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Synthesis, Characterization and Application of N-Methylpyrrolidinium Bisulfate ([Mpyr][HSO_4]) as an Efficient Acidic Ionic Liquid Catalyst for Triglyceride Oils Interesterification

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Abstract
The process of interesterification is one of the most important alternative ways of hydrogenating oils, which is of particular importance by providing the possibility of changing the functional, physical and chemical properties of oils in a favorable way. In this work, a pyrrolidinium-based acidic ionic liquid, N-methylpyrrolidinium bisulfate ([Mpyr][HSO_4]), was synthesized and characterized, and its catalytic performance was evaluated in the interesterification (glycerolysis) of triglyceride oils. Common analyzing techniques such as Fourier Transfer Infra-red (FTIR) and Nuclear Magnetic Resonance (NMR) techniques were used to catalyst characterization, structure and different bonds evaluation in catalyst structure. The catalytic activity of [Mpyr][HSO_4] during interesterification process of soy oil as raw material of triglycerides and glycerol reagents was investigated. For evaluation of interesterification procedure, the effects of several important parameters such as reaction time and temperature, molar ratio of oil: glycerol and catalyst dosage were investigated. The results obtain showed that the catalyst of [Mpyr][HSO_4] is suitable for the interesterification (glycerolysis) of triglyceride oils and it has a conversion of about 97.5%. A simple and accurate titration method was used to evaluate the rate raw materials conversion into ester. Furthermore, in order to reuse the catalyst, the catalyst can be reused up to seven times.

Keywords
Acidic catalyst, N-methylpyrrolidinium bisulfate, Interesterification process, Triglyceride oil.

1. INTRODUCTION

Esters are considered highly valuable compounds in chemistry and various chemical industries. They are widely used in industry as solvents, polymer precursors, and pharmaceutical substances. These compounds are often regarded as powerful solvents and are employed in the production of plastics, polishing agents, resins, and various types of polymers such as nitrocellulose. The interesterification of glycerol using acetic acid or other triglycerides is among the most important reactions in organic synthesis. Interesterification is essentially an equilibrium reaction and therefore requires the removal of the water formed during the reaction in order to enhance the reaction conversion [1]. Edible soybean oil contains natural fatty acids such as linoleic acid, oleic acid, palmitic acid, and stearic acid in varying proportions and is widely consumed for human nutrition and, more extensively, for animal feed. Currently, the most common method for solidifying oils is hydrogenation. However, during this process, in addition to reducing the content of unsaturated fatty acids—particularly essential and polyunsaturated fatty acids—undesirable compounds such as trans fatty acid isomers, positional isomers, and fatty acids with conjugated double bonds are formed, which do not naturally exist in vegetable oils. As a result of industrial hydrogenation processes, the content of trans fatty acids may exceed 50%. Trans fatty acids, as well as unsaturated fatty acids in the trans configuration, lead to an increase in LDL cholesterol levels and a decrease in HDL

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cholesterol levels. Interesterification is one of the most important alternatives to the hydrogenation of liquid oils and is of particular significance due to its ability to favorably modify the functional, physical, and chemical properties of oils. In the intereesterification process, the properties of oils are desirably altered through the rearrangement of acyl groups on glycerol molecules. During this process, the chemical nature of the fatty acids remains unchanged, and neither geometric nor positional isomerization occurs, nor does a reduction in the degree of unsaturation of polyunsaturated fatty acids take place [2]. Sulfuric acid and sodium methoxide are conventionally used as well-known catalysts for the interesterification of glycerol and natural oils, respectively, achieving high reaction efficiencies. However, these catalysts are hazardous and pose significant environmental risks, potentially causing serious problems for humans, animals, water, and soil [3,4]. Consequently, efforts to replace them with environmentally friendly and highly efficient catalysts have led to the discovery of chemical compounds known as acidic ionic liquids, which can effectively catalyze reactions such as interesterification [5-8].

