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Study on Effect of Extraction Method on Physio-Chemical Properties of Watermelon (Citrullus Lanatus) Seed Oil

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ABSTRACT: Seed oils are oils extracted from the seeds of various plants, such as sunflower, sesame, or pumpkin seeds which are mostly used nowadays. These oils are often used for cooking, cosmetics, and also in some cases as dietary supplements due to their nutritional properties. Each seed oil has its unique composition and characteristics, offering different flavors and health benefits. This study aimed to study the extraction of oil from watermelon seed by the Soxhlet method using petroleum ether and n-hexane as solvents at different feed solvent ratios and also the extraction using an oil expeller. Watermelon seed oil characterization was evaluated to determine whether this oil could be exploited as an edible oil. The extraction of oil from watermelon seeds was separated from the fruit, dried, and then subjected to extraction of the oil. This method preserves the oil's nutritional composition and natural properties, making it suitable for various applications such as cooking, skincare, and health supplements. Watermelon seed oil is rich in unsaturated fatty acids and antioxidants, making it a valuable resource for promoting health and well-being. Physical properties of the oil extracted using different are also studied in this paper giving a comparative analysis between them.

KEYWORDS: Watermelon seed oil, Soxhlet extraction, Expeller, Petroleum ether, Physicochemical properties

I. INTRODUCTION

[1] Vegetable oils have played a crucial role in meeting the world's nutritional needs and finding versatile applications in numerous industries. Over the centuries plant seeds have been harnessed as a valuable source of vegetable oil. Traditionally, oil from soybeans, cottonseed, peanuts, corn, palm seed, and sunflower has commercial dominance in production. [2] The world's supply of vegetable oil is currently more than 100 million metric tonnes (MMT). The demand is increasing at a rapid pace due to increasing demand for non-food use of vegetable oil, for example in cosmetics, biodiesel, lubricants, and pharmaceuticals. However, only 12 of the 500,000 known plant species are currently commercially exploited to produce vegetable oil to meet the world's increasing demand.

[3] Watermelon (Citrullus lanatus) is a flowering plant species of the Cucurbitaceae family and the name of its edible fruit. There are many different verities of watermelon, beyond their sweet and refreshing taste their seeds possess hidden potential as they contain essential nutrients such as carbohydrates, fats, insoluble fiber, and an array of minerals (calcium, zinc, sodium, iron, phosphorus, and magnesium) and vitamin A, B, C. [4] While the succulent flesh is the fruit is relished, its seed have been habitually discarded as waste. However, this research has unveiled the bioactive components present in watermelon seeds, elevating watermelon seed oil into a valuable resource. Watermelon seeds are nowadays used for oil extraction. [5] The seeds are dried and oil is extracted by pressing them. This practice is common in West Africa and the watermelon seed oil is popularly known as orange oil or Kalahari oil. Watermelon seeds are reported to worthwhile source of protein due to their high protein contents as well as their important medicinal constitution. The seeds are rich in oil (37.8-45.4%) and protein (25.2-37%) having precious amino acids.

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[6] Watermelon seed oil contains both linoleic and oleic acid in substantial level, which are super rich in various nutrients. These seeds have low-calorie content and are rich in micronutrients like zinc. [7] The oil from these seeds exhibits more potent antioxidant properties which helps to lower the chance of developing chronic ailments including cancer. [8,9] They also possess anti-inflammatory properties and demonstrate antimicrobial properties against various pathogens. [10] Watermelon seed oil contains a considerable amount of PUFAs which are very receptive to oxidation and other side reactions that cause deterioration of oil.

[11]R.L.Nibe and R.V.Hinge studied the oil yield from watermelon seeds using different ratios of feed to petroleum ether in the Soxhlet extractor. In this research, the watermelon seed powder to petroleum ether ratio is taken as 1:3,1:4,1:5 whose % yields are 32%, and 35%and37% respectively. From this, he concluded that petroleum ether is best suitable for the extraction of oil from watermelon seeds using a Soxhlet extractor. Petroleum ether can be separated easily after extraction and has a higher yield. It showed that the ratio of feed to solvent is also an important factor to be considered.

[12] Zahar Moaddabdoost Bboli researched the characteristics and composition of watermelon seed oil and solvent extraction parameters effect. In this paper he compared the properties of the extracted watermelon seed oil to the average properties of soybean and sunflower seed oil the yields of watermelon seed oil are appreciably higher than other oily vegetables such as palm (40%), soybean (18%), rapeseed (41%) and sunflower (40%).it stated that by increasing three parameters time, temperature and solvent federation, the oil extraction yield was improved.

