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Preparation of Methyl Esters of Solid Acids from Litsea Consimilis

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Abstract

This study focuses on the extraction and characterization of fatty acids from the seed oil of Litsea consimilis and their subsequent conversion into methyl esters. The oil demonstrated high iodine and Reichert-Meissl values, indicating a significant content of unsaturated and short-chain fatty acids. Glyceride analysis revealed that trilaurin constitutes over 90% of the triglycerides, confirmed through melting point analysis and hydrolysis yielding lauric acid as the sole component. The oil was fractionated into alcohol-soluble and -insoluble components, separating the solid and liquid fatty acids using lead acetate precipitation. The isolated solid acids were esterified to obtain methyl esters, which were then subjected to fractional distillation under reduced pressure. The esters were analyzed using iodine values and saponification equivalents to determine their composition. The major component identified was methyl laurate, with minor proportions of methyl oleate and traces of other fatty acid esters. Infrared spectroscopy confirmed the presence of characteristic functional groups in lauric acid. Chromatographic methods aided in distinguishing the individual acids present in minor quantities. The study highlights the high purity and yield of lauric acid derivatives from Litsea consimilis seeds, underscoring its potential as a valuable renewable resource for the production of medium-chain fatty acid esters. These findings have significant implications in biodiesel production, surfactants, and pharmaceutical intermediates, providing a scientific basis for the industrial utilization of this underexplored plant species.

Keywords: Methyl esters, solid acid catalysts, litsea consimilis, biodiesel synthesis, transesterification

INTRODUCTION

Litsea consimilis, a lesser-known species of the Lauraceae family, possesses seeds rich in oil with promising industrial potential. Figure 1 This study explores the extraction, separation, and chemical characterization of fatty acids present in its seed oil, focusing particularly on the solid acid fractions. Previous studies have highlighted the economic value of medium-chain fatty acids, especially lauric acid, in producing biodegradable surfactants, esters, and biofuels. By employing saponification, chromatography, and methyl esterification techniques, this research aims to isolate and quantify the major fatty acid constituents. The work further investigates the physicochemical properties of the oil and its esters, providing insights into the potential applications of *L. consimilis* as a sustainable source of high-purity lauric acid derivatives.[1-3]

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Physicochemical Characteristics

Almost all the important physicochemical characteristics were studied i.e. (i) specific gravity, (ii) refractive index, (iii) acid value, (iv) iodine value (v) saponiffcation value, (vi) ester value, (vii) acetyl value, (viii) hehner value and (ix) R.M. value.

Figure 1. Litsea consimilis.

The high iodine value found in the Litsea consimilies oil. The only reason for this can be that our seed samples were fresh and the work was started immediately. R.M. value (5.87) was also found to be exceptionally high.

Glyceride Structure

Most of the oil gets solidified at room temperature (20°), this was found to be due to high percentage (above 90%) of trilaurin. As trilaurin is sparingly soluble in cold 95% alcohol so, as much as possible: trilaurin was separated from the rest by first dissolving the oil in a boiling alcohol and them keeping overnight. Thus, the insoluble trilaurin alcohol to give fine needles of trilaurin m.pt. 45-46°C. There was no depression in m.pt. when it was mixed with an authentic sample of the glyceride. The oil, further on 130haracterizati followed by hydrolysis gave Lauric acid m.pt. 44°C as the only component acid. Thus the identy of the alcohol insoluble fat was confirmed(9-13) as trilaurin. [4-6]

The alcohol soluble fat was saponified and them free acids were generated by the addition of 10% H_2SO_4. For the 130haracterization of the free acids, reverse phase partition chromatography (paper only whatman no. 1) was applied. The mixed acids were found to be mainly a mixture of lauric acid with Rf. Value 0.66(14),

Paper chromatography was found to be a very good tool for the correct identification of higher fatty acids, even the negligible amount of acids can be easily detected.

Separation of solid from 'Liquid' Acids (Alcohol Soluble Fat)

The fatty acids (17.2 g.) so obtained were dissolved in about 90 ml of alcohol (95%). The solution was heated to boiling and to this hot solution 90 ml of lead acetate solution was added, (lead actate solution prepared by dissolving 7.5 g of lead acetate A.R., in 4 ml of acetic acid and then adding 125 ml. of alcohol). No immediate precipitate was formed, the contents were stirred by a glass rod and cooled till a white preicipitate was formed, and then kept over night. Next day contents were filtered to get insoluble solid acids and soluble "liquid acids"" and the precipitate washed thoroughly by alcohol. [7-9]

"Soild acids"

