267 g/mol), and Cu(NO$_3$)$_2$ (187,5 g/mol).

2.2. Methods

2.2.1. Determination of Maximum Wavelength MG

Before measuring the MG concentration, the maximum MG wavelength of 25 ppm was analyzed using a UV-Vis spectrophotometer in the wavelength range between 400 nm to 800 nm.

2.2.2. p variations in the MG Source Phase

The PIM was placed with normal thickness in the middle of the transport pipe. Then, 50 mL of 1 M HNO$_3$ solution was added as the receiving phase, and 25 ppm MG solution (source phase) 50 mL. Vary the pH of MG to 5; 6;

7, 8, and 9. The transport pipe was closed and stirred for 24 hours using a magnetic stirrer. After the transport process was complete, 5 mL samples were taken from the source phase and receiver phase. The UV-Vis spectrophotometer examined the concentration of MG in the source and receiving phases, utilizing the maximum wavelength for analysis. The transport process can be seen in Fig. 1.
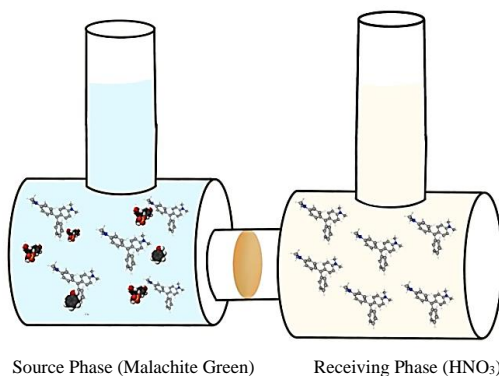

Fig.1. MG transport process

2.2.3. Variation of HNO$_3$ in the Receiving Phase

The normal thickness PIM was placed in the middle of the transport pipe. Then, HNO$_3$ solution (receiving phase) of 50 mL was added, and 25 ppm MG solution (source phase) with maximum pH. Variation of HNO$_3$ concentration in the receiving phase was 0.50; 0.75; 1.00; 1.25; and 1.50 M. The transport pipe was closed and stirred for 24 hours using a magnetic stirrer. After the transport process was completed, 5 mL samples were taken from the source phase and receiving phase. Both liquids (source and receiving phase) after transport were measured by UV-Vis spectrophotometer with maximum wavelength to know the concentration of both liquids.

2.2.4. Membrane Thickness Variations

PIM with varying thicknesses of thin (Th), normal (No), and thick (Tc) which had been placed in the middle of the transport pipe. The composition of the membrane thickness can be seen in Table 1. Next, 50 mL of HNO$_3$ solution (receiving phase) with optimum concentration was added, and 25 ppm MG solution 50 mL (source phase) with optimal pH. The pipe of transport was closed and stirred for 24 hours using a magnetic stirrer. After the transport process was completed, 5 mL samples were taken from the source phase and receiver phase. Both liquids (source and receiving phase) after transport were measured by UV-Vis spectrophotometer with maximum wavelength to know the concentration of both liquids.

Table 1
Comparison of polyeugenol-based membrane-forming components

Membrane	Polyeugenol (g)	Polyvinyl Chloride (g)	Dibenzylether (g)	Weight of Total (g)
Thin (Th)	0.027	0.087	0.156	0.27
Normal (No)	0.054	0.173	0.313	0.54
Thick (Tc)	0.108	0.346	0.626	1.08

2.2.5. Variation in Concentration of Carrier (Polyeugenol)

The normal thickness PIM was placed in the middle of the transport pipe. The variation in carrier is 0.01; 0.02; 0.03; 0.04; and 0.05M. Next, 50 mL of the optimum HNO$_3$ solution was added as the receiving phase, and 50 mL of the 25 ppm MG solution as the source phase with optimal conditions. The transport pipe was closed and stirred for 24 hours using a magnetic stirrer. After the transport process was complete, 5 mL samples were taken from the source phase and receiver phase. Both liquids (source and receiving phase) after transport were measured by UV-Vis spectrophotometer with maximum wavelength to know the concentration of both liquids.

2.2.6 Variations in Transport Time

The normal thickness PIM was placed in the middle of the transport pipe. Added HNO$_3$ 50 mL (receiving phase) solution with optimal concentration, and 25 ppm MG solution (the source phase) 50 mL with optimum pH. The

pipe of transport was closed and stirred for various durations of 6, 12, 18, 24, and 30 hours using a magnetic stirrer. After the transport process was complete, 5 mL samples were taken from the source phase and receiver phase. Both liquids (source and receiving phase) after transport were measured by UV-Vis spectrophotometer with maximum wavelength to know the concentration of both liquids.

