

The effect of organic solvent, temperature and mixing time on the production of oil from *Moringa oleifera* seeds

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Abstract. The effect of three different organic solvent, temperature and mixing time on the production of oil from *M.oleifera* seeds were studied to evaluate the effectiveness in obtaining the high oil yield based on the percentage of oil production. The modified version of Soxhlet extraction method was carried out to extract the oil from *M.oleifera* seeds by using hexane, heptane and ethanol as the organic solvent. Among the three solvents, it is found that heptane yield higher oil from *M.oleifera* seeds with maximum oil yield of 36.37% was obtained followed by hexane and ethanol with 33.89% and 18.46%, respectively. By using heptane as a solvent, the temperature (60°C, 70°C, 80°C) and mixing time (6 h, 7 h, and 8 h) were investigated to ensure the high oil yield over the experimental ranges employed and high oil yield was obtained at 60°C for 6 h with percentage oil yield of 36.37%. The fatty acid compositions of *M.oleifera* seeds oil were analyzed using Gas Chromatography/Mass Spectrometry (GC-MS). The main components of fatty acid contained in the oil extracted from *M.oleifera* seeds was oleic acid, followed by palmitic acid and arachidic acid, and small amount of behenic acid and margaric acid.

1. Introduction

Moringa oleifera tree origin is from North West India but nowadays it has been found all around the world and is naturalized in many locales. This might be because it possesses the special ability to endure in harsh environment and it grows in many different soil types as well as it is drought resistance [1]. The tree carries some physical characteristics such as it is filled with long and delicate-looking branches, which are covered with small and oval-shaped dark green leaves [1]. The height of the tree can reach up to 10 m naturally but for cultivation purposes, the tree often being cut back each year into 6 to 12 feet maximum so that the seeds and leaves can be harvested easily [2].

The most utilized part of the *M.oleifera* tree is the seeds which are used in the production of oil. The oil production from *M.oleifera* seeds are important because it have many advantages over other organic sources for example, it can be used to reduce blood pressure and lower glucose level, skin preparation and ointment, perfumes and also for cooking [3,4]. It also possesses the antimicrobial activity to effectively

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fight against common bacteria such as *Escherichia coli* and *Staphylococcus aureus* [5].

After the *M.oleifera* seeds are extracted for its oil, the seed cake can be used for other applications. According to a study made by Ali *et al.* (2010), *M.oleifera* seed cake can be used in water treatment as it acts as a natural coagulant for use in drinking water treatment [6]. Results showed that the turbidity removal using *M.oleifera* oil as the natural coagulant can be reached up to 96.23%. Emmanuel *et al.* (2011) proposed that *M.oleifera* seed cake can be added to the fertilizer mix as a decent natural, ecofriendly and biocompatible organic fertilizer [7]. The experimental results indicate the mineral content of the soil have increased when the *M.oleifera* seed cake mixed with the fertilizer which in turn improved the production of the harvest.

Oil can be extracted from *M.oleifera* seeds by traditional method. The seeds from mature pods are roasted, crushed and placed in boiling water for 5 minutes. After straining and left for overnight, the *M.oleifera* oil floats to the surface [3]. However carrying out this traditional method is not efficient as it is highly energy intensive and time consuming and only can produce low percentage of oil yield. Therefore, the researchers come up with different methods to maximize the oil recovery from *M.oleifera* seeds. Various methods have been used from the previous researchers to extract the oil from *M.oleifera* seeds such as cold press method [8], supercritical carbon dioxide extraction [9], aqueous enzymatic method [10] and solvent extraction method [1].

In this investigation, oil extraction using the method of solvent extraction was carried out using hexane, heptane and ethanol as the solvent. Solvent extraction has been reported by Mani *et al.* (2007) using hexane, petroleum ether and acetone [11]. Experimental Soxhlet method using hexane as the solvent, and also the effect of heat treatment on yield of oil expressed from *M.oleifera* seeds have been reported by Adejumo *et al.* (2013) [1]. However, there are limited data on the processing factors needed for the ideal extraction of *M.oleifera* oil especially for the type of solvent used. There are many factors influencing the oil extraction yield such as the method of extraction, temperature of extraction, seed particle size, ratio of solvent to the *M.oleifera* seed powder, pre-treatment conditions and residence time between the solvent and the seed [11]. However, this research are only focussing on the organic solvent, temperature of extraction and contact time of the solvent with the *M.oleifera* seeds to increase the oil extraction yield. Therefore, the main objective of this research is to evaluate the suitable set of parameters in obtaining the highest oil recovery within the studied range from *M.oleifera* seeds.

