Wax Ester from Vegetable Oils Extraction and Transesterification

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Abstract

Waxes are long-chain esters having at least 12 carbons in their chain that are generated from fatty acids and alcohols.The chemical-catalyzed approach for synthesising wax esters has various drawbacks, including the need for corrosive acids, handling risks, significant energy consumption, and degradation of synthesised esters. As a result, research into the synthesis of wax esters from natural sources, which are said to be environmentally beneficial, has received more attention. Animal and plant products including beeswax, sperm whale, jojoba seeds, sheep wool, seafowl feathers, etc. may be used to extract natural wax ester. The goal of the current research was to create wax esters by a transesterification process and analyse the oil content of three naturally occurring plant seeds: Annona reticulate, Parkia timoriana, and Citrus reticulata.

Keywords: Transesterification, vegetable oil seeds, and wax esters.

Introduction

A mixture of long-chain apolar lipids known as waxes forms a protective layer (cutin in the cuticle) on the leaves and fruits of plants as well as in animals (honeybee wax, insect cuticular lipids, sperm whale spermaceti, skin lipids, bird uropygial glands, depot fat of planktonic crustacea), algae, fungi, and bacteria. Some waxes come from minerals. In most cases, the term "wax" refers to a material that, when exposed to slightly higher temperatures, transforms from a solid to a low viscosity liquid. These 12 carbon or longer chain length fatty acids and alcohols are the source of these long chain esters. Garba and his colleagues investigated the fatty acid profiles, content, and physico-chemical characteristics of Citrullus vulgaris seed oils. Fatty acids, sterols, tocopherols, and trocotrienols have been examined in seed oils from Lophira lanceolata and Carapa procera. In order to save the fossil fuels that are being used less and less each day, a lot of effort has been put into producing biodiesel from seed oils. But no explicit initiatives have been undertaken to create synthetic wax ester from naturally occurring seed oils.

Animal and plant products including beeswax, sperm whale, jojoba seeds, sheep wool, seafowl feathers, etc. may be used to extract natural wax ester. Jojoba oil and spermaceti oil stand out among them for their commercial value, however the sperm whale was added to the endangered species list in 1970. Jojoba oil has certain characteristics with spermaceti oil and may be used as a replacement, however jojoba plant growth is limited to desert environments, and the cost of jojoba oil manufacturing is rather expensive. Natural unsaturated wax esters are hence too costly and uncommon for practical usage.

Aiming to replace natural wax esters, the manufacture of synthetic wax esters is necessary in light of the aforementioned factors. The chemical-catalyzed approach has a number of drawbacks, including the need for corrosive acids, handling risks, significant energy consumption, and degradation of synthesised esters. Because of their moderate reaction conditions and environmentally favourable design, lipase-catalyzed procedures have gained attention. In addition to lipase, Novozym 435 and Lipozyme RMIM were utilised in the production of wax ester.

For the manufacture of wax ester, plant oils are widely accessible and renewable sources. Plant oils (Jatropha oil) were extensively employed by numerous researchers for the synthesis of therapeutic soaps in addition to the creation of wax esters. Additionally, efforts were undertaken to enhance the quality of conventional soaps by studying the ashes extracted from plants. Vegetable oils have a sulphur component that is almost negligible, unlike hydrocarbon-based fuels, which means that the harm that sulphuric acid does to the environment is lessened. Research was done to produce hydrocarbon liquids from plant oils (castor oil) keeping this in mind. Furthermore, the manufacturing of plant oils removes more carbon dioxide from the environment than is released when they are burned. As a result, it reduces the atmosphere's rising carbon dioxide level.

Plant oils are essentially triglycerides, which are esters of three fatty acids and one glycerol, in terms of their chemical makeup. The amount of double bonds and the length of the carbon chains in the fatty acids varies. Vegetable oils often include the fatty acids stearic, palmitic, oleic, linoleic, and linoleic.

Wax ester synthesis as interpreted chemically

The essential family of chemical reactions known as transesterification, in which one ester is changed into another by switching the alkoxy moiety, is referred to as a transesterification reaction (Figure 1). Oils and fats are transesterified with methanol to produce glycerol as a byproduct and fatty acid methyl esters as a byproduct in the manufacturing of biodiesel. Instead of methanol, wax ester uses long-chain alcohols with a chain length of 12 carbons or more (Figure 2).

One mole of a triglyceride and three moles of the alcohol are needed for the reversible process known as transesterification. To boost the yields of the alkyl esters and enable their phase separation from the glycerol produced, an excessive quantity of alcohol is utilised. Transesterification really entails a series of subsequent, reversible processes. At each stage, one alkyl ester molecule is produced for every mole of glyceride, starting with the conversion of triglycerides to diglycerides, then to monoglycerides, and finally to glycerol (Figure 3). The goal of the current research was to investigate the oil content of three types of plant seeds that are readily available: Annona reticulata, Parkia timoriana, and Citrus reticulata. Additionally, wax esters were created using a transesterification process.

