

Production of Oil from Agro Seeds using Mechano-thermal Extraction: A Review of Chemical, Pharmaceutical, and Food Applications

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Abstract

The increasing demand for sustainable raw materials and renewable industrial resources has intensified research into the extraction of oils from agro seeds for chemical, pharmaceutical, and food applications. Agro-seed oils are valuable due to their biodegradability, nutritional composition, eco-friendliness, and wide industrial applicability. This review examines the production of oil from agro seeds using mechano-thermal extraction techniques with emphasis on extraction efficiency, physicochemical characteristics, and industrial utilization. Mechano-thermal extraction combines thermal conditioning and mechanical pressing to improve oil recovery by weakening cellular structures and enhancing oil release from seed matrices. The review further discusses enzyme-assisted aqueous extraction (EAAE), which employs enzymes such as proteases, cellulases, and pectinases to hydrolyze cell wall components and improve oil yield. Important process parameters including pH, temperature, hydrolysis time, enzyme concentration, particle size, and solid-to-solvent ratio significantly influence extraction performance and oil quality. Agro-seed oils possess desirable physicochemical and bioactive properties that support their applications in biodiesel production, pharmaceuticals, cosmetics, food processing, nutraceuticals, soaps, lubricants, and other engineering products. The utilization of agro-seed residues for oil production contributes to environmental sustainability by reducing agricultural waste and dependence on fossil-based resources. Furthermore, mechano-thermal extraction offers advantages such as lower solvent usage, reduced environmental impact, improved extraction efficiency, and adaptability to industrial-scale operations. The review concludes that mechano-thermal extraction and enzyme-assisted techniques provide sustainable and economically viable alternatives for agro-based oil production. Continued research into process optimization, refining technologies, and industrial commercialization is essential for maximizing the economic and environmental benefits of agro-seed oil production.

Keywords: Mechano-thermal extraction, agro-seed oils, enzyme-assisted aqueous extraction, sustainable processing, industrial applications

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INTRODUCTION

The growing global demand for renewable energy resources, sustainable industrial feedstocks, and environmentally friendly products has stimulated extensive research into the extraction and utilization of oils derived from agro seeds. Agro-based oils obtained from plant seeds and oleaginous biomass have become increasingly important due to their biodegradability, low toxicity, renewability, and broad industrial applicability. These oils are widely utilized in food industries, pharmaceuticals, cosmetics, biodiesel production,

soap manufacturing, lubricants, and several engineering applications. The increasing depletion of fossil fuel reserves, coupled with rising environmental concerns associated with petroleum-based products, has intensified the need for alternative renewable resources capable of supporting sustainable industrial development [1].

Agricultural activities across the world generate enormous quantities of agro residues and seed wastes that are often discarded without significant economic utilization. Many agro seeds contain appreciable amounts of lipids, proteins, carbohydrates, essential fatty acids, vitamins, and antioxidants that can be converted into valuable industrial products. The recovery of oils from these agro seeds not only provides renewable raw materials for industrial applications but also contributes to waste minimization and environmental sustainability. Consequently, efficient extraction technologies are essential for maximizing oil recovery and improving the quality of extracted oils for commercial utilization.

Conventional methods of oil extraction from agro seeds include solvent extraction, cold pressing, hydraulic pressing, and aqueous extraction techniques. Although these methods have been widely used, several limitations such as low extraction efficiency, high energy consumption, environmental pollution, solvent toxicity, and high operational costs have restricted their sustainability and industrial efficiency. In response to these challenges, mechano-thermal extraction has emerged as a promising alternative technique for agro-seed oil recovery. Mechano-thermal extraction combines the application of controlled thermal energy and mechanical force to facilitate the rupture of cellular structures within seed matrices, thereby enhancing oil release and extraction efficiency.

The mechano-thermal extraction process operates by subjecting agro seeds to thermal conditioning before mechanical pressing. Thermal treatment softens the seed structure, reduces oil viscosity, weakens intermolecular interactions, and improves oil mobility within the cellular network. Mechanical compression then facilitates efficient expulsion of the oil from the seed material. This integrated process has gained considerable attention because of its simplicity, reduced solvent dependency, lower environmental impact, and suitability for both small-scale and industrial-scale operations. Mechano-thermal extraction also minimizes contamination risks associated with organic solvents, making it highly attractive for food and pharmaceutical applications where product purity and safety are critical [2].

In addition to mechano-thermal extraction, enzyme-assisted aqueous extraction (EAAE) has emerged as an innovative and environmentally friendly approach for improving oil recovery from agro seeds. Enzymes play important roles in food and agro-processing industries by enhancing product yield and improving processing efficiency. According to Yusoff et al. (2022), [3] the addition of enzymes to oilseeds and oleaginous fruits promotes the degradation of cell wall materials and hydrolysis of structural polysaccharides, thereby enhancing oil release from plant tissues. Oil within plant cells is usually enclosed by complex macromolecular structures, and enzymatic hydrolysis helps to break down these barriers to facilitate efficient extraction.

Enzyme-assisted aqueous extraction commonly employs enzymes such as proteases, cellulases, pectinases, and hemicellulases to hydrolyze protein matrices and polysaccharide components surrounding oil bodies. The effectiveness of EAAE depends on several processing parameters including pH, temperature, enzyme concentration, hydrolysis duration, particle size, and solid-to-solvent ratio. Proper optimization of these factors significantly improves oil yield, extraction efficiency, and product quality. Compared with conventional solvent extraction methods, enzyme-assisted extraction offers several advantages including reduced solvent use, improved environmental safety, enhanced nutritional quality, and reduced processing hazards.

Agro-seed oils possess important physicochemical properties that determine their suitability for chemical, pharmaceutical, and food applications. Parameters such as viscosity, density, acid value, peroxide value, iodine value, moisture content, and saponification value are commonly evaluated to

assess oil quality and stability. Oils with favorable physicochemical characteristics can serve as valuable raw materials in biodiesel production, edible oils, cosmetics, pharmaceuticals, paints, detergents, bio-lubricants, and polymer industries. In pharmaceutical applications, agro-seed oils are utilized as carriers for drug formulations, ointments, emulsions, and nutraceutical products due to their bioactive compounds and antioxidant properties. In food industries, these oils contribute essential fatty acids, vitamins, and functional nutrients necessary for human health [4].

The industrial utilization of agro-seed oils aligns with global sustainable development goals aimed at promoting renewable resource utilization, reducing greenhouse gas emissions, and minimizing environmental pollution. The conversion of agricultural waste materials into valuable industrial feedstocks supports circular economy practices and creates opportunities for economic diversification, rural industrialization, and job creation. Developing countries with abundant agricultural resources particularly stand to benefit from the commercialization of agro-based oil production technologies [5].

Despite the growing interest in agro-seed oil extraction, several challenges remain regarding extraction efficiency, process optimization, product stability, and large-scale industrial implementation. Further research is therefore necessary to improve mechanothermal extraction systems, optimize enzyme-assisted processes, and develop cost-effective refining techniques that enhance oil quality and industrial applicability. This review examines the production of oil from agro seeds using mechanothermal extraction techniques and explores the chemical, pharmaceutical, and food applications of the extracted oils. The study also highlights recent developments in enzyme-assisted extraction technologies and discusses the role of agro-based oils in promoting sustainable engineering and industrial development.