2. Material and methods

2.1 Sample preparation

The dry *Moringa oleifera* seeds were purchased from Mitomasa Sdn. Bhd. The wings and coats of *M.oleifera* seed were removed manually. Only the good quality or undamaged seeds were selected to be used in the experiment. Solid to solvent ratio was 1:10, therefore 10g of the *M.oleifera* seeds were ground to a fine powder using an electrical grinder and hand-operated mortar.

2.2 Organic solvents for oil extraction

Hexane, heptane and ethanol (99% of purity) were used as organic solvents to leach out oils from the insoluble solid structure of the *M.oleifera* seeds. During the selection of organic solvents, the solid to solvent ratio was maintained as 1:10 for the oil extraction [12]. Temperature and mixing time were kept constant at 60°C and 6 hours respectively. The mixing rate was adjusted at 400 rpm for the whole experiment.

2.3 Oil extraction using solvent extraction method

The modified version of Soxhlet extraction method was used for oil extraction process. Extraction set up mainly consists of a condenser, retort stand, hot plate stirrer, 1 L beaker and 250 ml round bottom flask. The *M.oleifera* powder was placed in the round bottom flask which connected to the condenser. The ratio of solid to solvent that was used in this experiment was 1:10 [12], thus 100 ml of solvent was added round bottom flask containing 10 g of *M.oleifera* powder. A magnetic stirrer in the round bottom flask was used to mix up the powder with the solvent thoroughly. During this process, the round bottom flask was heated in the beaker filled with water and the solvent was evaporated and moved to the condenser where the steam was converted into liquids which then drip back into the round bottom flask. The solvent-oil mixture in the round bottom flask was collected at the end of the extraction process. Figure 1 shows two sets of Soxhlet apparatus used for solvent extraction.

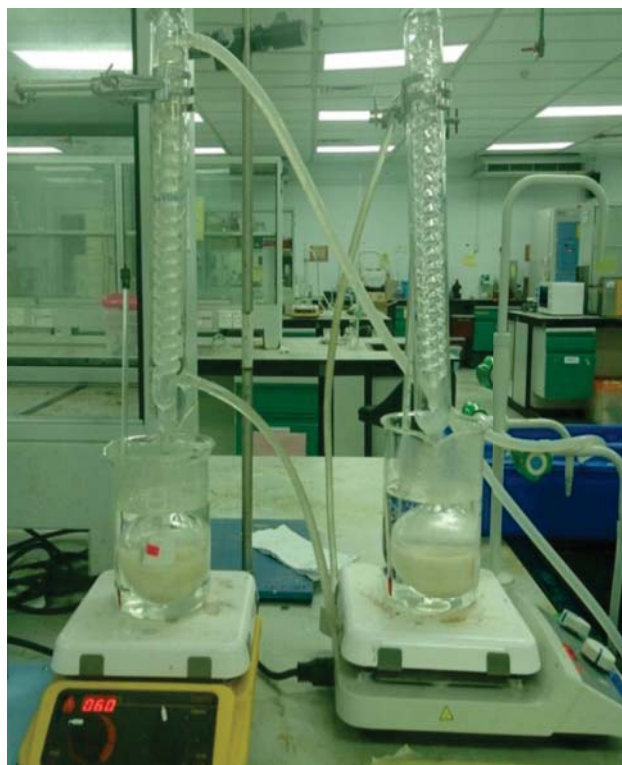


Figure 1. Modified Soxhlet extraction method for solvent extraction process

The flask containing a mixture of *M.oleifera* powder, solvent and oil were separated at the end of the extraction process. A filter paper was used to separate the powder from the solvent-oil mixture before it was dried in the oven overnight at 60°C. Then, the powder was being weighted for the determination of the percentage of oil yield. The oil was recovered by evaporating off the solvent using rotary evaporator where it was heated at the temperature higher than boiling point of the solvent until the solvent finally evaporated and leaving behind the extracted oil. All experiments were conducted in triplicate.

