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GREEN SYNTHESIS, CHARACTERIZATION, AND EVALUATION OF FATTY ACID ESTERS AS NATURAL EMULSIFIERS IN COSMETIC FORMULATIONS

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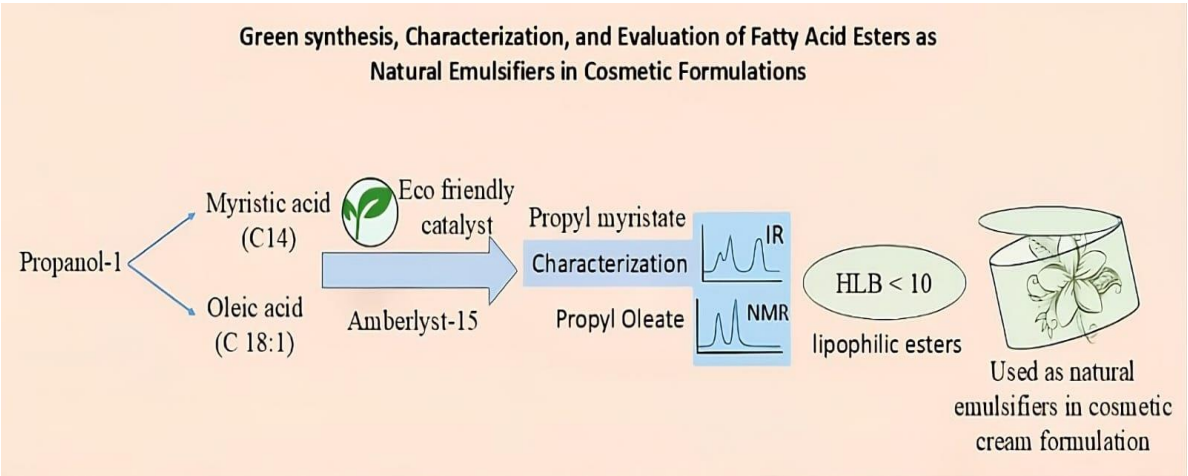
ABSTRACT

The synthesis of fatty esters is an important area in industrial chemistry due to their increasing use in cosmetic formulations as natural emulsifiers. Their surface-active properties enable the formation of stable emulsions, enhancing product stability and shelf life. In this study, an eco-friendly Fischer esterification method was employed to synthesize fatty esters from myristic acid and oleic acid using 1-propanol, catalyzed by the recyclable heterogeneous acid catalyst Amberlyst-15. The formation of propyl myristate and propyl oleate was confirmed by FTIR and ^1H-NMR spectroscopy, with yields of 72% and 69%, respectively. The emulsifying behavior of the synthesized esters was evaluated through hydrophilic–lipophilic balance (HLB) values, which indicated their lipophilic nature. Application tests in a cosmetic cream formulation demonstrated that blending both esters resulted in improved texture, consistency, and overall formulation performance compared to the individual esters.

KEYWORDS: myristic acid- Amberlyst 15- oleic acid -.HLB - Emulsions.



INTRODUCTION

In recent decades, the chemical industries have witnessed remarkable development enabling the production of numerous compounds with significant industrial and everyday applications; among the most prominent of these compounds are fatty esters.[1], The fatty acid esters possess a wide range of applications, including the production of cosmetics, food, pharmaceuticals, as well as paints and lubricants.[2.3], fatty esters include methyl esters, isoprppyl esters, wax esters, as well as ester oils.[4] The outstanding physicochemical properties of fatty acid esters have driven their use as surface tension-reducing

agents, with demand significantly increasing due to their well-known value as additives in cosmetiecs and their antimicrobial properties.[5]

