Effect of Process Parameters on the Oil Recovery of Maize Germ Oil

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Abstract- *The maize germ is valuable by-product of starch processing industry being rich in oil. The maize germ was separated from maize kernels using manual wet milling method. The present study was conducted to see the effect of extraction time and particle size on the oil recovery and physico-chemical characteristics of oil. The germ oil from maize PMH1 variety was extracted using solvent extraction method by varying the particle size of germ between 300 µm to 1.40 mm and time of extraction between 1.5 to 6 hrs. The different physico-chemical characteristics of extracted oil were determined viz., color, pH, unsaponifiable matter, saponification value, total phenolics content, acid number, density, peroxide value, flavor/odor and refractive index using standard methods. Maximum oil yield obtained were 29.25±0.76% for extraction time of six hour for 300 µm particle size. It was observed that as the particle size increased oil yield decreased linearly, whereas as the extraction time increased oil recovery increased linearly. The average values of extracted germ oil for colour (Red, Hue-52.9YR, Chroma-1.61), pH (6.49±0.07), unsaponifiable matter (3.63±0.15 %), saponification value (223.05±6.05 mgKOH/g), total phenolics (76.54±0.01 mg GAE/kg), acid number (3.73±0.03 mg KOH/g), free fatty acid (1.91±0.05 %), peroxide value (3.44±0.09 meq/kg), density (883.73±1.10 kg/m³), flavour/odor (mixed flavor of corn and solvent and the faint order of solvent) and refractive index (1.54±0.03) respectively.*

Keywords- Maize germ oil, solvent extraction method, particle size, colour, peroxide value

I. INTRODUCTION

Maize (Zea mays L.) is coarse grain and is now being accepted as staple diet and its demand increases year by year. Maize can play important role in crop diversification policy of the state (1). India ranks among the world's 10 highest maize producing countries, and produces about 22.97 million tonnes in 2014-15 (2). In Punjab, the maize production is estimated as 507 thousand tonnes in 2013-14 (3). To wean Punjab and Haryana farmers off water-and-labour-intensive paddy crop and to produce other crops viz., maize, cotton, vegetables and fruits, of Rs. 500 crore has been allocated for crop diversification(1). Maize can play important role in crop diversification policy of the state.

Maize germ, which is a by-product of maize processing industry, constitutes 5-14% of the weight of kernel and is a good source of key nutrients especially 18-41% of oil (4-5). Edible oils are vital, serving as important ingredient of many foods by imparting characteristics flavour and texture to finished food products (6). The maize oil is easy to digest, beneficial to human heart, reduce risk of chronic diseases, prevention macular degeneration and can be used for hair treatment. It can also be used for making of biodiesel, for wood conditioning and giving stainless steel a shine. Maize oil is high in polyunsaturated fat, which is a heart-healthy fat, and low in saturated fat. The American Heart Association suggests replacing saturated fat in one's diet with unsaturated fat in order to reduce cholesterol as well as reduce your risk of developing heart disease. Maize oil is comprised of about 86 % unsaturated fats and about 13 percent saturated fat, according to the Corn Refiners Association (7), maize oil is a good source of vitamin E, providing 15 % of the daily value per tablespoon.

Maize oil has many applications, including its use in pharmaceutical industry, manufacture of resins, plastics, lubricants and fuels. However, majority of oil produced is refined for direct consumption and for use by food industry. The reason for this is not far-fetched. Maize oil when refined has neutral flavour; relatively high smoking point and can therefore withstand heat. Also research has revealed that its consumption has health benefits (8). In developing countries, according to (9), most of the cultivation of corn is for human consumption.

Solvent extraction of oil from oilseeds is the most efficient and attractive method for oilseeds having low oil content (10-11). This is the most economical, efficient and widely used process for high oil content seeds (e.g. sunflower, peanut, canola) and also for medium oil content seeds (cottonseed and corn germ) (12-13). The objective of this study was to see the effect of particle size and extraction time on recovery of maize germ oil using solvent extraction method and to determine various quality characteristics of extracted oil.

II. MATERIAL AND METHODS

The Maize kernels of variety PMH1 were obtained from Punjab Agricultural University farm. The crop was cleaned and healthy kernels were selected, for the study.

