EXTRACTION OF *MORINGA* SEED OIL: KINETICS AND THERMODYNAMIC STUDIES

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Abstract: Extraction of oil from moringa seed was carried out in this work and the kinetic and thermodynamic parameters of the extraction process determined. The proximate analysis of moringa seed revealed that the moisture, ash, crude protein, crude lipid, carbohydrate, and crude fibre contents were 0.7, 1.4, 2.9, 39.6, 53.6, and 1.8%, respectively. The optimum parameters for the oil extraction from a 500 microns particle size were found to be 17.5 ml/g volume of solvent to mass ratio, extraction temperature and time were 343.15 K and 180 minutes respectively. The extraction kinetics was found to obey the fifth order with an activation energy, E_a, of 41.11 kJ/mol. The thermodynamic parameters at 343.15 K were found to be $\Delta H = 26.56\ \text{kJ/mol}$, $\Delta S = 0.1070 \frac{\text{kJ}}{\text{molK}}$ and $\Delta G = -10.14 \frac{\text{kJ}}{\text{molK}}$, respectively. Saponification value, specific gravity, iodine value, pH and a free fatty acid value of the extracted oil were 165.33, 0.8696, 66.86 g/mol, 6.1 and 8.26 mgKOH/g, respectively. Moringa seed is a great source of carbohydrate and contains a significant amount of oil.

Keywords: Activation energy, enthalpy, kinetics, *Moringa* oil, solvent extraction

Introduction

Moringa oleifera popularly called "Zogale" in the northern part of Nigeria is the most widely cultivated variety of the genus Moringa and belong to the family of Moringaceae. *Moringa oleifera* is a nutritious vegetable tree with a variety of potential uses. All parts of the *moringa oleifera* plant are used in a variety of traditional medicines (Anwar *et al*, 2007; Leone *et al*., 2015). The cake, obtained from oil extraction, is useful as a soil conditioner, or as a flocculent to purify water (Lea, 2010; Leone *et al*., 2016). The plants are grown as live fences and windbreaks (Assefa *et al.*, 2015). It is also used as fuelwood source after coppicing (cutting back the main stem to encourage side shoots), as an intercrop with other crops and the wood pulp may be used for paper-making. The green pods, fresh and dried leaves are used as a vegetable (Monica and Sharma, 2013).

Moringa oleifera seed has been reported to contain oil as its main component and represents between 35 - 48% of the seed weight (Tsaknis *et al.*, 1998; Onyekwelu and Olabiwonnu, 2010; Abdulkareem *et al*, 2011) and it is considered as a specie with great bioenergy potentials due to its high carbohydrate content (Bispo dos Santos *et al.*, 2015).

Extraction using solvents is generally conducted at a temperature near the boiling point of the solvent, which reduces the oil viscosity and improves its solubility in the solvent, thereby, ensuring the efficiency of the process (Johnson and Lusas, 1983; Ghandi *et al*., 2003; Lalas and Tsaknis, 2002). According to Saxena *et al*. (2011), n-Hexane is the most widely used solvent in the extraction of oil contained in seeds because of its low boiling point, extraction efficiency and ease of availability. Liauw *et al.* (2008) studied the extraction of neem oil using n-hexane and ethanol. They observed that more yield of oil was obtained with the use of n-hexane than with ethanol and the extraction process followed first order kinetics. The thermodynamic parameters showed that the process of extraction of oil from neem seeds is endothermic, irreversible and spontaneous. Also, Nwabanne (2012) in the study of the kinetics and thermodynamics of oil extraction from fluted pumpkin seed, observed that oil yield increased with increase in temperature, time and volume of solvent but decreased with increase in particle size. The kinetics of the process indicated a fourth order kinetic and thermodynamic parameters showed that the reaction is endothermic. Ibemesi and Attah (1990), extracted oil from the seeds of rubber and melon using different solvents at various temperatures. They reported that the oil yield increased by a factor of about 1.10 for every 10^0 rise in temperature and the enthalpy changes accompanying the extraction process were in the range of 4 – 13.5 kJ/mol.

