Microwave – Assisted Extraction of Castor Oil

L. Mgudu, E. Muzenda, J. Kabuba and M. Belaid

***Abstract*—**The effect of microwave pretreatment of castor beans on oil recovery and quality was investigated. The extraction parameters examined were moisture content, yield, refractive index, specific gravity, pH, free fatty acid and acid value. The results were a highest yield of 44.34% when beans were treated at 280W for 120 seconds, refractive index values ranging from 1.4712 – 1,4718 and a free acid value of 0.336.

***Keywords*—**Castor oil, extraction parameters, microwave pre-treating, oil yield

I. Introduction

MICROWAVE heating is worldwide procedure used for food preparation in domestic and catering operations. Microwave radiation causes molecular friction of electric dipoles, which results in heating. Lipids have a low specific heat, which makes them susceptible to this radiation [1]. Microwave preheating can be used to substitute conventional oven heating, resulting in quicker pretreating with less energy consumption. This generates permanent pores in the seed, resulting in a higher yield [2]. Microwave pretreatment also improves oil quality. The short exposure time to microwaves, as compared to oven cooking, preserves most thermolabile compounds from degradation reactions [3]. Reference [1] investigated the oxidative deterioration and thermal properties of refined peanut, high oleic sunflower and canola oil were affected by microwave treatment. The oils were treated at a power setting of 720W and 2450 MHz frequency for 1.5 to 15 minutes. It was found that canola was more extensively oxidized by microwave heating. Both peanuts and canola oil experienced changes in the heating profiles. Other properties such as free fatty acids and peroxide content were affected. Although the study was not on the pretreatment of seeds before extraction, it gives insight into how microwaves can affect the chemical composition of oil. Reference [4] substituted the conventional heat pretreatment with microwave radiation for hazel nuts. The nuts were pretreated at a frequency of 2450 MHz at power settings of 400W and 600W. The properties studied were fatty acids, *α-tocotrienol* content, oxidative stability as well as acid and peroxide values. Microwave pretreatment was found to increase the oil oxidative stability. Reference [2] investigated the effect of microwave pretreatment on oxidative stability and nutraceuticals content of oil from rapeseed. The rape seeds were radiated with an 800W microwave at a frequency of 2450MHz. The results were compared to oil extracted by mechanical pressing. Microwave pretreatment was found not only to increase the oil yield but also the oxidative stability as well thus increasing the shelf life.

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Reference [5] performed characterization study of the oil extracted from *Scenedesmusobliquus* using a continuous microwave system. The microwave settings were 1.2 kW at a frequency of 2450 MHz frequency. It was found that microwave pre-treated oil had a higher composition of unsaturated and essential fatty acids, thus a better oil quality. High oil yields, better extraction rates and good oil quality indicated that a continuous microwave system is feasible for a large variety of oil seeds [5]. Reference [6] studied soybean and rice bran oil extraction in a continuous microwave system. Seeds were radiated at 2450 MHz at varying power ranging from 3kW to 5kW. The authors' main focus was on yield optimization but oil quality analysis was also performed. Quality indicators such as, free fatty acids, phospholipids as well as iodine and acid values showed that the oil was of acceptable standards.

Reference [6] concluded that microwave extraction is a viable option for rapid oil recovery. On the other hand [7] didn't observe any significant effect of microwave assisted extraction on the oil quality from peanuts, olive and sunflower seeds. The authors compared the results of Soxhlet extraction using hexane and Microwave-Integrated Soxhlet (MIS) extraction. The microwaves were at a frequency of 2450MHz, with power varied from 430W-770W. Considering the fatty acid content, it was concluded that microwave assisted extraction saved time but did not have any significant effect on oil quality. Reference [8] also found no significant changes in quality with microwave extraction of oil from oregano. Processing time was reduced by 80% with negligible changes in composition and physical properties of oregano essential oils.

Reference [9] investigated microwave pretreatment of palm oil production. Microwave pretreatment was found to save time saves and the free fatty acid and moisture contents were desirably lower. Other qualities that were affected by time spend in the oven were vitamin E and carotene content. The success of microwave extraction on other seeds makes it an option for castor seeds. Reference [10] applied microwave assisted extraction for polyphenols from grape seeds and it was found to be more rapid and efficient option. Reference [11] successfully used microwave extraction for the bromine and caffeine from cacao. Their findings showed that optimum extraction was achieved when radiating at 210W for 5 minutes. Reference [12] also supported microwave-assisted extraction for olive seeds as it reduced extraction time from 8hours with conventional soxhlet extraction to 25 minutes with microwave assistance. Most past work on the microwave treatment of castor oil focused on oil quality hence little quantitative information is available. The aim of this study was to investigate the effect of the microwave treatment on the yield and quality of oil from castor beans. A wide range of parameters such as moisture content, yield, refractive index, specific gravity, pH as well free fatty acid and acid values were investigated.

