Results in Chemistry 17 (2025) 102659

Contents lists available at ScienceDirect

Results in Chemistry

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Homogenizer-intensified room temperature emollient ester isoamyl oleate production

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ARTICLE INFO

Keywords:
Emollient ester
Isoamyl oleate
Homogenizer
Room temperature

ABSTRACT

The present study reports a successful experiment to produce emollient ester isoamyl oleate (IAO) in room temperature conditions. The process was conducted under mild conditions without external heating. A factorial design was applied to study the effects of molar ratio, catalyst concentration, and rotational speed over different reaction times. The highest IAO conversion (99.3 %) was achieved within just 5 min at room temperature using a 1: 3 M ratio, 0.1 M catalyst concentration, and 4000 rpm rotational speed. The reaction was completed rapidly due to enhanced mass transfer and mixing. Compared to conventional reflux and microwave-assisted methods, the homogenizer-assisted process reduced energy consumption by up to 93 % and reaction time by over 90 %. This approach demonstrates a promising green alternative for ester production with significant energy and time savings.

1. Introduction

Isoamyl oleate is an emollient ester that has been used widely in the cosmetics industry as an addition to skin care formulations, moisturizers, creams, lotions, and serums to provide deep hydration, improve skin texture, and increase strength. This ester emollient compound can form a thin protective layer on the surface of the skin which effectively retains moisture and prevents water loss, thereby helping keep the skin hydrated, supple and nourished [1,2]. In the automotive industry, IAO compound is used as a biodegradable lubricant that can reduce friction between moving parts while providing a layer of protection to prevent corrosion [3]. In the pharmaceutical industry, this ester compound is a multipurpose solvent that can help dissolve active ingredients in oral and topical drug formulations [1–5]. The global market for cosmetic esters, including isoamyl oleate, is projected to grow significantly due to increased demand for natural emollients. The fatty acid ester market was valued USD 17.28 billion in 2024 and is expected to expand at a CAGR of 6.4 % through 2032 [6]. Commercial production of IAO is currently performed at high temperatures using acid or base catalysts, requiring temperatures as high as 90–240 °C [7,8]. The use of such high temperatures results often leads to thermal degradation of the product, formation of side products, and darkening of color, all of which compromise purity and sensory properties—key quality indicators for cosmetic-grade emollients [9].

To break down the problem, enzymatic esterification of emollient ester using enzyme lipase as a biocatalyst has been developed. Several attempts have been made and shown that enzyme lipase could catalyze esterification reactions in mild reaction temperatures [10–12]. Lage et al. (2016) used enzyme lipase from *Thermomyces lanuginosus* doped in the mesoporous compound and reported an IAO conversion of 85 % after a reaction time of 30 min [10]. However, some drawbacks such as the expensive price of the enzyme and the inactivation of the enzyme by alcohol constricted its utilization [13,14].

Therefore, it is necessary to find a new method for making emollient ester IAO that does not require heating at high temperatures. Homogeneous dispersing equipment (homogenizer) is controlled mixing

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https://doi.org/10.1016/j.rechem.2025.102659
Received 21 June 2025; Accepted 26 August 2025
Available online 27 August 2025
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equipment that has a stator. There is a gap varying from 100 to 3000 μm between the stirrer and the stator which can cause large turbulence [15]. The stirring force produced by this homogeneous dispersing device is known to be 3 times greater than that of a normal stirrer at the same speed [15]. The improved mixing provided by the homogenizer enhances contact between reactants, accelerating reaction rates. However, this acceleration also depends on the intrinsic kinetics of the esterification process [16]. Homogeneous esterification reactions are generally faster than their heterogeneous counterparts due to improved mass transfer and reaction kinetics. Conducting such reactions efficiently at room temperature could significantly reduce operational costs and energy consumption in industrial applications [17,18]. Our previous research results in the esterification of oleic acid with methanol using a sulphuric acid catalyst and homogeneous dispersing equipment showed that a methyl oleate conversion of 96.1 ± 0.4 % could be achieved within 30 min and at room temperature [19].