Extraction of germ

One kg of cleaned maize were batch steeped at 55ºC for 18 h in steeping solution i.e. 0.2% sulphur dioxide (SO_2) and 0.55% lactic acid (1,900 ml total solution) for germ separation (14) as shown in Figure 1. The sample was first grinded in grinder for 10 sec. The germ skimming process allowed the germ component and other kernel component to be separated by density differences. Germ skimming was done for 10 min followed by second grinding for 10 sec. The recovery of germ after second grinding was determined for the duration of 10, 20 and 30 min, each of which was mean of three replications. The skimmed germ was dried in hot air try dryer for 18 h upto 5% wb moisture content and then stored in refrigerator at temp of 4-5ºC.

Pre-treatment of sample

The dried sample of maize germ was collected, grinded and the sieve analysis using Standard Test Sieves (Accro-Tech Scientific Industries, New Delhi, India) of ground sample was done by arranging the sieves in descending order of pore aperture. The fractions of the sample retained on the various sieves were collected, dried in an oven set at temperature of 50ºC for 10 minutes and placed in different labelled containers (15).

Extraction of germ oil

The extraction of oil was done using soxhlet apparatus. About 12g of the dried grinded seed sample was weighed and put into the empty thimble. The thimble containing the sample was then weighed again and the value obtained recorded and put in a soxhlet apparatus. Two-third of capacity of round-bottomed flask petroleum ether was measured and poured into it. The electro thermal heating mantle was switched on and temp set at 40ºC. After (1.5, 3, 4.5 and 6) hour, the heating mantle was switched off (15). The residue was removed from the thimble and the produce repeated for the various separate fractions. On completion of extraction, the oil was heated for 20 min at 50ºC to enable solvent escape.

Determination of different physic -chemical characteristics of oil

Different physico-chemical characteristics of extracted oil were determined viz., color, pH, unsaponifiable matter, saponification value, total phenolics content, acid number, density, peroxide value, flavor/odor and refractive index using standard methods. Three readings were taken for each property and average as well as standard deviation value was calculated using the spreadsheet software program, Microsoft Excel (2007).

Color: The color of samples was measured by using Color Reader CR-10 colorimeter (Konica Minolta Sensing Inc.) (16). For determination of color, the oil sample was poured in a transparent glass bottle provided that no light is allowed to pass during the measuring process. The 'L', 'a' and 'b'values was recorded at D 65/10º. Numeric description of color using L, a and b in colorimeter. L (lightness or darkness) ranges from black (0) to white (100); a color direction in red ($a > 0$) or green ($a < 0$); b color direction in yellow ($b > 0$) or blue ($b < 0$).

$$\text{Chroma} = (a^2+b^2)^{1/2}$$

$$\text{Hue angle} = \tan^{-1}(b/a)$$

pH value: pH of oil was measured using pH meter using manual settings.

Unsaponifiable matter:- Unsaponifiable matter is defined as all the substance present in the products which, after saponification of the latter by potassium hydroxide and extraction by diethyl ether, are not volatile under the specified operating conditions (17).

$$\text{Unsaponifiable matter (\%)} = \frac{a}{m}\times 100$$

Where, a = weight of residue after oven drying (g)
m = weight of sample (g)

Saponification value: - Saponification value is defined as the weight of potassium hydroxide expressed in milligrams required to saponify 1 g of fat or oil (17).

$$\text{Saponification value} = \frac{(t_1 - t_2)\times 28.1}{W}$$

Where, t_1 = blank value
t_2 = sample value
W = weight of sample (g)

Total phenolics content: - Total phenolic content was determined using the modified Folin- Ciocalteu procedure (18-19).

Acid number: - Acid number may be defined as milligram of Potassium Hydroxide required for neutralizing the free fatty acid present in 1 g of oil (20).

$$\text{Acid number (mg KOH/g)} = \frac{5.6 \times (V_1 - V_2)}{W_1}$$

$$\text{Free Fatty Acids} = \frac{2.82 \times (V_1 - V_2)}{W_1}$$

Where, V_1 = Volume in ml of potassium hydroxide solution required for the sample

V_2 = Volume in ml of potassium hydroxide solution required for the blank

W_1= Weight of sample in g

Density: Density or specific gravity was determined using standard method (21).

$$\text{Density (g/m}^3) = \frac{\text{mass (g)}}{\text{volume (m}^3)}$$

Peroxide value: The sample is dissolved in acetic acid: chloroform mixture and potassium iodide is added. Peroxide oxygen liberates iodine from KI which is titrated with thiosulphate (22).

$$\text{Peroxide value (milliequiv.}\frac{\text{Peroxide}}{\text{kg}}\text{sample)} = \frac{(S - B) \times N \times 1000}{\text{sample weight (g)}})$$

Where, S = ml of 0.01 N $Na_2S_2O_3$ required by sample

B = ml of 0.01 N $Na_2S_2O_3$ required by blank

N = Normality of $Na_2S_2O_3$ solution

Refractive index:- A property of a material that changes the speed of light, computed as the ratio of the speed of light in a vacuum to the speed of light through the material is determined as Refractive index, was determined using Abbe's Refractometer (23).