The insoluble lead salt was transferred to a large porcelain basin followed by an addition of concentrated Hcland boiling water, addition of HCI and water was continued till whole of the fatty acids were regenerated. The acid layer along with some aqueous layer was taken in a separating funnel. After cooling, ether was added in portions to get fatty acids as a separate layer. The ethereal layer was taken out and washed several times with cold distilled water. Ether was distilled off and residue kept in a vacuum desiccator, overnight. Next day the sloid acids (saturated acids) were taken and weighed. Amount of solid acids= 9.18

"***Liquid acids":***

The alcohol from the solution of soluble lead salt of liquid acids was removed completely by distillation under reduced pressure, a little of HCL was added to decompose the lead salt into free fatty acids (normally the lead salts get automatically decomposed due to the presence of acetic acid which is added at the time when lead acetate is added but addition of HCl was found to be essential).

Then the liquid acids were extracted with ether, washed thoroughly with distilled water till the washings were free from lead salt. Ether was distilled off and the contents kept for a couple of days in a vacuum desiccator. Weight of the liquid acids = 9.1 g.

FRACTIONAL-DISTILLATION OF "SOLID" ACIDS

The solid acids were converted into their methyl esters and then the methyl esters were fractionally distilled and collected at various temperatures.

The amount of different esters in the mixture was calculated after determining the iodine value and the saponification equivalent of the fractions.

Preparation of Methyl Esters of Solid Acids

9.1 g of solid acids were taken and dissolved in 45 ml of methyl alcohol to which one ml. of sulphuric acid was already added. The contents were refluxed for 2 hours. About half of the alcohol was distilled off. The esters were then extracted with ether and the ether extract washed first with distilled water and by 10% KOH and then again with water till washing were neutral to litmus. Ether was distilled off and last traces of moisture were removed first by distillation under reduced pressure and the by desiccation process. [10-12]

Weight of methyl ester of "Solid acids" -8.91 g. 8.91 g of methyl ester of solid acids was taken in a 100 ml flask attached to the fractional distillation apparatus (under reduced pressure).

The flask was heated by a Bunsen burner and the fractions collected. The pressure of the apparatus due to vacuum pump was found to be 5 mm. (difference between the atmospheric pressure and the scale reading). Following are the fractions collected at various temperature: Table 1

The iodine value and the saponification equivalent of fractions Fi F2, Fs and Fe were determined by the methods described earlier, following are the results obtained. Table 2

Table 1 Iodine Value and Saponification Equivalent of Fractions

Fractions	Iodine value	Saponification equivalent
F_1	1.1	205.7
F_2	1.3	208.5
F_3	2.4	216.0
F_4	8.3	220.4
Residue	40.2	243.7

Table 2. Fractions Collected with Corresponding Temperatures and Weights

Fractions	Temperatures (°C)	Weight of the ester collected (g)
F_1	120–132	3.04 g
F_2	132–145º	4.41 g
F_3	145–160º	0.809 g
F_4	164–186º	0.403 g
Residue	– – –	0.190 g
Loss	– – –	0.058 g

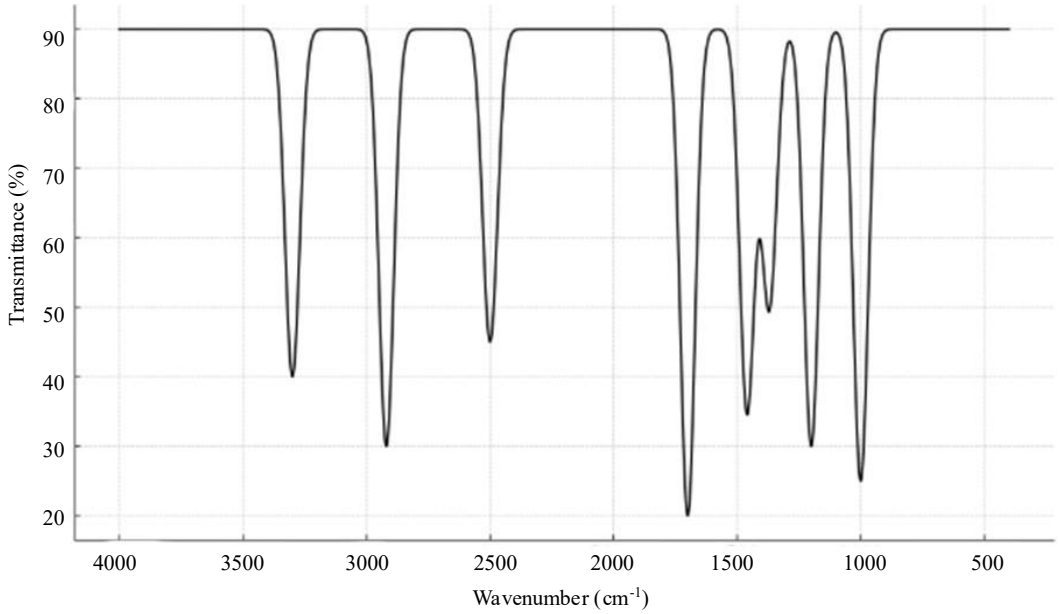

Figure 2. I. R. Spectrum of lauric acid.