Literature abound with studies on the kinetics and thermodynamics of oil extraction from different oilseeds such as Sunflower seeds (Topallar and Gecgel, 2000), *Jatropha curcas* seeds (Bispos dos Santos *et al*., 2015), Fluted pumpkin seeds (Nwabanne, 2012), *Daturametel Linn* seeds (Ramasamy and Nagarajan, 2016), Water-melon seeds (Olakunle *et al*. 2014), Castor seed cake (Amarante *et al*., 2014), *Thevetia peruviana* seeds (Jabar *et al*., 2015), with scanty information on the thermodynamics of the extraction of oil from moringa seed (Basuny and Al-marzouq, 2016; Premi and Sharma, 2013). The present study is therefore focused on the kinetics and thermodynamics of the extraction of oil from moringa seed. The kinetics of oil extraction is dependent on a number of factors like temperature, time of extraction and the polarity of the solvent used. Results obtained from this study will be helpful in maximizing oil yield, reducing the cost of extraction, reducing the amount of energy consumed over time and provide useful data for plant design for the extraction of oil from moringa seed.

Materials and Methods

Materials preparation and proximate analysis

The moringa seeds were procured from a local market in Sabon-Gari Local Government Area of Kaduna State on coordinates $11^0$9'48"N and $7^0$39'2"E. The proximate analysis of the seed was carried out according to the procedure of Association of Official Analytical Chemist (AOAC, 2006) to determine the moisture content, crude fibre, protein, crude fat, and carbohydrate content of moringa seed. The seeds were washed thoroughly with water to remove dirt and impurities, then dried in the oven at 60^0C until it reached constant moisture content. The seeds were ground and sieved through three different laboratory sieves to obtain particle sizes of 500, 1180 and 1700 microns, respectively, which were used for the extraction.

Oil extraction of moringa seeds

Effect of particle size on the process

20 g of the 500 microns ground moringa seed was weighed and inserted into a thimble fitted in the Soxhlet apparatus. 200 ml of the solvent (n-hexane) was heated at 40^0C in the round-bottom flask of the Soxhlet apparatus to effect the extraction of oil from the ground seeds. After the extraction with n-hexane, the set up (soxhlet extractor) was dismantled and the

miscella (a mixture of n-hexane and moringa seed oil) obtained was poured into the distillation flask and placed on the heating mantle. The heating mantle was set at 68^0C which is the boiling point of pure hexane. After distillation, the weight of the oil was determined. The same procedure was repeated for the 1180 and 1700 microns, respectively and the particle size which gave the highest oil yield was recorded as the optimum particle size.

Effect of solvent volume on the process

20 g of the optimum particle size ground moringa seed was weighed and inserted into a thimble fitted in the Soxhlet apparatus. 200 ml of the solvent was heated at 40^0C in the round bottom flask of the Soxhlet apparatus to effect the extraction of oil. After the extraction, the miscella obtained was poured into the distillation flask and heated in the heating mantle at 68^0C. The weight of the oil was determined after distillation. The same procedure was repeated using the solvent volume of 250, 300, 350 and 400 ml, respectively. The solvent volume with the highest oil yield was recorded as the optimum solvent volume.

Effect of time on the process

20 g of the optimum particle size ground moringa seed was weighed and inserted into the thimble fitted in the Soxhlet apparatus. The optimum solvent volume was measured and heated at 40^0C in the round bottom flask for 60 min to effect the extraction of the oil. After the extraction, the miscella obtained was poured into the distillation flask and heated in the heating mantle at 68^0C. The weight of the oil was determined after distillation. The same procedure was repeated using the extraction time of 90, 120, 150 and 180 min, respectively. The time which yielded the highest oil was recorded as optimum time.

Effect of temperature on the process

20 g of the optimum size ground moringa seed was weighed and inserted into the thimble fitted in the Soxhlet apparatus. The optimum solvent volume was measured and heated at 40^0C in the round bottom flask at the optimum time obtained to effect the extraction of the oil. After the extraction, the miscella obtained was poured into the distillation flask and heated in the heating mantle at 68^0C. The weight of the oil was determined after distillation. The same procedure was repeated using the extraction temperature of 50, 60, 70 and 80^0C, respectively.

Characterization of the extracted oil

Samples of the extracted oil were then characterized to determine its pH, saponification value, iodine value, free fatty acid value and the specific gravity.