LITERATURE REVIEW

Enzyme-assisted Aqueous Extraction (EAAE)

An important application of enzymes in food processing is to enhance the yield of products from the raw materials and make it more amenable to treatment. Addition of enzymes to oilseeds and oleaginous fruits promote the degradation of cell wall components and hydrolysis the lipid bodies and the structural polysaccharides, thus enhancing oil recovery. This is based on the hypothesis that oil in plant materials is usually inside the cells, linked with other macromolecules so that upon partial hydrolysis, oil extraction can be enhanced [6]. Owing to the structural complexity of plant materials, factors such as solid-to-solvent ratio, Ph, temperature, enzyme concentration and composition, hydrolysis time and particle size of the plant materials.

Aqueous enzymatic extraction of seed oil using different commercial enzymes (protease, pectinase, 11odelling and a-amylase) was reported by Puangsri *et al.* (2022) [7]. They mixed the seed powder with distilled water at a ratio of 1:10 (w/v) and boiled for 5 mm. Subsequently, 2% of enzyme was added and the mixture was incubated at 45°C with constant shaking for a day. Boiling distilled water was added on the next day to terminate the enzyme activities. The oil was then recovered using centrifugation. The highest oil yield was obtained when protease was used, subsequently followed by pectinase, Cl-amylase and 11odelling. Their study demonstrates the enzyme specificity on the cellular composition of Avocado seed. The existence of a higher amount of pectin in the cellular composition of Avocado seed suggests that the oil is liberated more rapidly from the cellular matrix by breaking down the pectin, which is achieved by the action of enzyme pectinase.

Aqueous enzymatic extraction is an environmental-friendly oil extraction technique. However, the application of this technique in oil extraction is still limited due to long processing time and high cost of enzymes. Also, emulsification can be formed in the oil, which requires the post-extraction de-emulsification process to enhance the oil yield [8].

Enzyme-assisted extraction is based on the addition of specific enzymes during the extraction process

to enhance the yield and recovery by breaking the cell wall and modelling the structural polysaccharides and lipid bodies. Cellulase, α -amylase, protease and pectinase are some examples of enzymes used to support the extraction and yield recovery [9]. The advantages of enzyme-assisted extraction include lower extraction temperature, no involvement of explosive solvents and no production of harmful wastes [10]. However, enzyme composition and concentration, the particle size of the sample, solid to water ratio as well as hydrolysis time are among the factors identified which might influence the efficiency of enzyme-assisted extraction [11].

Ultrasound-Assisted Solvent Extraction (UASE)

Utilization of ultrasound devices in the extraction of plant oil has recently drawn considerable attention in the food processing industry. Ultrasound is acoustic waves with frequencies higher than 20 kHz, which is above the threshold of human auditory range. In comparison to classical techniques (e.g. Soxhlet or solvent extraction), UASE allows shorter extraction time because it creates cavitation and contact surface between plant matrix and extraction solvent, which can break down the plant cell wall and the structure of oil emulsion, thereby liberating the intracellular compounds into the extracting solvents.

Extraction of seed oil using UASE technique was initially proposed [12]. They mixed Mango seed powder with hexane at the ratio of 1:8 (w/v) and sonicated in an ultrasonic water bath at 50°C for 30 min, followed by oil recovery using evaporation. The oil yield obtained by UASE was compared with the classical techniques. Results of their study showed the yield of Mango seed oil obtained by UASE (23.03%) was comparable with solvent extraction (24.03%), but lower than Soxhlet extraction (30.4%). This pilot study suggested that UASE can extract Mango seed oil at a relatively short extraction time as compared to the classical techniques. To obtain the highest recovery papaya seed oil with desirable quality, the researchers suggested the necessity experiment to optimize the UASE parameters. Subsequently, [13].

Conducted another experiment targeting on optimizing the UASE parameters (powder-to-solvent ratio, sonication power, sonication temperatures and sonication times) of seed oil extraction. Results of their study showed the optimum UASE parameters to obtain the greatest recovery of high oxidative stability and antioxidant activity seed oil were powder-to-solvent ratio of 1:7 (w/v), sonication power of 700 W, sonication temperature of 62.5°C and sonication time of 38.5 min. Under these conditions, the oil yield obtained was 23.30%. [14].

On the other hands, Li *et al.* (2020) compared the efficiency of ultrasound-microwave synergistic solvent extraction (UMSSE) and UASE in extracting seed oil. The UMSSE was performed using an ultrasonic horn transducer and a microwave oven, whereas the UASE was performed using an ultrasonic water bath. Both experiments were performed under different time (20-60 min) and temperatures (30-50 °C) using different solvent-to-powder ratio (10-30 ml g⁻¹), followed by oil recovery using evaporation. Under the fixed experimental conditions (time: 60 min; temperature: 50°C and powder-to-solvent ratio: 30 ml g⁻¹), oil yield obtained by UMSSE was about 3% greater than UASE indicating the combined use of microwave and ultrasound can expedite the extraction of seed oil. This is because the energy generated by microwave radiation is higher than other thermal treatments, due to the generation of efficient volumetric heating [15].

Prolonged sonication time (>30 min) could potentially increase oil yield, but this action might destroy the thermo-labile bioactive compounds. Thus, Senrayan & Venkatachalam (2019) proposed UASE of seed oil using short extraction times (<15 min). They evaluated the effects of UASE parameters (e.g. sonication temperature, sonication time, and amplitude and powder-to-solvent ratio) on the extraction efficiency of seed oil. It was reported that oil recovery elevated with increased amplitude level, probably the enhancement of the contact surfaces between seed powder and extracting solvent when the ultrasonic cavitation is increased. Moreover, their study demonstrated that the utilisation of higher powder-to-solvent ratio in UASE produced more oil yield, suggesting the usage of high volume solvent

improves the diffusion through the reduction in viscosity. After UASE, the SEM image of seed powder showed broken cell walls and perforations. This phenomenon indicates the destructive action of cavitation on the plant matrix. Their study showed the optimum UASE parameters to generate the greatest recovery of seed oil were 80% amplitude level, 7 mm sonication time, 48°C sonication temperature and 13 ml of solvent-to-powder ratio.

Besides, Zhang *et al.*, (2019) also measured the efficiency of UASE of seed oil using an ultrasonic water bath. To achieve the best extraction conditions, UASE parameters were optimised using a Box-Behnken experimental design. It was reported that the ultrasonic power had the greatest influence on oil extraction efficiency, followed by powder-to-solvent ratio and sonication time. The optimum extraction was set as follows: ultrasonic power: 250 W, powder-to-solvent ratio: 1:16 (w/v) and sonication time: 20 min. Application of UASE for seed oil extraction produced 7% more oil yield than the traditional Soxhlet extraction. The output of this research highlighted the feasibility of UASE as a simple, inexpensive and potential substitute to the traditional oil extraction method.

