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## Influence of moisture content and temperature on mechanical extraction of oil from watermelon (*Citrullus lanatus*) seeds

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#### Abstract

Watermelon fruit contains large quantities of seeds and the seed oil has got many benefits. The oil content of seeds was estimated using Soxtherm apparatus and found to contain 22 % of oil in it. The oil from seeds was extracted using mechanical screw press. To optimize the effect of temperature and moisture content on oil recovery completely randomized factorial design of experiments was employed. The oil was extracted at five different temperatures viz, 30, 40, 50, 60 and 70 °C and moisture contents viz, 4, 6, 8, 10 and 12 % combinations. The best combination for maximum oil recovery was observed to be at 50 °C temperature and 10 % moisture content. The extracted oil was evaluated for acid, iodine, saponification, free fatty acid, specific gravity, density and viscosity values. The determined values were 10.20 mgKOH/g, 154 gI<sub>2</sub>/100g, 190.80 mg/g, 5.32 mgKOH/g, 0.94, 1.39 g/cm<sup>3</sup> and 1.14 mm<sup>2</sup>/sec, respectively.

**Keywords:** Watermelon seeds, extraction, temperature, moisture content and oil recovery

#### 1. Introduction

Watermelon (*Citrullus lanatus*) belongs to the family of Cucumber (*Cucurbitaceae*), is large, oval, round or oblong in shape (Quek, Chok, & Swedlund, 2007)<sup>[16]</sup>. It is a vine-like flowering plant originated from southern Africa. In India, watermelon is cultivated in an area of 74,640 hectares with a total production of 1809.83 million tonnes and productivity of 24.2 t/ha (Anonymous, 2014)<sup>[1]</sup>. It is one of the major underutilized fruits grown in the warmer part of the world. The juice or pulp from watermelon is used for human consumption while rind and seeds are the major solid wastes (Mao, Jeong, Que, & Huber, 2006)<sup>[13]</sup>. The rind is utilized for products such as pickles and preserves as well as for extraction of pectin (Sadrnia, Rajabipour, Jafary, Javadi, & Mostofi, 2007)<sup>[17]</sup> whereas seeds are potential source of protein (Oyenuga & Fetuga, 1975)<sup>[15]</sup>; (Teotia & Ramakrishna, 1984)<sup>[20]</sup> and lipids (Kamel, Dawson, & Kakuda, 1985)<sup>[10]</sup>.

The dry seeds of watermelon contain on an average of about 32 g protein and 51.4 g fat per 100 g of sample (Kamel *et al.*, 1985)<sup>[10]</sup>. Watermelon seed oil is rich in Vitamin A, B and E. Magnesium, iron, copper, phosphorus, potassium and manganese compounds also present in the seed oil (Wani, Sogi, Singh, Wani, & Shrivhare, 2011)<sup>[21]</sup>. Watermelon seed oil provides essential amino-acids such as arginine and lysine which helps in improving the metabolism of the body and absorption of calcium for strengthening the bones and the tissues. The oil gives unsaturated healthy fats to the body which in turn reduces the blood pressure (Altuntas, 2008)<sup>[5]</sup>. The demand for vegetable oil is increasing at a rapid rate due to increasing population. Vegetable oils are an important component for both food (for feeding, margarine and canned food industry, bakery, confectionery) and non-food industry (production of detergents, paints, special varnishes, fatty acids, pharmaceuticals and cosmetics products, and painting (Koocheki *et al.*, 2007)<sup>[11]</sup>.

Currently, oil is being extracted by using different methods such as chemical, high pressure CO<sub>2</sub>, distillation and mechanical extraction methods. Although existing chemical methods of oil extraction is having high percentage of oil recovery it has several disadvantages such as high cost, chemical retainment in oil, not easily available for farmers etc. (Liauw *et al.*, 2008)<sup>[12]</sup>. A simple way to overcome the above said limiting factors is by using mechanical extraction method, as this method will be easily available for farmers and cost effective. Hence the experiment was conducted to find out the oil recovery in mechanical extraction under the influence of moisture content and temperature of water melon seeds.

