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Oil Extraction Optimization and Kinetics from Moringa Oleifera (PKM 1) Seeds

Monica Premi[1]* and H.K. Sharma[2]

*[1]Department of Food Technology, Jaipur National University,
Jagatpura- 302025, Jaipur, Rajasthan, India.
[2]Department of Food Engineering and Technology, Sant Longowal Institute of
Engineering and Technology, (Deemed to be University),
Longowal- 148016, Sangrur, Punjab, India.*

Abstract

Moringa oleifera, seed (PKM 1) was examined for extracted oil recovery under the different designed conditions using Central composite face centered design of Response surface methodology. The kinetics of extraction was investigated and its parameters were determined based on a second order model. The optimum conditions were found at 3.6 h reaction time, temperature, 67.84°C and solvent to solid ratio of 9:1. Among three process variables studied, solvent to solid ratio had the most significant effect on the oil percent followed by extraction temperature and time. The maximum extraction was found to be 33.30%. The characterization revealed that the oil had Iodine value 67.23, saponification value184.65, density 0.907 and refractive index 1.436 as respectively.

Keywords: Optimization, Solid liquid extraction, Kinetics, Characterization, Moringa seed, RSM.

1. Introduction

Moringa oleifera is the most widely cultivated species of the genus *Moringa*, which is the only genus in the family *Moringaceae* (Hsu et al. 2006) and it is considered very useful, as every part of it is used for food or other beneficial applications (Premi et al. 2010). In the tropics, it is used as forage for livestock, and in many countries, Moringa is basically used as a micronutrient powder to treat diseases. (Premi et al. 2012)

Drumstick seeds find traditional use as a folk remedy for the cure of various ailments such as diabetes, asthma, bronchitis, tuberculosis, dysentery. Drumstick seed oil is considered equivalent to olive oil in health benefits (Ramachandran et al. 1980).

Moringa seed oil content and its properties show a wide variation depending mainly on the species and environmental conditions (Ibrahim et al. 1974). The oil yield from the seeds depends on the nature of the solvent and oil, the temperature of extraction, seed particle size, contact (residence) time between the solvent and the seed and pre-treatment conditions (Sayyar et al. 2009). Hexane is often used for vegetable oil extraction mainly due to its efficiency and ease of recovery (Akaranta & Anusiem 1996).

No work has been reported to optimize practical operating conditions of solid/solvent ratio, temperature and time and its kinetics for the extraction of oil from *Moringa* seeds. Therefore, present study was undertaken to optimize process conditions for the maximum recovery of oil from *Moringa* seed and to study the kinetics and mechanism of solid liquid extraction of *Moringa* seeds based on a second order model.

2. Materials and Methods

Moringa oleifera seeds (PKM 1, variety) were procured from local market of Himayatnagar, District: Hyderabad, Andhra Pradesh, India. The chosen seed had an average diameter of 1 cm and weight of 0.3–0.4 g. Seed size 0.63 mm was found to be best for oil extraction from *M. oleifera PKM 1 variety* (Mani et al. 2007). Therefore, the particle size was fixed as 0.63 mm. A lab scale Soxhlet apparatus was used for extraction of oil from *Moringa* seeds. The extracted oil yield was expressed in percentage, which was calculated as weight of oil extracted over weight of the sample taken.

3. Experimental Design

The effect of five main factors which are type of solvent, temperature, solvent to solid ratio, particle size of the meal and reaction time were investigated to optimize the extraction operating conditions for achieving maximum oil percent.

Table 1: Independent variables and their coded and natural levels employed in a central composite face centred design for optimization.

Range **Variables**	**-1**	**0**	**+1**
Solvent: solid	5:1	7:1	9:1
Temperature, °C	50	60	70
Time, hrs	2	4	6

The experimental design for this study was divided into two major parts. Firstly, preliminary experiments and secondly, the optimisation of oil extraction was carried out using RSM and a second order polynomial model was developed. Central Composite Face centred design provides relatively high quality predictions over the entire design space and do not require using points outside the original factor range. The range of the experiments variables used in the coded and uncoded form in this study is given in Table 1.

4. Statistical Analysis and Optimization

The preliminary experimental results were analyzed using system software packages. Multiple linear regression analysis was performed by the software Design-Expert (version 6.0.2, trial version, State-Ease, Minneapolis, MN). The fitness of the polynomial model was expressed by the coefficient of determination R^2, and the statistical significances were evaluated by determining the F-value. The process conditions were optimized by using design expert software.

5. Kinetics of Extraction of Moringa Oleifera Oil

According to the literature (Meziane et al. 2006; Meziane & Kadi 2008), it is typical of a second order process to take place in two subsequent stages. Second order kinetic model is recommended as more suitable in predicting the rate of dissolution for the oil contained in the solid to solution; therefore second-order model was considered and undertaken.