Results and Discussion

Table 1 shows values obtained from the proximate analysis of moringa seed. It could be observed that moringa is a great source of carbohydrate and has a significant amount of oil. The low moisture content indicates that the activities of microorganisms on moringa seed would be reduced, thereby increasing the shelf life of moringa seed sample. The crude fibre content of moringa seed which was found to be 1.8% agrees with values obtained by Orhevba *et al*. (2013). Crude fibre content helps in bowel movement. Adequate intake of dietary fibre can lower cholesterol level and risk of hypertension (Ishida *et al*., 2000).

The effect of particle size on moringa seed oil yield is shown in Fig. 1. It could be observed that the oil yield increased from 10.5 to 14.5% as the particle size decreased from 1700 to 500 microns. A similar result was obtained by Nwabanne, (2012) while extracting oil from fluted pumpkin seed. He found that fluted pumpkin seed oil yield increased from 20 to 41% as particle size decreased from 7 to 2 mm. This observation is in agreement with theory and literature. According to Ebewele *et al*. (2010), the negative effect of particle size on oil yield could be attributed to the fact that smaller particles have a larger amount of surface area coupled with an increased number of ruptured cells resulting in a high oil concentration at the particle surface and little diffusion into the particles surface. Also, Sayyar *et al*. (2009) while extracting oil from jatropha seed suggested that large particles have a smaller amount of surface areas and are more resistant to the intrusion of solvent and oil diffusion. Therefore, a small amount of oil will be carried from inside the large particles to the surrounding solution.

Table 1: Proximate analysis of moringa seed

Proximate properties	Value (%)
Moisture content	0.7 ± 0.08
Ash content	1.4 ± 0.25
Crude lipid	39.6 ± 2.45
Crude protein	2.9 ± 0.35
Crude Fibre	1.8 ± 0.13
Carbohydrates	53.6 ± 3.15

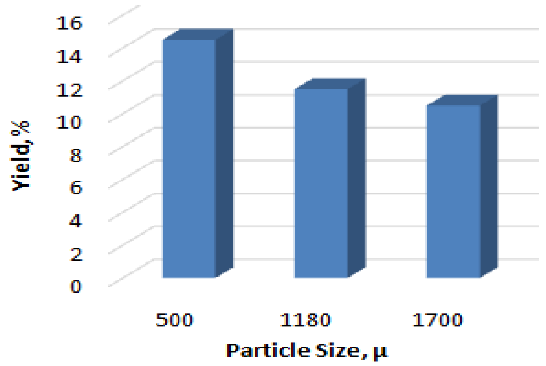

Fig. 1: Effect of particle size on moringa seed oil yield

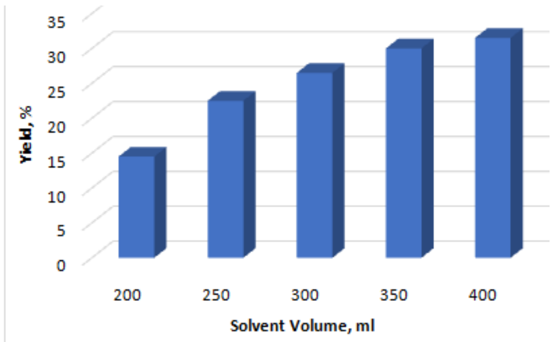

Fig. 2: Effect of solvent volume on moringa seed oil yield

Figure 2 shows the effect of solvent volume on moringa seed oil yield. It could be observed that oil yield increased from 13.5 to 31.5% as solvent volume was increased from 200 ml to 400 ml. The positive effect of solvent volume on oil yield was as a result of an increase in the concentration driving force as the volume of solvent increases.

It was also a result of increased washing of the oil extracted, away from the particle surface by the solvent as a result of increased volume (Meziance *et al.*, 2006). It is also observed that the increase in oil yield became less significant at 350 ml. Thus, 350 ml will be the optimal solvent volume as the higher solvent volume would result in a higher cost of extraction.

Table 2 shows the values of oil yield at different temperatures and time. It can be seen that oil yield increased as temperature increased at different times. The positive effect of temperature on oil yield is as a result of rupturing of oil cell walls which now create a void which serves as a migratory space for contents of the oil-bearing cells (Nwabanne, 2012). Oil yield also increased as time increased from 60 to 180 min. This same trend was reported by Olakunle *et al.* (2014) in their study of kinetic and thermodynamics of oil extraction from watermelon seeds. The highest percentage oil yield from moringa seed is recorded at 180 min and 343.15 K temperature.