In a previous study, a series of esters were prepared, including hexanoic acid, palmitic acid esters, and stearic acid esters. The study showed that these esters have good emulsifying ability and are likely oil-in-water type emulsifiers.[6] Additionally, isopropyl myristate ester was synthesized using lipase enzyme, achieving an 87% yield after 5 hours.[7,8] This ester, isopropyl myristate has been used as a natural oil substitute in cosmetic formulations due to its excellent spreading properties and ease of skin absorption.[9]

A previous study investigated the esterifications of oleic acid with ethanol using Amberlyst-15 as a catalyst, where the catalyst demonstrated high efficiency in the esterification process. Equilibrium was reached after approximately 6 hours, achieving a conversion rate exceeding 60%.[10] In a previous study, Oliec acid was esterified with methanol using Amberlyst-46 as a catalyst at 100C^{o} the reaction time was 120 minutes, and the yield exceeded 98%.[11]

Amberlyst-15 is considered one of the most important heterogeneous solid catalysts used in the esterification of fatty acids with alcohols, due to its high acidity, good selectivity, ease of separation from the reaction medium, and reusability. Few studies have utilized it; for example, it has been used in the esterification of short-chain acids, such as in the esterification of oleic acid with simple alcohols to produce biofuel. Attention has been directed toward the synthesis of fatty acid esters due to their biodegradability and non-toxicity. The hydrophobic fatty acid chain length and hydrophilic alcohol moieties confer these esters with distinct hydrophilic-lipophilic balance (HLB) values.[12,13]

In this research, the esterification of myristic acid with propanol-1 and subsequently oleic acid with propanol-1 was studied using Amberlyst-15 catalyst under the same conditions. Their emulsifying properties were then investigated by measuring the HLB values according to Griffin method.[14] and evaluating their effectiveness as natural emulsifiers in a cream formulation.it was found that blending the two esters produced a product with good skin absorption capacity and improved skin texture.

In the current era, the cosmetics market is experiencing substantial growth driven by consumer trends toward natural and sustainable products, with the natural emulsifiers market projected to reach USD 1.21 billion by 2032, at a compound annual growth rate (CAGR) of 4.6%.

Fatty esters, derived from natural fatty acids such as those found in plant oils, emerge as ideal alternatives to traditional synthetic emulsifiers due to their effective surface-active properties in stabilizing emulsions like creams and lotions, while minimizing skin irritation risks and environmental impacts. This reflects the urgent need for precise evaluation of these compounds to enhance stability and sensory performance in cosmetic formulations. The present study addresses this gap by characterizing fatty esters and assessing their efficacy as natural emulsifiers, with a focus on chemical and applicative aspects to support sustainable innovation in the industry.

Herbal cosmetics utilize plant-derived ingredients extracted directly from nature, free from harmful synthetic compounds. For instance, coconut oil serves as a commonly employed herbal ingredient renowned for its beneficial properties. Many consumers are increasingle concerned about the efficacy of toxic synthetic chemicals and mineral oils in conventional cosmetics, leading them to seek products containing traceable natural ingredients devoid of deleterious substances, while prioritizing both safety and efficacy.[15][16][17] In line with this trend, the present study evaluates prepared fatty esters as natural emulsifiers in the formulation of a cream-based cosmetic product.

Bandy et al. (2024) conducted a study on the formulation of a multi-herbal cosmetic cream, followed by an evaluation of its antimicrobial efficacy in treating symptoms of skin allergies. The assessment was performed under storage conditions ranging from 8-40C^{o} and 75% relative humidity, where the creams stability was evaluated, along with measurements of PH and total fatty matter content.[18]

Akash et al. (2023) synthesized a herbal cream for the treatment, nourishment, and moisturization of various skin types, incorporating aloe vera oil and basil into the formulation. Numerous studies were conducted on this cream to evaluate its physicochemical properties and therapeutic efficacy.[19]

Valarmathi et al. (2020) discussed the preparation of facial creams using diverse herbs, including powders from dried aloe vera, hibiscus, and coriander. These creams underwent evaluation based on several parameters, such as sensitizing properties, PH, stability, homogeneity, and viscosity, with the study demonstrating satisfactory results across all formulations.[20]

