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Research Article

Effect of Reaction Conditions on the Transesterification Reaction Between Methyl Hexanoate and Trimethylolpropane Using SrO/MgO–CaO Catalysts

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ABSTRACT

Introduction: Petroleum resources are non-renewable energy sources that pose significant environmental risks. The transesterification of vegetable oil into biolubricants is recognized as a potential alternative to mineral-based lubricants. In this study, a model reaction between methyl hexanoate (representing diesel derived from vegetable oil; MH) with trimethylolpropane (TMP) was investigated using heterogeneous SrO/MgO–CaO catalysts to determine the optimal reaction conditions for the production of biolubricants from diesel.

Method: SrO/MgO–CaO catalysts were synthesized using a co-precipitation method and analyzed using X-ray diffraction. The transesterification of MH and TMP was evaluated in a batch reactor under different reaction conditions. The products were analyzed using gas chromatography (GC) and gas chromatography–mass spectrometry (GC–MS).

Results: The conversion of MH increased with reaction temperature, while the conversion and yield of diesters and triesters were greatest at a catalyst loading of 3 wt.%, a MH:TMP molar ratio of 3:1, and a reaction time of 3 h. SrO/MgO–CaO (NH_4OH) and SrO/MgO–CaO (KOH, K_2CO_3) catalysts achieved MH conversions exceeding 76%, while the SrO/MgO–CaO (NH_4OH, K_2CO_3) catalyst exhibited the greatest diester and triester selectivity (>93%).

Conclusions: SrO/MgO–CaO catalysts were successfully synthesized and demonstrated high diester and triester yields of 65–68% following the transesterification reaction between MH and TMP. The high yield of the transesterification reaction highlights the strong potential of SrO/MgO-CaO catalysts in transesterification reactions, such as the production of biolubricants from biodiesel.

Key words: Lubricant, methyl hexanoate, SrO/MgO–CaO, transesterification, trimethylolpropane

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INTRODUCTION

Lubricants are often referred to as the "blood" of mechanical systems due to their essential role in reducing friction, wear, and heat generation in machines. By forming a protective film between contacting surfaces, lubricants reduce friction and facilitate the cooling and cleaning of these surfaces, thereby reducing wear and potential damage to the moving parts[1]. Conventional lubricants are predominantly derived from crude oil. However, increasing environmental concerns and the limited nature of fossil resources have increased the demand for sustainable and environmentally friendly alternatives[2].

Biolubricants, although not as widely used as mineral oil–based lubricants, have gained increasing attention in recent years due to extensive research and development efforts[3]. Among renewable feedstocks, vegetable oils—naturally derived from plant sources—have emerged as promising candidates. In particular, palm oil has demonstrated significant potential due to its high availability, favorable fatty acid composition, and relatively low production cost, making it an attractive base material for biolubricant synthesis[4,5].

Biolubricants can be synthesized from biodiesel, which is typically produced through the transesterification of triglycerides with monohydric alcohols (e.g., methanol) in the presence of a catalyst[6,7]. Biolubricants can also be produced through the transesterification of biodiesel (methyl esters) with polyhydric alcohols such as glycerol in a process that is effectively the reverse of biodiesel synthesis. First, a methyl ester reacts with one hydroxyl group in a polyol to form a monoester (ME) and methanol. A second methyl ester then reacts with the remaining hydroxyl group to produce a diester (DE) and methanol, followed by a third reaction yielding a triester (TE) and yet another methanol molecule.

Common polyols used in transesterification reactions include trimethylolpropane (TMP), neopentyl glycol (NPG), and pentaerythritol (PE). In this study, TMP

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was selected as the polyol of choice due to its physicochemical properties. TMP provides a balanced combination of viscosity, thermal stability, and oxidative stability comparable to those of PE- and NPG-based biolubricants, while achieving higher overall stability. In addition, TMP exhibits a relatively low melting point (58 °C) and a high boiling point (298 °C), compared with the higher melting points of NPG (129 °C) and PE (260 °C), making it more practical for processing and other applications[8].

Although homogeneous catalysts often demonstrate high catalytic efficiency, their use is associated with significant practical and environmental challenges. In particular, the separation of homogeneous catalysts from reaction mixtures is difficult; consequently, these catalysts are generally not recoverable, limiting their reuse and contributing to waste generation[9]. In accordance with green chemistry principles, heterogeneous catalysts represent a more sustainable alternative. These catalysts allow for simpler and more cost-effective separation, greatly minimize waste production, and can be reused over multiple reaction cycles, thereby improving the overall sustainability and efficiency of the process[10].