Previously, many studies have been devoted to investigating the extraction efficiency of ultrasound devices, especially UASE, in manufacturing seed oil. Although these studies suggest that UASE as a promising non-conventional technology for seed oil extraction, it still has limitations. For instance, this technology has been only applied using laboratory-scale equipment (e.g. ultrasonic water bath), and until now, no research has been carried out in a commercial scale edible oil industry. Also, most literature on UASE of seed oil focused on the examination of sonication temperature, sonication time, sonication power, powder-to-solvent ratio, and extraction yield and oil quality. However, no research has been carried out to examine the economic cost of the proposed process and the amount of energy consumed per kg per ton of seed powder used, in comparison to conventional methods.

Ultrasound, within the range of 20 kHz to 100 MHz, is a special sound wave which beyond human hearing. It creates expansion and compression cycles when passes through a medium. Ultrasound could improve the mass transfer, induce a greater penetration of the solvent into cellular materials and also facilitate the release of contents through the disruption of biological cell walls. This extraction method is recommended for thermo labile compounds that tended to be altered or neglected in the solvent extraction method.

The advantages of ultrasound-assisted extraction are simple, inexpensive equipment, reduction of extraction time, temperature, energy and solvent used. Nevertheless, there are some factors that might influence the efficiency and effectiveness of ultrasound-assisted extraction, which includes moisture content of the sample, particle size, solvent selection, temperature, pressure, frequency and time of sonication.

Sub- and Supercritical Fluid Extraction

Sub- and supercritical fluid technologies are eco-friendly because the solutes dissolved in the fluids can be completely separated using a depressurization system. The common fluids (solvents) used in both systems are carbon dioxide (CO₂), butane, pentane and water. When a fluid is forced to temperatures and pressures above the critical point, it becomes a supercritical fluid. It is a state where the gas and liquid are indistinguishable from each other and become compressible.

Subcritical fluid extraction operates in the same manner as supercritical fluid extraction, except it operates below the critical point of the fluids.

More than 300 plant species have been extracted using supercritical fluids system over the past decades. Among these, isolation of plant oil using supercritical fluids extraction has been the core area of research since the year of 2021. Samaram *et al.*, (2020) attempted to extract Native pear seed oil using supercritical fluid technology, but they found that the yield was low along with excessive amount of impurities detected in the oil. High amounts of impurities (e.g. free fatty acids, waxes and

phospholipids) can affect the quality and flavour of the edible oil. In contrast to supercritical fluid extraction, the impurities of plant oil obtained by subcritical fluid extraction is usually lesser. Hence, Zhang *et al.*, (2018) conducted research to compare the physicochemical properties of Native pear seed oil obtained by subcritical butane extraction (SBE) and supercritical CO₂ extraction (SCE). Both the techniques utilised different extraction conditions (35°C, 0.4 Mpa and 40 mm for SBE and 45°C, 30 Mpa and 3 h for SCE). Under SEM, the cell walls of seed powder showed minor invagination after SBE whereas honeycomb holes observed on the cell walls of native pear seed powder after SCE. These observations suggest both extraction techniques can break down the cell walls to various degrees, which can aid in releasing the oil from the seed. On the other hands, the oxidative stability indexes (peroxide, p-anisidine, total oxidation, free fatty acid and aldehyde contents) of SBE-extracted seed oil was lower than SCE-extracted seed oil, probably due to the greater levels of lipophilic bioactive compounds. The researchers concluded that the SBE may be an alternative way to the fractionation of seed oil.

Commercially, decaffeination and hop extraction are the primary applications of sub- and supercritical fluids in the food industry. Owing to expensive equipment installation, oil extraction using both systems is mostly accomplished in laboratory-scale. The yield and quality of oil obtained from sub- and supercritical fluid extraction are greatly influenced by the operating parameters. In a recent work by Vibha & Shabina (2019), the SCE parameters such as pressure, temperature, flow rate, co-solvent and particle size on the yield of seed oil were optimised using a central composite design. It was reported that increasing the pressure increased the extraction rate, possibly due to the increment of solvent density. A decrease in the particle size of samples enhanced the extraction yield, which could be due to the increment of surface area to contact with the CO₂. Moreover, the addition of polar co-solvent (e.g. ethanol) during SCE could enhance the CO₂ strength, thus improved the yield of seed oil. In contrast to the results reported by Samaram *et al.* (2020), Vibha & Shabina (2019) found the oil recovery of seed oil produced using the optimised SCE parameters was high. Further study shall be conducted to investigate the chemical characteristics of seed oil extracted using SCE.

High Hydrostatic Pressure-Assisted Solvent Extraction (HHPASE)

High hydrostatic pressure is a low or non-thermal technique for processing food products. The pressure range utilized in HHP is 100–1000 Mpa, which is greater than the pressure (E100 Mpa) used in the supercritical fluid system. HHP creates large differential pressures in between the exterior and interior of the plant cells, causing protein denaturation and cell membranes destruction. This condition allows the solvent to rapidly permeate through broken membranes into the inner of cells, and thus, more lipid components are extracted out of the cells. As compared to thermally produced oil, HHP produced oil has better nutritional value and flavour.

The feasibility of crude oil production from the mountain Avocado seed using HHP-assisted solvent extraction was examined by Briones-Labarca *et al.* (2020). The seed powder was mixed with hexane, hermetically sealed in a polyethylene bag and pressurised at 500 Mpa for 15 mm. Oil yield obtained from HHP-assisted solvent extraction (40.5%) was greater than Soxhlet extraction (32.1%), suggesting the commercialisation potential of this new emerging technique for seed oil extraction. Further investigations are needed to identify and quantify the lipid classes (e.g. neutral lipids, phospholipids and glycolipids) of seed oil extracted using both techniques.

Extraction of functional oil using HHP-assisted solvent method has been well-explored. It requires low amounts of organic solvent and the extraction process is fast. However, the parameters such as type and amount of solvent, number of cycles, time and pressure can significantly affect the quality of oil. High levels of impurities in the oil are retained if the operating conditions of HHP assisted solvent technique are not chosen judiciously. Therefore, it is crucial to optimize the operating conditions using RSM to get high-quality papaya seed oil.

The research/ knowledge gap in the Table 1 above showed that my new method of extraction/

distillation process (Methanothermal) is more efficient with optimal yield for oil extraction from agro seed compared to any other method.

METHODOLOGY

Determination of Crude Fibre of Agro Seed Oil

Crude fibre is the insoluble and combustible organic residue which remains after the sample has been treated consecutively with light petroleum spirit, boiling dilute sulphuric acid, boiling dilute sodium hydroxide, alcohol. Crude fibre consists largely of the cellulose, lignin and hemicellulose.