## 2. Materials and Methods

The present investigation on “Influence of moisture content and temperature on mechanical extraction of oil from watermelon (*Citrullus lanatus*) seeds” was carried out in laboratories of Biofuel Center and Department of Agriculture Engineering, UAS, GVK, Bengaluru. The ‘Arka manic’ variety of watermelon seeds were procured from IIHR, Hesargatta, Bengaluru.

### 2.1 Estimation of oil content in watermelon seeds

The oil content in watermelon seeds was determined using the Soxtherm apparatus (Model: Gerhardt, Germany) which works on the principle of solvent extraction. Dried seeds were powdered in a mixer grinder. Three grams of powder was weighed with precision to three decimal places using an electronic digital balance (Model PS 200/2000/C/2-RADWAG, Poland). Powdered seed samples were taken in a cotton thimble and plugged with cotton and placed in pre-weighed soxtherm jars containing boiling stones. After adding 100 ml of petroleum ether into the jars, the jars were placed in the soxtherm apparatus. Oil was extracted by running the preprogrammed soxtherm apparatus for 4 h 35min. After completion of extraction, the remaining petroleum ether and moisture was removed by keeping it in hot air oven at 110°C for 1 hour. The jars were removed from the oven and placed in desiccators containing calcium carbonate for 1 h to remove the moisture content. The oil content in the seeds was determined using the following formula (Matthäus & Brühl, 2001)<sup>[14]</sup>.

$$\text{Oil content (\%)} = \frac{W_2 - W_1}{W} \times 100$$

#### Where

$W$  = weight of powdered seed sample

$W_1$  = weight of the soxtherm jar along with boiling stones

$W_2$  = Final weight of the soxtherm jar along with the boiling stones and extracted oil.

### 2.2 Sample preparation

The initial moisture content of the dried watermelon seeds was determined using a standard oven method (AOAC, 2002) and it was found to contain 4.02 % moisture content. The watermelon seed sample of desired moisture levels were prepared by adding calculated amount of distilled water through mixing and then sealing in separate plastic bags. The samples were kept at 5 °C in a refrigerator for at least a week to enable the moisture to distribute uniformly throughout the sample. Before starting a test, the required quantities of seeds were allowed to warm up to room temperature. The quantity of distilled water was estimated from the following equation. Temperature of the samples were varied by keeping the samples in the hot air oven to desired temperature for 24 h (Koocheki *et al.*, 2007)<sup>[11]</sup>

$$W_2 = W_1 \frac{M_1 - M_2}{100 - 1}$$

#### Where

$W_2$  = Mass of distilled water added (kg)

$W_1$  = Initial sample mass (kg)

$M_1$  and  $M_2$  are the initial and desired moisture content of sample (w.b %).

### 2.3 Design of experiments

The experiments were carried out by varying temperature and moisture content.

1. Temperature ( $T_1$ -30 °C,  $T_2$ -40 °C,  $T_3$ -50 °C,  $T_4$ -60 °C and  $T_5$ -70 °C)
2. Moisture content ( $M_1$ -4,  $M_2$ -6,  $M_3$ -8,  $M_4$ -10 and  $M_5$ -12 %)

The experimental design contains 25 treatments which were the combinations of temperature and moisture contents using Factorial Completely Randomized Design (FCRD).

Treatments: 25; Sample size: 1 kg of seeds per treatment per replication.

### 2.4 Oil extraction from watermelon seeds

The oil was extracted from watermelon seeds using a mechanical screw expeller, a motorized oil extraction device (0.5 hp and 2 kg/h capacity) designed and fabricated by Bio fuel center, Department of Forestry and Environmental sciences, UAS, GVK, Bengaluru. In this method of oil extraction, raw materials were squeezed under high pressure in a single step of operation. The screw present in the extractor, presses oil seeds through a perforated caged barrel. Due to friction, pressure and compression forces, seed oil seeps through the small opening. Oil extracted in this method is called cold-pressed oil. The waste material obtained from this screw press can be used for preparing manure by composting. The oil obtained should be filtered to remove larger impurities and allowed to settle for 2-3 days to remove smaller impurities and measured. Obtained clean oil can be used for further analysis

### 2.5 Physico-chemical characteristics of watermelon seed oil

Extracted oil was subjected to evaluation for biochemical properties using the method followed by (Sarma, Konwer, & Bordoloi, 2005)<sup>[18]</sup> The details of the properties determined and the methods followed for the determination are described below.

