Reprint **ISSN 1997-2571 (Web Version)**

Journal of Innovation & Development Strategy (JIDS)

(J. Innov. Dev. Strategy)

Volume: 7 **Issue: 2** **August 2013**

J. Innov. Dev. Strategy 7(2): 5-9 (August 2013)

STUDIES ON THE EFFECT OF DIFFERENT SOLVENTS AND OPTIMIZATION OF TEMPERATURE, TIME AND PARTICLE SIZE FOR THE EXTRACTION OF CORN OIL

M.M. UDDIN, M.S. RAHMAN, S. YEASMIN, M.M. RAHMAN AND M.I.H. MONDAL

GGF
Nature is Power

An International Scientific Research Publisher
Green Global Foundation©
Publication and Bibliography Division
100 Leeward Glenway
Apartment # 1601
M3c2z1, Toronto, Canada
E-mails: publication@ggfjournals.com, editor@ggfjournals.com
http://ggfjournals.com/ejournals/current issues

JIDS** issn 1997-2571, HQ:19-10 central place, saskatoon, saskatchewan, s7n 2s2, Canada

ISSN-1997-2571 (Online)
J. Innov. Dev. Strategy 7(2):5-9(August 2013)

STUDIES ON THE EFFECT OF DIFFERENT SOLVENTS AND OPTIMIZATION OF TEMPERATURE, TIME AND PARTICLE SIZE FOR THE EXTRACTION OF CORN OIL

M.M. UDDIN[1], M.S. RAHMAN[2*], S. YEASMIN[1], M.M. RAHMAN[1] AND M.I.H. MONDAL[2]

[1]BCSIR Laboratories, Binodpur Bazar, Rajshahi-6206, Bangladesh; [2]Department of Applied Chemistry and Chemical Engineering, Rajshahi University, Rajshahi-6205, Bangladesh.

*Corresponding author & address: Md. Saifur Rahman; E-mail: saifur_13bd@yahoo.com
Accepted for publication on 15 July 2013

ABSTRACT

Uddin MM, Rahman MS, Yeasmin S, Rahman MM, Mondal MIH (2013) Studies on the effect of different solvents and optimization of temperature, time and particle size for the extraction of corn oil. *J. Innov. Dev. Strategy.* 7(2), 5-9.

Effect of three different solvents namely n-hexane, chloroform and ethanol (absolute) for the extraction of oil from ground corn were investigated. Dried ground corn of three different particle sizes 123, 150 and 180 µm were taken for extraction. Extractions were carried out at different temperatures 25, 50 and 68°C for 1 to 8 hours with 1 hour interval. Maximum yield were 5, 4.5 and 3.8% for n-hexane, chloroform and ethanol respectively. It was found that the amount of oil extracted from 123 µm was the highest (5%) at the temperature 68°C for 5 hours extraction time. Considering the yield of oil, the optimal conditions were: (i) extraction time 5 hours, (ii) temperature 68°C and (iii) particle size 123 µm. Under these conditions the extraction were then carried out using three different solvents for observing the effect of extracting solvents on physico-chemical characteristics, lipids and glyceride compositions and fatty acids of the oil. The total lipids were fractionated into lipid classes by silicic acid column chromatography. The neutral lipids were found to be 86.31, 86.98 and 87.06%, glycolipids 9.97, 9.69 and 9.39% and phospholipids 3.71, 3.32 and 3.54% for n-hexane, chloroform and ethanol, respectively.