Table 1. Knowledge gap

S/N	Different Raw Materials for Oil Extraction Studies	Methods	Findings	Author	Research Gap
1a	Mango – Batek Batu Specie	Extracted Oil Using Extrusion Method	Volume of Oil Extracted From 250g of The Seed At 70°C in 60min was 30.70ml	Pusnom <i>etal</i> (2022) Malesia	The maximum operational temperature is only 70°C and 60mins with 30.70ml vol
1b	Mango – Baccon Specie	In Research, I Extracted Oil Using Mechanothermal Extraction/Distillation Method	Volume of Oil Extracted From 250g of The Seed At 60°C in 60min was 80ml which means that the Extraction was efficient with high yield using my new method	Kingdom M Umah 2024	The maximum operational temperature is 60°C and 90mins maximum time with 80ml vol.
2a	Native Pear Seed Oil-Gold Specie	Extracted Oil Using Solvent(Haxane) Soxhlet Method	Volume of Oil Extracted From 150g of The Seed At 70°C in 50min was 20ml	De Melo & De Susa (2022) Brazil	The maximum operational temperature is only 70°C and 60mins with 20ml vol.
2b	Native Pear Seed Oil - Saphindale Specie	In Research, I Extracted Oil Using Mechanothermal Extraction/Distillation Method	Volume of Oil Extracted From 150g of The Seed At 60°C in 50min was 28ml which means that the Extraction was efficient with high yield using my new method	Kingdom M Umah 2024	The maximum operational temperature is 60°C and 90mins maximum time with 28ml vol.
3a	Avocado Pear Seed Oil – Hass Specie	Extracted Oil Using Steam Distillation Method	Volume of Oil Extracted From 200g of The Seed At 70°C in 50min was 23.90ml	Priyawadee (2022) Hongkong	The maximum operational temperature is only 70°C and 60mins with 23.90ml
3b	Avocado Pear Seed Oil-Hass Specie	In Research, I Extracted Oil Using Mechanothermal Extraction/Distillation Method	Volume of Oil Extracted From 150g of The Seed At 60°C in 50min was 58ml which means that the Extraction was efficient with high yield using my new method	Kingdom M Umah 2024	The maximum operational temperature is 60°C and 90mins maximum time with 58ml vol.

Principle

The starch and the protein are dissolved by boiling the samples with sulphuric acid and then with sodium hydroxide. The residue is washed, dried and weighed (fibre). The residue is ashed and the weight of the ash is subtracted from the weight of the fibre. The method is empirical and therefore the details of the procedure must be strictly adhered to.

Wash down the residue with hot distilled water for about 3 times.

Alkaline Hydrolysis

The process involve washing of the residue with 25ml of 1.25 NaOH solution with the acid 100ml placed in a beaker. The solution mixture was boiled of 2-3 minutes and allowed to cool for purpose of filtration. The obtained was filtered with filter paper and a residual solution was obtained, which was further heated with distilled water. The filtered solution was over-dry for a period of 1 hour at a temperature of 105 degree centigrade and the obtained solution weight and the result recorded. The filter paper was ashed and the residual material added in one container and the obtained mixture heated up at temperature of 550 degrees centigrade for time period of 3 hour and the obtained result recorded as per the weight result of the ash as well as the computation procedure which involve the calculation procedures of crude fibre.

Crude fibre (g) = Weight of Residue — Actual Ash

$$\% \text{ Crude Fibre} = \frac{\text{Crude Fibre}}{\text{Sample Weight}} \times \frac{100}{1}$$

Calculation

Weight of residue (g) = (Wt. of Filter paper + residue) — (wt. Of filter paper)
= 1.056 — 0.994 = 0.062g

Weight of Ash (g) = (Wt. of crucible + Ash) — (Weight of crucible)
= 25.698 — 25.675 = 0.023g

Actual Ash (g) = Weight of Ash — Blank (Ash wt. of empty filter paper) Actual Ash (g) = 0.023g — 0.002g = 0.021g

Crude fibre (g) = weight of Residue — Actual Ash
= 0.062 — 0.021 = 0.041g

$$\% \text{ Crude Fibre} = \frac{\text{Crude Fibre}}{\text{Sample Weight}} \times \frac{100}{1}$$

Determination of Nitrogen Content of 3 Varieties of Agro Seed Cake

Food proteins consist of amino acids possessing various functional groups. The kjeldahi procedure is one of the most suitable methods for the direct estimation of protein.

Principle (Micro Kjeldahl)

The kjeldahl method is based on wet. Combustion of the sample by heating with concentrated sulphuric acid in the presence of metallic and other catalyst to effect the reduction of organic nitrogen in the sample to ammonia which is retained in solution as ammonium sulphate. The digest having been made alkaline, is steam distilled or distilled to release the ammonia which is trapped and titrated.

Reagents/Apparatus

Procedure

The process of determining the nitrogen content of 3 varieties of Agro seed cake involve measuring 0.5g of the seed cake into a NO 1. Watchman filter paper and place it in a **kjeldahl** digestion flask. 3gm of sodium sulphate (Na₂SO₄) and 3gm of cupper sulphate (CuSO₄) which serve as a catalyst was added and 12ml of Conc. Sulphuric acid (H₂SO₄) were added into the flask. The flask was heated in an inclined position in a form **cupboard** until frothing of the solution ceased. The solution was boiled briskly in the fun chamber until the digest is clear. The cleared digest was allowed to cool for 10min after which it was made up to 100ml volume with distil water using measuring cylinder. 20ml of the digest was measured into another 500ml distillation flask, 20ml 40% sodium hydroxide solution was also added into the distillation flask, simple distillation processes was carried out to for form a distilled alkaline-Ammonia solution. 10ml of folic acid was added to the distilled ammonia-alkaline solution was in another 200ml conical flask and shake properly. 150ml of the ammonia alkaline solution collected into a beaker and filtrated against O. INHCL.

The blank solution was carried out using the same processes except the sample in question.

Calculation

$$\% \text{ Nitrogen} = \frac{\text{Sample Titre} - \text{Blank} \times N + CL \times 14}{W + \text{. of Sample}}$$

$$\text{Crude protein} = \% N \times \text{protein conversion factor}$$

Where

N = Normality of acid used = 0. IN

Dilution factor = 0.2

ANALYSIS OF FATS AND OILS

Determination of Specific Gravity

Introduction

For specific gravity, measurements are made at 20°C but where the fat is not liquid at that temperature, measurements are made at 40°C or 60°C. Hydrometers (e.g. Pycnometer are used in measuring the density and specific gravity).

Materials

- Specific Gravity bottle (Pycnometer)
- Water bath
- Analytical, balance

Procedure

The process of determining specific gravity of 3 varieties of Agro seed cake involved using pycnometer method. In this process, the pycnometer was clean using a dry agent to get rid of contaminant. The empty weight of the pycnometer or density bottle was recorded. The density bottle was filled with water to the brim and the mass of the water plus the empty bottle was also recorded at room temperature of 25°C. After this the water in the pycnometer was discharged and the bottle was filled up to one quarter of (1/3) level of the density bottle and the remaining part of the bottle was refilled with water and reweighed. The mass was recorded.

Calculation

$$\frac{W_2 - W_1}{W_4 - W_3} \times 100$$

Determination of Refractive Index

The Abb Refractometer may be used to determine the refractive index. The temperature of oils should be 20°C or 25°C and that of fat at 40°C. The instrument should be placed such that diffused daylight or artificial light can be used as illumination.

Determination for the Determination of Iodine Value of the Oil

Introduction

The iodine number of a fat is the number of grams of iodine that can be absorbed by 100g of fat. It is a measure of the degree of unsaturated acid present in the fat. The Wij's method of determination is used. The oil/fat is treated with an excess iodine monochloride that is also estimated by treating with potassium iodide. The liberated iodine is titrated with sodium thiosulphate solution.