The rate for oil extraction process can be written as in Equation 1;

$$\frac{dY_t}{dt} = kY_t^n \text{ ....1}$$

where; Y_t is the per cent oil yield, t is the time of extraction, k is the extraction constant and n is the order of the process.

Taking the natural logarithm as applicable in the differential method of analysis of reaction rate (Levenspiel,1999), Equation 1 leads to Equation 2;

$$ln\frac{dY_t}{dt} = lnk + nlnY_t \text{...........2}$$

A plot of $ln\frac{dY_t}{dt}$ against lnY_t for each temperature value as shown in Fig. 3, gives the values of the n, the order of the process, as the slope and lnk as intercept from which the values of k, the rate constant can be evaluated.

Table 3 gives the order, the rate constants and the R^2 values of the extraction process at various temperatures as determined from Fig. 3.

Table 2: Oil yield at various extraction temperature and time (Particle size = 500-micron, Solvent volume = 350 ml)

Time	Oil yield (%)			
(min)	313.15 K	323.15 K	333.15 K	343.15 K
60	30.0	30.0	31.5	32.5
90	31.0	31.5	32.0	33.0
120	32.5	33.0	33.5	34.5
150	34.5	35.0	35.5	36.5
180	37.0	37.5	38.0	38.5

Table 3: Process order and rate constants for moringa seed oil extraction at various temperatures

T (K)	N	k $(dm^3/mol)^4S^{-1}$	R^2
313.15	5.0484	1.07×10^{-9}	0.9517
323.15	4.8959	1.73×10^{-9}	0.9634
333.15	4.8192	2.1×10^{-9}	0.9712
343.15	4.5430	4.66×10^{-9}	0.8677

From Table 3, it is observed that the rate constant at 343.15 K is the highest, this agrees with the trend in literature that increase in temperature leads to increase in percentage oil yield (Ibemesi and Attah, 1990; Olakunle *et al.*, 2014; Nwabanne, 2012). Also, the process of extraction of oil was found to obey fifth order kinetic.

The activation energy of the extraction process can be calculated using the Arrhenius equation;

$$k = Ae^{-E_a/RT} \text{..........................3}$$

Where k is the reaction rate (extraction) constant, A is the Arrhenius constant or frequency factor, E_a is the activation energy, R is universal gas constant, and T, the absolute Temperature.

Taking the natural logarithm of both sides, equation 3 becomes;

$$lnk = lnA - \frac{E_a}{R} \times \frac{1}{T} \text{....................4}$$

A graph of ln k against $\frac{1}{T}$ gives $-\frac{E_a}{R}$ as the slope and ln A as intercept as shown in Fig. 4. From Fig. 4, the activation energy and Arrhenius constant are 41.11 kJ/mol and 0.00725 s^{-1} , respectively. High activation energy lowers the rate of the extraction process while low activation energy increases the rate of the extraction process.

The thermodynamic parameters for moringa seed oil extraction can be determined using the Van't hoff expressions as seen in Equations 5 and 6 (Topallar and Gecgel, 2000).

$$\ln K = \frac{-\Delta G}{R}\frac{1}{T} = -\frac{\Delta H}{R}\frac{1}{T} + \frac{\Delta S}{R} \text{ ....5}$$

and $$K = \frac{Y_T}{Y_U} \text{..............................6}$$

Where K is the equilibrium constant; Y_T is the per cent oil yield at temperature T; Y_U is the per cent unextracted oil; ΔH is the enthalpy change; ΔS is the entropy change, and ΔG is the free energy or Gibb's energy.

