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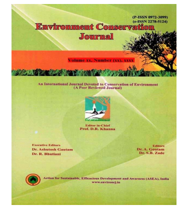

Effects of different aqueous extraction techniques on physicochemical quality and oil recovery of sesame oil

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ABSTRACT

Sesame is the oldest oilseed crop in agriculture, and it produces more oil than any other crop on the planet. This research aimed to investigate the impact of different oil extraction procedures on sesame seed oil physicochemical quality and oil recovery. The oil was extracted from the clean and healthy seeds using four extraction methods: aqueous, enzyme-assisted aqueous, ultrasound-assisted aqueous and solvent extraction using the Soxhlet apparatus. It was observed that ultrasound-assisted aqueous extracted oil had maximum saponification value and minimum acid value, refractive index, and lower free fatty acid value, compared with aqueous extracted oil and enzyme-assisted aqueous extracted oil. Ultrasound-assisted aqueous extraction method also yielded maximum oil, retrieval followed by enzyme-assisted aqueous extraction and aqueous extraction.

Introduction

Food and bio-fuel industries rely on oil extraction from oil-bearing crops and materials. The vegetable oil industry's most common oil extraction process is solvent extraction. Vegetable oil producers have been seeking new ways to improve the efficiency of their extraction operations to reduce production costs and waste. Sesame oil extraction using ultrasound-assisted aqueous which has the calibre to exchange the solvent extraction. The findings demonstrate that the physicochemical quality of sesame oil extracted using an ultrasound-assisted aqueous technique was equivalent to that of oil extracted using an enzyme-assisted aqueous method. Annual flower plant sesame is the oldest oilseed crop in agriculture and the most geriatric crop known to mankind. Sesame (*Sesamum indicum*) is a widespread and famous crop which is known by the local names as til or gingely or sim sim or ellu. Sesame is cultivated in tropical and subtropical areas of 3 different continents like Asia, Africa, and South America in the summer Kharif and semi-rabi seasons (Berhe Gebremedhn *et al.*, 2017). Sesame seed is available in different color like white, black, and yellow vary with the variety of seeds that grows for cultivation. Sesame is a premium oil known for its high nutritional value, economical value, and medicinal properties due to its oil composition and oil content in sesame seeds vary between 45-60% (Pathak *et al.*, 2014),which is high amount than the other oil-producing crops in the world. In 2019, Sudan, Myanmar, India, Tanzania, and China were the leading producers of sesame crops, harvesting over 5.0 million metric tonnes of sesame seeds from an aggregate of 9.6

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million hectares (Anonymous, 2021a). India is the world's third-largest producer of sesame oilseeds, and the industry plays a significant role in the agricultural economy. India's total sesame acreage was 1.6 million hectares in 2019-20, with a total output of 0.69 million metric tonnes (Anonymous, 2021a). In India, the sesame crop is majorly cultivated in West Bengal, Andhra Pradesh, Gujrat, Tamil Nadu, Telangana, Punjab, Madhya Pradesh, and Maharashtra. In Punjab, the total area under sesame was 2.6 thousand hectares and total production was 0.1 million metric tons (Anonymous, 2021b). Sesame seeds are a chain of nutritional benefits such as fibre, minerals, protein, vitamins, carbohydrates, fats, fatty acids, etc. With these several health benefits, sesame is used in making pharmaceuticals, cosmetics, perfumes, bath oil, snacks, and beverages. Sesame seeds are used to prepare and garnish various Indian sweets, baking products, and dishes that act as a flavouring agent. In north India, it is used in different forms like seeds, oil, paste, flour, and so on. Oil extraction from seeds is a crucial component in the commercialization of these products. The extraction technique directly influences the amount and quality of protein and oils produced. Cold pressing, mechanical screw pressing, solvent, supercritical fluid, and aqueous extraction are all ways of extracting oilseeds (Çakaloğlu *et al.*, 2018). The most frequently used method of extracting oil from oilseeds is mechanical screw pressing. About 90% of the total oilseeds produced in India are crushed using this method. The use of heat and pressure to extract oil from seeds is known as mechanical extraction. In India, however, mechanical screw presses (oil expellers) are inefficient, leaving 8 to 14 percent of the remaining oil in the cake. Several scholars have undertaken research on increasing oilseed oil recovery (Boutin & Badens, 2009). Solvents for oil recovery have numerous downsides, including significant safety risk, high energy input, low-quality oil, environmental danger, toxicological consequences, and low-quality food (Chemat *et al.*, 2019).