Procedure

The process for the determination of Iodine value of the Agro seed oil involved the weighing of the seed 10.0g into a 250ml conical flask with stopper 10ml of CCl_4 was added to dissolved in the oil. 20ml Weyl's reagent solution was added. The mixture was allowed to stand in a dark cupboard for 30min, after which 15ml of 10% KI and Starch solution was added into the conical flask containing the mixture and stirred vigorously. 0.1N $\text{Na}_2\text{S}_2\text{O}_3$ was used for titration. In the process, the blank solution was also prepared in the same manner.

Calculation

$$\text{Iodine value (g/100g)} = \frac{(b-a) \times N \times 12.69}{\text{Sample wt. (g)}}$$

Determination of Peroxide Value

Peroxides are the first products of oxidation of unsaturated fats and oils. When the concentration of peroxides reached a certain level, complex changes occur with the formation of ketones. Aldehydes and hydroxyl groups volatile and mainly responsible for the off flavours and odours. The peroxide value is indicative of oxidation during early stages of lipid deterioration. It is expressed as peroxide per kg of fat or in millequivalent. It depends on the reaction of oil with fat to liberate iodine in the presence of acetic acid which is later titrated (decolourized) by sodium thiosulphate.

Procedure

The process of determining the peroxide value of the agro seed, oil involved weighing 5mgs of the oil into a clean 250ml beaker, 30ml of acetic acid and 10ml chloroform was also added into the beaker containing the oil. The mixture was swirled until there was a complete dissolution of the oil, 0.5ml of saturated KI solution was also added in the dissolved oil. The solution was allowed to stand for 30min with stirring at every 5min and 30ml of distilled water was added after the expiration of 30min. 0.01N of $\text{Na}_2\text{S}_2\text{O}_3$ was used for titration until the colour changes yellow that emerged disappears. Another 0.5ml of starch solution indicator was added to the sample with gradual agitation for 1min between 0.10ml after which, the mixture was filtrated again 0.01N $\text{Na}_2\text{S}_2\text{O}_3$ solution until the emerged blue colour disappeared.

Calculation

$$\text{PV (m.eq/kg)} = \frac{(S-B) \times N \times 100}{\text{wt. of Sample}}$$

Determination of Acid Value and Free Fatty Acid

The acid value is a measure of the extent to which the glycerides in the oil have been decomposed by lipase or other reactions. The decomposition is accelerated by heat and light. As rancidity is usually accompanied by free fatty acid formation, the determination is often used as a general indication of the condition and edibility of oils. Solution of a known quantity of fat is to be dissolved in a mixture of ethanol and diethyl ether, followed by titration of the free fatty acids present with an ethanolic solution of potassium hydroxide.

The acidity is a conventional expression of free fatty acids percentage of the fat.

Procedure

The process of the analysis of acid value and free fatty of agro seed oil involved measuring 10gms of the oil into 250ml conical flask, 25ml of diethyl ester, 25ml of 98% alcohol and 1ml of 1% phenolphthalein indicator was added to the oil in the conical flask containing the oil. The mixture was carefully filtrated with 0.1mol NaOH mixture until there was a colour change to pink it stable for 15sec which is the end point.

MATERIALS AND METHOD FOR HPLC/MS GAS CHROMATOGRAPH

Determination of Aromatic Compound of the Agro Seed Oil

Sample Preparation

Sample was extracted with petroleum ether using the Soxhlet method. Approximately 5 g of dried and 80 ml of petroleum ether (60–90°C) were used in each Soxhlet extraction, which was realized at 80°C for 8 h. After the extraction procedure, the solvents were evaporated under vacuum, and the samples were subsequently stored at 4°C.

Procedure

Lipids obtained after the extraction of the samples were converted to the corresponding FAMES. In this procedure, 40 ml of the was placed into 10 ml centrifuge tubes to which 0.7 ml of potassium hydroxide (10 M) solution and 5.3 ml of methanol were added. The reaction was performed at 55°C for 1.5 h with mixing for 5 s every 20 min. After cooling to room temperature, 0.58 ml of 19odelling acid (10 M) solution was added and the reaction was continued at 55°C for 1.5 h with mixing for 5 s every 20 min. After cooling to room temperature, 3 ml of n-hexane was added and mixed for 5 min. Subsequently, the tubes were centrifuged for 5 min and the extracts were removed for GC analysis.

GC–MS analysis was carried out using an Agilent 6890 gas chromatograph with a 5973 MS detector equipped with 60 m×0.25 mm, i.d. 0.25 µm/MS DB-WAX capillary column (Agilent). The following temperature ramp was used: injector at 250 °C, oven initially at 200°C, held for 1 min and heated to 230°C (1.5°C min⁻¹, then held for 10 min). The characterization and identification of FAMES from the sample was completed in the SCAN mode with the m/z range varied from 35 to 450. The flow rate of the nitrogen as carrier gas was 1 ml min⁻¹; manual injection; the injection volume was 1 ml. The fatty acid composition of the FAMES from the sample was determined using an Agilent 6820 gas chromatograph equipped with a Supelco capillary column (hp-innowax, Agilent, 100 m×0.25 mm, i.d. 0.20 µm), injection port. The initial oven temperature was 200 °C, which was held for 1 min, subsequently increased to 230°C at 1.5°C min⁻¹ and then held for 1 min. The injector was set at 250°C, and the detector at 280°C. Nitrogen was used as the carrier gas at a flow rate of 1 ml min⁻¹. The split ratio was 50:1, and the sample size was 1 ml.

RESULTS AND DISCUSSION

Figure 1 shows the theoretical evaluation of the extract recovered from native pear seed oil of 400g initial mass (Eludicote species) upon the effect of time. The result demonstrate decrease in the initial feed mass of 350g of the native pear seed oil with increase in the oil extracted from the process with respect to times. However, the theoretical regression equation of each behaviour is shown in figure 1 as $Y_{IC} = -0.0261x^2 - 1.626x + 250.18$ (initial concentration by mass g), $Y_{RC} = 0.0109x^2 + 0.1328x - 0.0734$ (Recovered volume in ml) and $Y = -0.0053x^2 + 0.4137x + 0.0847$ (final concentration glan) in terms of polynomial expression of quadratic equation and the best fit of the square root of the polynomial expression in terms of its reliability are $R_{IC}^2 = 0.9795$ (99.98%), $R_{RC}^2 = 0.9979$ (99.98%) and $R_{FC}^2 = 0.9342$ (96.36%). The above polynomial expression of the quadratic equation define the rate of initial native pear seed mesh conversion with extracted oil. This has further revealed the significance of the extraction model adopted to monitor the initial concentration, volume of oil recovered and the final concentration using the mechanochemical approach in oil production for domestic application, pharmaceutical, chemical and industrial utilization in Table 2 and Table 3.

Table 2. Functional parameters of extract recovered from native pear seed oil with respect to initial concentration by mass (g), Time (min.), volume recovered (ml) and final concentration (g/cm³) At 250g For (Eludicote species).