2.4 Extraction parameters to enhance the oil yield

The best condition of extraction parameters such as temperature (60°C, 70°C and 80°C) and mixing time (6, 7 and 8 hours) were evaluated to increase the oil recovery from *M.oleifera* seeds using the best solvent from previous result with the highest percentage of oil yield. The details of the experiment are described in table 1.

Table 1. Variables of the different parameters in the oil extraction

Experiment	Constant variable	Manipulated Variable
1	Temperature and mixing time	Solvents
2	The best solvent and mixing time	Temperature
3	The best solvent and temperature	Mixing time

2.5 Determination of oil yield percentage

The percentage of oil yield was calculated using following formula:

$$X = \frac{\text{Weight of oil extracted (g)}}{\text{Weight of } M.oleifera \text{ powder before extraction (g)}} \times 100\% \quad (1)$$

where X indicates the percentage of oil yield (%) and weight of oil extracted from *M.oleifera* seed can be calculated by subtracting the weight of *M.oleifera* powder before and after the extraction [1].

2.6 Gas chromatography analysis

The fatty acid profiles of *M.oleifera* oil were analyzed using Gas Chromatography/Mass Spectrometry (GC-MS) after each extraction using Agilent Technologies (G3171A, China). The samples were injected into capillary column HP- 5 (30 m length, 0.25 mm ID). Helium is used as the carrier gas with a flow rate of 1 ml/min. The inlet temperature and the detector temperature were kept constant at 250°C and 280°C respectively with temperature programming of 100°C for 3 minutes with increase at 10°C per minutes up to injector temperature 250°C [13].

3. Results and discussion

3.1 The effect of organic solvent

The *M.oleifera* seeds were extracted with different solvents which are hexane, heptane and ethanol at constant temperature of 60°C for 6 hours to evaluate the effectiveness of the solvents in producing the maximum oil yield from *M.oleifera* seeds. The result of the effect of organic solvent on the oil yield of *M.oleifera* seeds were compared with the other researches [1,9,12] as presented in table 2. As can be noticed in table 2, heptane showed a better solvent compared to hexane and ethanol. According to Conkerton *et al.* (1995), the yield and quality of the oil extracted by heptane were similar to that extracted by hexane [12]. This is might be due to the fact that heptane and hexane are both non-polar solvent while ethanol is a polar solvent. Ethanol yields about 20% lower than heptane and hexane. This result is similar to the research conducted by Nwabueze & Okocha (2008) [14]. They also find that the non-polar solvent is better solvent for extracting oil from African breadfruit (*Treculia africana*) seed oil.

Table 2. The effect of organic solvents on the oil yield of *M.oleifera* seeds

Organic Solvents	Experimental data (%)	Adejumo <i>et al.</i> (2013) ^a	Palafox <i>et al.</i> (2012) ^b	Conkerton <i>et al.</i> (1995) ^c
Heptane	36.26±2.48	-	-	30.4±0.19
Hexane	35.58±10.08	33.7	29	28.4 ± 0.36
Ethanol	16.94±3.06	-	35	-

^a Heated at 100°C for 6 hours.^b Heated at 60°C for 6 hours.^c Heated at 88-95°C for 4 hours.

3.2 The effect of temperature

Figure 2 shows the effect of temperature by using heptane as the solvent. The result shows that the *M.oleifera* seeds heated at 60°C have the highest percentage yield of 36.3% while the lowest oil yield of *M.oleifera* is at 80°C. The results also showed that there is a reduction in percentage oil yield when the seed is heated above 60°C. This finding was also found in the study reported by Adejumo *et al.* (2013) where the oil yield from *M.oleifera* seed decreased when the temperature increased from 100°C until 150°C [1]. Mani *et al.* (2007) also reported the same finding when hexane is used as the solvent for extracting *M.oleifera* oils at temperature range of 40°C until 80°C. The study also revealed that the oil yield was constant up to 60°C but slowly decreased as temperature increases [11].