This study reports the outcomes of the homogenizer-intensified room temperature production of IAO using sulphuric acid as a catalyst. The effect of processing parameters such as catalyst concentration, molar ratio of oleic acid to isoamyl alcohol and rotational speed were investigated in different reaction times using factorial design. Further, the reusability study is conducted to evaluate the catalyst performance. The novelty of this study lies in the production of emollient ester in room temperature conditions.

2. Materials and methods

2.1. Materials

Oleic acid (≥99 %, Sigma-Aldrich), isoamyl alcohol (≥98 %, Merck), and sulphuric acid (95–98 %, Merck) were used without further purification.

2.2. Room temperature esterification production of IAO

The esterification reaction of oleic acid with isoamyl alcohol was conducted in a homogenizer flask. The homogenizer consists of a rotor-stator with a diameter of 25 and 35 mm, respectively. The reaction was carried out using 40 mL of oleic acid (≈0.13 mol) and appropriate amounts of isoamyl alcohol (based on molar ratio). Sulphuric acid (0.1 M) was added in volumes of 0.13–0.39 mmol depending on conditions. The reaction mixture was stirred according to the specified stirring speed and reaction time. After time elapsed, the reaction was quenched in an ice bath. The reaction mixture was extracted with n-hexane to separate the product and facilitate purification. The IAO ester compound obtained was separated, washed with distilled water, dried and then the n-hexane solvent was evaporated, weighed and stored in a desiccator for conversion analysis. All experiments will be carried out up to 3 repetitions to obtain reliable data.

2.3. The IAO formation and conversion analysis

The spectrophotometer FT-IR was used to identify the IAO formation. The wavelength of the FT-IR spectra was recorded using the Perkin Elmer FT-IR 100 in the range of 4000–400 cm^{-1}. The IAO conversion was calculated based on the acid value of the initial and final sample using the AOCS Cd 3d-63 procedure [20,21].

The acid value is expressed as:

$$AV = \frac{V \times N \times 56.1}{W}$$

where AV is the acid value (mg KOH/g sample), V is the consumed volume of KOH (ml), N is the concentration of KOH (mol/L) and W is the mass of sample (g). The conversion of IAO (C) was determined by:

$$C = \frac{AV_i - AV_f}{AV_i}$$

where AV_i and AV_f are the initial and final acid values of the samples, respectively.

2.4. Data analysis

A factorial design approach was employed to investigate the influence of key processing parameters on the conversion of IAO. The parameters studied included: molar ratio of oleic acid to isoamyl alcohol (1:1, 1:3, 1:6, and 1:9), catalyst concentration (0, 0.01, 0.05, 0.1, 0.2, and 0.3 M), and homogenizer rotational speed (3000, 4000, and 5000 rpm). These variables were examined across a range of reaction times (1, 5, 15, 30, 45, 60, and 75 min). This factorial design allowed for the identification of individual and combined effects of each parameter on the esterification process efficiency. The Statistica v13 software was used with a significance level set to $\alpha = 0.05$.

3. Results and discussion

3.1. Effect of molar ratio

Four different molar ratios of oleic acid to isoamyl alcohol of 1:1, 1:3, 1:6 and 1:9 were studied to determine the effect of molar ratio on IAO conversion. The reaction time was varied from 1 to 75 min with a catalyst concentration of 0.3 M and rotational speed of 4000 rpm. As shown in Fig. 2, the molar ratio of 1:1 which is the stoichiometric value of this esterification reaction produces the lowest IAO conversion. Although a 1:1 M ratio represents the theoretical stoichiometry for esterification, using excess alcohol shifts the equilibrium forward, resulting in higher conversions. Our previous study on methanolysis of high-FFA oils showed similar conversion limitations at low alcohol volumes [19]. Using the stoichiometric molar ratio of 1:1, the reaction resulted in an average IAO conversion of 74.1 %, which was notably lower than reactions conducted with excess isoamyl alcohol. Interestingly, an addition of isoamyl alcohol in the reactant significantly increases the conversion. The IAO conversion of >97 % was routinely obtained when using a molar ratio above 1:1. In fact, that high IAO conversion was achieved in a short reaction time. The IAO conversion of 98.1 ± 0.6 %, 97.7 ± 0.5 and 98.2 ± 0.1 % were achieved using molar ratios of 1:3, 1:6 and 1:9, respectively at a reaction time of 1 min. Notably, increasing the reaction time had little impact on IAO conversion when using excess isoamyl alcohol, suggesting the reaction approached completion rapidly under these conditions. The plateau line of IAO conversion was achieved for all the reaction time parameters tested. Although an excess of isoamyl alcohol could potentially dilute the catalyst, the high turbulence and efficient mixing provided by the homogenizer appear to compensate for this, maintaining high conversion rates. In contrast, some researchers reported a decline in ester conversion when using an excessive volume of alcohol. The decrease is due to dilution of the limiting reactant concentration and inhibition catalyst active site, particularly when using a biocatalyst [19,22,23]. Further, the significant effect of changing the isoamyl alcohol amount and reaction time on the IAO conversion was determined using analysis of variance and showed that the significance was driven by low IAO conversion at a molar ratio of 1:1. (See Fig. 1.)