III. RESULT AND DISCUSSION

The maize germ was extracted using wet milling technology and germ was dried using hot air dryer at 55ºC for 18 h upto when the moisture content of germ reached 5% wb. The recovery of germ after first grinding was 3.93±0.06% and the maximum recovery of germ was determined about 6.54±0.04% of maize kernels after second grinding. The germ oil was extracted using solvent extraction method. The effect of process parameters on recovery of oil and different quality parameters of oil are described as under:

Effect of particle size of maize germ on oil yield

The effect of particle size on recovery of maize germ oil was evaluated. It was observed that as the particle size increased (300 µm to 1.40 mm) oil yield decreased (29.17% to 21.67%) during 3 hour duration of oil extraction as shown in Table 1 and Figure 2, because the smaller particle size gave more yield because of the higher surface area to volume ratio that in turn enhanced the contact between the solvent molecules and germ particles during the extraction process (24). When the size of the particle is small, it results in relatively large surface area, hence increasing the contact area in between the solvent (25). Size reduction also helps for easier accessibility of the soluble substrates that are otherwise located deep inside the plant matrix (26). The linear relationship between the oil yield and particle size of grinded maize germ was developed, linear regression equation was found to fit for predicting oil yield as shown in Table 2.

Effect of duration of oil extraction process on oil yield

The effect of extraction time on recovery of maize germ oil was evaluated. It was observed from analysis that the oil yield increased linearly with increase in extraction time upto 3 hour of oil extraction process duration but after 3 hour the variance noted was not wide as shown in Table 1 and Figure 3. The yield of oil increased with the duration linearly because for longer duration the particle and solvent will be in contact and thus the particles release more oil. Form the figure it can be observed that the fine particles size (300 to 600 µm) yield appreciably oil in first 3 hour of extraction duration and beyond 3 hour the oil extraction was quite slow. It might be due to the fact that diffusion of oil was fast due to high initial oil content. This diffusion rate decreased significantly when the oil content of sample decreased (27), whereas large particle size (850 µm to 1.4 mm) and unsieved sample yield appreciable amount of oil in all range of oil extraction duration due to the coarse particle size require more extraction duration for diffusion of oil present in them. The polynomial relationship between the oil yield and oil extraction duration was developed, polynomial regression equation was found to fit for predicting oil yield as shown in Table 3.

Physico-chemical characteristics of maize germ oil

Different quality characteristics of the extracted maize germ oil and its comparasion with the standard values were presented in Table 4.

Color is an important food evaluation indicator for rapid monitoring of the quality of oil i.e. the level of lutein, zeaxanthin and other carotenoids (28). Chroma (saturation or vividness) determine the color as chromaticity increased, a color becomes more vivid; as it decreased, a color becomes more dull. Hue (tint of color) determine an angular measurement in which 0º equal to red and 90º equal to yellow (29).The average value of 'L', 'a' and 'b' were 25.7, 1.9 and 3.1. The visually observed colour of extracted oil was brownish red, this may be due to the ability of the solvent to extract pigments and the determined the value of 'L' indicate colour near black, 'a' near red and 'b' near yellow. The colour of the oil was determined as Hue as 52.9 YR and Chroma as 1.61.

The maize germ oil does not have any pH because it does not release hydrogen or hydroxide ions in water. The pH of oil was found to be around 6 close to neutral pH.

Tocopherols are particularly important functional constituent of the unsaponifiable fraction of vegetable oils. These compounds display antioxidant properties and are active as vitamin E, which make them particularly essential for human nutrition (30). The unsaponifiable matter of oil was determined as 3.50 % to 3.80 %.

Saponification is a process that produces soap, usually from fats and lye. Vegetable oils and animal fats are the main materials that are saponified. These greasy materials, triesters called triglycerides, are mixtures derived from diverse fatty acids. Triglycerides can be converted to soap (31). The saponification value of oil was determined as 216.63 to 228.64 (mgKOH/g).

Phenolic compounds are important plant constituents, because they exhibit antioxidant activity by inactivating lipid free radicals or preventing decomposition of hydroperoxides in to radicals (32). The Total phenolics content of oil was determined as 76.54 to 76.55 (mg GAE/kg).