II. Materials and Method

A. Castor Seeds Preparation

- **Cleaning:** The seeds were cleaned by hand-picking to remove foreign objects.
- **Dehulling & Winnowing:** A pestle and mortar was used to crack the seeds, the shell was hand removed.
- **Drying:** The seeds were oven dried at 90°C for 6 hours. 100g of seeds were oven heated, weighed every hour to constant weight. The moisture content of the beans was determined using (1).

$$\text{Moisture Content} = \frac{W_i - W_o}{W_o} \times 100\% \quad (1)$$

In (1) W_i and W_o are the initial and final weights before and after drying respectively

B. Microwave Pre-Treatment

Seven 100g samples were treated as shown in Table 1.

TABLE I
MICROWAVE PRETREATMENT SETTING

Sample	Time (sec)	Power (W)
1	0	0
2	30	119
3	60	119
4	120	119
5	30	280
6	60	280
7	120	280

- **Size Reduction**

All the samples were crushed into a paste using a pestle and mortar.

- **Solvent extraction**

100ml of hexane was poured into a round-bottom flask. The flask was placed inside the heating mantle. Cotton wool was placed inside the round soxhlet extractor. 50g of each sample was placed inside the extractor, the condenser was connected to the extractor, the water tap was opened and finally the heating mantle was turned on. The hexane was boiled and evaporated. The vapour was condensed and dropped onto the castor seeds. The hot hexane caused the castor seed paste to disintegrate into a powdery form. The liquid hexane dripped back into the round-bottom flask, where it was evaporated. This evaporation-condensation process continued for 45 minutes resulting in oil extraction. After 45 minutes, the heating mantle was switched off and the apparatus was allowed to cool. The cold hexane-castor oil mixture was transferred into a beaker and oven heated at 70°C to remove hexane.

- **Oil Recovery Determination**

After removing the hexane, the beaker containing the oil was cooled and weighed. The amount of oil was found as the difference in weight between the dry clean beaker and that with oil. The amount of oil extracted was calculated (2).

$$\text{Oil Extracted} = \frac{\text{Oil}}{\text{Sample Size}} \text{x}100\% \quad (2)$$

- **Acid Value and Free Fatty Acids Determination**

A mixture of 12.5ml diethyl ether and 12.5ml ethanol was prepared in a beaker. This mixture was added to 5g of oil in a conical flask. A few drops of phenolphthalein were added. The mixture was titrated with 0.1M NaOH with consistent shaking. The end point was recognized by a dark pink colour. The volume of 0.1M NaOH (V_o) was noted. The free fatty acid (FFA) content was calculated using (3).

$$\text{FFA} = \frac{V_o}{W_o} \times 2.83 \times 100\% \quad (3)$$

In (3) 100 ml of 0.1M NaOH is equal to 2.83g of Oleic acid, W_o is sample weight and acid value = 2 x FFA.

- **Specific Gravity Determination**

A clean dry 10ml conical flask was weighed (W_o). 10ml of oil was added into the flask and then weighed (W_1). The flask was emptied, washed and dried. 10ml of water was then added into the flask and then weighed (W_2). The specific gravity which is the ratio of the mass of the substance to mass of an equal volume of water was calculated using (4).

$$SG = \frac{W_1 - W_0}{W_2 - W_0} \quad (4)$$

- **Refractive Index Determination**

A few drops of the oil were poured on the glass window of the refractometer and the refractive index was noted. This value was read 3 times for each sample and the window was wiped thoroughly after each test. The final refractive index was a mean of 3 readings.

- **pH Measurement**

2g of the oil was poured into a beaker and hot distilled water was added. The mixture was stirred slowly then cooled to 25°C using a cold-water bath. The pH electrode was first standardized with a buffer solution and then submerged onto the oil –water mixture.

III. Results and Discussion

A. Moisture Content

Table 2 shows the moisture content of the beans. The moisture content ranged from 3.16 – 3.72 % compared to the 5 – 7% reported in literature [13]. The reason could be that the beans investigated were from the Mpumalanga province in South Africa with high temperatures and low humidity leading to moisture removal as a result of natural evaporation.

TABLE II
CASTOR BEANS MOISTUE CONTENT

Sample	Mass (g)	Moisture Content (%)
1	100.03	3.28
2	100.04	3.35
3	100.03	3.53
4	100.02	3.2
5	100.04	3.09
6	100	3.72
7	100	3.16

B. Oil Yield

Fig. 1 shows the yield at each microwave setting. The beans were all treated at a frequency of 2450 MHz. The yield was in the range of 40 – 44% which is within the 40 – 45% range [14]. Microwave treated beans gave better results compared to the untreated.