Expermintal

Chemicals used

High-purity chemical materials were used in the study. The system included myristic acid, which had a purity of 98% and was obtained from Merck. Also, Oliec acid with a purity of 99% was used, obtained from Merck. Additionally, 1-propanol (primary alcohol) with 99% purity was used, obtained from Sigma-Aldrich. Toluene with 99% purity from the same company was also used, along with Amberlist-15 of high purity from Merck. Chloroform with 99% purity from Sigma-Aldrich was included as well. Furthermore, anhydrous sodium sulfate

with 98% purity from Merck was used to ensure the removal of moisture from the samples.

Apparatus and Characterization Methods

The product was characterized using an FT-IR-4100 infrared spectrometer from Shimadzu, Japan, and a proton NMR spectrometer with a frequency of 400 MHz from Bruker, Switzerland. Additionally, some physical properties of the prepared esters were measured, including density using a pycnometer, and kinematic viscosity using an ostvald viscometer. Furthermore, the hydrophilic-lipophilic balance (HLB) of the prepared esters was measured according to Griffin method.

Procedure

Preparation of fatty acid ester

Myristic acid (MA) was first esterified, followed by esterification of Oleic acid (OA) with alcohol (1-propanol), using the Fischer esterification method, using Amberlyst-15 as a heterogeneous catalyst in toluene under reflux. The molar ratio used was 1:1 for (1-propanol/ fatty acid). All conditions are presented in table 1. After completion of the reactions, the catalyst was separated by filtration, the solvent was evaporated, Residue was solved in chloroform and washed four times using hot water. Afterwards, the ester layer separated using a Separating funnel. and dried using anhydrous sodium sulphate.[21] The reaction schemes were shown in figure1.

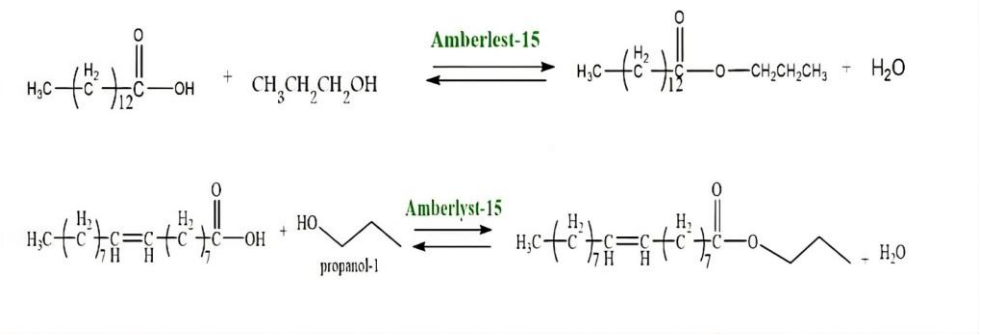


Figure 1: esterification reactions of myristic acid and oleic acid derivatives.

Table 1: Condition esterification of the synthesized fatty acid esters.

Acide	Alcohol	Ratio (Acid/Alcohol)	Catalyst(w/w) %	Temperature °C	Duration of reactionh	Yield%
MA	1-propanol	(1:1)	Amberlyst-15 (5W%)	110	7	72
OA	1- propanol	(1:1)	Amberlyst-15 (5W%)	110	12	69

RESULTS AND DISCUSSION

Characterization of the prepared esters

The identity of the synthesized esters was confirmed by FT-IR spectroscopy. As shown in Figure 2, the spectrum of pure myristic acid exhibits the characteristic broad O–H stretching band (≈ 2500–3300 cm^{-1}) and the sharp C=O stretching of the carboxylic acid (≈ 1700–1715 cm^{-1}). In contrast, the spectra of the ester derivative of propyl myristate display the disappearance of the O–H band and a shift of the carbonyl stretching vibration toward lower wavenumbers (≈ 1735–1750 cm^{-1}), which is typical of ester functionalities. Moreover, the appearance of a strong absorption band at ≈ 1200 cm^{-1} corresponding to the C–O stretching vibration provides further evidence of ester bond formation. These spectral changes clearly demonstrate the successful transformation of myristic acid into its ester derivative propyl myristate.