3.2. Effect of catalyst concentration

Finding an appropriate catalyst concentration is necessary as a low concentration can lead to an incomplete esterification reaction and a high concentration could increase the viscosity of the reactants reducing the ester conversion [24]. Hence, the effect of catalyst concentration on IAO conversion was evaluated by adjusting the concentration of

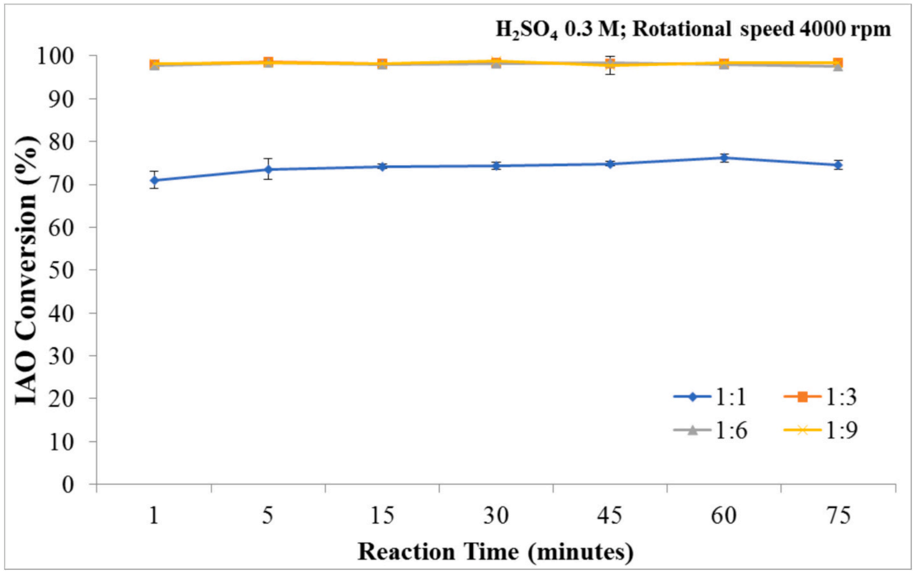

Fig. 2. The effect of the molar ratio of oleic acid to isoamyl alcohol on IAO conversion at various reaction times.