6. Second-order Mechanism Model

Considering a second-order rate law, the rate of dissolution for the oil contained in the solid to solution can be described by the following Eq.:

$$\frac{dC_t}{dt} = k\,(C_s - C_t)^2 \tag{1}$$

Where:
k = The second-order extraction rate constant (L g^{-1} min^{-1})
Cs = The extraction capacity (concentration of oil at saturation in g L^{-1})
Ct = The concentration of oil in the solution at any time (g L^{-1}), t (min)

7. Calculation of Activation Energy

For a second order system, the rate constants increases with temperature and may be described by the Arrhenius law:

$$k = A\,exp\left(\frac{-E}{RT}\right) \tag{2}$$

Where:

k = The extraction rate constant (L g^{-1} min^{-1}); A = The temperature independent factor (Lg^{-1} min^{-1}); E = The activation energy (J moL^{-1}); R = The gas constant (8.314 J mo$L^{-1}K^{-1}$) and T = The absolute suspension temperature (K)

8. Characterization of the Extracted Moringa Oil

The extracted Moringa oil was determined for iodine value, saponifcation value, acidity, peroxide value, density and refractive index as per the standard AOCS methods (AOCS, 1989).

9. Results

9.1 Preliminary Experiments

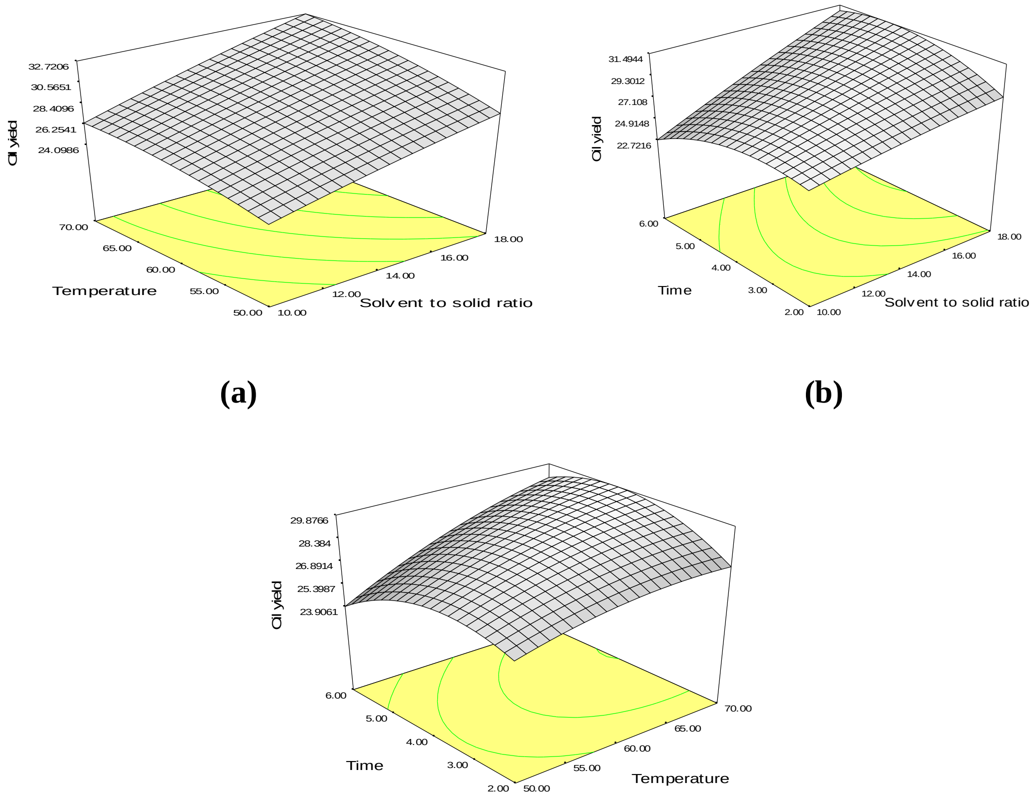
Mani et al. (2007) and Sayyar et al. (2009) found hexane to be the best solvents and seed size 0.63 mm was reported the best for oil extraction from *M. oleifera*, PKM 1. Therefore, all experiments were performed at constant particle size (0.63 mm) by using the hexane as the solvent for oil extraction. Range of extraction was selected on the basis of these preliminary results in order to obtain maximum oil recovery (Table 1).

9.2 Oil Recovery and Adequacy of the Model

The extracted oil from the seed of *Moringa* using hexane was 19.29 and 33.3% under different extraction conditions, solvent to solid ratio, temperature and time (5:1, 50 °C and 6 hrs) and (9:1, 70 °C and 6 hrs) respectively. The variation in oil content of *Moringa* seed may be due to the species, varietal difference and the environmental conditions.