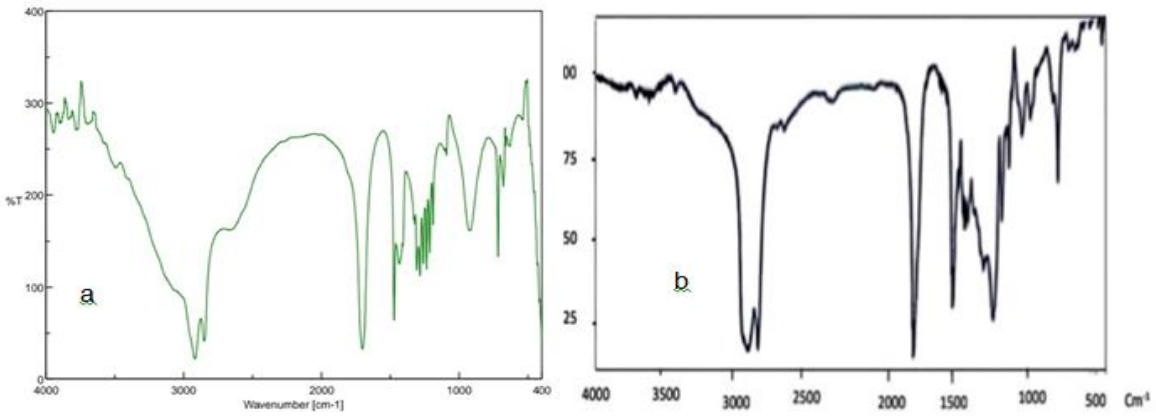


Figure 2: (a) FT-IR spectra of myristic acid, (b) FT-IR spectra of myristate propyl.

the spectrum of pure oliec acid exhibits the characteristic broad O–H stretching band (≈ 2500–3350 cm^{-1}) and the sharp C=O stretching of the carboxylic acid (≈ 1700–1711 cm^{-1}). In contrast, the spectra of the ester derivative of propyl myristate display the disappearance of the O–H band and a shift of the carbonyl stretching vibration toward lower wavenumbers (≈ 1735–1741cm^{-1}), which is typical of ester functionalities. Moreover, the appearance of a strong absorption band at ≈ 1200 cm^{-1} corresponding to the C–O stretching vibration provides further evidence of ester bond formation. These spectral changes clearly demonstrate the successful transformation of oliec acid into its ester derivative propyl oleate. As shown in figure 3.