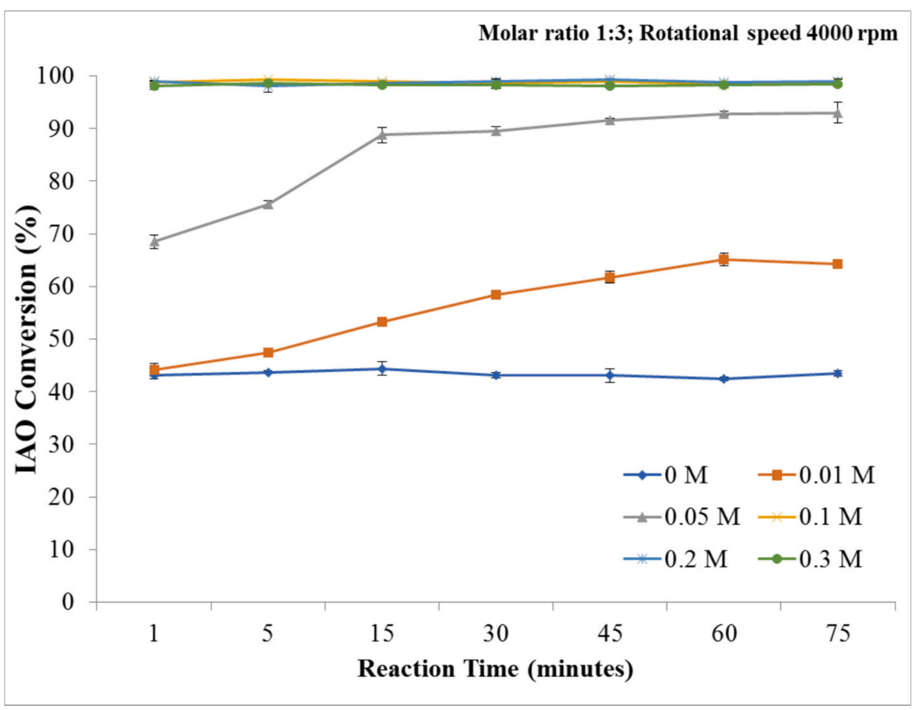

Fig. 3. The effect of catalyst concentration (molar) on IAO conversion at various reaction times.

sulphuric acid from 0.01 M to 0.3 M and reaction time from 1 to 75 min under reaction condition of molar ratio oleic acid to isoamyl alcohol of 1:3 and rotational speed of 4000 rpm. Fig. 3 shows that there was a significant increase in IAO conversion with increasing catalyst concentration. At a reaction time of 1 min, the IAO conversion of 44.1 $\pm$ 1.2 % was achieved using catalyst concentration of 0.01 M and was raised to 98.8 $\pm$ 0.2 % when using catalyst concentration of 0.1 M. Further, increasing catalyst concentration did not significantly affect the IAO conversion. This is presumably due to the esterification reaction having reached the equilibrium state [24,25]. Interestingly, the reaction time only affected the IAO conversion when using low catalyst concentration. At catalyst concentration below 0.1 M, the IAO conversion was increased in the prolonged reaction time and reached a plateau reaction curve proposing the maximum conversion was achieved. In contrast, the reaction time did not have a significant effect on IAO conversion when using a catalyst concentration above 0.1 M. At higher catalyst concentrations, the reaction quickly reaches equilibrium due to rapid protonation and ester formation, limiting further conversion even with extended time. This result is in line with our previous research in biodiesel production from esterification oleic acid with methanol using sulphuric acid as a catalyst and a homogenizer to facilitate the reaction at room temperature [19]. Similarly, Zhang et al. (2024) stated that a high catalyst concentration neither increases the conversion nor the availability of the catalyst active site [26]. A factorial analysis of variance determined a significant effect of the reaction time and catalyst concentration on the IAO conversion.

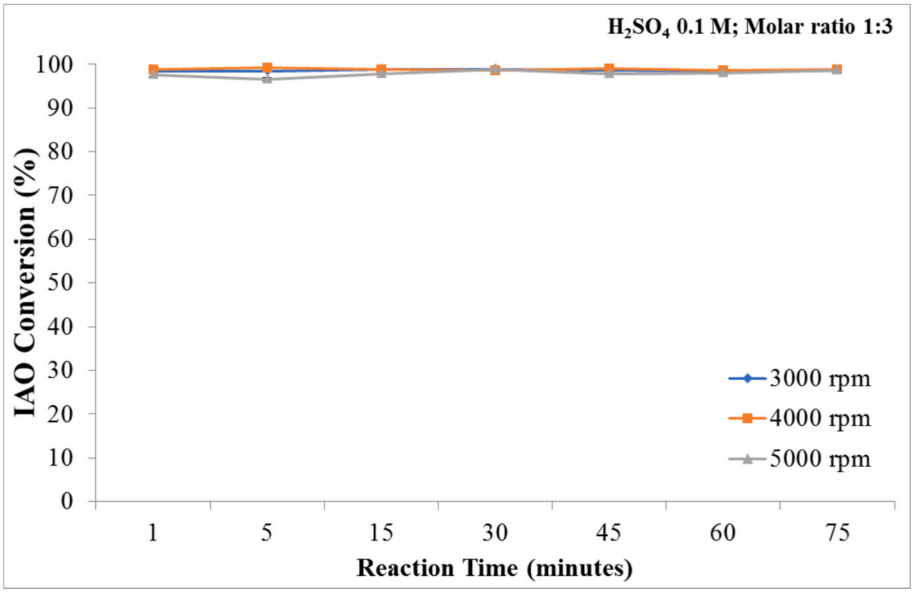

Fig. 4. The effect of rotational speed on IAO conversion at various reaction times.