The growing trend of avoiding hazardous organic solvents has rekindled interest in alternate extraction methods. Although the oil yield was poor, aqueous oil extraction has emerged as a viable approach for oil extraction. This procedure is determined to be the most acceptable option for solvent extraction. Enzymatic pre-treatment has recently developed as a unique and effective method of improving oil recovery. Several scientists have investigated the use of enzymes in oil extraction (Abdulkarim *et al.*, 2005). In the oil extraction process, enzymes are primarily used to hydrolyze the structured polysaccharides present in the cell wall. When compared to hexane extracted oils, the quality of the oil produced by enzyme treatment is rather excellent (Jeevan Kumar *et al.*, 2017). Considering the use of benefits of alternative extraction techniques for extraction of oils. The study aimed to evaluate the effect of different extraction methods on the oil recovery and the physicochemical quality of sesame seed oil.

Material and Methods

The sesame seeds were procured from a local farmer from Jalandhar, Punjab, India. The cleaning was done with the help of a sieve shaker. Clean and healthy seeds were selected for the extraction of oil. The experimentation was conducted in the various laboratories of the SGTB Khalsa College, Shri Anandpur Sahib, Punjab, India. Figure 1 shows the flowchart of extraction of oil from sesame seeds.

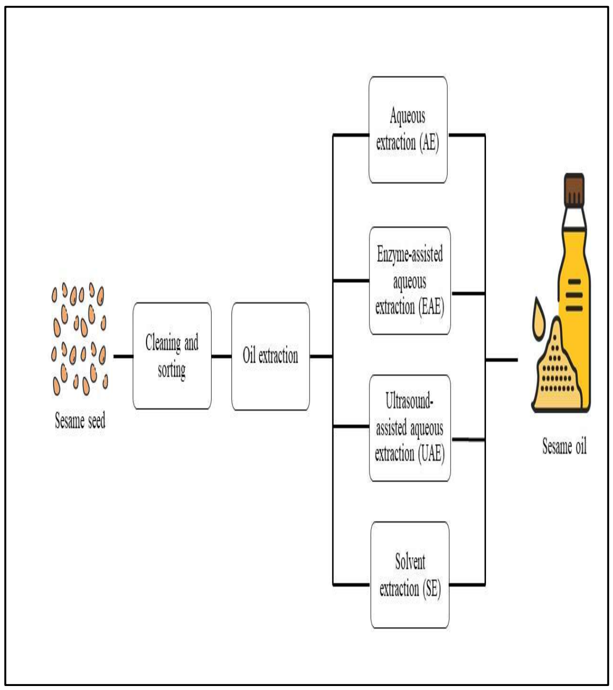

Figure 1: Process flowchart for extraction of sesame oil using different aqueous methods

Oil extraction methods

Aqueous extraction (AE)

240 ml of distilled water was combined with 40 g of seed sample. 0.5 N NaOH and 0.5 N HCl were used to adjust the pH of the mixture to 5. Grind the mixture with a high-speed grinder (Tandem 10 W) for 20 minutes in a hot water bath. "Churn" the mix in a rotating water bath at 65ºC for 20 hours, with tubes shaking horizontally at 160 rpm. Allow tubes to cool for 30-60 minutes at room temperature. In a centrifuge, spin for 15 minutes. Using a pipette, remove the top oil layer. To eliminate the remaining water, the oil was put in a 70ºC oven for 30 minutes (Moreau *et al.*, 2004).