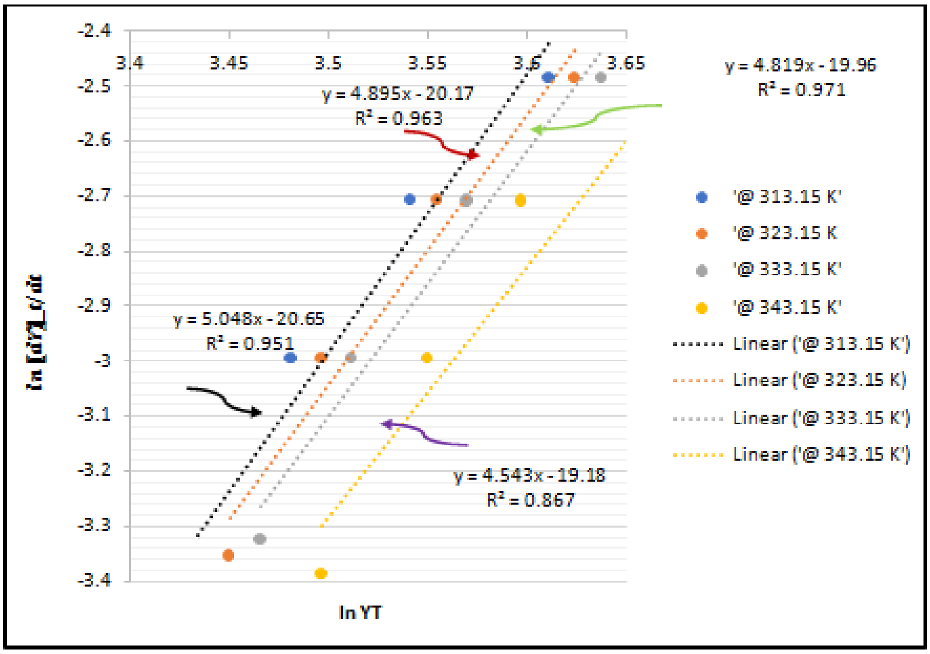

Fig. 3: $ln\frac{dY_t}{dt}$ **against** lnY_t

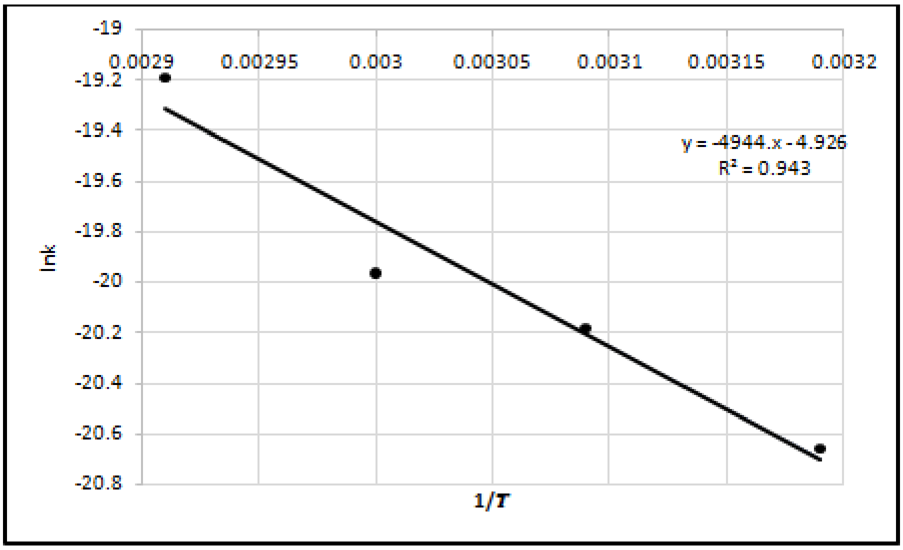

Fig. 4: Graph of lnk against 1/*T*

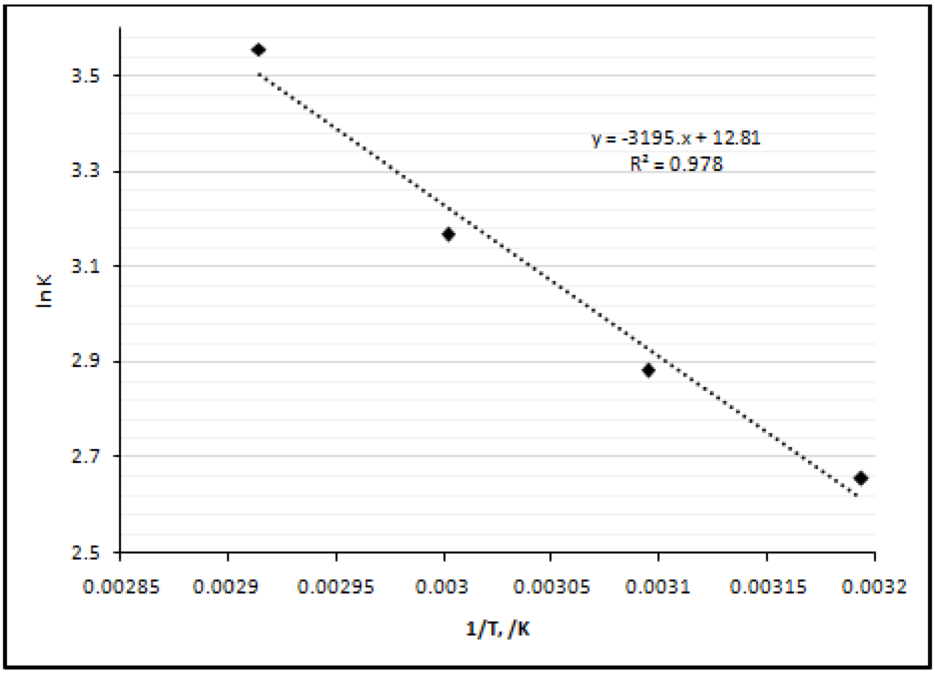

Fig. 5: Graph of ln K against 1/T

A graph of ln K against 1/T yields $-\frac{\Delta H}{R}$ as the slope and $\frac{\Delta S}{R}$ as intercept as shown in Fig. 5. From Fig. 5 slope of the graph is -3195.3, and the value of ΔH was calculated to be $26.56\ kJ/mol$ which is higher than the range of 4 – 13.5 kJ/mol reported by Ibemeshi and Attah (1990) for rubber and melon seeds using different solvents at various temperatures. Also, Topallar and Gecgel (2000) reported an enthalpy value of 11.5 kJ/mol for oil extraction from sunflower seed. Also, the positive value of enthalpy change indicates that the reaction is endothermic and required energy during the process. The higher enthalpy value reported in this work did indicate a higher energy requirement for the oil extraction from the seeds of *Moringa oleifera*. This could be justified also by the fact that longer duration gave a higher yield of the oil.

From the results obtained in Table 4, it could be observed that the values of entropy change are greater than zero while values of free energy or Gibb's energy are less than zero. These negative values indicate the feasibility and spontaneous nature of the process.

Table 4: Equilibrium constant (K), entropy change (ΔS) and free energy (ΔG) at various temperature

T (K)	K	$\Delta S\ (kJ/mol)$	$\Delta G\ (kJ/mol)$
313.15	14.23	0.1069	-6.91
323.15	17.86	0.1061	-7.74
333.15	23.75	0.1060	-8.77
343.15	35.00	0.1070	-10.14

Table 5: Physicochemical properties of moringa seed oil

Physicochemical properties	Values
pH	6.10
Saponification Value (mg/g)	165.33
Iodine value (g/mol)	66.86