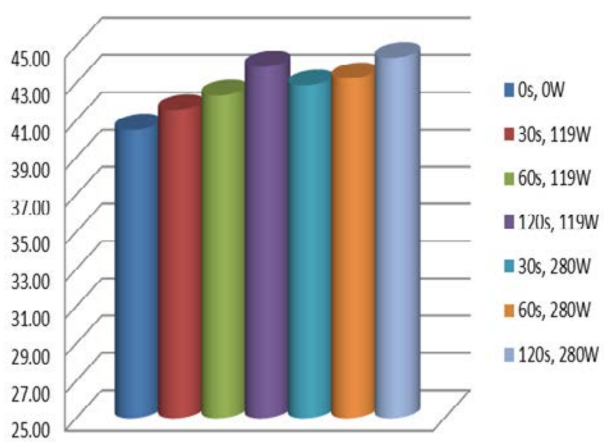

Fig. 1 Variation of oil yield with microwave setting

The highest yield of 44.34 % was obtained when beans were treated at 280W for 120 seconds. Microwave treatment decreases the moisture content of the beans, thereby making them more fragile. As a result, the tissues rapture easily, creating a higher porosity which leads to a higher yield. As discussed [15], [16], microwave pretreatment reduces moisture content making the internal pressure to increase. The release of the water causes disintegration of the cell membranes, which increases the porosity of the beans. All the samples used were crushed into a paste. The untreated beans remained as small granules throughout the extraction while the microwave treated beans turned into powder. The powdery beans gave higher yield as the contact surface was increased.

C. Refractive Index

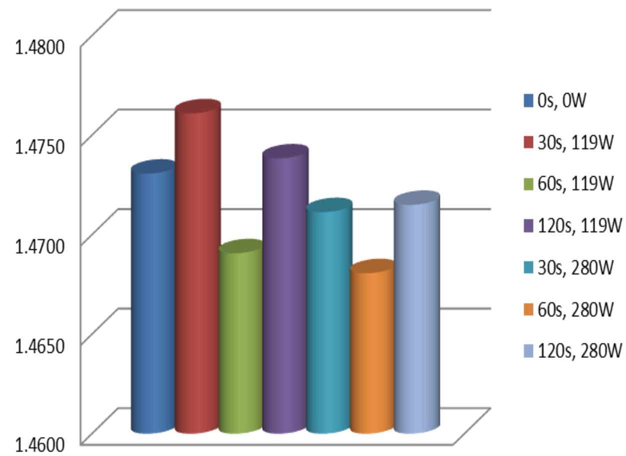

Fig. 2 Variation of refractive index with microwave setting

The refractive index varied with microwave treatment from 1.4712 to 1.4718 although no particular trend was observed, Fig. 2. These values are slightly lower than the range of 1.475 – 1.480 [13], giving a 1% variation.

D. Specific Gravity

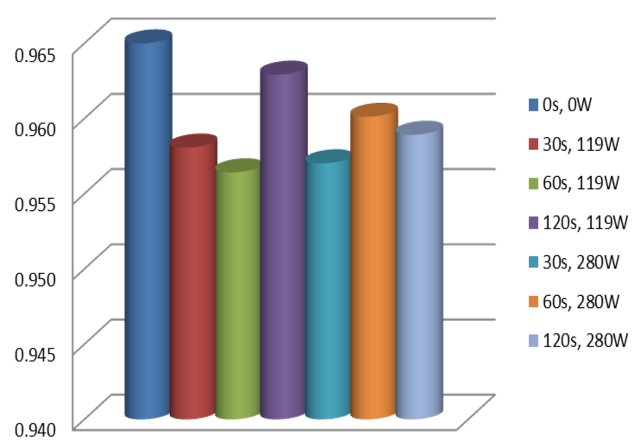

Fig. 3 Variation of oil specific gravity with microwave setting

The specific gravity is expected to be in the range of 0.950 – 0.965 at 25^{o}C [13]. This characteristic relates the ratio of the weight of the oil to the weight of an equal volume of water. In this work, the specific gravity was found to be in the range of 0.956 – 0.965 at 25^{o}C. Microwave pretreatment has an insignificant effect on specific gravity.