Enzyme-assisted aqueous extraction (EAE)

40 g dry seed samples and 240 ml buffer were placed in Borosil tubes with a capacity of 300 ml (0.05 M Na acetate, pH 4.0). In a food processor, the ingredients were ground for a minute at high speed (make Tandem 10 W). Cellulase enzyme was added at a dosage of 0.025 percent (w/w). Churning was done in a shaking water bath for 4 hours at 50 degrees Celsius, with tubes shaking horizontally at 160 rotations per minute. Churning for another 16 hours at 65°C in a revolving water bath with tubes rotating horizontally at 160 rpm. Tubes were allowed to cool for 30-60 minutes at room temperature. After that, the samples were centrifuged for 15 minutes in a hot centrifuge. The top oil layer was scraped with a pipette to remove any remaining water, and the oil was then baked for 30 minutes at 70°C (Moreau *et al.*, 2004).

Ultrasound-assisted aqueous extraction (UAE)

For ultrasound preparation, an ultrasonic cleaning bath was employed. 40 g crushed dry material was combined with 240 ml distilled water. The pretreatment with ultrasound lasted 60 minutes. 0.5 N NaOH and 0.5 N HCl were used to adjust the pH of the solution to 5. The mixture was incubated at 60°C for 2 hours. After incubation, the mixture was spun in a hot centrifuge for 15 minutes at 3000 rpm using centrifuge equipment. In a hot water bath, mill the mixture for 20 minutes using a high-speed grinder (Tandem 10 W). "Churn" the mixture for 20 hours at 65°C with tubes shaking horizontally at 160 rpm in a spinning water bat. Allow tubes to cool at room temperature for 30-60 minutes. Spin for 15 minutes in a centrifuge. A pipette was used to remove the top oil layer. The oil was kept in the oven for 30 minutes at 70°C to remove any leftover water (Khoei & Chekin, 2016).

Solvent extraction (SE)

In the soxhlet extractor, the 40 g powdered seed sample was placed in a thimble composed of thick filter paper and fed into the main chamber. The Soxhlet extractor was placed on top with the extraction solvent in a distillation flask. The apparatus was then fitted with a condenser. The solvent was then heated to boiling point. A distillation arm raised solvent vapour, which rose into the solid thimble chamber. In the condenser, the solvent vapour cooled and fell back into the chamber containing the solid substance. The solid material was gradually poured into the compartment containing the warm solvent. The heated solvent caused some of the desired chemicals to dissolve. A syphon side arm automatically emptied the Soxhlet chamber when it was nearly full, returning the solvent to the distillation flask. In this way, the thimble ensured that no solid materials were carried into the still pot by the quick velocity of the solvent. The cycle was repeated several times over the course of several hours. A portion of the nonvolatile component is dissolved in the solvent during each process**.** Many cycles of distillation were required to concentrate the required component in the flask. A liquid or solid phase is extracted by transferring its components to another liquid phase. The extraction process takes advantage of differences in the solubility of components. Extracting a solute from a liquid solution involves contacting it with another liquid solvent that is somewhat miscible with the solution (liquid-liquid extraction or solvent extraction). Solvents are liquids that are added to a solution to allow it to be extracted. A solvent layer is called extract, and a raffinate layer is called raffinate.

Physico-chemical properties of sesame oil

Different physicochemical properties of extracted sesame oil viz., refractive index, acid value, saponification value, and free fatty acid were determined using standard methods as discussed below:

Refractive index

For the determination of the Refractive Index, Abbe's Refractometer was used (AOAC, 2000).