Materials and Methods

To make the catalyst, the trunk of the banana plant was thinly chopped into pieces and let to dry in the sun for several days. A dry object was lighted, allowed to burn spontaneously, and then allowed to cool to normal temperature. The length of time it takes for a substance to burn and naturally cool depends on how much of it is eaten. While cooling down takes longer, burning is often predicted to be accomplished in a half-hour. The catalyst enhances the post-harvest plant's value and operates in a heterogeneous manner.

The investigational seeds were ground in a grinder. A magnetic stirrer was employed to perform the reaction and extract the oil. Buchi rotavapour R-200 was used to evaporate the solvents. NMR spectrometer: Bruker 300 MHz. The product's 1H NMR analysis in CDCl3 is carried out using a 300 MHz instrument, while the 13C analysis in CDCl3 is done with a 600 MHz instrument. Additionally, IR spectra were recorded using a Perkin-Elmer FT-IR IR-Spectrometer. Three seeds in all were gathered in Rajasthan, India, in order to extract the oil. Two were gathered in Jaipur and Ajmer. Citrus reticulata and Annona reticulata are a few of examples. Parkia timoriana, a seed variety from Kota, was the last one. Transesterification was carried out using cetylalcohol that was acquired from Himedia. Ash from the Musa balbisiana stem was the catalyst used in this process.

The banana plant's trunk was thinly cut into pieces and allowed to dry in the sun for several days in order to prepare the catalyst. Dry material was lit and then allowed to burn naturally and cool to room temperature. The amount of material consumed determines how long it takes to burn and cool naturally. Burning is often anticipated to be finished in a half-hour, but cooling down takes longer. The catalyst functions in a heterogeneous way and is the post-harvest plant's method of value addition.

$$\underset{\text{Ester}}{RCOOR^1} + \underset{\text{Alcohol}}{R^2OH} \xrightleftharpoons{\text{Catalyst}} \underset{\text{Ester}}{RCOOR^2} + \underset{\text{Alcohol}}{R^1OH}$$

Figure 1. General Transesterification equation

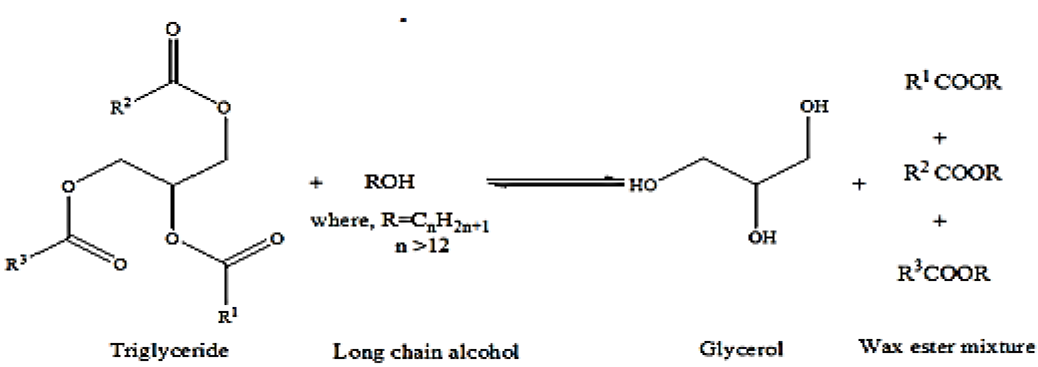

Figure 2. Wax ester produced by transesterifying a trigyceride.

$$\text{Triglyceride} + \text{ROH} \rightleftharpoons \text{Diglyceride} + R^1\,\text{COOR}$$
$$\text{Diglyceride} + \text{ROH} \rightleftharpoons \text{Monoglyceride} + R^2\,\text{COOR}$$
$$\text{Monoglyceride} + \text{ROH} \rightleftharpoons \text{Glycerol} + R^3\,\text{COOR}$$

Figure 3 Esters and glycerol produced during the transesterification of vegetable oil with alcohol.

Catalyst composition: Atomic absorption spectroscopy, flame photometry, and chemical analysis were used to evaluate the catalyst's chemical composition. Along with eleven additional metals, including Al, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Cd, and Pb, which are only present in trace levels (ppm level), the main components present are K, Na, CO3, and Cl. Metals may be found as their oxides, chlorides, or carbonates. Additionally, there exist tiny carbon particles (Figure 1).