As the amount of Myristic, Linolic, and Unidentified acid was found to be only in traces in the 'solid' acid so it was assumed for calculation that both the fractions Fa and Fe contain only Oleic and Lauric acids.

Considering the iodine value and the saponification equivalent of Fi and F2, it was very clear that both the fractions contain (almost) only one component of fatty acid ester i.e. Lauric acid ester.

Therefore in fraction Fi, amount of Lauric acid ester-3.04 g. In fraction F_2 the amount of Lauric acid ester = 4.41g . Methods used for calculations are as prescribed by Hilditch. [13-16]

The iodine value of Lauric acid ester is zero so the calculations were made directly from the iodine vale formula. Figure 2

CALCULATIONS

Fraction Fs:-
Weight of the fraction Fa = 0.809(w)
lodine value of fraction Fa = 2.4(I_{w})
lodine value of Lauric acid ester = 0(I_{e})
lodine value of Oleic acid ester = 85.8(ly)
Let the weight of Lauric acid ester be xg.
And the weight of Oleic acid ester y g.

Therefore
All the results are tabulated and shown in Table 3

Table 3. Distillation data of methyl esters with S.E., I.V., weight, and methyl laurate/oleate content.

Fractions with B.P. (°C)	S.E.	I.V.	W/g	Methyl laurate	Methyl oleate
F_1 (120 – 132°)	205.7	1.1	3.040	3.040	-
F_2 (132 – 145°)	208.5	1.3	4.410	4.410	-
F_3 (145 – 160°)	216.0	2.4	0.809	0.7864	0.0226
F_4 (164 – 185°)	220.4	8.3	0.403	0.3594	0.0436
Residue	243.7	40.2	0.190	0.1010	0.0890
Loss	-	-	0.058	-	-
Total	-	-	8.910	8.6968	0.1552

According to the formulae:
$x + y = w$
$x.I_x + y.Iy = w.I_w$

$x + y = 0.809$
$x.0 + y.85.8 = 0.809 \times 2.4$

Therefore
$Y = \frac{0.809 \times 2.4}{85.8} = 0.226g$
And $x = 0.8090 = 0.0226g = 0.7864g$

Weight of Lauric acid ester thus determined = 0.78648
Weight of Oleic acid ester thus determined 0.0226 g.
Fraction Fe:
Weight of the fraction F_{4} : 0.403 g. (w)
lodine value of fraction F_{4} : 8.3 (Iw)

Let the weight of Lauric acid ester be: x g.
And the weight of Oleic acid ester y g.
x + y = 0.403x.0 + y. 85.8 = 0.403 x 8.3

Therefore y = (0.403 * 8.3)/85.8 = 0.0436/g * gand -0.403-0.0436 g * = 0.3594g .
Weight of Lauric acid ester thus determined 0.3594 g.
Weight of Oleic acid ester thus determined 0.0436 gResidue:
In residue it was assumed that there was no unsaponifiable matter because that portion was removed before the liberation of free fatty acids. [17]
Weight of the residue 0.190 (w)
Iodine value of the residue 40.2 (I)

There fore x + y = 0.190
And= 0.190 x 40.2 / 85.8 = = 0.0890. g.

Therefore x = 0.190-0.0890
= -0.1010 g.
Weight of Lauric acid ester thus determined = 0.1010 g.
Weight of Oleic acid ester thus determine = -0.0890 g.

CONCLUSION

The present study demonstrates the effective extraction, separation, and characterization of fatty acids from Litsea consimilis seed oil, with a particular focus on the preparation of methyl esters from the solid acid fraction. Analytical results confirmed lauric acid as the predominant component, primarily in the form of trilaurin, with minor traces of oleic and other fatty acids. The successful conversion of these fatty acids into methyl esters and their detailed analysis through fractional distillation and spectroscopic techniques underline the potential of L. consimilis as a viable source of medium-chain fatty acids. These findings offer promising prospects for the use of this oil in biofuel production and other industrial applications requiring high-purity fatty acid esters.

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