Saponification value

The saponification value is defined as the amount of potassium hydroxide needed to saponify one gram of oil (AOAC, 2000). 1.5 g of oil was placed in a flask. The mixture was then refluxed at 50ºC for 1 hour with 25 ml of alcoholic potassium hydroxide solution added. It was then allowed to cool. Using approximately 1.0 mL of phenolphthalein indicator solution, 2 to 3 drops of phenolphthalein were added. The excess potassium hydroxide was titrated with 0.5 N hydrochloric acid until a pink colour appeared. A burette measurement was made at the beginning and end to determine the amount of HCl solution. The saponification value is calculated using Equation 1:

$$\text{Saponification value (mg KOH/g oil)} = 56.1 \times \frac{(B-S)N}{W} \quad (1)$$

Where,
B = Volume of HCl required in ml for titration of a blank sample
S = Volume of HCl required in ml for titration of oil sample
N = Normality of HCl solution
W = Weight of oil taken in grams

Free fatty acid

The oil was placed in a conical flask along with 50 ml of freshly neutralized ethyl alcohol and 1 ml of phenolphthalein indicator solution in a 250 ml flask. Titration was performed with N/10 potassium hydroxide after boiling for roughly five minutes and continuing until a slight pink colour appeared. To determine the amount of potassium hydroxide solution, the initial and final burette measurements were recorded (AOAC, 2000).

$$\text{Free fatty acid (mg KOH/g oil)} = \frac{28.2\ VN}{W} \quad (2)$$

Where,
V = Volume in ml of potassium hydroxide solution used
N = Normality of the potassium hydroxide solution used
W = Weight of sample in g

Acid value

According to AOCS (1996), the acid value was calculated. Two and a half grams of oil were placed in a 250 ml conical flask, 50 ml of freshly neutralized hot ethyl alcohol and about 1 ml of phenolphthalein indicator solution. It was then titrated with N/10 potassium hydroxide for approximately five minutes until a slight pink colour was obtained. Calculation of the amount of potassium hydroxide solution was done using equation 3 based on the initial and final burette measurements.

$$\text{Acid Value (mg KOH/goil)} = \frac{56.1\ V \times N}{W} \quad (3)$$

Where,
V = Volume in ml of potassium hydroxide solution used
N = Normality of the potassium hydroxide solution used
W = Weight of sample in g

Oil recovery

The oil recovery was calculated using the equation 4 (Olaniyan & Oje, 2011)

$$\text{The oil yield (Y)} = \frac{W_{OE}}{W_S} \times 100 \quad (4)$$

Where,
W_{OE} = weight of oil expressed (g)
W_S = weight of the sample before expression (g)

Statistical analysis

The statistical programme R version 4.0.5 was used for the analysis of variance (ANOVA) and mean comparisons at a 5% level of significance.

Results and Discussion

Physico-chemical properties of sesame oil using different extraction techniques

Refractive index

The refractive index is a physical parameter during oil processing from various oil extraction methods. The refractive index indicates the transparency or amount of impurities present in the oil and method described by Pearson in 1976. The Refractive index with the lowest value indicated the highest transparency and fewer impurities. Hence, it should not exceed 1.4559 for edible oil (FAO/WHO, 2009). The refractive index of sesame oil under different extraction methods was found to be 1.46 ± 0.02 for the AE sample, 1.45 ± 0.01 for the UAE sample, 1.46 ± 0.01 for the EAE sample, and 1.47 ± 0.02 for the SE sample. The refractive index was found minimum in UA extracted oil and maximum in aqueous, solvent and EA extracted oil as shown in Figure 2. As the refractive index is affected by the wavelength of light, hence it can be increased due to the presence of impurities in the oil (Katkade *et al.*, 2018). Therefore, it was concluded that the UAE sample had fewer impurities causing it to have a lower refractive index value.