This research paper aims to dry the watermelon seed and oil extracted using Soxhlet extraction and oil expeller, to perform a comparative study on the properties of the watermelon seed oil extracted using petroleum ether as a solvent and expeller and to analyze the physiochemical properties of watermelon seed oil.

II MATRIAL AND METHODS

The watermelon seeds used for this work are purchased at Ayurvedic Medicinal Shop in Sulur, Coimbatore, Tamil Nadu, India. The equipment's are Soxhlet extractor and an oil expeller for oil extraction. Petroleum ether is used as a solvent in Soxhlet extractor.

A **Soxhlet extractor** is a piece of laboratory apparatus invented in 1879 by Franz von Soxhlet. It was originally designed for the extraction of a lipid from a solid material. Typically, Soxhlet extraction is used when the desired compound has limited solubility in a solvent, and the impurity is insoluble in that solvent. It allows for unmonitored and unmanaged operation while efficiently recycling a small amount of solvent to dissolve a larger amount of material

Soxhlet extractor has three main sections: a percolator (boiler and reflux) which circulates the solvent, a thimble (usually made of thick filter paper) which retains the solid to be extracted, and a siphon mechanism, which periodically empties the condensed solvent from the thimble back into the percolator**.**

Fig. 1 Watermelon seed

Fig 2. Soxhlet Extractor

An oil extraction machine uses high pressure and heat to squeeze the oil out of a plant product. Those products include seeds, nuts, vegetables, and fruit through this mechanical process which is done without adding chemicals. They are also known as oil expellers and oil presses. Oil extraction machines typically use screw presses. Some products like ground nut, and watermelon seeds need to be shelled or peeled before moving through the screw press. Oil extraction machines are very versatile.

The conventional solvent extraction (CSE) process is amongst other factors based on the ability of the solvent to dissolve oils and extract them from the seeds. Therefore, the solvent must be able to solubilize the oil for an effective extraction. It is the most commonly used method usually carried out either a batch or continuous process. The three major steps involved in solvent extraction are

- Oil seed cleaning and conditioning
- Oil extraction and
- Separation of miscella

Crude oil and meal quality depend mostly on the type of solvent used, reaction temperature, and type of pre-treatment given to the oil seed [10,11].

Take 100gm watermelon seed dried in oven at 105c to remove moisture. The dried seeds are crushed to form powder. Take 1:3, 1:4 ratio of watermelon seed powder (100gm) to petroleum ether (400ml) respectively. Take filter paper and watermelon seed powder in the filter paper Put a filter paper in thimble of Soxhlet extraction apparatus contain seed powder Take400ml of petroleum ether as solvent in round bottom flask of Soxhlet. The mixture was heated at 35-40c (B.P. solvent) for 1.5-2 hrs. After extraction remove the round bottom flask from Soxhlet apparatus. Seed oil to be removed from the solvent using simple distillation. Separation by simple distillation carried out at temperature 35-40c.In distillation petroleum ether recovers as the top product and oil as a bottom product.

The dried watermelon seeds are fed into the screw press, which comprises a screw inside a high-pressure cylinder chamber. The seeds move through the screw, generating friction and heat. While the high pressure squeezes out the oil, the presences of heat denature some proteins in the oil making it easier to move by decreasing the viscosity. As the oil is pressed out, it seeps through a filter to ensure no solids or fibers move with it. The leftover pressed seeds from a hard cake are removed from the machine and the oil flows out into separate containers.

III . DETERMINATION OF PHYSIOCHEMICAL PROPERTIES OF WATERMELON SEED OIL

Oil yield:

Oil yield refers to the amount of oil that can be extracted from an oil seed. Oil yield determination is a base for many other calculations.[3]

Percentage yield= (weight of extracted seed oil)/(weight of dried seeds)×100

Acid–value (A.V) determination

An Erlenmeyer flask was dried and filled with 2.5g of watermelon seed oil, 50 ml of absolute ethanol, and 1ml of phenolphthalein. the mixture was heated for 3 hrs and titrated with 0.1 KOH to determine the acid AV of the oil, which was subsequently used to calculate the free fatty acid content of the oil.[4]

Acid value= (ml of KOH ×N ×56)/(Weight of sample used) mg of KOH

FFA was calculated by applying AV % free fatty acid = (Acid value)/2

Determination of saponification value (S.V)

A conical flask was filled with 2g of oil. Then, 25 ml of an alcoholic potassium hydroxide solution was added to the flask. The flask was heated for one hour while repeatedly shaken in boiling water with a reflux condenser attached. Neat, 1 ml of a 1% solution of phenolphthalein was added. The hot excess alkali was then titrated with 0.5 M hydrochloric acid and the volume of hydrochloric acid used in titration was recorded as ‘a’ (in ml). Simultaneously, a black titration is conducted, and the volume of hydrochloric acid used in black titration is recorded as ‘b’ (in ml). [4]