A wide range of solid base catalysts has been investigated, including mixed metal oxides (SiO_2–MgO, SiO_2–CaO, MgO–Al_2O_3, MgO–La_2O_3), alkali or alkaline earth oxides supported on zeolites, and alkali compounds (KF, K_2CO_3, NaOH, and KOH) supported on materials such as anion exchange resins, zeolites, clays, and phosphates[11]. These catalysts are particularly effective for the transesterification of high-purity oils with low free fatty acid (FFA) content and generally exhibit higher catalytic activity than acid-based catalysts[12].

In this study, the SrO/MgO–CaO (SMC) catalyst was selected due to the synergistic reactions between its strontium, magnesium oxide, and calcium oxide components. Although Ca-based catalysts are simpler, less expensive, and more widely available than Sr-based systems, their catalytic activity is lower than that of Sr-based systems[13]. Furthermore, SMC exhibits higher activity compared with other alkaline earth oxides, such as BaO, which is both toxic and highly soluble in methanol[14]. Consequently, the incorporation of SrO, MgO, and CaO offers a promising cost-effective, efficient, and sustainable heterogeneous catalyst for biolubricant production. Methyl hexanoate and trimethylolpropane were employed as model compounds for the reaction between biodiesel and polyols, respectively.

MATERIALS AND METHODS

Materials

Trimethylolpropane (98%), n-dodecane ($\geq$ 95%), ethanol (99.8%), ammonium hydroxide (99%), magnesium nitrate hexahydrate (99%), potassium carbonate (99.5%), and potassium hydroxide (99%) were purchased from Fisher Chemical (USA). Calcium nitrate tetrahydrate (99%), phosphoric acid (99%), and methanol (99.9%) were purchased from Xilong Scientific Co., Ltd. (China). Strontium chloride hexahydrate (99%) was supplied by Zhanyun Chemical Co., Ltd. (China). Methyl hexanoate (99%) was obtained from Aladdin Bio-Chem Technology Co., Ltd. (China).

Catalyst Synthesis

The SrO/MgO–CaO catalyst was prepared by a co-precipitation method, modified from a previously reported procedure[15]. A catalyst with a Sr:Ca:Mg molar ratio of 0.15:0.25:1 was synthesized using both NH_4OH and K_2CO_3 as precipitating agents. Solution I was prepared by dissolving 2.6662 g $SrCl_2\cdot 6H_2O$, 3.9538 g $Ca(NO_3)_2\cdot 4H_2O$ and 17.0933 g $Mg(NO_3)_2\cdot 6H_2O$ in 50 mL of deionized (DI) water. Solution II was prepared by dissolving 18.0594 g K_2CO_3 in 50 mL DI water. Solution II was then gradually added to Solution I under continuous stirring at 1000 rpm while maintaining a temperature between 60– 70 °C. The pH of the mixture was adjusted to 11 using 2M NH_4OH. The resulting suspension was stirred for 2–3 h and subsequently aged at 60 °C for 20 h. The mixture was filtered, washed with DI water until it reached a neutral pH, then dried at 110 °C for 24 h. Finally, the mixture was calcined at 1000 °C for 5 h at a heating rate of 5 °C/min. The resulting catalyst was denoted as SrO/MgO–CaO (NH_4OH, K_2CO_3). Catalysts synthesized using the same procedure but using NH_4OH as the sole precipitating agent were denoted as SrO/MgO–CaO (NH_4OH), while those synthesized using KOH and K_2CO_3 (with a KOH/K_2CO_3 molar ratio of 0.3) were denoted as SrO/MgO–CaO (KOH, K_2CO_3).

Experimental method

A total of 4 g of methyl hexanoate (MH) was introduced to a 100 mL round-bottom flask together with TMP, n-dodecane (an internal standard), and the catalyst. The reaction system was connected to a reflux condenser and a vacuum pump to maintain a low-pressure, airtight environment. The reactor was placed in a thermal oil bath to control the reaction temperature and stirred with a magnetic stirrer to ensure homogeneity. Upon completion of the reaction, the reactor was removed from the oil bath and left to cool, after which the products were stored in Falcon tubes. The sampled solutions were then analyzed by gas chromatography (GC) to determine conversion and selectivity, and gas chromatography–mass spectrometry (GC–MS) was used to identify the products.