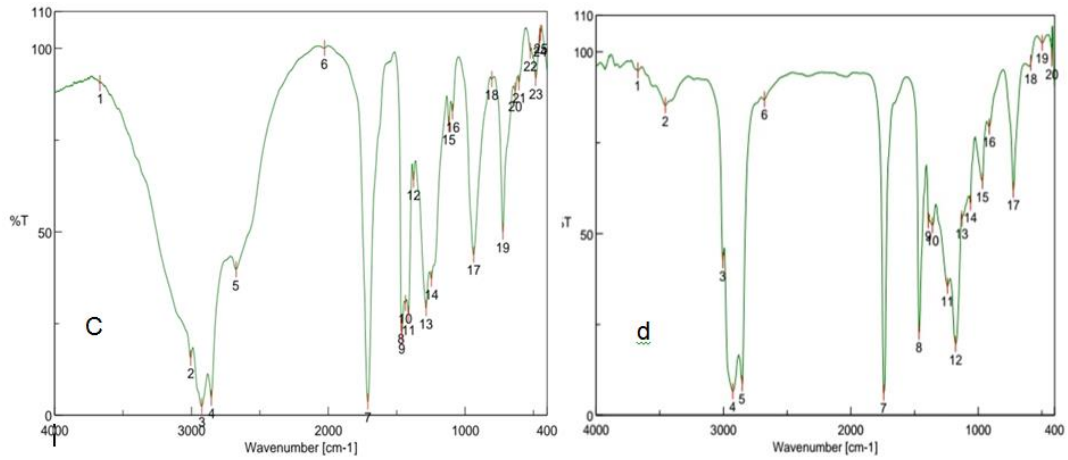


Figure 3: (c) FT-IR spectra of oliec acid, (d) FT-IR spectra of oleate propyl.

The structures of the synthesized ester derivatives were further confirmed by ^{1}H-NMR spectroscopy. As shown in Figure 4, the spectrum of propyl myristate exhibits a distinct resonance at ~4.0 ppm, assigned to the methylene protons adjacent to the ester oxygen (–CO–O–CH_2–). While the spectrum of propyl oleate exhibits a distinct resonance at ~4.1 ppm, assigned to the methylene protons adjacent to the ester oxygen (–CO–O–CH_2–). As shown in figure 5, A key feature in both spectra is the absence of hydroxyl proton resonances, which indicates the complete consumption of the alcohol and confirms successful esterification. Furthermore, the proton integration values are in good agreement with the theoretical proton counts of the synthesized esters, providing additional validation of the proposed molecular structures.

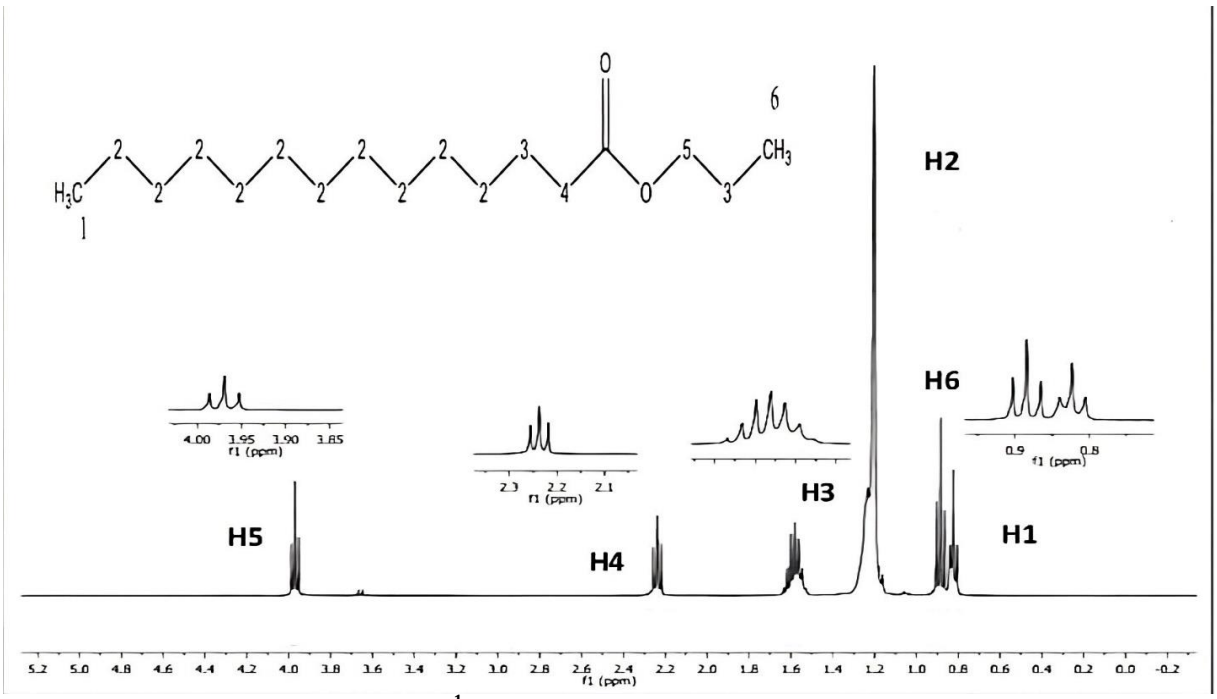


Figure 4: ^{1}H NMR spectra of myristate propyl.