#### 2.5.1 Acid value and Free Fatty Acid (FFA)

Acid value is the amount of potassium hydroxide in mg that would neutralize the free acid in a gram of the sample and is expressed in (mgKOH/g). 25 ml of petroleum ether was mixed with 25 ml of ethanol and 4 drops of phenolphthalein indicator into a conical flask containing 2 g of the watermelon seed oil sample and was titrated with aqueous 0.1 M KOH and shaken properly until a pink colour which persist for 15 seconds was obtained.

**Free Fatty Acid:** It is a number that is usually calculated as oleic acid, also expressed in (mg KOH/g).

#### Calculation

$$\text{Acid value(mgKOH/g)} = \frac{\text{Titre (ml)} \times 0.1 \times 56.10}{\text{Weight of the sample used(g)}} \quad \text{---3.10}$$

$$\text{FFA(mgKOH/g)} = \frac{\text{Acid value}}{2} \quad \text{---3.11}$$

#### 2.5.2 Iodine value

This is a measure of the degree of unsaturation in any

vegetable oil or animal fat. It is the weight of iodine absorbed by 100 parts by weight of the sample and is expressed in (mg/g). Exactly 0.3 g of oil was transferred to a clean dry iodine glass containing 10 ml of chloroform/carbon tetrachloride, mixed well and 25 ml of iodine monobromide (IBr) was added to it. One conical flask was kept as a blank without oil. Both the flasks were shaken well and were placed in dark. After this, stirring was done for each flask for every 5 min for about 30 min. 50 ml of water and 10 ml of 10 % potassium iodide solution was added to each flask. Then the solutions were titrated against standard  $\text{Na}_2\text{S}_2\text{O}_3$  solution using 2 ml of starch as indicator with end point blue to colorless. Iodine number was calculated using the following formula.

$$\text{Iodine value} = \frac{(V_1 - V_2) \times N \times 126.9}{W \times 1000} \times 100 \quad \text{---3.12}$$

Where,  $V_1$  is the volume of  $\text{Na}_2\text{S}_2\text{O}_3$  used for blank,  $V_2$  is the volume of  $\text{Na}_2\text{S}_2\text{O}_3$  used for oil,  $N$  is normality of  $\text{Na}_2\text{S}_2\text{O}_3$ ,  $W$  is the weight of the oil in gram.

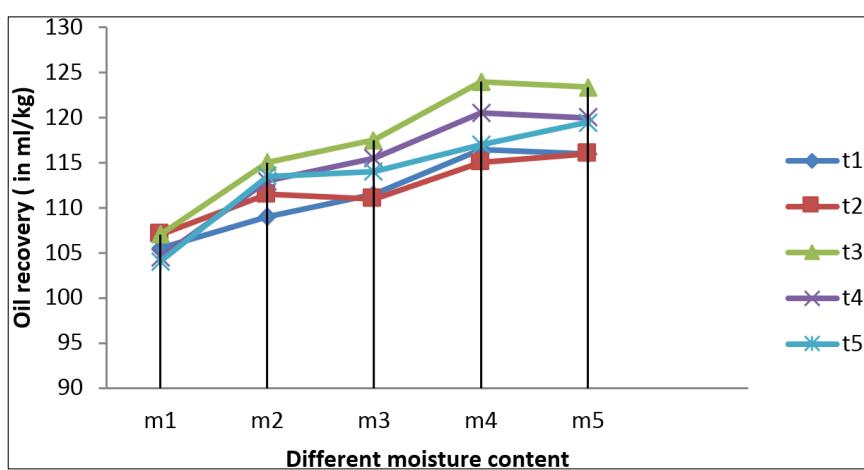
### 2.5.3 Specific gravity and Density

25 ml of pycnometer bottle was washed, dried and weighed. The bottle was filled with Toluene ( $\text{C}_6\text{H}_5\text{CH}_3$ ) and weighed. The bottle was emptied, dried and filled with the extracted watermelon seed oil and weighed.