The products were analyzed using an Agilent Technologies 7890A gas chromatograph equipped with an Agilent FID detector and an HP-5 column (30 m × 0.320 mm × 0.25 μm). Nitrogen was used as a carrier gas. The oven temperature program was as follows: 35 °C (2 min hold), 25 °C/min ramp rate until 280 °C, followed by a 10 min hold. The injector temperature was set at 275 °C, the detector temperature was set at 300 °C, and the split ratio was 50:1 with a 2 μL injection volume. The cumulative run time was 21.8 minutes. Acetone and methanol (three times each) were used to clean the syringe before and after every run. Before injection, the samples were centrifuged at 13,300 rpm for 5 minutes to remove the catalyst, and the clear phase was dissolved in ethanol. MH conversion was estimated based on the equation below:

$$X\% = \frac{m_{MH\ initial} - m_{MH\ after}}{m_{MH\ initial}} \times 100\%$$

The selectivity of ester products was calculated using the following formulas:

$$Selectivity(ME) = \frac{Area(ME)}{\sum Area\ of\ all\ products} \times 100\%$$

$$Selectivity(DE) = \frac{Area(DE)}{\sum Area\ of\ all\ products} \times 100\%$$

$$Selectivity(TE) = \frac{Area(TE)}{\sum Area\ of\ all\ products} \times 100\%$$

The yield was determined by combining conversion and selectivity, calculated as follows:

$$Yield = \frac{X\% \times Selectivity}{100}$$

Characterization

XRD study

Powder X-ray diffraction (XRD) was conducted using a Shimadzu 6100 diffractometer (Japan) operated at 40 kV and 30 mA with CuKα radiation (λ = 0.15406 nm). The scan rate was 0.05 °/s, and the step size was 0.02° over a 2θ range of 10°–70°. The crystallite size was estimated using the Scherrer equation:

$$d = \frac{K\lambda}{\beta\cos(\theta)}$$

where d is the average crystallite size (nm), K is the shape factor (K = 0.94), λ is the X-ray wavelength (λ = 0.15406 nm), β is the full width at half maximum (FWHM) of the diffraction peak (rad), and θ is the Bragg angle corresponding to the peak position (rad).

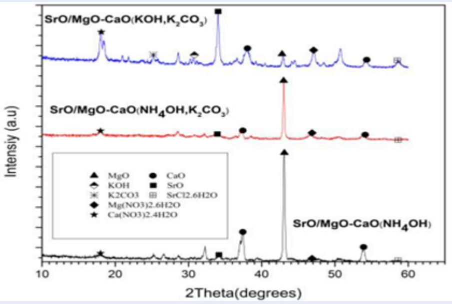


Figure 1: The XRD spectra of the SrO/MgO-CaO catalysts.