3.3 The effect of mixing time

Figure 3 demonstrated the effect of mixing time on the oil yield of *M.oleifera* seeds. The results revealed that 6 hours of extraction resulted in the highest oil yield for *M.oleifera* seeds which is 36.3% of oil yield. This observation shows that the longer the mixing time, the lower the oil yields that can be extracted from *M.oleifera* seeds. This result agrees to the finding reported by Mani *et al.* (2007) that any further increase passes 6 hours in extraction time did not increase the oil yield of *M.oleifera* seeds [11]. This phenomenon is due to low solvent density contained left in the sample after 6 hours [15]. However, the oil yield increased dramatically when the *M.oleifera* seeds were heated from 1 hour until 6 hours because the diffusivities of the oil and solvent increases, which result in high oil yield [11]. Therefore, the maximum oil yield could be achieved even at shorter residence time with an optimal extraction temperature.

Oil yield comparison of the extracted seeds is presented in table 3. It is possible to observe that *Jatropha curcas* has a great ability to extract oil up to 86.1% compared to *M.oleifera*, sunflower and soybean. The ranges of processing parameters investigated by the authors such as applied pressure, moisture contain, preheating time, shell removal and particle size explains the higher yield obtained from *Jatropha curcas*. As can be noticed in table 3, the percentage of oil yield is lower when ethanol used as a solvent even for different temperature and mixing time for sunflower. This observation is supported by the fact that the lower selectivity of ethanol towards oil makes this solvent difficult to extract the oil as much as possible from the seeds [16].

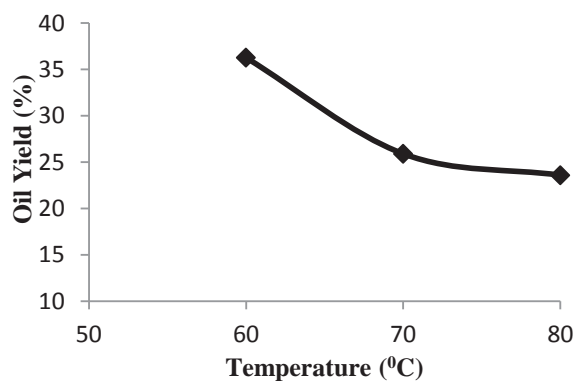


Figure 2. The effect of temperature on the oil yield of *Moringa oleifera*

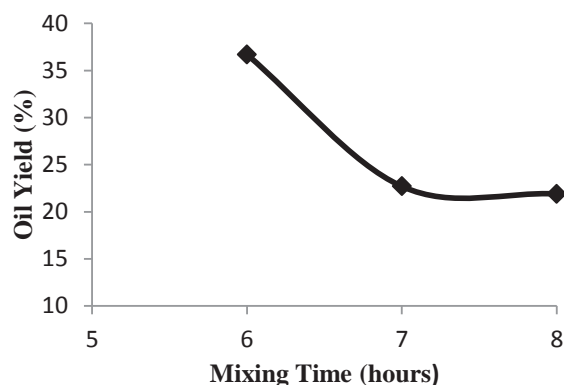


Figure 3. The effect of mixing time on the oil yield of *Moringa oleifera*

Table 3. Comparison of oil yield with the other seeds obtained by Soxhlet.

Seed	Solvents	Temperature (°C)	Mixing time (hour)	Oil yield (%)	Ref.
<i>M.oleifera</i>	Heptane	60	6	36.26±2.48	
<i>M.oleifera</i>	Hexane	60	6	35.58±10.08	
<i>M.oleifera</i>	Ethanol	60	6	16.94±3.06	
Sunflower	Ethanol	60	16	24.5±0.1	[16]
Sunflower	Hexane	130	4	46.2±0.7	[17]
<i>Jatropha curcas</i>	Hexane	60	24	86.1	[18]
Soybean	Ethanol	55	3	25	[19]
Soybean	Ethanol	60	3	25.6±0.2	[20]