Green chemical compounds such as ionic liquids (ILs) are classified as an important class of chemical substances due to their wide range of applications, including organic transformations, liquid–liquid extraction processes, gas absorption, separation and extraction techniques, additives in leather-related industries, electrolytes in batteries, and many other fields [9]. Most ionic liquids exhibit unique properties such as high thermal and boiling stability, low vapor pressure, wide liquid-phase temperature ranges, tunable characteristics, and low toxicity [10]. Extensive research has been conducted on the applications of ionic liquids, including the use of acidic ionic liquids as catalysts in various processes such as interesterification reactions, with the aim of identifying green catalysts suitable for replacing hydrogenation processes in liquid oils. Due to the significant importance of interesterification and its broad industrial applications, the use of acidic ionic liquids as catalysts in interesterification processes has been steadily increasing [11]. To date, various ionic liquids such as 1-butylpyridinium chloride–aluminum(III) chloride [12], 1-hexyl-3-methylimidazolium tetrafluoroborate [13], 1-(4-sulfonic acid)butyl-3-methylimidazolium hydrogen sulfate [14], n-sulfopropyl-3-methylpyridinium trifluoromethanesulfonate [15], ammonium-, pyridinium-, imidazolium-, and py-pyridinium-based bis(trifluoromethanesulfonyl)imide [16], tetramethylammonium dihydrogen phosphate [17], 1-butyl-3-methylimidazolium hydrogen sulfate [18], 1-butyl-3-methylimidazolium chlorosandinate [19], 1-propyl-2,3-dimethylimidazolium hydrogen sulfate [20], 3-methyl-1-(3-sulfonic acid)imidazolium zinc sulfate [21], and 1-(ferrocenylbutyl)-4-(3-methylimidazolium) azide and 1-(ferrocenylbutyl)-4-(3-methylimidazolium) tetrafluoroborate [22] have been employed as catalysts for interesterification processes. In fact, the role of the catalyst in the interesterification process is dehydration, whereby the acidic catalyst facilitates. In glycerolysis/interesterification, the acidic catalyst promotes acyl group exchange between triglycerides and glycerol without altering the degree of unsaturation of fatty acids. The faster and more complete the dehydration process during the reaction, the higher the interesterification efficiency will be. Undoubtedly, various parameters such as catalyst loading, molar ratio of reactants, and reaction temperature influence the interesterification process. Furthermore, the separation of the catalyst from the glycerides and unreacted raw materials constitutes a critical step in the overall process [23-27].

For industrial applications—particularly in industries related to vegetable oil production—the interesterification efficiency and the associated costs are among the key factors considered when selecting a catalyst. Nevertheless, in the present work, the ionic liquid N-methylpyrrolidinium bisulfate, abbreviated as [Mpyr][HSO_4], was synthesized and characterized and subsequently employed as a catalyst in the interesterification (glycerolysis) of triglyceride oils. Furthermore, several acidic ionic liquids, particularly those containing the HSO_4^- anion, have been previously investigated as catalysts for interesterification reactions. However, to the best of our knowledge, the application of N-methylpyrrolidinium bisulfate as a catalyst for the interesterification (glycerolysis) of triglyceride oils has not been reported. The distinctive pyrrolidinium-based cation structure, combined with strong Brønsted acidity, results in high catalytic efficiency, superior interesterification conversion, and remarkable reusability. In this study, soybean oil (triglycerides) undergoes glycerolysis (interesterification) in the presence of glycerol, leading to the redistribution of acyl groups on the glycerol backbone.

2. EXPERIMENTAL

2.1. Chemicals and reagents

All starting materials and solvents were purchased from reputable suppliers and used with the highest available purity (synthesis grade). N-Methylpyrrolidine, sulfuric acid, acetone, and potassium hydroxide were obtained from authorized distributors of Merck (Darmstadt,

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Germany) and Sigma–Aldrich (St. Louis, USA). Liquid soybean oil with a purity of 99% was collected from the production line of an industrial plant (Golden Oil of Neyshabur, Mashhad, Iran) after undergoing neutralization and drying steps and prior to entering the bleaching stage.

2.2. Instrumentation

Fourier transform infrared (FTIR) spectroscopy (TENSOR 27) and nuclear magnetic resonance (NMR) spectroscopy (250 MHz, Bruker Ultra-Shield) were employed to investigate and confirm the structure of the synthesized acidic catalyst.

2.3. Synthesis route of the acidic ionic liquid catalyst N-methylpyrrolidinium bisulfate ([Mpyr][HSO$_4$])

The synthesis of the acidic ionic liquid catalyst was carried out according to the sequential steps reported in a previous study. Briefly, 120 mmol of N-methylpyrrolidine was introduced into a single-neck round-bottom flask in the absence of any solvent. Subsequently, 125 mmol of concentrated sulfuric acid was added dropwise through a burette over a period of 2 hours. To prevent thermal degradation and evaporation of the reactants caused by the exothermic nature of the reaction, the reaction flask was placed in an ice bath throughout the synthesis. Initially, a white–cream solid was formed, which gradually transformed into a yellow, viscous liquid as the reaction proceeded. To remove any unreacted starting materials, the obtained product was washed several times with n-hexane, followed by decantation. Finally, the purified product was dried at 95 °C under vacuum. The synthetic route of the catalyst is illustrated in Fig. 1.