The acid value is considered in the food industry as an indicator of the quality of the oil and the degree of its degradation during heating. An increase in the acid value leads to the development of unpleasant tastes and odors in oils. The increase in acid value attributed to the hydrolysis of TAG (triacylglycerol) and/or cleavage and oxidation of fatty acid double bonds (33). The acid value and free fatty acid content of oil was determined as 3.69 to 3.75 (mg KOH/g) and 1.88 to 1.97 (%).

The number of peroxides present in edible fats and oils is an index of their primary oxidative level. The peroxide value test used is an index of its status of preservation. In fact, the lower the peroxide value, the better the fat or oil quality and its status of preservation (34). The peroxide value of oil was determined as 3.33 to 3.50 (meq/kg).

The solvent residue in oil develop undesirable odor in oil and mild flavor of maize with bitter taste of peteroleum ether. During the refining process of oil deodorization of oil was required (Chiacchierini et al. 2007). The density and refractive index of oil were determined as 883.73 to 885 (kg/m³) and 1.53 to 1.55.

IV. CONCLUSIONS

The maize germ oil was extracted using solvent extraction method and the effect of extraction time and particle size on oil content of maize germ was determined. It was observed that as the particle size increased oil yield decreased, similarly, the oil yield increased linearly with increase in extraction time. The colour of extracted oil was dark red with a value 1.9 and L value varies between 24 to 26 near black, suspended particles and had mixed flavor/odor of solvent thus not fit for human consumption. The unsaponifiable matter value of oil was observed high due to the ability of the solvent to extract other lipid-associated substances such as sterols, fat-soluble vitamins, hydrocarbons and pigments. Total phenolic contents produce scavenging capacity and inhibition of linoleic acid peroxidation of the oils. The increased acid value of oil was due to degradation of oil at higher temperature during oil extraction process similarly peroxide value also increases at higher temperature.

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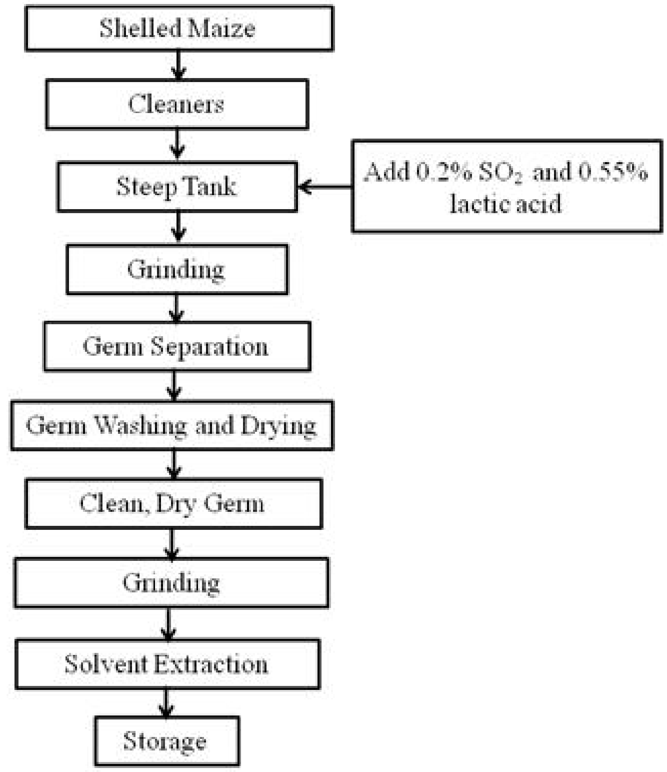

Figure 1: Process flow chart for extraction of maize germ oil.