Calculation:

$$\text{Specific gravity} = \frac{\text{weight of 25ml of oil(g)}}{\text{weight of 25ml of water(g)}} \quad \text{---3.13}$$

$$\text{Density } \left( \frac{\text{g}}{\text{cm}^3} \right) = \frac{\text{weight of 25ml of oil(g)}}{\text{volume of water}(\text{C}^3)} \quad \text{---3.14}$$



**Fig 1:** Oil recovery at 5 different temperature and moisture content combinations

The results of oil recovery at various combinations are represented in the fig 1. From the fig. 1 it was observed that, oil recovery at all combinations increased initially, reaches to maximum and subsequently reduced with further increase in temperature. The similar trend was observed in the findings of (Gikuru & Moriasi, 2007) [9] for grounded soybeans. The maximum and minimum oil recovery (124 ml) was obtained at  $T_3M_4$  ( $50^\circ\text{C}$ , 10 %) and  $T_5M_1$  ( $70^\circ\text{C}$ , 4%) combinations respectively. This increased oil recovery might be due to the increase in temperature which in turn increases the oil mobility as it facilitates the rupturing of oil cells by creating voids and serves as a migratory space for oil. (Adeeko & Ajibola, 1990) [4]. Oil recovery was reduced by further

### 2.5.4 Viscosity

Viscosity of watermelon seed oil was measured using Viscometer (Cannon-Fenske Viscometer No. 200 with nominal constant 0.10). The sample was introduced into the viscometer by immersing tube 'A' into the sample and suction was applied to 'I', till the sample raised to the etched line 'E'. Then the viscometer was made to normal position and tube A was cleaned. The viscometer was then inserted into a holder and placed in water bath maintained at constant temperature of  $40^\circ\text{C}$ . Sample from tube A was brought into the tube B, a short distance above the mark C and the time to pass from A to B was recorded. The kinematic viscosity was measured by multiplying the time with a viscometer constant.

### 2.6 Statistical analysis

The results of the oil extraction using different moisture content and temperature combinations were analyzed using Factorial Completely Randomized Design (FCRD) with two replications by using AGRES software. The mean of the observation was subjected to two-way ANOVA and significance of the means were tested using F-test at ( $p<0.05$ ) among the design and operational parameters, the best treatment combination was selected based on their treatment mean values and significance level (Fisher & Yates, 1963) [7].

## 3. Results and Discussion

The oil content was estimated using soxholt apparatus which was found to be 22 % and the time for the extraction was 4 hour and 35 min. After extraction, the petroleum ether and the moisture remained in the oil was separated by keeping in oven at  $110^\circ\text{C}$  for 1 h and thus the total time required was 5 h and 35 min.

### 3.1 Effect of temperature and moisture content on oil recovery of seeds (ml)

increase in temperature (above  $50^\circ\text{C}$ ) as samples were hardened due to moisture loss and there was frequent choking and jamming of screw as cake becomes hard and choke the outlet which might be the reason for reduction of oil recovery (Schembre & Kovscek, 2004) [19]

From the figure it was observed that, oil recovery increased as moisture content increased from 4 % and reaches to maximum at 10 % at all 5 temperatures but further increase in moisture content not shown any desirable effect on oil recovery. The reason might be due to the increase in temperature causes hardening of samples but increase in moisture content might have avoided hardening of samples hence higher percentage of oil was observed. Further increase

in moisture content (above 10 %) at lower temperature causes seeds to become soft with absorption of moisture and hence less force is applied by screw on the seed due to which the seed slide along with screw. Moisture acted as lubricant in the barrel which resulted in insufficient friction and reduced level of compression and contributed to less oil recovery. Decrease in moisture content and increase in temperature causes seeds to get hardened and hence frequent choking and jamming of screw reduces the oil recovery. The results were found similar to the finding of (Adebanjo & Kehinde, 2013)<sup>[3]</sup> for *moringa oleifera* seeds at different temperatures.