RESULTS

XRD analysis

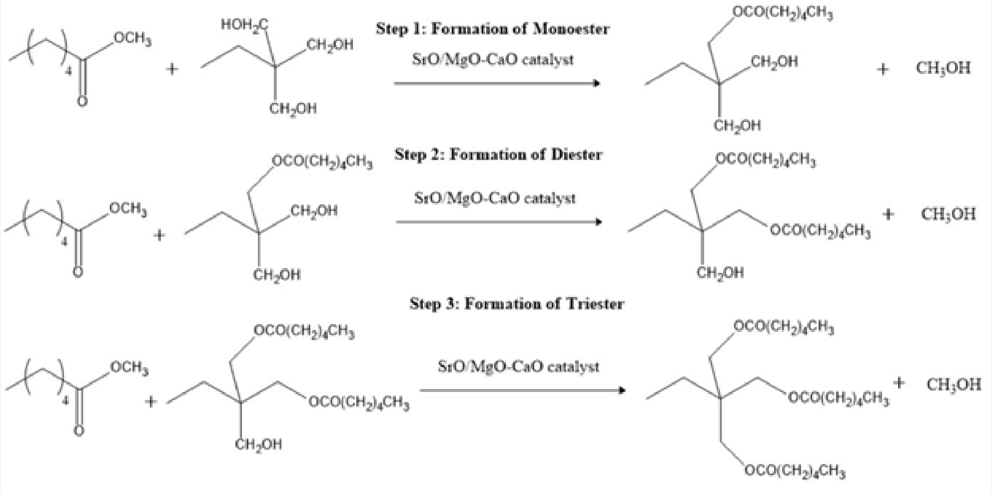
The XRD spectra of all catalyst samples exhibited distinct crystalline peaks associated with SrO, MgO, and CaO at 2θ angles of 34.454°, 42.475°, and 37.158°, respectively. An additional CaO peak was observed at 2θ = 53.562° (Figure 1)[16]. The most intense diffraction peak was observed at 2θ = 33.2°, indicating a high degree of crystallinity in the synthesized materials. SrO/MgO–CaO (KOH, K_2CO_3) catalyst exhibited residual peaks that were related to unreacted precursors. The presence of these impurities implies the incomplete removal of salts during synthesis. In contrast, the SrO/MgO–CaO (NH_4OH, K_2CO_3) did not exhibit any impurities, indicating more effective washing and filtration of unreacted species. SrO/MgO–CaO (NH_4OH) also exhibited a low level of contamination, likely due to the ease of removing NH_4OH throughout washing and heating. Table 1 shows that the crystal sizes of SrO and MgO in the SrO/MgO–CaO (NH_4OH, K_2CO_3) and SrO/MgO–CaO (NH_4OH) catalysts were smaller than those of the SrO/MgO–CaO (KOH, K_2CO_3) catalyst. These findings suggest that the choice of precipitating agent and washing procedure plays a critical role in determining the crystalline structure and, consequently, the catalytic performance of SrO/MgO–CaO systems.

Identifying the Products of the Transesterification Reaction

The transesterification of MH and TMP was studied using SrO/MgO-CaO catalysts under a range of conditions. The reaction occurs in three steps (Scheme 1), beginning with the reaction of MH with the first hydroxyl group of TMP, followed by the reaction of MH with subsequent hydroxyl groups until the formation of triesters. The ester products were identified using

Table 1: **Average crystal sizes of SrO, MgO, and CaO.**

Crystal phase	Crystal size (nm)		
	Precipitating agent: KOH, K_2CO_3	**Precipitating agent: NH_4OH, K_2CO_3**	**Precipitating agent: NH_4OH**
SrO	24.4	9.5	9.5
MgO	50.3	39.2	39.2
CaO	11.9	19.4	19.4



Scheme 1: Reaction scheme showing the transesterification of methyl hexanoate with trimethylolpropane in the presence of a SrO/MgO–CaO catalyst.

GC-MS data (Figure 2): the chromatograms reveal the presence of MH and TMP reactants, the internal standard, and three ester products.

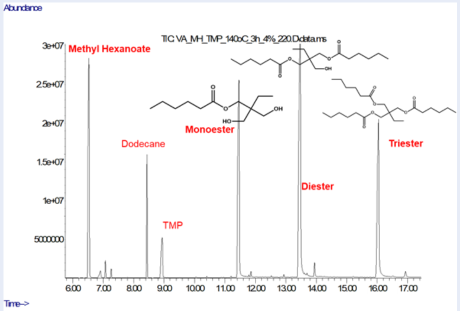


Figure 2: GC-MS spectra showing the products of the transesterification reaction between methyl hexanoate and trimethylolpropane in the presence of a SrO/MgO–CaO catalyst.

The effect of temperature

Figure 3 shows that the extent of the MH conversion and its selectivity toward TEs improved as the reaction temperature increased. The conversion of MH increased from 130 °C to 150 °C, reaching a maximum value of 76.18% at 150 °C. Increasing reaction temperatures leads to an increase in the rate of molecular motion and collision frequency, which accelerates the transesterification reaction. Nevertheless, reaction temperatures should not exceed the boiling point of MH (151 °C) as the evaporation of MH could promote the reverse reaction and reduce the overall conversion. In terms of selectivity, the highest selectivity toward DE and TE (87.35%) was achieved at 150 °C, corresponding to the maximum catalytic conversion achieved at this temperature.