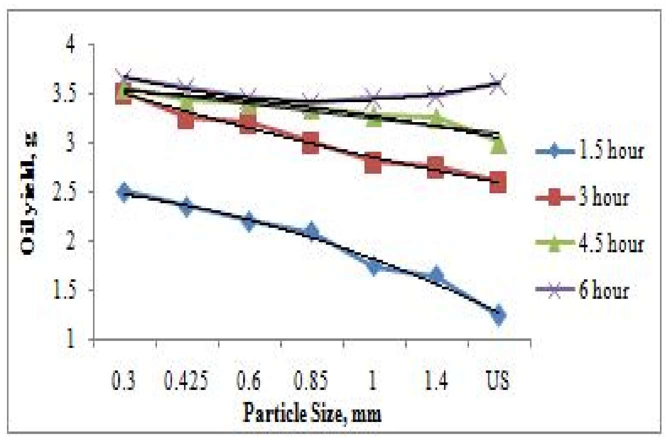

Figure 2: Effect of duration of oil extraction on oil yield

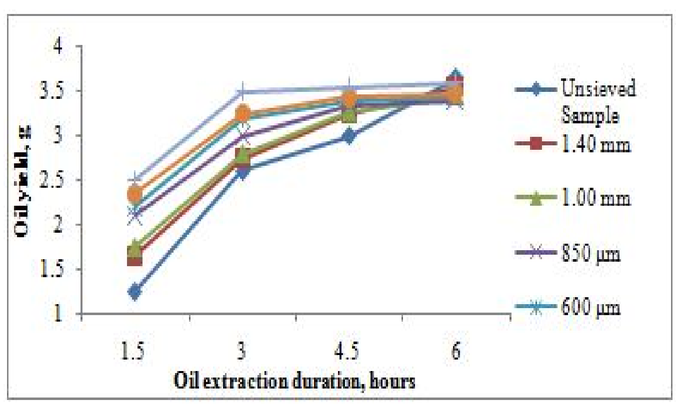

Figure 3: Effect of Particle size on oil yield

Table 1: Effect of Process Variables on Oil yield.

Particle Size	1.5 hour		3 hour		4.5 hour		6 hour	
	Weight(g)	% yield	Weight(g)	% yield	Weight(g)	% yield	Weight(g)	% yield
Unsieved Sample (US)	1.25	10.42	2.60	21.67	3.00	25.00	3.45	30.42
1.40 mm	1.65	11.67	2.75	22.92	3.25	27.08	3.55	29.58
1.00 mm	1.75	14.58	2.80	23.33	3.27	27.25	3.45	28.75
850 µm	2.10	17.50	3.00	25.00	3.35	27.92	3.40	28.33
600 µm	2.20	18.33	3.20	26.67	3.40	28.33	3.45	28.75
425 µm	2.35	19.58	3.25	27.08	3.43	28.58	3.47	28.92
300 µm +Pan	2.50	20.83	3.50	29.17	3.55	29.58	3.60	30.00
Average	1.97±0.44	16.13±3.99	3.01±0.32	25.12±2.66	3.32±0.17	27.67±1.45	3.51±0.09	29.25±0.76

Solvent used: Petroleum ether (40-60 ºC).

Table 2: Regression equations for oil yield at different particle sizes of maize germ

S.No.	Particle Size	Linear Regression Equation	R - Square
1.	Unsieved Sample	$y = 0.76x + 0.725$	0.937
2.	1.40 mm	$y = 0.62x + 1.25$	0.919
3.	1.00 mm	$y = 0.557x + 1.425$	0.889
4.	850 µm	$y = 0.425x + 1.9$	0.831
5.	600 µm	$y = 0.395x + 2.075$	0.759
6.	425 µm	$y = 0.354x + 2.24$	0.756
7.	300 µm	$y = 0.335x + 2.45$	0.675

Where x is the Particle size of maize germ (mm)

Table 3: Regression equations for oil yield at different duration of oil extraction.

S.No.	Duration	Polynomial Regression Equation	R - Square
1.	1.5 hour	$y = -0.018x^2 - 0.057x + 2.557$	0.985
2.	3 hour	$y = 0.006x^2 - 0.194x + 3.671$	0.983
3.	4.5 hour	$y = -0.008x^2 - 0.0136x + 3.533$	0.927
4.	6 hour	$y = 0.023x^2 - 0.197x + 3.833$	0.970

Where x is the oil extraction process duration (hour)

Table 4: Comparison of the characteristic values of extracted crude maize germ oil with standard values

Oil property	**Standard Value**	**Value**
Oil yield (%)	18-41%	24.86±0.70
Time required for extraction (hourr)	3.00	3.00±0.00
Colour	Pale Yellow	Red, Hue-52.9YR, Chroma-1.61
pH	close to neutral pH	6.49±0.07
Unsaponificable matter (%)	1.18	3.63±0.15
Saponification value (mgKOH/g)	189-195	223.05±6.05
Total phenolics (mg GAE/kg)	81.4 to 198.5	76.54±0.01
Acid number (mg KOH/g)	0.005	3.73±0.03
Free fatty acid (FFA) (%)	0.01	1.91±0.05
Peroxide value (meq/kg)	Not more than 10	3.44±0.09
Density (kg/m^3)	867-918	883.73±1.10
Flavour/odor	Slight corn slight nutty/buttery	Mixed flavor of corn and solvent and the faint order of solvent
Refractive index	1.470-1.474	1.54±0.03

(Source: CRA 2006; Tuan 2011; Abdulkadir and Abubakar 2011; Latif 2009)