2.2.7. Characterization of PIM

The characterization of PIM consisted of FTIR and SEM for membranes before and after transport.

3. Results and Discussion

3.1. pH Variations in the MG Source Phase

pH was a crucial parameter in this research because diffusion could occur in MG transport due to the difference in proton concentration in the source and receiving phase. Variations in the MG source phase of pH consist of pH 5, 6, 7, 8, and 9. It means this variation showed that the number of protons in the source phase would increase further. The MG transport process was carried out with the source phase containing 25 ppm MG solution while the receiving phase contained 1M HNO_3 solution and was carried out at 24 hours for the transport time. The solution after transport was analyzed using a UV-Vis Spectrophotometer to determine the concentration of the source phase and receiver phase.

This optimum condition for MG pH variations was obtained at pH 7. Based on the results, the %removal transported was 85.63% with %Cp as large as shown in Fig. 2. The hypothesis of an optimum condition of pH 7 could be caused by MG being in a molecular form which allowed a greater number of hydrogen bonds to occur. In addition, the tendency of the target compound to dissolve in the organic phase would increase the π-π interactions and hydrogen bonds between the target compound (MG) and the carrier in the membrane phase. This increased interaction causes the target compound (MG) to be transported to increase [18].
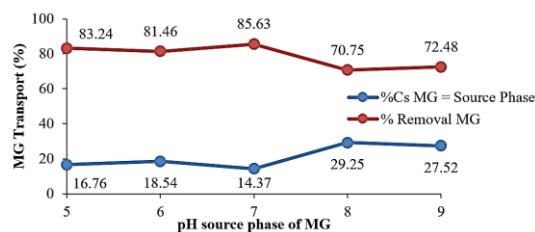
Fig. 2. Effect of MG pH on %MG transported

3.2. Variation of HNO3 Concentration in the Receiving Phase

Concentration variations carried out in the MG transport process used an optimum pH of 7 with a transport time of 24 hours. The concentration variations were used 0.50; 0.75; 1.00; 1.25 and 1.50 M. If the concentration of the receiving phase, namely HNO_3, was higher, it was hoped that it would have a greater influence on the MG transport process so that it could increase the percent of MG transported to the receiving phase.

This optimum condition for HNO_3 concentration in the receiving phase was an HNO_3 concentration of 0.75M with %removal of 86.29% with %Cs MG of 13.71%. This optimum condition occurs because the release of nitrate ions NO_3^- and hydronium ions (H_3O^+) in the receiving phase is equivalent to the release of MG into the receiving phase. The transport results of this variation are shown in Fig. 3.

Research by Ling and Suah (2017) [1], the concentration of HNO_3 in the receiving phase was 1M which resulted in an extraction percentage of >96% using the carrier compound bis-(2-ethylhexyl) phosphate (B_2EHP) using the PIM method. Based on this variation, the greater the concentration of the HNO_3 receiving phase, the more difficult it was for the target compound (MG) to be transported to the receiving phase because its acidic nature is greater, so the release of the target compound (MG) was not proportional to the NO_3^- nitrate ion and hydronium ion (H_3O^+) in the receiving phase.
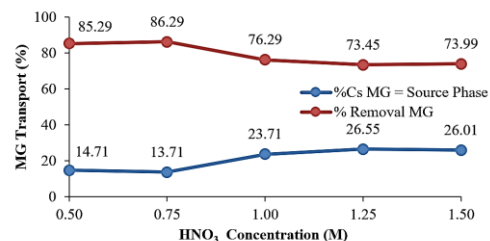
Fig. 3. Effect of HNO_3 concentration on %MG transported

3.3. Variations in Membrane Thickness

Variations in membrane thickness were expected if there was a lot of carrier in the membrane, namely –OH groups, the baser polymer (PVC), and DBE as a plasticizer, it was hoped that the transport process would be more effective. PIMs were made with three thickness variations, namely thin (Th), normal (No) and thick (Tc). This variant of transport uses optimum MG pH conditions of 7, HNO_3 concentration of 0.75M with a transport time of 24 hours.