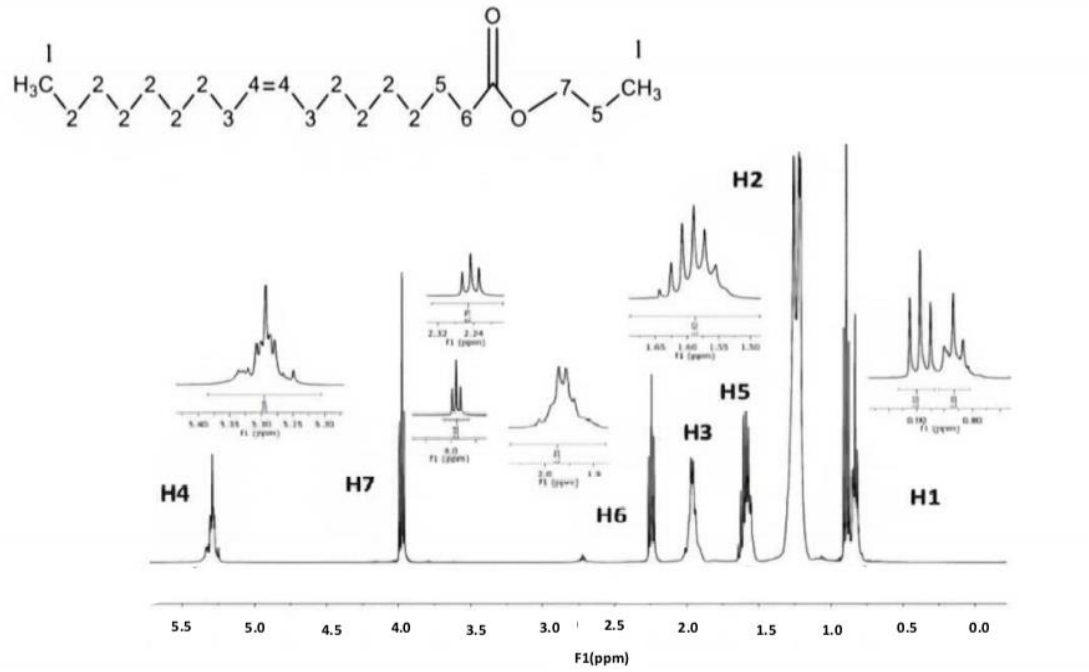


Figure 5: 1H NMR spectra of oleate propyl.

-Calculation of the Hydrophilic-Lipophilic balance (HLB) of the prepared esters

the hydrophilic-lipophilic balance values of the two resulting compounds were calculated according to Griffin method using the following equation.

HLB=20*Mh/M

Mh: molecular weight of the hydrophilic (water-attracting) portion of the molecule.

M: molecular weight of the entire compound.

The calculated HLB values for the propyl myristate ester (4.36) and the propyl oleate ester (3.64). It was observed that the HLB values of both compounds are less than 10, indicating that these compounds are oil-soluble and water-insoluble, which is attributed to the increased length of the carbon chain.

-A study on the application of synthesized fatty acid esters as emulsifiers in cosmetic products

a cosmetic formulation was prepared containing (2 gr) of propyl myristate ester and (2 gr) of propyl oleate ester, combined with supporting ingredients to achieve a creamy texture and effective moisturizing properties. To ensure formulation stability, beeswax and coconut oil were selected in carefully studied proportions, resulting in the following optimal ratios.