9.3 Effect of Variables on Oil Recovery

Response surface plot of oil percent using hexane for various combinations of solvent to solid ratio, temperature and residence time is shows in Figure 1. Increasing solvent to solid ratio may increase the oil recovery since the concentration gradient between the solid and the liquid phase becomes greater which possibly favours good mass transfer. As extraction temperature increased, the diffusivities of the solvent increased, which resulted in higher oil recovery. By increasing the temperature approaching to the boiling point of the solvent, both the diffusion coefficient and the solubility of the oil in the solvent were possibly enhanced, thus improved the extraction rate. Maximum oil extraction percentage could be achieved even at shorter residence time with an optimal extraction temperature. The combined effect of solvent to solid ratio and extraction temperature on oil percent was higher than that of residence time.



(a) **(b)**

(c)

Figure 1: Response surface plots of percent oil extracted at different conditions.

9.4 Optimization of Processing Parameters for the Maximum Recovery of Oil and Kinetics

The numerical optimization based on the maximum oil recovery was carried out by using design expert software. The maximum recovery of the oil was observed at 9:1, solvent to solid ratio, 67.8 ^{0}C, temperature and 3.6 hrs, time (Table 2). Under the optimum conditions, the oil recovery was predicted as 32.5% with desirability of 98.2%. The optimum conditions, obtained were tried and the experiment was performed to look at the variation. Under the optimum conditions, the oil recovered was 33.3%, slightly higher than the predicted value.

Table 2: Optimal extraction conditions and the predicted and experimental value for oil yield.

Variables	**Optimum Value**	**Responses**	**Experimental Value**	**predicted Value**	**CV %**
Solvent : solid	9:1				
Temperature (□C)	67.84				
Time (hrs)	3.6	**Oil yield (%)**	33.3	32.5	1.69

Table 3: Linearization of second order kinetic model of solid liquid extraction of Moringa seed for at different temperatures using hexane as solvent, solvent to solid ratio 9:1 and coarse size seeds.

T (K)	C_S (g L^{-1})	k (L g^{-1} min^{-1})	h (g L^{-1} min^{-1})	R^2
323	37.04	1.28 X 10^{-3}	1.76	0.998
333	38.46	1.40 X 10^{-3}	2.07	0.997
343	40	1.53 X 10^{-3}	2.45	0.999

The kinetics of oil extraction from *Moringa* seeds was studied at different temperatures (Table 3). At the beginning of the operation, the rate of extraction was very fast, followed by a slower rate for the remaining of the extraction period until it reaches the plateau after 300 min of extraction. When the maximum amount of extractable oil is obtained, the oil yield level remains invariable even by extending the reaction time. The saturated extraction capacity, C_s, increased from 37.04–40.0 gL^{-1} when the temperature increased from 323-343 K. Similar trends was also observed for the extraction rate constant, k and the initial extraction rate, h, when temperature changed from 323 to 343 K. The values increased from 1.28 to 1.53 × 10^{-3} $Lg^{-1}min^{-1}$ and 1.76 to 2.45 $gL^{-1}min^{-1}$ respectively. The saturated extraction capacity, Cs, the extraction rate constant, k and the initial extraction rate, all increased with temperature. The extraction temperature had a direct effect on all the independent variables.

10.Activation Energy

The energy level of the molecule to initiate a chemical reaction is generally represented as activation energy. From the data, it can be concluded that solid liquid extraction of *Moringa* seeds is an endothermic process since the activation energy is positive. Similar finding have been reported by Sayyar et al. (2009) for Jatropha seeds.

11.Characterization of the Extracted Moringa Oil

The oil obtained was analysed for various parameters as shown in Table 4. The oil had iodine value 67.23, saponifcation value 184.65, density 0.907 and refractive index 1.436. The results are in agreement with the findings of Abdulkarim et al. (2005) except the small variations in the compositional parameters. The variation may be due to the differences in variety of plant, cultivation climate, ripening stage and the harvesting time of the seeds.

Table 4: Characterization of *Moringa oleifera* oil.

Parameters	**Composition**
Acidity (% as oleic acid)	1.09± 0.05
Saponification value(mg of KOH/g of oil)	184.65± 1.10
Iodine value(g of I/100 g of oil)	67.23± 1.00
Peroxide value (meq/kg of oil)	0.98± 0.02
Refractive index (40^0C)	1.436± 0.001
Density (g/cm^3) 24^0C	0.9073± 0.001

[a]mean ± standard deviation

12.Conclusion

The optimum processing conditions were solvent to solid ratio (9:1), temperature (67.84°C) and residence time (3.6 hrs) for the maximum recovery of oil from *Moringa* seeds (PKM 1 variety). The maximum oil, 33.30% was yielded using hexane. The extraction temperature had a direct effect on the variables. The solid liquid extraction of *Moringa* seeds was observed as an endothermic process since the activation energy was positive. The characterization revealed that the oil could be utilized as a source of edible oil for human consumption.

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