The optimum conditions for variations in membrane thickness obtained were normal thickness with membrane thickness: 0.24 mm with %removal of 88.19% and %Cp of 11.81%. The optimum results are shown in Fig. 4. The optimum conditions for normal thickness were obtained because the number of MG molecules that interacted with the carrier compound was very effective so the resulting membrane was neither too thick nor too thin.
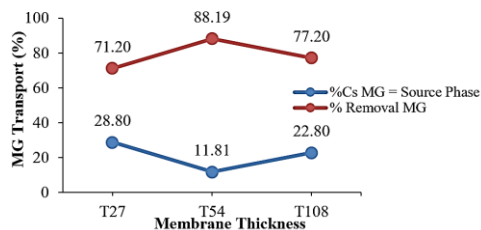
Fig. 4. Effect of membrane thickness on %MG transported

3.4. Variations in Polyeugenol Concentration as a Carrier

Variations in the carrier (polyeugenol) to determine the effect of the carrier (polyeugneol) in the liquid membrane transport process on MG compounds. Variations in the concentration of polyeugenol carrier used were 1×10^{-2}, 2×10^{-2}, 3×10^{-2}, 4×10^{-2}, and 5×10^{-2}M. The increasing concentration of polyeugenol was expected to cause the amount of MG transported to be greater. MG transport was carried out with a chamber containing the source phase, namely 50 mL MG with an optimum pH of 7, and the receiver phase containing HNO_3 with an optimum concentration of 0.75 M using a normal membrane thickness for 24 hours.

The optimum results of MG transport at varying carrier concentrations, namely at a concentration of 4×10^{-2} M, obtained a %removal of 88.93% with a %Cp of 11.07%. The results of the transport of various carriers can be seen in Fig. 5. The ooptimum conditions were obtained because the number of polyeugenol particles increased, meaning there were more –OH active sites, so that the MG-polyeugenol interaction hydrogen bonds increased, it was polyeugenol concentration of 0.04M.
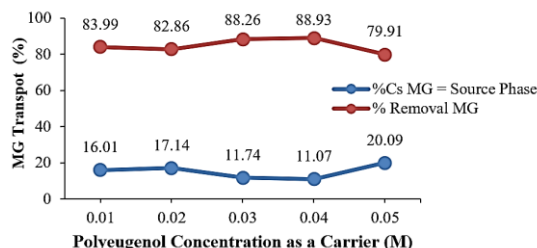
Fig. 5. Effect of polyeugenol carrier concentrations on %MG transported

3.5. Variations in Time Transport

Time had an effect on MG transport which was directly related to the length of interaction between MG in the source phase and the carrier on the membrane which would occur through hydrogen bonds and π-π interactions. This transport process was carried out under optimum conditions with a pH of MG 7, an HNO$_3$ concentration of 0.75M, a membrane with normal thickness, and a carrier concentration of 0.04M. The variations in transport time used were 6, 12, 18, 24 and 30 hours. Variations in transport time were expected, the longer interaction contact time between MG and the carrier on the membrane would increase the transport results (%removal).

The optimum results for MG transport at varying times, namely 24 hours of transport time, obtained a %removal of 89.09% and a %Cp of 12.78%. The results of the variation in transport time are shown in Fig. 6. This is supported by the optimum conditions of concentration of the source phase, receiver phase, membrane thickness, and carrier which interacted to form hydrogen bonds, and the π interaction occurs effectively.
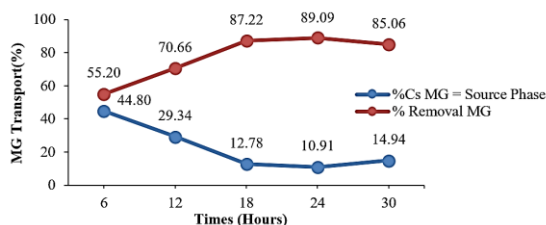

Fig. 6. Effect of polyeugenol transport time on % MG transported

3.6. Liquid Membrane Loss (LM Loss)

LM loss was the difference between the weight of the PIM before and after MG transport. Determination of LM loss was carried out to determine the amount (%) difference in weight of the PIM-containing polyeugenol carrier. The percentage of LM loss results would be used as a parameter for the stability and durability of a membrane. The durability of the membrane was assumed be have the ability to withstand leaks and the ability of the membrane to be used repeatedly. The greater the LM loss value indicated that more membrane components were lost.

The lost membrane component could be a carrier, namely polyeugenol, PVC as the base polymer, or DBE as a plasticizer. The increasing amount of LM loss would cause a decrease in the percentage of MG that was transported to the receiving phase. Fewer active sites of polyeugenol on the membrane result in less MG being transported to the receiving phase. The result of ML loss meant that in the transport process, there were components that had been lost so that the cross-section and surface of the membrane became porous. The LM loss results obtained by MG under conditions of optimum pH variation MG 7, HNO$_3$ variation 0.75M, normal thickness, polyeugenol concentration 0.04M, and 24-hour transport can be seen in Fig. 7.
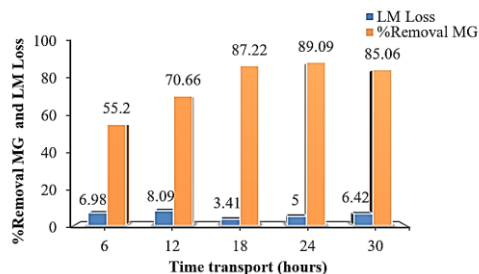