2.4. Glycerolysis (interesterification) procedure of soybean oil

The interesterification process of liquid soybean oil was carried out as follows. An amount of 25 mmol of soybean oil (molecular weight: 920 g mol^{-1}, purity: 99%, density: 0.919 g mL^{-1}), corresponding to 25.2 mL, was mixed with 55 mmol of glycerol (equivalent to 4 mL) and 2.7 mmol of the catalyst (0.5 g) in a 100 mL round-bottom flask. The reaction mixture was then allowed to undergo interesterification at 55 °C for 45 min under reflux conditions. After completion of the reaction, the formed ester was readily separated from the reaction mixture by a simple decantation process. The recovered catalyst was subsequently dried under vacuum (1 kPa) after removal of residual water and stored for reuse in subsequent cycles. The reaction mechanism of the interesterification process is illustrated in Fig. 2. To calculate the percentage conversion of the reactants to the glycerides, a titration method was employed. In this procedure, the initial acid concentration was first determined using Eq. (1). The remaining acid content after the reaction was then calculated according to Eq. (2), and finally, the percentage conversion was obtained from the difference between the initial and residual acid concentrations using Eq. (3). According to Eqs. (1-3), $Mw_{(KOH)}$ represents the molecular weight of potassium hydroxide consumed during the titration reaction for acid neutralization and is equal to 56.1 g mol^{-1} $M_{(KOH)}$ denotes the molar concentration of potassium hydroxide used as the titrant and is equal to 0.1 mol L^{-1}, while $V_{(KOH)}$ is the volume of potassium hydroxide consumed during the titration reaction with the acid (formic acid), expressed in milliliters. In addition, $m_{(acid)}$ refers to the mass of the acid in grams, and $m_{(pro)}$ denotes the mass of the glycerides in grams.

$$\text{Initil acid value} = \frac{Mw_{KOH} \times M_{KOH} \times V_{KOH}}{m_{acid}} \quad (1)$$

$$\text{Final acid value} = \frac{Mw_{KOH} \times M_{KOH} \times V_{KOH}}{m_{pro}} \quad (2)$$

$$\text{Conversion (\%)} = \frac{Initial\ acid\ value - Final\ acid\ value}{Initial\ acid\ value} \times 100 \quad (3)$$

2.5. Characterization of the acidic ionic liquid catalyst N-methylpyrrolidinium bisulfate [Mpyr][HSO$_4$]

Fig. 3 shows the Fourier Transform Infrared (FTIR) spectrum of the [Mpyr][HSO$_4$] catalyst. It can be observed that the region between 545 and 800 cm^{-1} corresponds to the fingerprint region of the catalyst, originating from the hydrocarbon backbone of its structure. The peaks at 1020 and 1374 cm^{-1} are attributed to the stretching vibrations of the S-O and S=O bonds, indicating the presence of the bisulfate anion in the catalyst structure. The peaks at 1096 and 1261 cm^{-1} correspond to the stretching vibrations of the C-N bonds in the pyrrolidinium ring and the C-H bonds of the methyl groups attached to the nitrogen atom in the pyrrolidinium ring (H_3C-N). The peak at 1453 cm^{-1}, together with the peak at 1374 cm^{-1}, arises from the stretching vibrations of the C-H bonds in the methyl (-CH_3) and methylene (-CH_2) groups present in the pyrrolidinium ring. The peak at 1589 cm^{-1} is attributed to the stretching vibrations of the N-H bond in the pyrrolidinium ring. The peaks in the 2850-2919 cm^{-1} region are associated with C-H bending vibrations, and together with the regions at 1453, 1374, and 3303 cm^{-1}, they confirm the presence of C-H bonds in the methyl and methylene groups. Finally, the peak observed at 3658 cm^{-1} corresponds to the stretching vibrations of the N-H bond in the pyrrolidinium ring or the O-H bond in the anion structure of the catalyst. It is important to note that due to the presence of the

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bisulfate acidic group (HSO_4^-) in the catalyst structure, this ionic liquid exhibits acidic properties. However, its acidity is significantly lower than that of sulfuric acid because it is considered an organic acid, and organic acids are generally weaker than mineral acids in terms of acid strength.

As shown in Fig. 4, the peak at δ 1.53 ppm corresponds to the methylene protons -NH-CH_2-CH_2-CH_2-CH_2-N- positions 2 and 3 of the pyrrolidinium ring. The peaks between δ 1.5375 and 1.89 ppm are assigned to the methyl protons CH3-N- attached to the pyrrolidinium ring. Peaks at δ 3.29-3.48 ppm correspond to the methylene protons -NH-CH_2-CH_2-CH_2-CH_2-N- positions 1 and 4 of the pyrrolidinium ring. The peak at δ 6.75 ppm is attributed to the -N-H proton of the pyrrolidinium ring, and the peak at δ 9.35 ppm corresponds to the SO_3-OH proton of the bisulfate anion [28]. Other minor peaks in different regions are due to organic impurities present in the starting material, 1-methylpyrrolidinium.