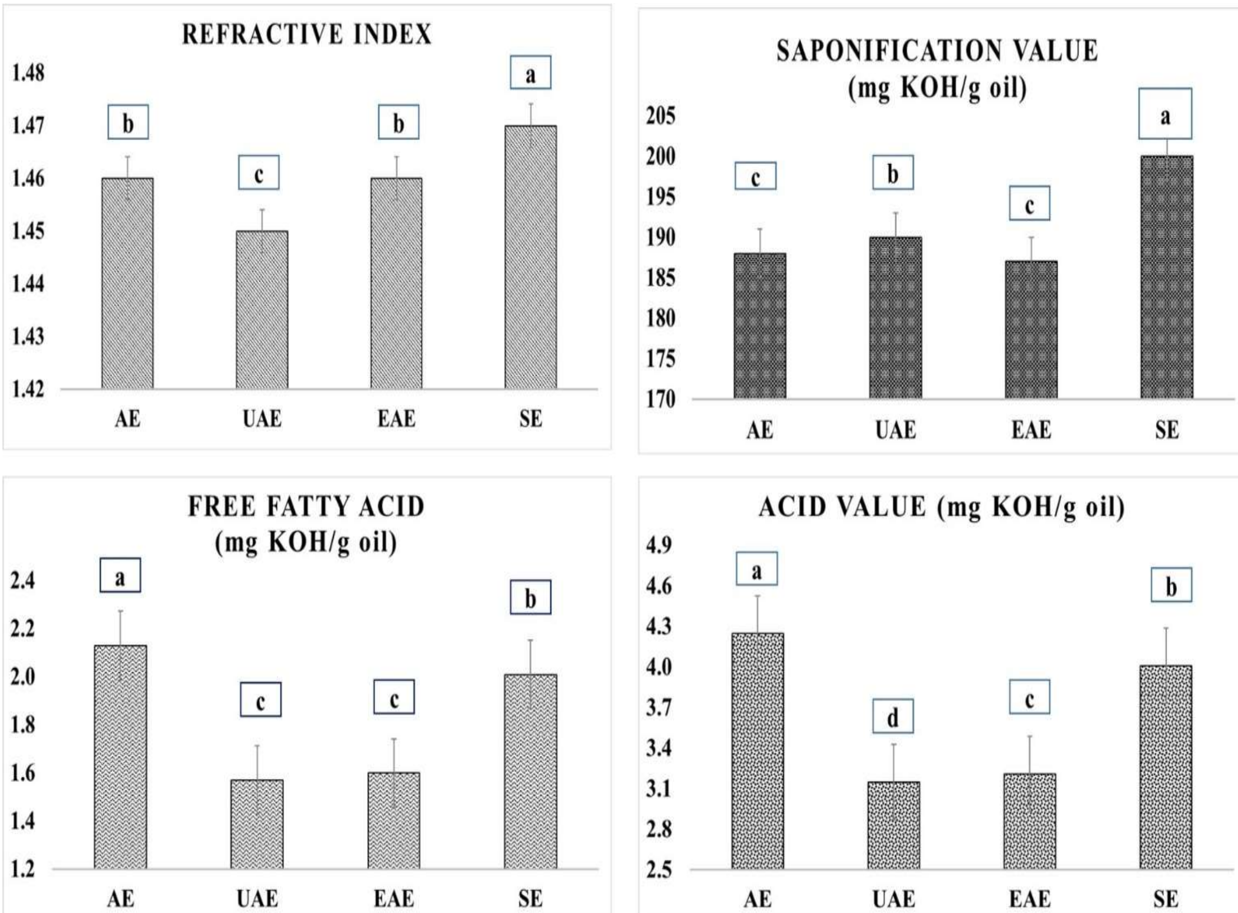

Figure 2: Effect of different extraction methods on the physicochemical quality of sesame oil (AE- Aqueous extraction, UAE- Ultrasound-assisted aqueous extraction, EAE-Enzyme-assisted aqueous extraction and SE-Solvent extraction) Note: Similar letters have no significant difference

Saponification value

The saponification value of sesame oil under different extraction methods was found to be 190 ± 3.15 mg KOH/g oil for the AE sample, 186 ± 3.01 mg KOH/g oil for the UAE sample, 189 ± 3.33 mg KOH/g oil for the EAE sample whereas it was 200 ± 3.89 KOH/g oil for solvent extracted oil. It was significantly ($p<0.05$) affected by different extraction methods. The maximum value was found for SE oil, followed by AE oil and minimum in EAE oil (Figure 2). The lower saponification value shows this might be due to the high amount of impurities present. Similar results were found for a refractive index of coconut oil (Odoom & Edusei, 2015).