Fig. 7. LM Transport Loss MG

3.7. PIM Characterization

The PIM before transport and after transport was characterized by FTIR and SEM. FTIR characterization to determine the hydroxy functional groups (-OH) contained in the PIM. Characterization of the PIM before and after transport can be seen in Fig. 8.
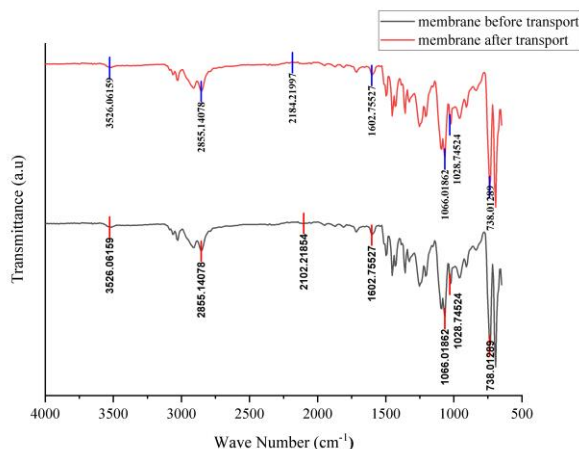

Fig. 8. Membrane FTIR results before and after transport

Fig. 8 showed the results of a comparison of the membrane before and after transport, that the wave number is 3526 cm^{-1}, namely the absorption of the hydroxyl group (-OH) on the membrane before transport for a wave number shift which indicated there was an interaction between groups in MG and the -OH groups in the carrier in the membrane during transport time. There was a C-O strain that before and after transport produced a wave number of 1028.7 cm^{-1}. Apart from that, there is a shift in the C≡C strain group where before transport it produced a wave number of 2102.2 cm$^{-1,}$ and after transport, it produced a wave number of 2184 cm^{-1}. The wave number of the -OH group could occur because some of the active sites in polyeugenol on the membrane were lost during the transport process. The loss of active sites on the membrane after transport could be seen in the results of the PIM characterization using SEM. The results of SEM characterization at 500x can be seen in Fig. 9.
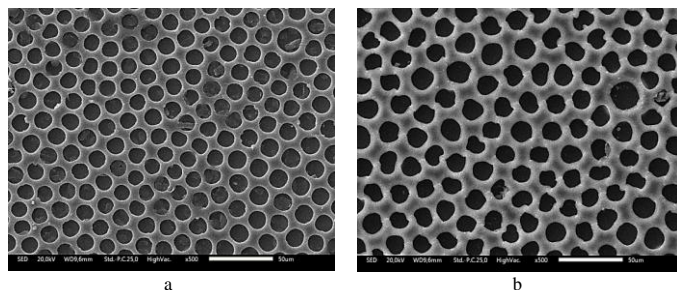

Fig. 9. PIM magnification 500x (a) before transport and (b) after transport

In the membrane before transport (Fig. 9a) at 500x magnification the surface of the membrane is still covered by plasticizer as the liquid medium, meaning that the membrane pores look homogeneous. The PIM after MG transport (Fig. 9b) with a magnification of 500x has an uneven surface and the membrane had pores, meaning that during transport, membrane components were lost (leaching). The use of membranes for transport allows leaching of the membrane components to occur. Leaching membrane components would cause the surface of the PIM to become porous and non-homogeneous. Leaching components could come from the loss of carriers, base polymers, or plasticizers.

4. Conclusions

The conclusions in this research consist of:
1. MG transport was obtained in an optimum condition with a pH of the MG source phase of 7, an optimum receiver concentration of the HNO$_3$ 0.75 M with normal thickness using polyeugenol concentration 0.04 M and 24 hours transport with the amount of MG transported from membrane phase (%removal) was 89.09%.
2. The results of the characterization SEM of the PIM after transport had a smooth surface and the membrane had pores because there were membrane components that were lost during the transport

process (leaching). This was proven by tests of qualitative FTIR which showed a shift in the absorption band and a decrease in intensity.

Data availability

Data are available upon request.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

CRedit authorship contribution statement

A.A. Kiswandono: Conceptualization; Supervision; Methodology; investigation, data curation, writing original draft, review and editing.
A.V. Sindiani: Data curation, writing manuscript and editing
R.Kh. Khotimah: Investigation; Methodology
M.B. Rabbani: Formal analysis; Project administration
B. Kurniawan: Investigation; Project administration
R. Rinawati: Data curation; writing - Review and editing
H E. Putra: Review and editing

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