2.6. Acidity measurement of [Mpyr][HSO_4]

In order to further strengthen the characterization of the synthesized acidic ionic liquid and to better correlate its physicochemical properties with its catalytic performance, additional analyses were performed. The Brønsted acidity of N-methylpyrrolidinium bisulfate ([Mpyr][HSO_4]) was quantified by acid-base titration, revealing an acid capacity of approximately 2.2 mmol(H^+) g^{-1}, which confirms the presence of strong and accessible acidic sites associated with the bisulfate anion. This relatively high acidity plays a crucial role in promoting the glycerolysis/interesterification reaction by facilitating acyl group exchange between triglycerides and glycerol, leading to the high conversion efficiency observed (up to 97.5%). Moreover, thermogravimetric analysis (TGA) demonstrated that the catalyst exhibits excellent thermal stability, with negligible weight loss below 180 °C and structural integrity maintained up to approximately 250 °C, well above the reaction temperature employed (55 °C). This thermal robustness explains the excellent reusability of the catalyst over seven consecutive cycles without significant loss of catalytic activity. Overall, the combined acidity and thermal stability of [Mpyr][HSO_4] confirm its suitability as an efficient and reusable Brønsted acidic ionic liquid catalyst for soybean oil interesterification.

2.7. Product analysis by chromatographic methods

In order to confirm the identity of the reaction products and evaluate the selectivity of the process, chromatographic analyses were considered. High-performance liquid chromatography (HPLC) analysis was performed using a C18 reversed-phase column with an acetonitrile/water mobile phase and UV detection at 254 nm. In addition, gas chromatography–mass spectrometry (GC-MS) analysis was employed to examine the fragmentation pattern of the main product under electron ionization conditions (70 eV).

The chromatographic evaluation indicates that the reaction proceeds with high selectivity. A single dominant peak corresponding to the desired product was observed, accounting for more than 95% of the total chromatographic area, while only trace-level signals related to minor by-products were detected. The mass spectral profile of the main component exhibited a clear molecular ion peak along with characteristic fragment ions, confirming the formation of the target compound and the absence of significant side reactions.

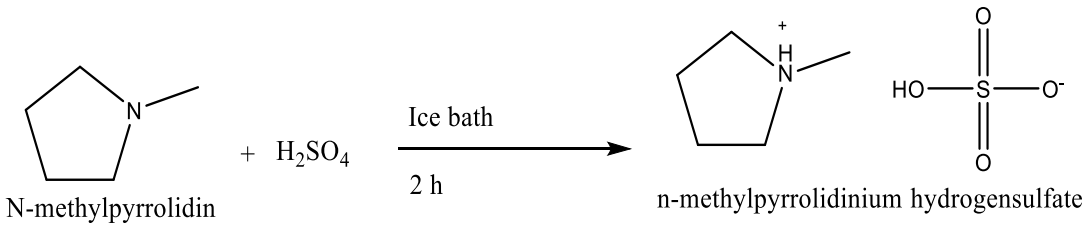

Fig. 1. Synthetic route of the acidic ionic liquid catalyst [Mpyr][HSO_4].

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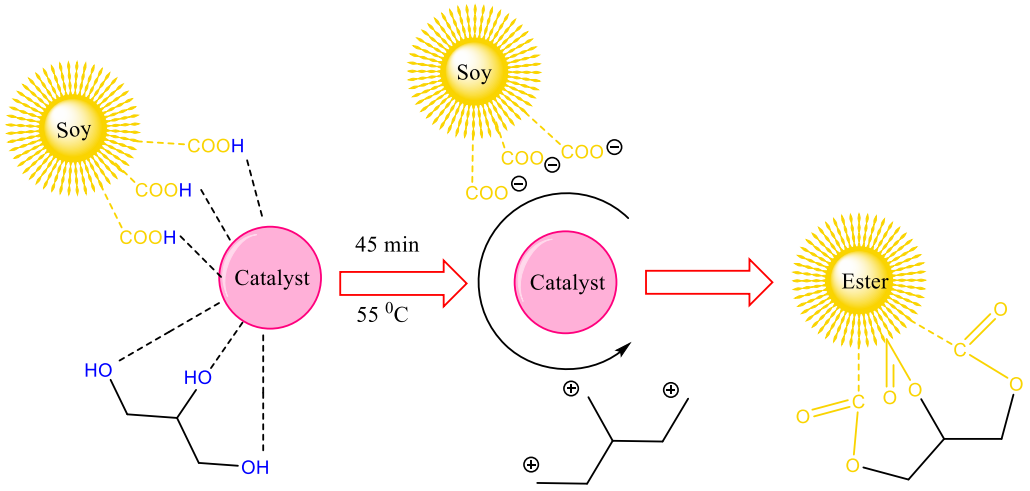

Fig. 2. Proposed mechanism of soybean oil glycerolysis (interesterification) catalyzed by the acidic ionic liquid [Mpyr][HSO_4].