E. Free Fatty Acids and Acid Value

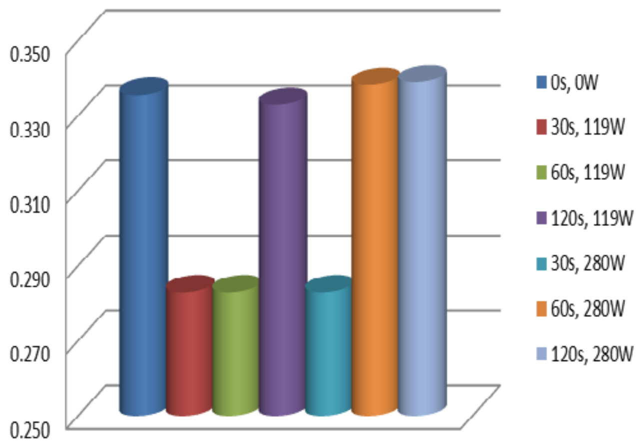

Fig. 4 Variation of free fatty acid content with microwave setting

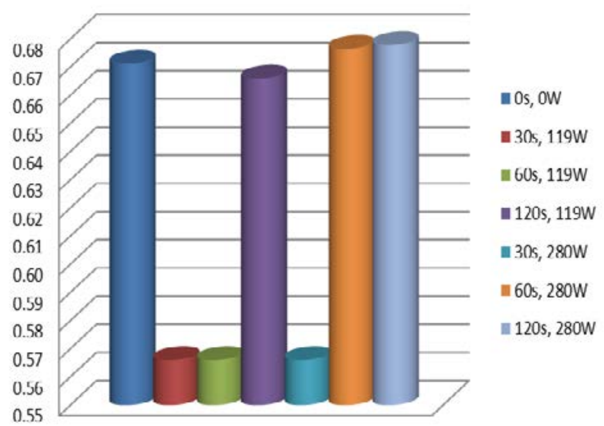

Fig. 5 Variation of acid values with microwave setting

The American Society for Testing and Materials (ASTM) specifications for castor oil are free fatty acids (FFA) in the range of 0.2 – 2.0 [13]. The FFA, as seen in Fig. 4, was found to be in the range of 0.283 – 0.339, which is acceptable. Microwave treatment at 280W has a pronounced effect on the FFA content as it increased from 0.336 for the untreated sample to 0.339 for that treated at 280W for 120 sec. The acid values for castor oil are in the range of 0.4 – 4.0 [13], and this work has shown acid values in the range of 0.57 – 0.68, Fig.5. There was a small increase in acid value as a result of microwave treatment and this may be due to the hydrolysis of triacylglycerol treatment producing fatty acids [17]. Ideally, oil should have a low content of free fatty acids as they affect the shelf-life. Refining may be used to remove these FFAs.

F. pH

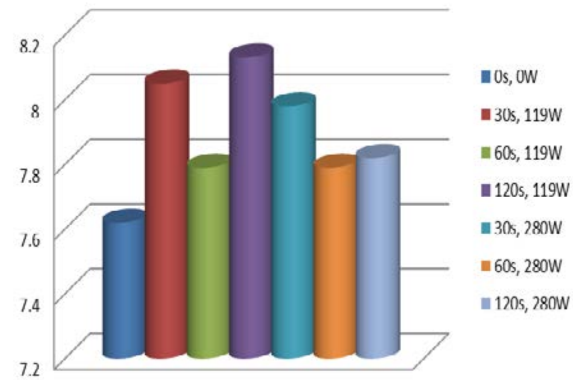

Fig. 6 Castor oil pH variation with microwave setting

After removing free fatty acids, the oil pH ranged from 7.62 to 8.13.

G. Oil Colour

Fig. **7** Oil colour variation with microwave setting

Fig. 7 shows castor oil colour variation with microwave setting. The settings were as follows: sample 1 at 0W, 0s; sample 2 at 191W, 30s; sample 3 at 280W, 120s; sample 4 at 191W, 120s; sample 5 at 280W, 30s; sample 6 at 280W, 60s and sample 7 at 191W, 60s. The neutralization process reduced the FFA content of crude oil from 19% to 1% resulting in the desired pale oil colour shown in Fig. 7, sample 3 and a refractive index of 1.4718. The pale yellow colour can also be attributed to the low acid value.

TABLE III
ASTM CASTOR OIL QUALITY SPEECIFICATION

Moisture content	3.16-3.72	07-May
Refractive Index	1.475-1.400	1.471-1.472
Specific gravity @25^{0}C	0.950-0.965	0.956-0.965
FFA	0.2-2.0	0.283-0.339
Acid value	0.4-4.0	0.57-0.58

IV. Conclusion

This work has shown that microwave pretreatment of castor beans resulted in better oil recoveries with the highest yield of 44.34% at the treatment of 280W for 120 seconds. The values for moisture content, refractive index, specific gravity, free fatty acids and acid values were found to be within the standard specified by the American Society for Testing and Materials.

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