S. V=((b-a)×28.05)/(weight of sample)

Determination of iodine value (V.I)

The iodine value was determined using a method described in reference [13]. To begin, 0.3 g of dry matter melon oil was accurately measured and placed in a 250 ml flask. Next, 25 ml of carbon tetrachloride was added to the flask, and the contents were thoroughly mixed. The weight of the watermelon oil sample was checked to ensure an excess of 50-60% of the required viscosity. subsequently, 25 of the wijs solution was pipetted into the mixture, and the glass stopper was reinserted after dampening it with potassium iodide (KI) solution. The mixture was then thoroughly mixed and placed in a dark cabinet for 30 min. A blank test was conducted without adding watermelon oil. After the 30 min, incubation period, 15ml of KI solution is added, followed by 100ml of freshly boiled and cooled water. The stopper was rinsed, and the released Potassium iodide solution was titrated with sodium thiosulfate. Solution using a starch indicator. The titration process continued until the blue color

disappeared with vigorous shaking.[5]
I.V=((12.69(B-S)N))/W
where,
B = volume of sodium thiosulfate solution used for blank titration,
S = volume of sodium thiosulfate solution used for the sample,
N = normality of sodium thiosulfate solution,
W = weight of the watermelon oil sample.

Determination of peroxide value (P.V)

Approximately 2g of oil sample was measured and placed into a clean, dry flask. To the flask was added 30ml of a mixture of glacial acetic acid (CH3COOH) and chloroform (CHCL3) in a ratio of 3:3. Thereafter, drops of standard potassium iodide (KI) solution were added to the flask and thoroughly mixed. The solution was left in the dark cupboard for 5 minutes. A standardized 0.1N iodine (I) solution was used to titrate the released iodine until a persistent yellow color was achieved. Following that, 1 ml of the starch indicator solution was added, and the titration was carried out till the blue color mixture disappeared. A blank titration was performed using the same procedure but without a watermelon oil sample.[5]
P.V(meq/kg) =((V×N×1000))/W
where,
V=Volume of iodine solution used
N=Normality of iodine solution (0.1N)
W=Weight of oil sample(g)

Determination of refractive index (R.I)

Abbe refractometer was employed to determine the refractive index (I.R). The lower prism of the instrument was coated with watermelon oil, and then the prism was sealed. The light was directed through the refractometer machine using an angled mirror, causing the reflected light to create a dark background. Refractometer telescope tubes were adjusted until the absence of shadow, which was seen as the centre dark region in the cross-wire indicator, was attained. The refractive index (RI) of oil was then read off from the refractometer scale.

Specific Gravity (S.G.) determination

The 50 ml pycnometer bottle was carefully cleaned using soap and water; and then rinsed with petroleum ether. Thereafter, the pycnometer bottle was weighed to determine its empty weight. The 50 ml bottle was subsequently filled with purified water, and its weight was once more measured. The 50 ml bottle was filled with the oil sample after being dried and then weighed to determine its weight.[6]
S.G =(weight of the specific volume of oil sample)/(weight of an equal volume of distilled water)

pH determination

By first employing a buffer solution, the pH electrode was calibrated. After the calibration was finished, the pH electrode was put into the sample of watermelon oil to measure its pH level, and the readings were recorded.

IV.RESULT AND DISCUSSION

Table 1 represents the oil yield at different solvent ratios and methods. Table 2 represents the physicochemical properties of watermelon seed oil including pH refractive index, specific gravity, free fatty acid, etc.
Let us consider the following,

- Sample 1 Oil extracted from petroleum ether at a ratio of 1:4
- Sample 2 Oil extracted from petroleum ether at a ratio of 1:3
- Sample 3 Oil extracted using oil expeller

TABLE 1 OIL YIELD AT DIFFERENT EXTRACTION METHODS

S.NO	EXTRACTION METHOD	OIL YIELD PERCENTAGE
1.	Extraction with petroleum ether for feed-to-solvent ratio 1:4	26%
2.	Extraction with petroleum ether for feed-to-solvent ratio 1.3	19%
3.	Extraction using oil expeller	43%

TABLE 2 PHYSIOCHEMICAL PROPERTIES OF EXTRACTED OIL

S.NO	PARAMETERS	SAMPLE1	SAMPLE2	SAMPLE3

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1.	Acid value	4.37	4.52	3.8
2.	Saponification value	196.7	307.8	316.3
3.	Iodine value	134.5	123.6	125.5
4.	Peroxide value	4.25	5.23	4.1
5.	Refractive index	1.450	1.398	1.452
6.	Specific gravity	0.933	0.928	0.934
7.	Ph	4.06	4.09	4.01