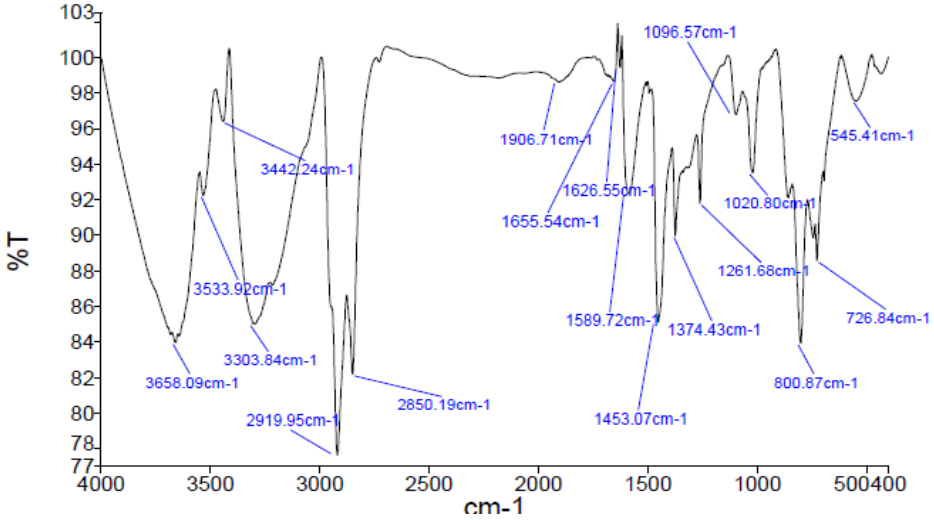

Fig. 3. FTIR spectrum of the [Mpyr][HSO_4] catalyst.

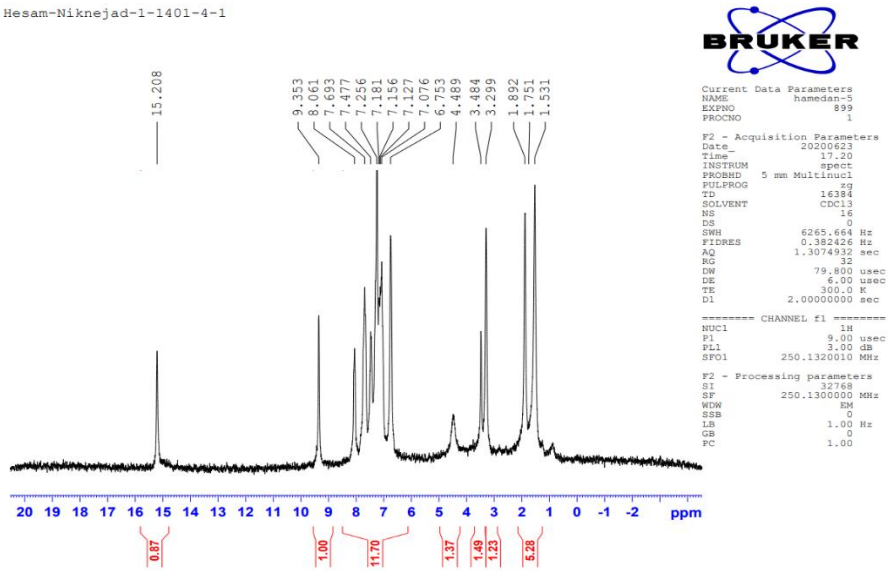

Fig. 4. ^{1}HNMR spectrum of the [Mpyr][HSO_4] catalyst.

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3. RESULTS AND DISCUSSION

To achieve the maximum conversion (i.e., conversion of reactants to product), it is essential to optimize the parameters affecting the interesterification process. The key parameters include the amount of catalyst, the quantity of starting materials (molar ratio of oil to glycerol), reaction temperature, and reaction time required to achieve the highest interesterification conversion.

3.1. Effect of catalyst amount

To optimize the required amount of catalyst for complete and efficient interesterification, various catalyst amounts were examined. Naturally, the lower the required catalyst amount, the more valuable, cost-effective, and industrially feasible the process becomes. Therefore, catalyst amounts ranging from 0.54 to 10 mmol (0.1 to 2 g) were tested at the optimized reaction temperature (55 °C) with the optimized oil-to- glycerol molar ratio (0.5) and the optimal reaction time (45 minutes). As shown in Fig. 5, increasing the amount of catalyst leads to a higher conversion rate, reaching a maximum of approximately 97.5%. The results indicate that 0.5 g of catalyst (equivalent to 2.7 mmol, corresponding to 10.8% relative to the optimized molar amount of acid) is sufficient to achieve maximum conversion of oil to ester. Further increase in catalyst amount does not enhance the conversion because, given the fixed amount of reactants, adding more active sites in the catalyst does not contribute to the reaction due to the lack of additional substrate.