3.3 Fatty acid composition of extracted oil

The fatty acid compositions for *M.oleifera* were analyzed by GC-MS using Agilent Technologies Chromatograph. In this present study, the major component of *M.oleifera* oil was oleic acid (C18:1), followed by palmitic acid (C16:0) and arachidic acid (C20:0) and a small amount of behenic acid (C22:0) and margaric acid (C17:0). The result also shows a higher oleic acid composition compared to the values reported by Gaikwad *et al.* (2011) and Abdulkarim *et al.* (2005) [10,13]. According to Adejumo *et al.* (2013) and Ogunsina *et al.* (2014), the high oleic acid content indicates the oil has an excellent quality similar to olive oil which also provides good stability and good thermal stability to *M.oleifera* seed oil [1,21].

4. Conclusion

Extraction of oil from *M.oleifera* seeds by using heptane as the organic solvent showed better results when compared with other solvents studied. The temperature of 60⁰C for 6 hours of extraction time gave a maximum oil yield percentage which is 36.37%. In this study, the results also showed the higher percentage of oleic acid oil when compared to previous researches. In addition, based on the fatty acid composition, *M.oleifera* seeds can extract the good quality of oil for further applications.

5. References

- [1] Adejumo B A, Alakowe A T and Obi D E 2013 *Int. J. Eng. Sci.* **2**(1) 232
- [2] *Moringa Oleifera* 2014 In *I Love Moringa: The Fast-growing, nutritious, delicious gift from god.* <http://www.ilovemoringa.com/MoringaOleifera.html>
- [3] Price M L 2007 *The Moringa Tree* (North Fort Myers: ECHO Staff).
- [4] Moringasource 2013 *Moringa oleifera - one of nature's best-kept health and nutrition secrets.* <http://www.moringasource.com/>
- [5] Lalas S, Gortzi O, Athanasiadis V, Tsaknis J and Chinou I 2012 *Molecules* **17**(3) 2330
- [6] Ali E N, Muyibi S A, Salleh H M, Alam M Z and M. Salleh M 2010 *Water Resour. Prot.* **2**(3) 259
- [7] Emmanuel S, Zaku S, Adedirin S, Tafida M and Thomas S 2011 *Agric. Biol. J. North Am.* **2**(9) 1289
- [8] Lalas S and Tsaknis J 2002 *J. Food Compos. Anal.* **15**(1) 65
- [9] Palafox J O, Navarrete A, Sacramento-Rivero J C, Rubio-Atoche C, Escoffie P A and Rocha-Urbe J A 2012 *Am. J. Anal. Chem.* **3** 946
- [10] Abdulkarim S, Long K, Lai O, Muhammad S and Ghazali H 2005 *Food chem.* **93** 253
- [11] Mani S, Jaya S and Vadivambal R 2007 *Food Bioprod. Process.* **85** 328
- [12] Conkerton E, Wan P and Richard O 1995 *J. Am. Oil Chem. Soc.* **72**(8) 963
- [13] Gaikwad M, Kale S, Bhandare S, Urunkar V and Rajmane A 2011 *Int. J. PharmTech Res.* **3**(3) 1567
- [14] Nwabueze T and Okocha K S 2008 *Afr. J. Food Sci.* **2**(10) 119
- [15] Treybal R E 1980 *Mass Transfer Operations* (New York, USA: McGraw Hill).
- [16] Baumler E R, Carrin M E and Carelli A A 2016 *J. Food Eng.* Article in Press.
- [17] Ravber M, Knez Z and Skerget M 2015 *Food Chem.* **166** 316
- [18] Subroto E, Manurung R, Heeres H J and Broekhuis A A 2015 *Ind. Crops Prod.* **63** 303
- [19] Dagostin J L A, Carpine D and Corazza M L 2015 *Ind. Crops Prod.* **74** 69
- [20] Toda T A, Sawada M M and Rodrigues C E C 2016 *Food Bioprod. Process* **98** 1
- [21] Ogunsina B S, Indira T, Bhatnagar A, Radha C, Debnath S and Gropala Krishna A 2014 *J. Food Sci. Technol.* **51**(3) 503

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