The effect of catalyst loading

The influence of catalyst loading on the transesterification reaction between MH and TMP was investigated over a catalyst loading range of 2–7 wt.% (Figure 4). The conversion increased with catalyst loading

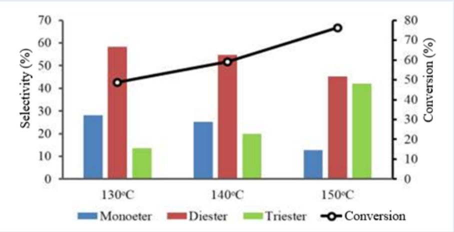


Figure 3: The effect of temperature on the conversion of MH and ester selectivity in the presence of SrO/MgO-CaO (NH_4OH, K_2CO_3). Reaction conditions: MH:TMP molar ratio of 3:1, 3 wt.% catalyst loading, 3 h reaction time, 25 mbar vacuum pressure and 500 rpm stirring speed.

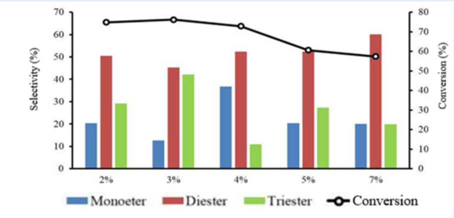


Figure 4: The effect of catalyst loading on the conversion of MH and ester selectivity in the presence of SrO/MgO-CaO (NH_4OH, K_2CO_3). Reaction conditions: MH:TMP molar ratio of 3:1, 3 wt.% catalyst loading, 3h reaction time, 25 mbar vacuum pressure, and 500 rpm stirring speed.

up to 3 wt.%, which can be attributed to the greater availability of active sites for the reaction[17].

Beyond this optimal loading, both the MH conversion rate and TE selectivity decreased. This decline is likely due to mass transfer limitations arising from the heterogeneous nature of the catalyst, with excessive catalyst concentrations hindering effective reactions between MH and TMP[18]. Furthermore, higher catalyst loadings result in saponification, leading to lower viscosities and reduced product yields. The results indicate that a catalyst loading of 3 wt.% provides an optimal balance between active surface area and effective mass transfer, ensuring the greatest conversion and selectivity in ester production.

The effect of the reactant molar ratio

The molar ratio of MH to TMP significantly influenced both conversion and product selectivity (Figure 5). A 3:1 molar ratio of MH:TMP yielded the best conversion and selectivity to DEs and TEs, which is consistent with the stoichiometric requirements for the complete reaction between the three hydroxyl groups in TMP with MH. Increasing the ratio to 5:1 resulted in significant changes to conversion and TE selectivity, which can be attributed to excess MH occupying active sites on the catalyst surface, thereby inhibiting TMP adsorption and reducing product formation[19]. Conversely, decreasing the ratio to 1:1 resulted in incomplete esterification and produced large amounts of MEs. These findings show that the molar ratio of the reactants is an important factor that controls the balance between the availability of reactants and the effectiveness of catalysts. A molar ratio of 3:1 MH:TMP was found to be the optimal ratio for achieving the highest conversion and greatest selectivity toward the desired esters.

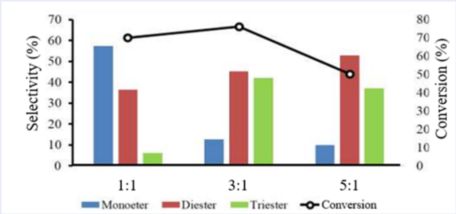


Figure 5: The effect of reactant molar ratio (MH:TMP) on conversion and selectivity using SrO/MgO-CaO (NH_4OH, K_2CO_3). Reaction conditions: 150 °C reaction temperature, 3 wt.% catalyst loading, 3 h reaction time, 25 mbar vacuum pressure, and 500 rpm stirring speed.

The effect of reaction time

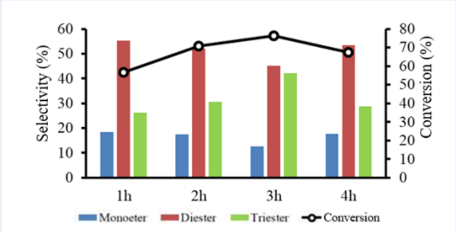


Figure 6: The effect of reaction time on conversion and selectivity using SrO/MgO-CaO (NH_4OH, K_2CO_3). Reaction conditions: MH:TMP molar ratio of 3:1, 150 °C reaction temperature, 3 wt.% catalyst loading, 3 h reaction time, 25 mbar vacuum pressure, and 500 rpm stirring speed.