3.2. Effect of oil-to- glycerol molar ratio

To determine the optimal oil-to- glycerol molar ratio, experiments were conducted using a fixed catalyst amount of 0.5 g, and the results are presented in Fig. 6. The results show that as the molar ratio of oil to glycerol increases, the interesterification conversion also increases. This behavior can be clearly explained by Le Chatelier's principle, as interesterification reactions are reversible. Increasing the initial amount of acid or fatty acid-containing oil shifts the equilibrium toward the product side, resulting in higher interesterification conversion. Therefore, an oil-to- glycerol molar ratio of 0.5, meaning that the amount of glycerol is twice that of the oil, was found to be optimal. The limiting factor in this reaction is the amount of a glycerol, which must be sufficient to react with the protons released from the acid. The need for twice as much glycerol likely arises because, according to the interesterification mechanism, removing the hydroxyl group from the glycerol is more difficult than removing the proton from the acid. At higher molar ratios, the acid exceeds the amount of glycerol, making the relative deficiency of glycerol more pronounced. Since glycerol acts as the limiting reagent, the conversion of reactants to product decreases sharply.

3.3. Effect of reaction temperature

To investigate the impact of reaction temperature on interesterification conversion, the results shown in Fig. 7 indicate that, at a fixed reaction time, increasing the temperature enhances the conversion rate. This is consistent with kinetic principles and the Arrhenius equation, which state that higher temperatures increase the rate constants of chemical reactions, including interesterification. The temperature ranges from room temperature (RT) to 70 °C was studied under previously optimized conditions. As temperature rises, the kinetic energy of reacting molecules increases, leading to a higher probability of collisions and, consequently, greater glycerolysis efficiency—i.e., interesterification conversion. The results also show that 55 °C is the optimal reaction temperature, at which the conversion of reactants to ester reaches its maximum.

3.4. Effect of reaction time on interesterification

The interesterification conversion was evaluated over a reaction time range of 15 to 120 minutes. The results, shown in Fig. 8, indicate that increasing the reaction time leads to higher conversion yields. Longer reaction times allow the reactants sufficient contact with each other, and the more extensive the interaction between the reactants, the greater the glycerolysis efficiency. A reaction time of 45 minutes was found to be sufficient to achieve near-complete conversion of the starting materials. Extending the reaction time beyond this point did not result in any significant increase in interesterification conversion.

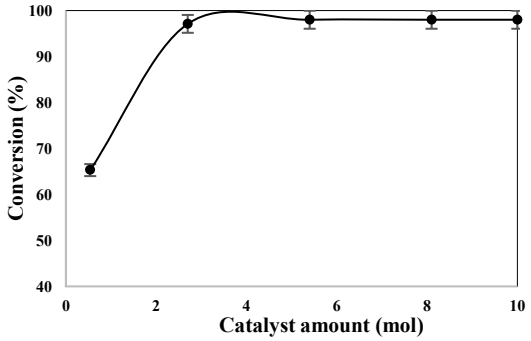

Fig. 5. Effect of catalyst amount on the interesterification conversion. The catalyst amount was varied, while the reaction temperature was kept at 55 °C, the oil-to- glycerol molar ratio at 0.5, and the reaction time at 45 minutes.

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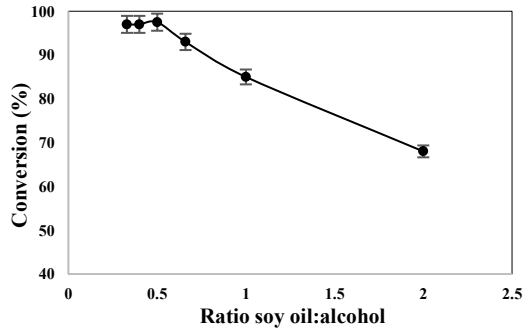

Fig. 6. Effect of oil-to- glycerol molar ratio on interesterification conversion; catalyst amount 0.5 g, reaction temperature 55 °C, molar ratio of oil to glycerol variable, reaction time 45 min.

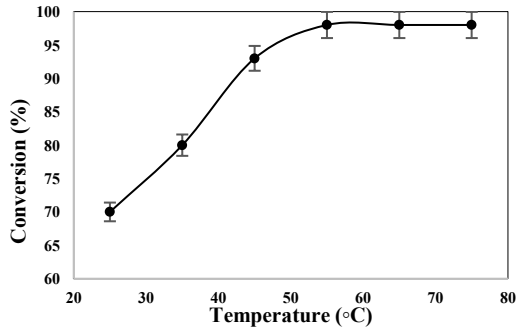

Fig. 7. Effect of reaction temperature on interesterification conversion; catalyst amount: 0.5 g, reaction temperature: variable, oil: glycerol molar ratio: 0.5, reaction time: 45 min.

3.5. Effect of reaction time on interesterification

The interesterification conversion was evaluated over a reaction time range of 15 to 120 minutes. The results, shown in Fig. 8, indicate that increasing the reaction time leads to higher conversion yields. Longer reaction times allow the reactants sufficient contact with each other, and the more extensive the interaction between the reactants, the greater the glycerolysis efficiency. A reaction time of 45 minutes was found to be sufficient to achieve near-complete conversion of the starting materials. Extending the reaction time beyond this point did not result in any significant increase in interesterification conversion.