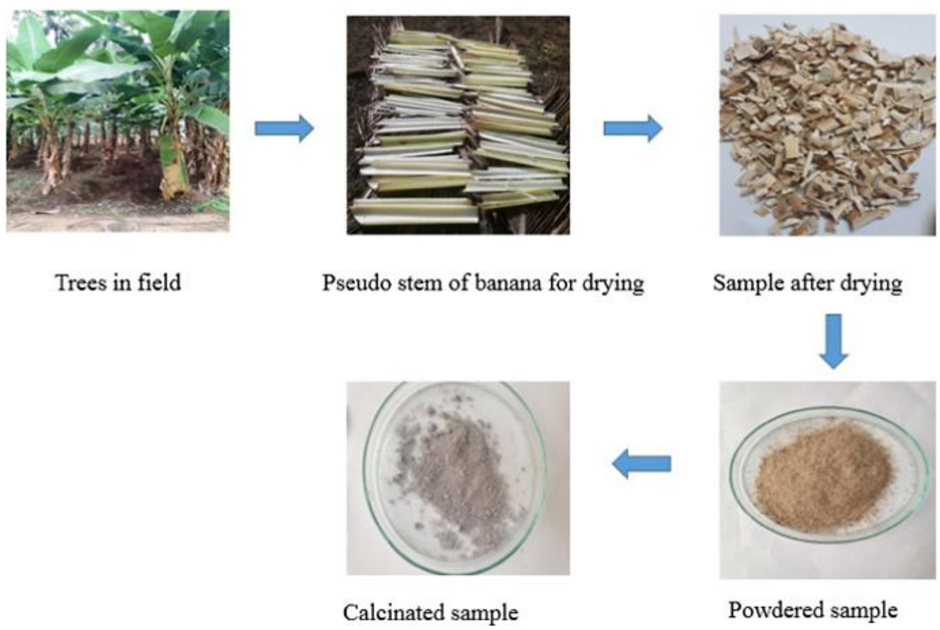

Figure 4. Preparation of catalyst

General Instructions for Oil Extraction:

The solvent extraction technique was used as the method for extracting the oil. The seeds were first dried in the sun before being ground in a grinder. These crushed seeds were combined with petroleum ether (40-60C, 10 mL/g) at room temperature and swirled magnetically for roughly 4 hours. To get the crude oil, the solid residue was filtered out using a Buchner funnel, and the solvent was drawn out of the filtrate using a rotary vacuum evaporator. To extract the majority of the oil, the procedure was repeated using new solvent and the seed cake. Finally, employing column chromatography over silica gel (60-120 mesh) and a solution of mild petrol (40-60C) and ethyl acetate (2%), the extracted oil was refined.

Experiment specifics: Annona seed no. 1, reticulate Known locally as Seetaphal 10 grammes of seeds were measured, with a 28% oil content after extraction. Parkia timoriana, seed number 2, Yougchak, Timor-Leste 12 g of seeds were collected, with a 22% oil content after column. Citrus reticulata, seed no. Known locally as: mandarin orange Taken seed weight is 10g, and the oil content after column is 22%.

The Findings and Discussion

Three distinct seeds, which are listed in Table 1, were looked at in quest of a suitable non-traditional source of oil. These seeds were mostly gathered in India's north-eastern area.

Table-1 Names of the plants whose oil content was measured and their names

Common name	Scientific name	Oil content before purification (%)	Oil content after purification (%)
Seetaphal	Annona reticulate	33%	28%
Yougchak	Parkia timoriana	35%	22%
Mandarin orange	Citrus reticulate	30%	22%

Transesterification of oils to long chain wax esters:

Optimising different reaction conditions for the production of wax ester using soya oil: Using an alternative solvent Cetyl alcohol weight is 726 mg (3 mmol equivalent), soy oil weight is 872.33 mg (1 mmol equivalent), and catalyst weight is 175 mg (20% of oil). Thus, under all reaction circumstances, the ratio of oil to cetyl alcohol remained at 1:3.

Thus, it was discovered that the oil/alcohol ratio of 1:3, the catalyst (20% of oil), the reaction temperature of 110^{0}C, and petroleum ether as the solvent are the most suited conditions for the synthesis of wax ester. At a temperature of 110°C, wax esters were created utilising cetyl alcohol as the solvent and acetone as the solvent, together with a catalyst made from the trunk of Musa balbisiana.

Table-2 Transesterification with cetyl alcohol

Name of plant	Reaction time	Yield of wax ester before purification (%)	Yield of wax ester after Purification (%)
Annona reticulata	3	98	95
Parkia timoriana	3.5	97	94
Citrus reticulata	3	98	96

Conclusion

In the current study, the oil content of seeds collected from various Rajastrhan areas of India was assessed and analysed using the best available laboratory methods. The Annona reticulata, Parkia timoriana, and Citrus reticulata plant seeds that were tested for the inquiry all had a healthy quantity of oil content (28%, 22%, and 22%, respectively). The oil content of the seeds under research was used to synthesise wax esters, which is the most essential outcome of evaluating the most optimised conditions for wax ester manufacture. The distinctive peaks were also found by the spectral analysis of oil and wax ester.

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