Key words: *corn, extraction, lipids, glyceride, fatty acid, n-hexane*

INTRODUCTION

Corn is one of the principal cereal crops of the world. In 2011 about 885289935 MT of corn were produced throughout the world (Food and Agri. stat. of UN 2011). It grows well in the climatic conditions of Bangladesh and the annual production is about 1850656 MT (Food and Agri. stat. of UN 2012).The cultivation of corn is tremendously increased in recent years in Bangladesh (Bennetzen and Hake, 2009). About 80% of the corn is used as poultry and livestock feed in Bangladesh (Mowlah 1990). It is well documented that deoiled kernel or meal of corn is better than that of whole grain of corn for poultry. If the oil remains in kernel, it is disintegrated in glycerin and free fatty acids; free glycerin acts as purgative in the stomach and intestine of poultry birds and cattle. Free fatty acids are also equally harmful for stomach and intestine (Mowlah 1990). So extraction of oil from corn gives a harmless grain for cattle and poultry feeds. Embryo or germ of corn contains about 4-6% of oil and with 85% of polyunsaturated fatty acid (Reiners 1978). Its good keeping qualities make it desirable edible oil (Swern 1964). The chemical composition of corn revealed that it contains about 3-6% lipids (Woods 1907). In the conventional corn milling process (wet milling) corn oil is recovered exclusively from corn germ by degerminating system using ethanol as an extracting solvent but the lipids of the endosperms do not recover by this process (Chien *et al.* 1990). Corn starch is another valuable product derived from corn and it is also found that a defatted corn gives a good quality corn starch. If corn oil could be extracted from whole corn using a suitable solvent, it would add significant revenue.

A number of organic solvents can be used for extracting oil but all of them are not recommended as suitable solvents for the extraction of edible oil. Several attempts have been made in the past to develop such processes (Chen and Hoff, 1987; Chien *et al.* 1990; Hojilla-Evangelista *et al.* 1992). Chloroform has been extensively used for the extraction of plant and animal lipids. But recently throughout the world the use of chloroform as extracting solvent has been restricted due to its hepatoxic action (IARC 1979). Ethanol is comparatively harmless as well as cost effective but it is difficult to recover from oil due to its higher boiling point (78.37°C). n-hexane is the most popular and recommended solvent which is less toxic, less harmful as well as easy to recover from extract for its low boiling point (68°C). So we have attempted to evaluate the efficiency of three solvents for the extraction of oil from dried ground corn and optimize the time of extraction, temperature and particle size of ground corn.

MATERIALS AND METHODS

Collection of Corn

Fresh and ripe corns (with cob) were collected from Bangladesh Rice Research Institute (BRRI) Regional Station, Rajshahi. The seeds were dehulled by conventional method and sorted manually to remove defective seeds. The fresh and clean seeds were sundried to reduce moisture to 4% and moisture was determined according to (ICOMR 1971) standard method.

Grinding and making different particle sizes

Whole corn was ground into different particle sizes using a disk mill (model: RXCO FFC-5). Particle size distribution of milled corn was determined by using a rotary screen shaker (RO-TAP:W.S.Tylerluc.Mentor OH) and US Standard sieves. The ground corn was used for the extraction experiment immediately after grinding and

the variable parameters were i) particle sizes of ground corn 123,150 and180 µm ii) time of extraction 1 to 8 hours with 1 hour interval iii) temperature-25 (room temperature), 50 and 68°C and iv) solvent-to-solids ratio (ml/g) was 4:1. The solvents namely n-hexane, chloroform and ethanol (absolute) (E Mark Germany) were used for extraction.

Extraction with different solvents

The extractions were performed in one liter Erlenmeyer flasks each contains 400 ml solvent and 100 gram of ground corn on a hot plate (temperature controlled) using a magnetic stir bar. Mouths of flasks were sealed properly so that solvents cannot be leaked out.

Batch extraction at the different temperatures 25, 50 and 68°C of particle size 123,150 and 180 µm were performed serially for 1-8 hrs and yield of oil were observed after every one hour. The slurry of extracts were filtered by using Whatman filter paper no.1 (11-µ mean pore size) The oil were then recovered from the filtrate by evaporating the solvent using a rotary vacuum evaporator model (IKA-WERKE) under reduced pressure and the percentage of oils were calculated for different particle size, temperature, and time of extraction for optimization of the process.

The physico-chemical characteristics of the extracted oils were determined by the American oil chemist's society (AOCS 1980) and International Union of Pure and Applied Chemistry (IUPAC 1967) standard methods. After optimizing the conditions (temperature 68°C, time 5 hrs and particle size 123 µm) soxhlet method (AOAC 1984) with n-hexane was used for continuous extraction.