**Table 1:** Efficiency of mechanical screw press at different treatments (%)

Treatments	M <sub>1</sub>	M <sub>2</sub>	M <sub>3</sub>	M <sub>4</sub>	M <sub>5</sub>
T <sub>1</sub>	58.61	60.55	61.94	64.72	64.44
T <sub>2</sub>	59.44	61.94	61.66	63.88	64.44
T <sub>3</sub>	59.44	63.88	65.27	68.88	68.55
T <sub>4</sub>	58.05	62.77	64.16	66.94	66.66
T <sub>5</sub>	57.77	63.05	63.33	65.00	66.38

## Where

T-Temperature (°C)	T <sub>1</sub> - 30	T <sub>2</sub> - 40	T <sub>3</sub> - 50	T <sub>4</sub> - 60	T <sub>5</sub> - 70
M- Moisture content (% w.b)	M <sub>1</sub> - 4	M <sub>2</sub> - 6	M <sub>3</sub> - 8	M <sub>4</sub> - 10	M <sub>5</sub> - 12

## 3.2 Biochemical characterization of oils at optimized treatments

Biochemical analysis for the watermelon seed oil obtained at optimized treatment (10 % moisture content and 50 °C) was determined. The acid value of extracted watermelon seed oil was found to contain 2.80 mgKOH/g. Free Fatty Acid (FFA) was determined by knowing acid value and the values of FFA ranged from 5.32 mgKOH/g. The amount of catalyst required for trans esterification process if the oil is used for biodiesel purpose can be calculated based on FFA values and the obtained values are given in the Table 2. The higher acid values could be due to incomplete formation of triglycerides or due to hydrolysis of triglycerides by the action of enzymes. The results were similar to the findings of Oladeji (2015) for egusi melon seed oil.

The iodine value obtained was 154 gI<sub>2</sub>/100g and the result is given in the Table 2. Iodine value is the measure of unsaturated fatty acids in the oil. It helps to indicate the oxidation stability and provide information about the fuel's tendency to form the sludge, affect lubricant quality and may cause corrosion. Higher iodine number represents lower oxidation stability, also the presence of polyunsaturated fatty acids that may polymerize at higher temperatures and form sludge and affect the performance of engine. The results were similar to the findings of (Fröhlich & Rice, 2005)<sup>[8]</sup> for *camelina sativa* oil.

**Table 2:** Biochemical properties of watermelon seed oil.

Sl. No	Properties	Watermelon seed oil
		Mean
1	Acid value, mgKOH/g	2.80
2	Free Fatty Acid(FFA), mgKOH/g	5.32
3	Iodine value, gI <sub>2</sub> /100g	154
4	Saponification value, mg/g	190.80
5	Specific gravity	0.94
6	Density, g/cm <sup>3</sup>	1.39
7	Viscosity, (millipoise)	112.32

The values of specific gravity were found to range from 0.94 which was slightly lesser than the specific gravity of water. The density of oil was found to be 1.39 g/cm<sup>3</sup>. The results were similar to the finding of (Berchmans & Hirata, 2008)<sup>[6]</sup> for *Jatropha* seed oil.

The values of viscosity for oil obtained was 112.32 millipoise. The results were found similar to the findings of the (Fröhlich & Rice, 2005)<sup>[8]</sup> for *Camelina sativa* oil. Since viscosity is the rate of opposition of flow of a liquid, it makes it to be a very important property of oil.

## Conclusion

Watermelon (*Citrullus lanatus*) seeds contains desirable essential amino acids and mineral compounds hence it is best suited for food applications. In that aspect oil was extracted using screw press and the best combination for maximum oil recovery was observed to be at 10 % moisture content and 50 °C temperature. The extracted oil was evaluated based on properties such as acid value, iodine value, saponification value, free fatty acid value, specific gravity, density and viscosity. The values obtained for these properties were 10.20mgKOH/g, 154 gI<sub>2</sub>/100g, 190.80 mg/g, 5.32 mgKOH/g, 0.94, 1.39 g/cm<sup>3</sup> and 1.14 mm<sup>2</sup>/sec, respectively.

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