Free Fatty Acids

The free fatty acid value in sesame oil varied from 1.57 to 2.13 mg KOH/g oil. The maximum free fatty acid value was found for AE oil (2.13 ± 0.62 KOH/g oil), followed by SE oil (2.01 ± 0.22 mg KOH/g oil) and minimum in UAE oil (1.57 ± 0.41 mg KOH/g oil) as shown in Figure 2. The higher value of the free fatty acid in oil makes it more susceptible to oxidative ageing, hence quicker the oil becomes rancid. Therefore, a higher level of free fatty acids decreases the overall quality of oil (Katkade *et al.*, 2018).

Acid Value

The acid value is a chemical parameter carried out on the oil of sesame seeds. The Acid value is a measurement of the free fatty acids in oil which is directly connected to the superiority of the oil. According to AOAC (1990), the acid value does not exceed the 5.0386 mg KOH/g. Therefore, the acid value of sesame oil extracted using different methods was lower value than prescribed by AOAC (1990) viz. 4.25±0.6mg KOH/g oil for the AE sample, 3.21±0.25 mg KOH/g oil for the EAE sample, 3.15±0.13 mg KOH/g oil for the UAE sample, and 4.01 ± 0.22 mg KOH/g oil for the SE sample. It examined that the acid value was significantly ($p< 0.05$) pretentious by the method used for oil extraction from sesame seeds. The acid value of the oil sample extracted using the AE method had maximum value, followed by SE then EAE method, and minimum in UAE method as shown in Figure 2. It can be attributed that oil had a lower free fatty acid value in the oil, causing a lower acid value (Negash *et al.*, 2019).

Oil recovery and extraction time

The sesame oil recovery and extraction time using various methods i.e., AE, SE, UAE, and EAE are presented in Table 1. Although Controlled sample oil extracted by SE had a higher extraction time as compared to the other three methods, the oil recovery was found to be maximum in SE followed by UAE and minimum in AE. This finding suggested that the compression and rarefaction of the elastic mechanical vibration wave of ultrasonic can drive the medium, resulting in changes in liquid pressure and a significant number of vacuum bubbles. When bubbles rupture and produce high immediate pressure, this is known as the "cavitation effect". Cells from sesame seeds were sheared and crushed. This activity aided sesame oil extraction, increasing the rate of oil production (Khoei & Chekin, 2016).

Table 1: Extraction of oil by various methods

Method of extraction	Total time taken (h)	Oil yield (%)
Aqueous extraction	22.5	27±0.02
Ultrasound assisted aqueous extraction	23.5	41±0.04
Enzyme assisted aqueous extraction	21.5	37±0.08
Solvent extraction	12	45±0.03

Note: Readings were taken in triplicate and readings represent in the form of Mean ± SD

Conclusion

The influence of four distinct oil extraction procedures on the physicochemical quality of sesame oil was investigated in this study: solvent extraction, aqueous extraction, ultrasound-assisted aqueous extraction, and enzyme-assisted aqueous extraction. When compared to oil extracted by AE, EAE, and SE, UA extracted oil had a lower refractive index, acid value, and free fatty acid value, maximal saponification value, extraction time, and oil recovery. It was concluded that oil extracted from sesame seeds using UAE method had better quality retention than other methods.

Conflict of interest

The authors declare that they have no conflict of interest.

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