Initial concentration by mass (g)	Time (min)	Volume recovered (ml)	Final concentration (g/cm ³)
250	0	0	0
273	45	27	9.26
266	50	34	7.353

260	55	40	6.25
251	60	49	5.102
246	65	54	4.63
238	70	62	4.0321

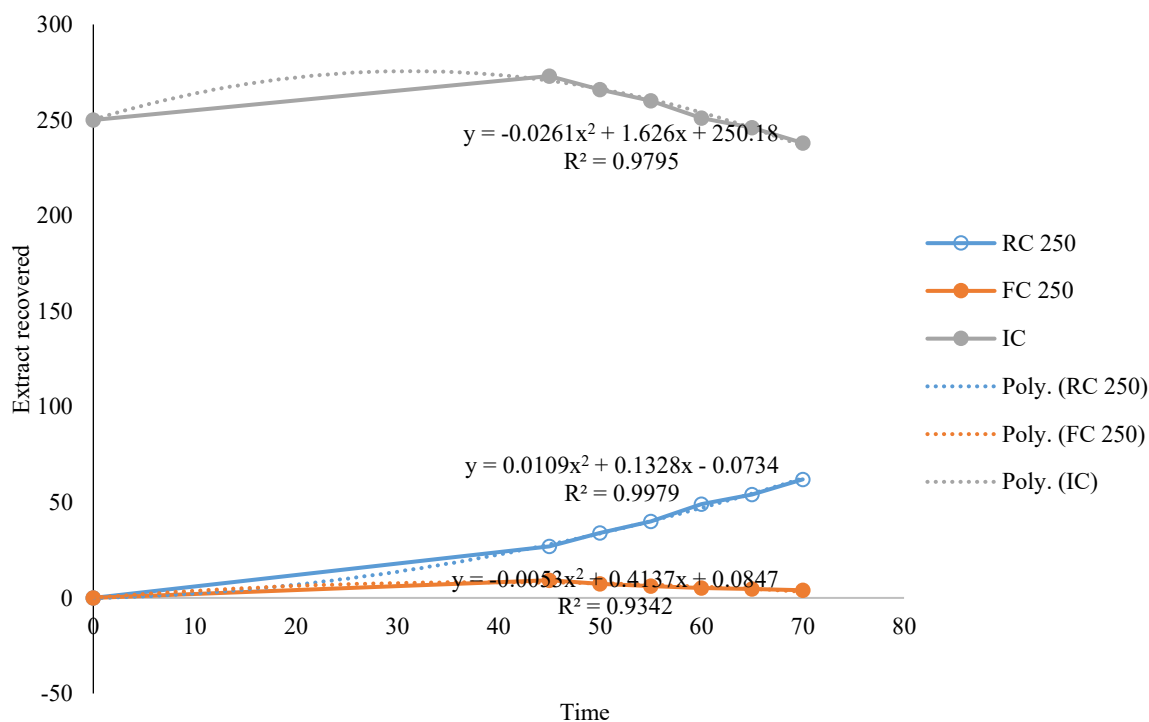


Figure 1. Graph of functional parameters of extract recovered from native pear seed oil with respect to initial concentration by mass (g), Time (min.), Volume recovered (ml) and final concentration (g/cm^3) At 250g For (Eludicote species).

Table 3. Functional parameters of extract recovered from native pear seed oil with respect to initial concentration by mass (g), Time (min.), Volume Recovered (ml) and Final Concentration (g/cm^3) At 200g For (Eludicote species).

Initial Concentration by mass (g)	Time (min)	Volume Recovered (ml)	Final Concentration (g/cm^3)
200	0	0	0
82	45	18	11.11
67	50	33	6.061
64	55	36	5.56
58	60	42	4.762
51	65	49	4.082
48	70	52	3.85

Figure 2 shows the theoretical evaluation of the extract recovered from native pear seed oil of 400g initial mass (Eludicote species) upon the effect of time. The result demonstrate decrease in the initial feed mass of 350g of the native pear seed oil with increase in the oil extracted from the process with respect to times. However, the theoretical regression equation of each behaviour is shown in figure 2 as $Y_{IC} = 0.0101x^2 + 0.0793x - 0.2997$ (initial concentration by mass g), $Y_{RC} = 0.0202x^2 - 3.5968x + 200.08$ (Recovered volume in ml) and $Y = -0.0058x^2 + 0.438x + 0.162$ (final concentration g/ml) in terms of polynomial expression of quadratic equation and the best fit of the square root of the polynomial expression in terms of its reliability are $R_{IC}^2 = 0.9691$ (99.98%), $R_{RC}^2 = 0.9986$ (99.98%) and

$R_{FC}^2 = 0.7707$ (96.36%). The above polynomial expression of the quadratic equation define the rate of initial native pear seed mesh conversion with extracted oil. This has further revealed the significance of the extraction model adopted to monitor the initial concentration, volume of oil recovered and the final concentration using the mechanochemical approach in oil production for domestic application, pharmaceutical, chemical and industrial utilization.

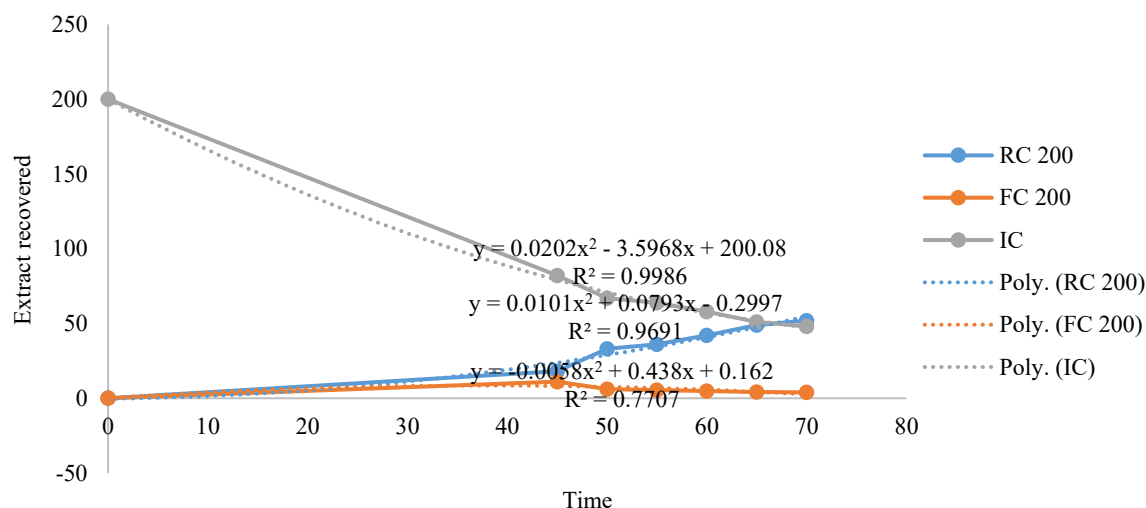


Figure 2. Graph of functional parameters of extract recovered from native pear seed oil with respect to initial concentration by mass (g), Time (min.), Volume recovered (ml) and final concentration (g/cm^3) At 200g For (Eludicote species).