Beeswax: at a concentration ranging from 10% to 20% of the ester weight to enhance the cohesion and creamy texture of the formulation.

Coconut oil: at a concentration ranging from 80% to 90% of the ester weight to provide high moisturization and rapid skin absorption.

These ingredients were combined by gentle heating until the beeswax completely melted with the coconut oil. Propyl myristate and propyl oleate esters were continuously stirred during mixing to ensure a homogeneous formulation. Then, the formulation was allowed to cool gradually with continuous stirring to achieve a homogeneous and stable creamy texture. Two drops of the preservative benzalkonium chloride, which has an effective antimicrobial and antibacterial role, were added. Finally, a few drops of rose oil were incorporated to impart a pleasant and natural fragrance, enhancing the overall quality of the product.

The results showed that combining the two esters, propyl myristate and propyl oleate, produced a formulation with favorable properties. Propyl myristate demonstrated good skin penetration and improved skin texture, while propyl oleate provided effective moisturization.thus, their combination imparts more consumer-acceptable characteristics. The PH of the formulation was measured at 5, which is ideal as it closely matches the skin natural acidity, helping to maintain the skin barrier balance and reduce irritation and dryness.

-Measurement of the density of the resulting esters

The densities of the prepared esters, propyl myristate and then propyl oleate, were measured using a 25ml pycnometer by accurately weighing the volume of the solutions, and used the following equation.

Density=mass of the substance /displaced volume.

Table 2: Results of density measurements of the prepared esters.

Prepared ester	Empty pycnometer weight (gr)	pycnometer weight with solvent (gr) (chloroform)	Sample weight (gr)	pycnometer weight with sample solution (gr)	Sample density
Propyl myristate	24.4018	62.3770	0.25	62.2910	0.744
Propyle oleate	24.4018	62.3770	0.25	62.2742	0.708

- Measurement of the dynamic viscosity of the prepared esters using an Ostvald viscometer: the sample solution was prepared in a 25ml volumetric flask by weighing 0.25gr of each sample separately, then the volume was completed to 25ml with chloroform solvent while stirring until complete dissolution, resulting in homogeneous solutions with a concentration of 10g/l.

15ml was added to the viscometer for each sample separately, and the viscometer was placed in a water bath for 15 minutes at 40C^{o}. The time required for the solution to flow through the viscometer was measured and then multiplied by then instrument constant, thus obtaining the viscosity value according to the following equation.

$\eta = t \times Q$

t:the time required for the solution to flow through the viscometer.

Q:the instrument constant whose value is 0.033580 at a temperature of 40C^{o}.

Table 3: Results of dynamic viscosity measurements of the prepared esters.

Prepared esters	Temperature C^{o}	Device constant	Time required for the solution to flow (sec)	Kinematic viscosity (m^2.s)
Propyl myristate	40C^{o}	0.033580	18.576	0.623
Propyle oleate	40C^{o}	0.033580	20.34	0.683

CONCLUSIONS

This study demonstrated the successful green synthesis of propanol esters starting from myristic acid followed by oleic acid, using Amberlyst-15 as a heterogeneous acid catalyst through the fischer esterification process. The catalyst proved effective in preparing these esters, as confirmed by fourier –transform infrared (FTIR) spectroscopy and proton nuclear magnetic resonance (^{1}H-NMR), which verified the identity of the esters and the success of the esterification reaction.

The hydrophilic-lipophilic balance (HLB) of the prepared esters was measured and found to be less than 10, indicating that they likely form oil-in-water emulsion. The resulting esters, propyl myristate and propyl oleate, demonstrated their effectiveness as natural emulsifiers in producing a creamy formulation with desirable consumer properties, combining moisturizing, pleasant texture, and high quality. Additionally, the densities of the prepared esters were measured, followed by their kinematic viscosities using an Ostvald viscometer. It was found that propyl myristate has a higher density but lower viscosity compared to propyl oleate.

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