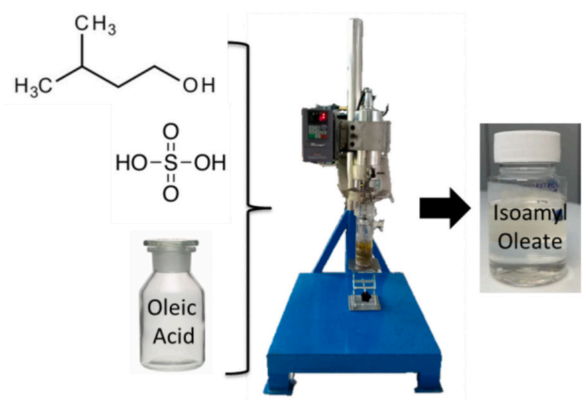

Fig. 1. The schematic image of the homogenizer-intensified IAO production.

3.3. *Effect of rotational speed*

A high agitation is required to increase the miscibility and mass transfer between oleic acid and isoamyl alcohol. The limited miscibility between oleic acid and isoamyl alcohol, influenced by differences in polarity and molecular interactions, can reduce mass transfer efficiency in conventional mixing systems [4]. A homogenizer apparatus provides a high mixing intensity that can increase the reaction rate [15]. In this study, the influence of rotational speed on the IAO conversion was investigated at different rotational speeds from 3000 to 5000 rpm and reaction time (1 to 75 min) at a constant molar ratio of 1:3 and catalyst concentration of 0.1 M. As shown in Fig. 4, the IAO conversion is very similar for all the rotational speeds tested. The IAO conversion is routinely achieved at >96 % for all rotational speeds. The high IAO conversion can be attributed to the fact that high turbulence generated by a homogenizer could facilitate the mass transfer between reactants [15,27,28]. Similar results were reported in some previous research in homogenizer-intensified biodiesel production using a heterogeneous catalyst [24,25]. The significant effect of changing rotational speed and reaction time on IAO conversion was detected by a factorial analysis of variance.

3.4. *Comparison of isoamyl fatty acid production using various methods*

Esterification production of ester fatty acids has been frequently reported, but only a few publications refer to the isoamyl fatty acid ester, particularly isoamyl oleate. Table 1 shows the comparison of isoamyl fatty acid ester production using various methods and catalysts. It can be noted that the reaction condition had a different effect on the ester conversion. Most of the esterification reactions in that table were conducted at a high reaction temperature except for this study. The lowest ester conversion of 85 % was achieved using enzyme lipase while other catalysts produced a conversion of >96 %. This result can be explained by the low reaction rate of the enzymatic reaction [29]. Another reason is explained by Lage et al. (2016) who used a stoichiometric molar ratio of fatty acid to isoamyl ester which is not sufficient to drive the enzymatic reaction to ester production [10]. In terms of this molar ratio, this present study uses an excessive amount of isoamyl alcohol (molar ratio of 1:3) than other methods. The high volume of isoamyl alcohol is required to provide large turbulence between reactants which can increase the reaction rate in the esterification production using a homogenizer device [25,30]. When compared to previously reported isoamyl oleate production using reflux conditions, our homogenizer-based method offers drastically shorter reaction time and lower energy consumption while maintaining high conversion. However, this present study using a homogeneous acid catalyst which requires neutralization and purification of the product which therefore could

Table 1
Summary of isoamyl fatty acid production using different catalysts and methods.

Product / Catalyst	Reaction condition (molar ratio of fatty acid: isoamyl alcohol, catalyst concentration, reaction time, temperature)	Ester conversion (%)	Refs.
Isoamyl oleate Sulphuric acid	1:2, 1.25 wt%, 60 min, 90 °C, magnetic heater and stirrer	97.3	[8]
Isoamyl oleate Sulphuric acid	1:3, 0.1 M, 5 min, room temperature, homogenizer rotational speed 4000 rpm	99.3	This study
Isoamyl oleate Enzyme lipase from *Thermomyces lanuginosus*	1:1, 20 wt%, 30 min, 45 °C, orbital shaker	85	[10]

increase the production time in total.