Separation of lipid classes by column and thin layer chromatography

The total lipids extracted with three different solvents were fractionated into three major lipid classes on a silicic acid column. The silicic acid (E.Merck Darmstadt, W.Germany, 70-230 mesh) was washed with water and methanol to remove fine particles and impurities. It was activated at 120°C over night and again for 1 hr immediately before the column was prepared. For each column 25 gm of silicic acid was washed with 250 ml of chloroform methanol (7:1 v/v), 120 ml of chloroform – methanol (15:1 v/l) and 160 ml chloroform. Slurry of 25 gm of silicic acid in chloroform was poured into the column (2.2 cm. id). After the column washed with 100 ml diethyl ether and 32.5 ml 4% diethyl ether in petroleum ether (b.p 60-70°C), 150 gm of total lipids were dissolved in 5 ml chloroform and quantitatively transferred to column (Hirsch and Ahrens, 1958).

The neutral lipid was eluted by 80 ml of chloroform, glycolipids by 200 ml of acetone and phospholipids 175 ml of methanol. The elution was controlled with a flow rate of 1.5-2 ml/min. The elution of each fraction was monitored by micro slide thin layer chromatography (TLC) to ensure uniformity of separation of each lipid class during silicic acid chromatography and eluted solvents were collected in a weighed flask. The fractions thus obtained were evaporated in a rotary vacuum evaporator and were under pressure before being weighed. The purity of the lipid classes was further checked by TLC on 20 cm × 20 cm plates coated with a layer (0.5mm) of silica gel. G.

Neutral lipids were separated further by high performance preparative TLC using hexane –diethyl ether –acetic acid (80:20:2) as the developing solvent system and individual lipid class and their fatty acids were quantitatively determined by GLC with added internal standard (Christie 1982).

The fatty acid composition of the triglyceride (TG) fraction of each batch (extraction using three different solvents) was analyzed by borontriflonride methanol method (William and Smith, 1964). The analysis was carried out by a gas liquid chromatography (GLC) instrument equipped with a flame ionization detector. (Analysis was carried out at isothermal column temperature of 190°C, injector and detector temperature for all GLC analysis was 200°C).

RESULTS AND DISCUSSION

Extraction of oil from ground corn of three different particle sizes 123, 150 and 180 µm by using three different solvents namely n-hexane, chloroform and ethanol were observed. At room temperature (25°C) the amount of extracted oil was recorded after each 1 hour and the yields were depicted in table 1. It was observed that the amount of oil extracted from particle size 123 µm was the highest for n-hexane and after 6 hrs it was leveled off. In case of chloroform it was almost same but in case of ethanol it was remarkably lower than that of n-hexane and chloroform.

At 50°C the yields were recorded in table 2. Table 2 indicates that as the temperature raises the yield of oil increases. In case of n-hexane it was 2.87% in first1 hr which is near about 50% of total yield and it was leveled off after 5%. In case of chloroform it was slightly lower than n-hexane (4.5%) Yield of oil by ethanol (3.8%) is much lower than those of other two solvents and it is similar to the reported results of (Kwiatkowski and Cheryan, 2002). They reported that when anhydrous ethanol was used to repeatedly extract fresh corn, moisture was absorbed linearly by ethanol from the corn in successive stages, which in turn decreased oil yield.

Table 3 shows the yield at temperature 68°C. At this temperature about 75% of oil was extracted in 1st hour and after 3 hrs it was leveled off (5%). In case of chloroform the rate of increase of yield was same (4.5%) but total yield was somewhat less. In case of ethanol it was significantly less (3.8%).

From these three tables (1, 2, 3) it is clear that the yield is the highest in n-hexane. The smaller particle (123 µm) gave higher yield of oil because of the particles came more intimately contact with solvent than those of larger particles (150, 180 µm). The results are in good agreement with Chien *et al.* (1990). The authors used only ethanol as an extracting solvent but we used three different solvents to get the better yield. After optimizing the conditions, extraction time 5 hrs, temperature 68°C and particle size 123µm, the physico-chemical properties of the extracted oil with n-hexane, chloroform and ethanol were observed and the results were presented in the table 4. From table 4 it is observed that the characteristics are not remarkably affected by the solvents and these are in good agreement with the reported results of Uddin *et al.* (2007). The values are also similar to that of other popular vegetable oil of good quality (Mowlah 1990). Total lipids were fractionated into three major lipid classes, neutral lipids, glycolipids and phospholipids by silicic acid column chromatography.