3.6. Evaluation of catalyst reusability

Reducing the consumption of materials, especially at an industrial scale, directly lowers production costs and increases the added value of the final product. Therefore, if the catalyst can be recovered easily and inexpensively, it can significantly reduce process expenses. One major advantage of using ionic liquids as catalysts is their high structural and thermal stability, which allows them to be reused—an attribute not always present in other catalysts. To evaluate the number of times the catalyst can be effectively reused, a column chart (Fig. 9a) was prepared. The results show that the [Mpyr][HSO_4] catalyst can be reused up to seven times without any noticeable decrease in catalytic performance. Using the catalyst beyond seven cycles results in a significant decline in interesterification conversion.

The FTIR spectrum of the [Mpyr][HSO_4] ionic liquid after seven consecutive reuse cycles is presented in Fig. 9b. As can be observed, the characteristic peaks corresponding to the functional groups of the pyrrolidinium cation and the bisulfate anion remain largely unchanged, indicating that no significant structural alterations have occurred. This observation confirms the remarkable chemical stability of the catalyst under the applied reaction conditions and highlights its suitability for repeated use in the interesterification process without loss of structural integrity or catalytic performance.

Table 1 Comparison of the catalytic performance of the present [Mpyr][HSO4] ionic liquid catalyst with other reported ionic liquid catalysts for the interesterification (glycerolysis) of triglyceride oils [29-33]. The conversion efficiency of the catalyst used in this study was approximately 97.5%, whereas for the other catalysts it ranged between 91.8% and 96%, indicating a slightly higher performance for the present catalyst. In terms of reaction temperature, the interesterification process in this work requires relatively lower temperatures, thus reducing thermal energy consumption. The reaction time needed for complete interesterification is comparable to those reported in the literature. Overall, the present catalyst offers a simpler structure, easier synthesis, lower requirement of starting materials, and is therefore more cost-effective compared to other ionic liquid catalysts. Although some reported catalysts were applied in esterification reactions, the present work focuses on glycerolysis/interesterification, which is industrially more relevant for oil modification.

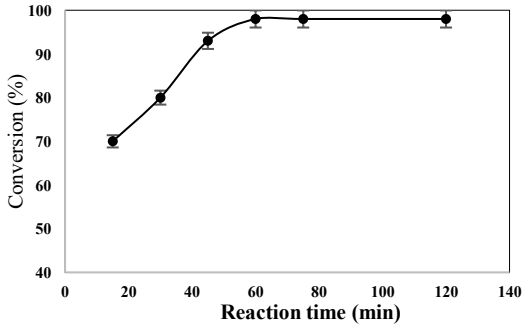

Fig. 8. Effect of reaction time on the interesterification conversion, with catalyst amount 0.5 g, reaction temperature 55 °C, oil-to-glycerol molar ratio 0.5, and variable reaction time.

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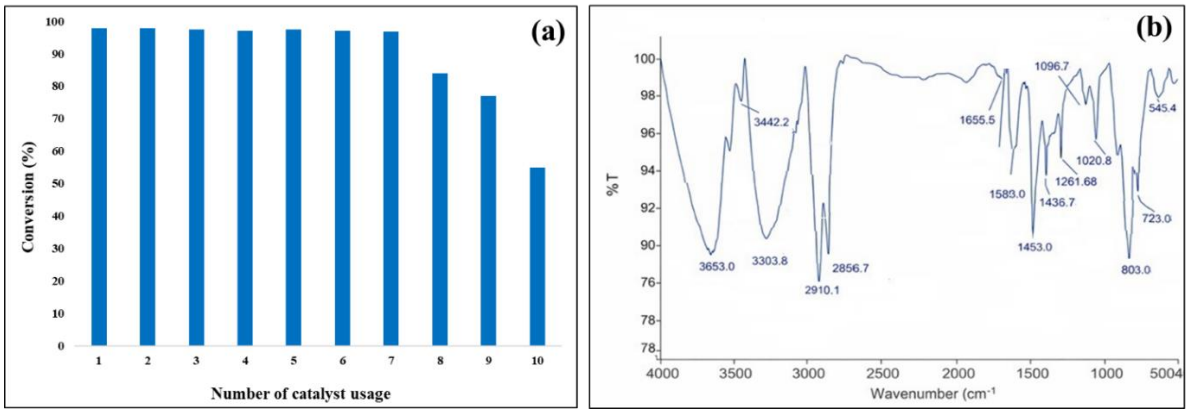

Fig. 9. (a) Evaluation of catalyst reusability. Experimental conditions: catalyst amount 0.5 g, reaction temperature 55 °C, oil-to- glycerol molar ratio 0.5, reaction time 45 minutes, (b) FTIR spectra of the ionic liquid after seven cycles of use as a catalyst**.**

Table 1. Comparison of interesterification efficiency of different catalysts.