The effect of reaction time on the transesterification reaction was evaluated over 1–4 h at 150 °C, a catalyst loading of 3 wt.%, a MH:TMP molar ratio of 3:1, a vacuum pressure of 25 mbar, and a stirring speed of 500 rpm (Figure 6). The conversion of MH increased steadily from 1 to 3 h, reaching a maximum of 76.18% at 3 h. (Figure 6). This increase is attributed to prolonged contact between reactants and catalyst, allowing for more complete esterification and the formation of higher esters.

However, extending the reaction time to 4 h resulted in a decrease in conversion to 67.48%. This reduction may be due to catalyst deactivation over prolonged reaction periods, which reduces the number of available active sites[20]. Furthermore, prolonged reaction times can favor reversible reactions or the degradation of intermediate products, thereby decreasing the yield of the target esters.

The effect of different types of catalysts

All synthesized catalysts exhibited comparable performance in terms of conversion and selectivity (Figure 7). SrO/MgO-CaO (NH_4OH) exhibited the highest conversion, while SrO/MgO–CaO (NH_4OH, K_2CO_3) showed the greatest selectivity to DEs and TEs (combined 93.26%).

These differences could be attributed to residual ions of the precipitating agents, which can cover or compete for active sites on the SrO/MgO–CaO oxides, resulting in lower catalytic performances[21]. Overall, these results highlight the importance of catalyst preparation methods in determining catalytic efficiency.

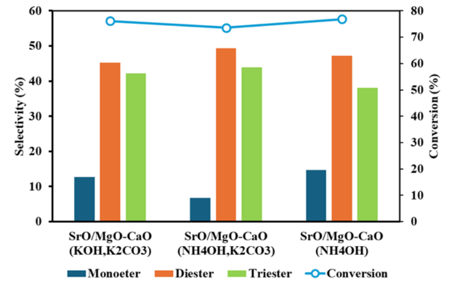


Figure 7: The effect of catalyst type on conversion and selectivity. Reaction conditions: MH:TMPmolar ratio of 3:1, 150 °C reaction temperature, 3 wt.% catalyst loading, 3 h reaction time, 25 mbar vacuum pressure, and 500 rpm stirring speed.

CONCLUSIONS

In this study, SrO/MgO–CaO catalysts were successfully synthesized using a co-precipitation method and evaluated in the transesterification reaction between MH and TMP. XRD analysis confirmed the formation of crystalline SrO, MgO, and CaO phases, as well as the presence of impurities depending on the synthesis conditions. Reaction conditions played an important role in the catalytic conversion and product selectivity of the transesterification reaction. Among the catalysts tested, SrO/MgO–CaO (NH_4OH) and SrO/MgO–CaO (KOH, K_2CO_3) exhibited the highest MH conversion (>76%) at 150 °C, 3 wt.% catalyst loading, a 3:1 molar ratio of MH:TMP, and a reaction time of 3 h. In addition, SrO/MgO–CaO (NH_4OH, K_2CO_3) exhibited the greatest DE and TE selectivity (>93%). Overall, the yield of DEs and TEs over the three SrO/MgO-CaO catalysts ranged between 65–68%, demonstrating that SrO/MgO-CaO catalysts are promising candidates for the sustainable production of biolubricants from biodiesel via transesterification.

LIST OF ABBREVIATIONS

DE: diester
GC: Gas Chromatography
GC-MS: Gas Chromatography-Mass Spectrometry
ME: monoester
MH: Methyl Hexanoate
NPG: neopentyl glycol
PE: pentaerythritol
SMC: SrO/MgO-CaO (Strontium Oxide / Magnesium Oxide - Calcium Oxide)
TE: triester
TMP: Trimethylolpropane
XRD: X-ray Diffraction

COMPETING INTERESTS

The author(s) declares that they have no competing interests.

AUTHORS' CONTRIBUTIONS

Thang Vu Pham: Writing—original draft preparation, Investigation, Conceptualization, Methodology, Data curation, Visualization; Van-Anh Vo Nguyen: Writing—original draft preparation, Investigation, Conceptualization, Methodology, Data curation, Visualization; Phat Dinh Duong: Conceptualization, Methodology, Data curation; Tong Thien Ngo: Data curation; Thanh Khoa Phung: Supervision, Writing—review and editing.

All authors have read and agreed to the published version of the manuscript.

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