Table 4. Functional parameters of extract recovered from native pear seed oil with respect to initial concentration by mass (g), Time (min.), volume recovered (ml) and final concentration (g/cm^3) At 150g For (Eludicote species).

Initial Concentration by mass (g)	Time (min)	Volume Recovered (ml)	Final Concentration (g/cm^3)
150	0	0	0
118	45	32	4.69
112	50	38	3.95
109	55	41	3.67
104	60	46	3.26
98	65	52	2.89
92	70	58	2.59

Figure 3 shows the theoretical evaluation of the extract recovered from native pear seed oil of 400g initial mass (Eludicote species) upon the effect of time. The result demonstrate decrease in the initial feed mass of 350g of the native pear seed oil with increase in the oil extracted from the process with respect to times. However, the theoretical regression equation of each behaviour is shown in figure 3 as $Y_{IC} = -0.0043x^2 - 0.5202x + 149.97$ (initial concentration by mass g), $Y_{RC} = 0.0043x^2 + 0.5202x + 0.0308$ (Recovered volume in ml) and $Y = -0.0024x^2 + 0.2041x + 0.0271$ (final concentration g/ml) in terms of polynomial expression of quadratic equation and the best fit of the square root of the polynomial expression in terms of its reliability are $R_{IC}^2 = 0.9988$ (99.98%), $R_{RC}^2 = 0.9988$ (99.98%) and $R_{FC}^2 = 0.9729$ (96.36%). The above polynomial expression of the quadratic equation define the rate of initial native pear seed mesh conversion with extracted oil. This has further revealed the significance of the extraction model adopted to monitor the initial concentration, volume of oil recovered and the final concentration using the mechanochemical approach in oil production for domestic application, pharmaceutical, chemical and industrial utilization in Table 4 and Table 5.

Method B

Applying the extraction mass transfer equation

$$C = C_s \left[1 - \exp \left[-\frac{KA}{Vb} t \right] \right] \quad (1)$$

e – Exponential

t – Extraction Time

A – Area of Extract = $3.4159 \times L^2$

L – Length of Cylinder Containing the Extract (30mm)

V – Volume of Extract

b – Thickness of Extract

CS – Initial Concentration of Feed (g)

K – Diffusion Coefficient = $1.12 \times 10^{-8} \text{ m}^2$

Taking the natural logarithm of equation 1

$$\ln C = \ln \left[C_s \left(1 - e^{-\frac{KA}{Vb} t} \right) \right]$$

$$\ln C = \ln C_s + \ln \left(1 - e^{-\frac{KA}{Vb} t} \right)$$

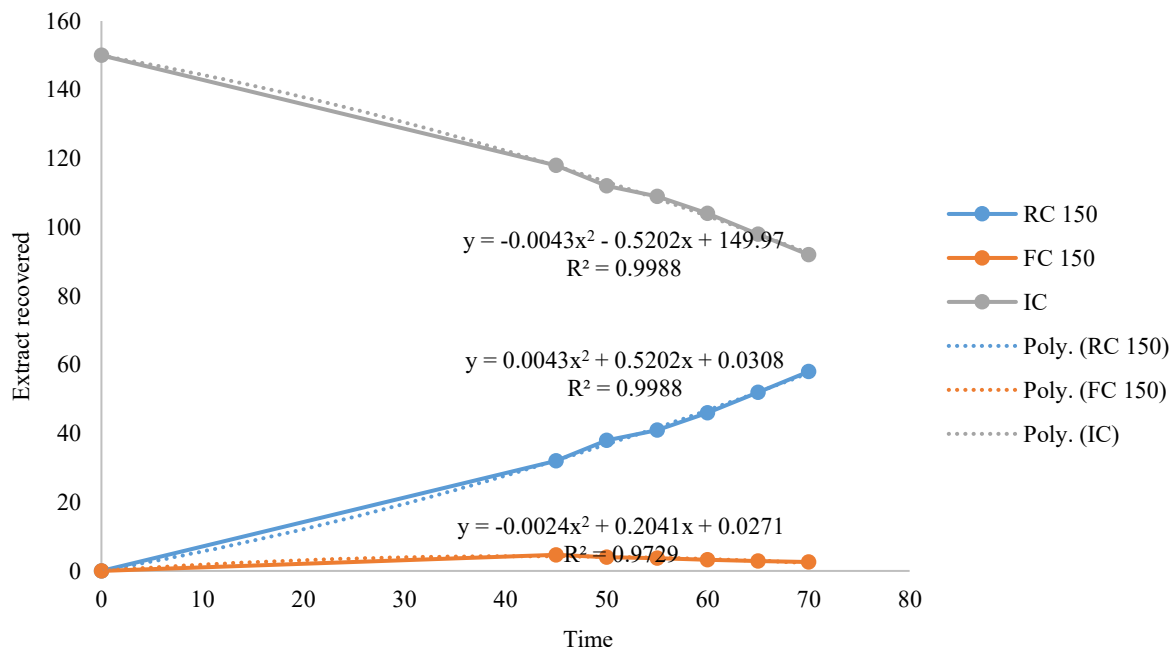


Figure 3. Graph of functional parameters of extract recovered from native pear seed oil with respect to initial concentration by mass (g), Time (min.), Volume recovered (ml) and final concentration (g/cm^3) At 150g For (Eludicote species).

$$\ln C = \ln C_s + \ln 1 - \ln e^{-\frac{KA}{Vb} t} \quad (2)$$

But $\ln 1 = 0$

Equation (4.2) yields

$$\ln C = \ln C_s - \ln e^{-\frac{KA}{Vb} t} \quad (3)$$

Mathematical analysis of Equation (3) gives

$$\ln C = \ln C_s + \frac{KAt}{Vb} \quad (4)$$

Upon variable separation or re-arrangement of equation (4)

$$\ln C_s = \ln C - \frac{KAt}{Vb} \quad (5)$$

Therefore, a plot of $\ln C_s$ against t using Equation (5) gives a straight line graph of intercept in $\ln C_s$ and equation $\ln C$ and the slope of the plot equals $-\frac{KAt}{Vb}$

Method C

Applying the extraction mass transfer equation

$$C = C_s \left[1 - \exp \left[-\frac{KA}{Vb} t \right] \right] \quad (6)$$

e – Exponential

t – Extraction Time

A – Area of Extract = $3.4159 \times L^2$

L – Length of Cylinder Containing the Extract (30mm)

V – Volume of Extract

b – Thickness of Extract

CS – Initial Concentration of Feed (g)

K – Diffusion Coefficient = $1.12 \times 10^{-8} \text{ m}^2$

Taking the natural logarithm of equation 1

$$\begin{aligned} \ln C &= \ln \left[C_s \left(1 - e - \frac{KAt}{Vb} \right) \right] \\ \ln C &= \ln C_s + \ln \left(1 - e - \frac{KAt}{Vb} \right) \\ \ln C &= \ln C_s + \ln 1 - \ln e - \frac{KAt}{Vb} \end{aligned} \quad (7)$$

But $\ln 1 = 0$

Equation (7) yields

$$\ln C = \ln C_s - \ln e - \frac{KAt}{Vb} \quad (8)$$

$$\text{Mathematical analysis of Equation (8) gives } \ln C = \ln C_s + \frac{KAt}{Vb} \quad (9)$$

Upon variable separation or re-arrangement of equation (9)

$$\ln C_s = \ln C - \frac{KAt}{Vb} \quad (10)$$

Therefore, a plot of $\ln C_s$ against t using Equation (10) gives a straight line graph of intercept in $\ln C_s$ and equation $\ln C$ and the slope of the plot equals $-\frac{KAt}{Vb}$

From the plot of $\ln C_s$ against time (t)

The slope (8) = -0.0047

Table 5. Applying the experimental data of C_s against extraction time.