3.5. Energy consumption and CO_2 emission

Energy consumption and the environmental impact of the isoamyl fatty acid ester production method are important to consider for commercialization. Table 2 shows the electricity consumption and CO_2 emission of various ester production methods. The microwave device consumed 200 watts to facilitate the esterification production which is less than a regular magnetic stirrer hotplate and a homogenizer device which required 510 and 1500 watts, respectively. However, the electricity consumption of the microwave apparatus is the highest compared to the other esterification-intensification devices due to the longer reaction time to complete the esterification reaction. The total energy required to produce 1 kg of isoamyl oleate at room temperature using the homogenizer device was 3.5 kWh kg^{-1} which is equivalent to 12.6 MJ kg^{-1} and emitted 2.8 kg CO_2 to the atmosphere. Compared to the reflux method, which consumes 5.1 kWh/kg, the homogenizer method (3.5 kWh/kg) saved approximately 31 % energy. In comparison with the microwave irradiation method, which uses 47.1 kWh/kg, the energy savings reached 93 %. In addition, in terms of reaction time, the homogenizer-intensified room temperature isoamyl oleate production is time-wise. The homogenizer method also reduced reaction time substantially. Compared to the reflux method (60 min) and the microwave method (120 min), the 5-min reaction using a homogenizer resulted in time savings of approximately 92 % and 96 %, respectively. While CO_2 emissions were not directly measured in this study, the significant differences in electricity consumption highlight the homogenizer method's lower energy footprint, especially compared to microwave systems. Hence, esterification production using the dispersion method is a suitable green process to produce isoamyl oleate.

4. Conclusion

A novel procedure to produce IAO in room temperature conditions using a homogeneous acid as the catalyst and a homogenizer device has been developed. The factorial design experiment showed the significant effect of catalyst concentration, molar ratio of isoamyl alcohol to oleic acid and rotational speed of the homogenizer device. The maximum conversion of 99.3 $\pm$ 0.1 % was achieved in reaction time of 5 min, molar ratio of 1:3, catalyst concentration of 0.1 M and rotational speed of 4000 rpm. A preliminary energy consumption assessment showed that the homogenizer-intensified room temperature esterification reaction is energy efficient and could save 31–93 % of energy compared to other methods. This process is also time-efficient which can save >90 % reaction time and is environmentally friendly. The homogenizer-assisted process enables rapid, high-conversion IAO synthesis at room temperature with significantly reduced energy input compared to conventional methods, offering potential advantages for industrial-scale green production.

CRediT authorship contribution statement

Juliati Br. Tarigan: Writing – original draft, Supervision, Methodology, Funding acquisition, Conceptualization. **Aman Santoso:** Resources, Methodology. **Eko K. Sitepu:** Writing – review & editing, Supervision, Formal analysis. **Minto Supeno:** Methodology, Formal analysis. **Sabarmin Perangiangin:** Project administration, Formal analysis. **John P. Sihotang:** Methodology, Investigation. **Gloria M. Tarigan:** Investigation, Formal analysis. **David Chaidir:** Project administration, Methodology, Investigation. **Y.H. Taufiq-Yap:** Writing – review & editing, Supervision.

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.

Table 2
Comparison of electricity consumption and CO_2 emission of different esterification production methods.

Method	Electricity consumption (kWh/kg)	References
Reflux	5.1	[8]
Microwave	47.1	[7]
Homogenizer	3.5	This study

Acknowledgments

This work was supported by EQUITY Project released by the Ministry of Higher Education, Culture, Research, and Technology of Republic of Indonesia (Grant No. 59/E/HK.02.02/2022), LPDP-Indonesia Endowment Fund for Education Agency of the Ministry of Finance of Republic of Indonesia (Grant No. RJP-24/LPDP/2022), Directorate General of Higher Education, Research and Technology (Grant No. 3792/E3/DT.03.08/2023) and Universitas Sumatera Utara (Grant No. 8154.1/UN5.1.R/KPM/2023).

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.rechem.2025.102659.

Data availability

Data will be made available on request.

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