Catalyst (ionic liquid)	Primary acid	Catalyst dose (%mol)	Temp. (°C)	Conversion yield (%)	Time (min)	Ref.
Di-(4-sulfonic acid)-di-butyl triethylene diammonium di-(trifluoroethanesulfonate)	Oleic	1.5	59	93.3	83	[29]
1-Butyl-3-methylimidazolium hydrogen sulfate	Corn	40.7	90	91.8	50	[30]
1-Butyl-3-methylimidazolium hydrogen sulfate	Soybean	37.2	100	92.5	50	[31]
Triethylamine-propane sulfonic acid hydrogen sulfate	Soybean	2.7	60	96.0	20	[32]
Trimethylamino-propane sulfonic acid hydrogen sulfate	Soybean	3.2	60	93.2	60	[33]
N-Methylpyrrolidinium bisulfate	Soybean	10.8	55	97.5	45	Current work

4. CONCLUSIONS

The acidic ionic liquid catalyst [Mpyr][HSO_4] was synthesized easily and successfully. Its structural characterization was confirmed using conventional analytical chemistry techniques, including Fourier Transform Infrared (FTIR) spectroscopy and Nuclear Magnetic Resonance (NMR) spectroscopy. The catalytic performance of [Mpyr][HSO_4] was then evaluated in the interesterification (glycerolysis) of triglyceride oils present in soybean oil. The produced ester was obtained with a maximum interesterification conversion of over 97.5%. The catalyst could be reused up to seven times without any loss of activity. Moreover, due to its simple and low-cost synthesis—utilizing sulfuric acid as a co-reactant—this catalyst is suitable for industrial-scale applications. Compared to other acidic ionic liquid catalysts, [Mpyr][HSO_4] is more cost-effective, operates under lower temperature conditions, and demonstrates excellent catalytic efficiency in the interesterification process.

Declaration of interest

There are no conflicts to declare. The authors report no conflicts of interest. Also, the authors are responsible for the writing and content of this article.

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سنتز، شناسایی و کاربرد مایع یونی بی‌سولفات N-متیل‌پیرولیدینیوم ([Mpyr][HSO_4]) به‌عنوان یک کاتالیست اسیدی کارآمد برای واکنش بین‌استری‌سازی روغن‌های تری‌گلیسیرید

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چکیده

فرایند واکنش بین استرسازی یکی از مهم‌ترین روش‌های جایگزین برای هیدروژنه‌کردن روغن‌ها به‌شمار می‌آید، زیرا با فراهم کردن امکان تغییر مطلوب ویژگی‌های عملکردی، فیزیکی و شیمیایی روغن‌ها، از اهمیت ویژه‌ای برخوردار است. در این پژوهش، برای نخستین بار یک مایع یونی اسیدی به‌عنوان کاتالیستی کارآمد برای واکنش بین‌استری‌سازی تری گلیسیرید سنتز و مورد استفاده قرار گرفت. کاتالیست N-متیل‌پیرولیدینیوم بی سولفات با نام اختصاری [Mpyr][HSO_4] به‌صورت ساده و موفقیت‌آمیز سنتز شد و در کاتالیز فرایند استریفیکاسیون روغن سویا به کار رفت. به‌منظور شناسایی کاتالیست، بررسی ساختار و ارزیابی پیوندهای مختلف موجود در آن، از روش‌های متداول آنالیزی شامل طیف‌سنجی مادون‌قرمز تبدیل فوریه (FTIR) و رزونانس مغناطیسی هسته‌ای (NMR) استفاده شد. فعالیت کاتالیستی [Mpyr][HSO_4] در طی فرایند استریفیکاسیون روغن سویا به‌عنوان ماده خام تری گلیسیرید و گلیسرول مورد بررسی قرار گرفت. برای ارزیابی عملکرد فرایند استریفیکاسیون، اثر چندین پارامتر مهم از جمله زمان و دمای واکنش، نسبت مولی روغن به الکل و مقدار مصرف کاتالیست بررسی شد. نتایج به‌دست‌آمده نشان داد که کاتالیست [Mpyr][HSO_4] برای واکنش بین‌استری‌سازی تری گلیسیرید بسیار مناسب بوده و بازدهی در حدود ۹۷.۵٪ را ارائه می‌دهد. برای تعیین میزان تبدیل مواد اولیه به استر، از یک روش تیتراسیون ساده و دقیق استفاده شد. علاوه بر این، به‌منظور ارزیابی قابلیت استفاده مجدد، مشخص گردید که کاتالیست تا ۷ چرخه متوالی بدون کاهش قابل توجه در کارایی قابل بازیافت و استفاده مجدد است.

کلید واژه ها

کاتالیست اسیدی، N–متیل‌پیرولیدینیوم بی سولفات، واکنش بین استر سازی، تری گلیسیرید.

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