C_s (g)	T(min)	$\ln C_s$
150	40	5.011
142	50	4.956
132	60	4.883
128	70	4.852

123	80	4.812
118	90	4.771

Table 6. Applying the experimental data of C_s against extraction time.

C_s (g)	T(min)	$\ln C_s$
150	40	5.011
142	50	4.956
132	60	4.883
128	70	4.852
123	80	4.812
118	90	4.771

$$\text{Thus, } 5 = -\frac{KA}{vb} = -0.0047$$

$$\text{Also, } \ln C = 5.18928$$

$$C = e^{5.18928}$$

$$C = \exp(5.18920)$$

$$C = 179.339$$

From the plot of $\ln C_s$ against time (t)

$$\text{The slope (8)} = -0.0047$$

$$\text{Thus, } 5 = -\frac{KA}{vb} = -0.0047$$

$$\text{Also, } \ln C = 5.18928$$

$$C = e^{5.18928}$$

$$C = \exp(5.18920)$$

$$C = 179.339$$

Figure 4 shows the theoretical evaluation of the extract recovered from native pear seed oil of initial mass (Eludicote species) upon the effect of time. The result demonstrate decrease in the initial concentration of the native pear seed oil with increase in the oil extracted from the process with respect to times. However, the theoretical regression equation of each behaviour is shown in Figure 4 as $Y = 0.004x + 5.1892$ (initial concentration by mass g), (Recovered volume in ml) and (final concentration g) and the best fit of the square root of the polynomial expression in terms of its reliability is $R^2 = 0.9821$ (99.21%). The above polynomial expression of the quadratic equation define the rate of initial native pear seed mesh conversion with extracted oil. This has further revealed the significance of the extraction model adopted to monitor the initial concentration, volume of oil recovered and the final concentration using the mechanochemical approach in oil production for domestic application, pharmaceutical, chemical and industrial utilization in Table 6.

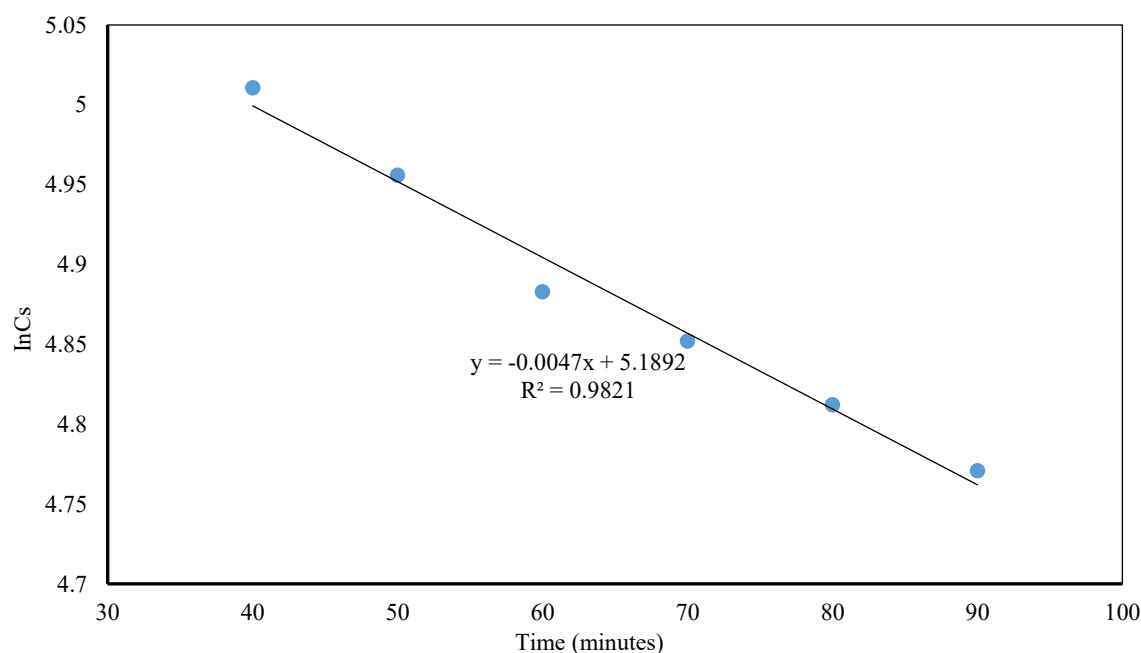


Figure 4. Plot of concentration of extracted oil recovered versus time.

CONCLUSION

The production of oil from agro seeds using mechanochemical extraction has emerged as a sustainable and efficient approach for the recovery of renewable industrial oils with wide-ranging applications in chemical, pharmaceutical, and food industries. This review has demonstrated that agro seeds constitute valuable sources of bio-oils containing important lipids, fatty acids, antioxidants, and bioactive compounds suitable for numerous industrial processes. The increasing demand for environmentally friendly and biodegradable raw materials has further enhanced the significance of agro-based oils as alternatives to petroleum-derived products.

Mechanochemical extraction has proven to be an effective method for improving oil recovery through the combined application of thermal conditioning and mechanical pressing. The process enhances cellular rupture, reduces oil viscosity, and facilitates efficient oil release from seed matrices while minimizing the dependence on harmful organic solvents. Compared with conventional extraction methods, mechanochemical extraction offers advantages such as lower environmental impact, improved process safety, operational simplicity, and adaptability to both small-scale and industrial-scale operations.

The review also highlighted the importance of enzyme-assisted aqueous extraction (EAAE) as an environmentally friendly extraction technology. The use of enzymes such as proteases, cellulases, and pectinases enhances the degradation of structural polysaccharides and improves oil extraction efficiency. Optimization of process variables including temperature, pH, hydrolysis time, enzyme concentration, and particle size is essential for maximizing extraction performance and product quality.

Agro-seed oils possess desirable physicochemical and nutritional properties that support their utilization in biodiesel production, pharmaceuticals, cosmetics, nutraceuticals, soaps, edible oils, lubricants, and other engineering applications. Their utilization contributes significantly to waste valorization, environmental sustainability, renewable energy development, and circular economy practices. Furthermore, the commercialization of agro-based oil extraction technologies can stimulate rural industrialization, create employment opportunities, and reduce environmental pollution associated with agricultural waste disposal.

In conclusion, mechanochemical extraction and enzyme-assisted extraction technologies provide economically viable and environmentally sustainable alternatives for agro-seed oil production. Continued research into process optimization, quality enhancement, and industrial scale-up is necessary to improve the commercial competitiveness and global adoption of agro-based oils in modern industrial applications.

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