Free fatty acid (mgKOH/g)	8.26
Specific Gravity	0.8696

Values obtained from the characterisation of the moringa seed oil are shown in Table 5. All values obtained agree with values obtained in the literature by Orhevba *et al*. (2013). Iodine value which is the measure of unsaturation of the oil was found to be 66.86. Higher iodine value indicates higher unsaturation of fats and oils (Adegbe *et al*., 2016).
The saponification value and percentage of free fatty acids were found to be 165.33 and 8.26, respectively. These values also show consistency with literature values.

Conclusion
Proximate analysis showed that moringa seed is a great source of carbohydrates and contains a significant amount of oil. Optimum particle size for oil extraction was found to be 500 microns, optimum solvent volume was 350 ml and optimum time and temperature were 180 mins and 343.15 K, respectively. Moringa seed oil extraction obeys fifth order kinetics. The activation energy E_a was found to be 41.11 KJ/mol and the enthalpy value was $26.54\ kJ/mol$ and other thermodynamic parameters at an optimum temperature of 343.15 K were $\Delta G = -10.14\ kJ/mol$ and $\Delta S = 0.1070$ kJ/mol. All values obtained from the characterisation of moringa seed oil shows some consistency with literature values, and deviations in some cases, indicating the uniqueness of the moringa plant. From the results obtained in the characterisation, it can be seen that moringa seed oil contains high unsaturated to saturated fatty acids due to its high iodine and saponification values and can be an acceptable substituent for high saturated oil such as olive oil in diets. The production of useful oils from the seed could also be of economic importance.

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