The volume of the oil was found to be 80 ml. The percentage of the oil produced is calculated. From the different methods of extraction carried out on watermelon seed samples, the following comparative analysis can be made shown in table 2. The acid value of watermelon seed oil and its acid value ranges from 0.5 to 3mg KOH/g (Figure 4). This value indicates the amount of free fatty acid present in the oil. The saponification value is highest for the oil extracted using an oil expeller followed by the oil extracted using petroleum ether 1:3 ratio (Figure 5). This indicates the alkaline value of extracted oil. The iodine value of watermelon seed oil falls between 100 to 140 g (Figure 6). This value reflects the degree of unsaturated fatty acid present in the oil. The peroxide value of watermelon seed oil ranges from 0.5 to 10 meq/kg. This value indicates the extent of oxidation in the oil, reflecting its freshness and stability.

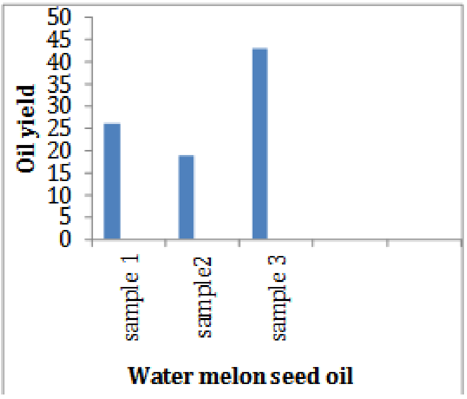

Fig. 3 Oil yield at different extraction methods

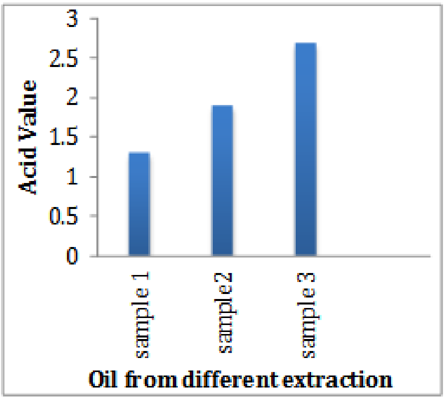

Fig. 4 Acid value

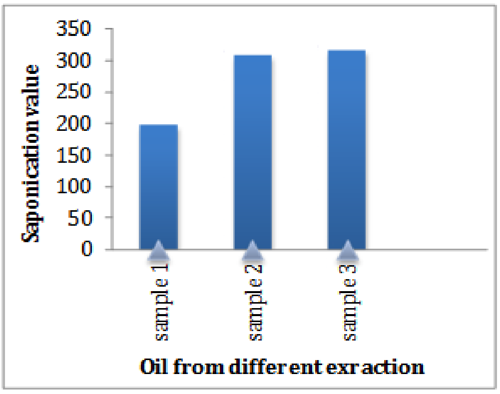

Fig. 5 Saponification value

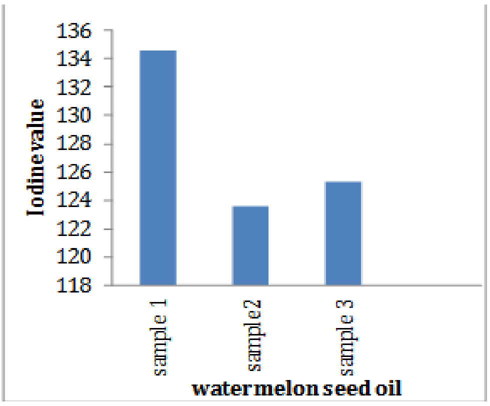

Fig. 6 Iodine value

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V. CONCLUSION

In this research, watermelon (Citrullus lanatus) oil is successfully extracted and characterized. Also revealing is favorable physicochemical properties and oil yield at different methods. solvent extraction is one of the traditional techniques of extracting vegetable oil. but the usage of chemicals and the apparatus setup gives trouble. Whereas the oil expeller is easy to work and maintain. the oil extraction is easily filtered too. Watermelon seeds could be utilized successfully as a source of edible oil for human consumption. The high yield and valuable properties of watermelon seed oil make it a promising candidate for application in various industries. The oil's ether solubility and soot-free flame quality enhance its utility in various formulations. They might be an acceptable substitute for highly unsaturated oil. These results will be helpful in the utilization of watermelon seed for oil production could provide extra income and at the same time help to minimize waste production.

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