The effect of three different solvents on the lipid compositions was observed and the results are shown in table 5. It is observed that the quantity of total lipids extracted with n-hexane is the highest, lipids extracted with chloroform did not differ significantly with n-hexane but the quantity extracted with ethanol is much lower than that of two solvents. From table 5 it is also observed that higher amount of neutral lipid and lower amount of glycolipids and phospholipids extracted by n-hexane compared to those of other two solvents.

Fractionation of neutral lipids was observed and the results are presented in table 6. It was found that the level of triglyceride fraction was slightly higher and the level of fatty acid fraction was lower in the neutral lipids extracted by n-hexane than those of other two solvents. The amount of triglyceride and fatty acid fraction extracted by chloroform and ethanol were almost similar. Fatty acid compositions of corn oil extracted by three different solvents were observed and the results are presented in table 7. The fatty acid compositions of the lipids showed no significant difference for the different extracting solvents in the fatty acid profiles.

Table 1. Extraction of oil from dried ground corn of three different particle sizes at room temperature (25°C) with three different solvents

Time, hr	% yield of oil (w/w) at different particle sizes (µm)								
	Extracting solvent, n-hexane			Extracting solvent, chloroform			Extracting solvent, ethanol		
	Particle size			Particle size			Particle size		
	123, µm	150, µm	180, µm	123, µm	150, µm	180, µm	123, µm	150, µm	180, µm
1	0.52	0.43	0.10	0.46	0.31	0.08	0.24	0.14	0.07
2	0.79	0.79	0.18	0.64	0.42	0.18	0.32	0.21	0.17
3	1.13	0.97	0.27	0.83	0.57	0.24	0.45	0.38	0.23
4	1.56	1.21	0.43	1.03	0.81	0.47	0.67	0.51	0.38
5	1.79	1.46	0.64	1.28	1.10	0.78	0.83	0.79	0.47
6	1.85	1.61	0.89	1.52	1.24	0.93	1.09	0.91	0.61
7	1.85	1.84	1.06	1.55	1.26	0.96	1.11	0.94	0.64
8	1.85	1.84	1.07	1.55	1.28	0.96	1.13	0.97	0.67
Mean ± SD	1.42±0.53	1.27±0.51	0.58±0.39	1.11±0.43	0.87±0.40	0.57±0.37	0.73±0.36	0.61±0.34	0.40±0.23

Table 2. Extraction of oil from dried ground corn of three different particle sizes at 50°C temperature with three different solvents

Time, hr	% yield of oil (w/w) at different particle sizes (µm)								
	Extracting solvent, n-hexane			Extracting solvent, chloroform			Extracting solvent, ethanol		
	Particle size			Particle size			Particle size		
	123, µm	150, µm	180, µm	123, µm	150, µm	180, µm	123, µm	150, µm	180, µm
1	2.87	2.30	1.88	2.62	2.12	1.68	2.16	1.83	0.88
2	3.74	2.92	2.33	3.06	2.54	2.04	2.51	2.23	1.02
3	4.27	3.31	2.69	3.75	2.92	2.41	2.93	2.82	1.26
4	4.74	3.86	2.98	4.05	3.30	2.86	3.37	3.29	1.69
5	5.00	4.28	3.50	4.50	3.79	3.30	3.80	3.64	2.14
6	5.00	4.71	4.00	4.50	4.06	3.98	3.80	3.80	2.72
7	5.00	5.00	4.61	4.50	4.50	4.36	3.80	3.80	3.26
8	5.00	5.00	5.00	4.50	4.50	4.36	3.80	3.80	3.46
Mean ± SD	4.45±0.79	3.92±1.01	3.37±1.10	3.93±0.74	3.47±0.89	3.12±1.05	3.27±0.66	3.15±0.78	2.05±1.01

Table 3. Extraction of oil from dried ground corn of three different particle sizes at 68°C temperature with three different solvents

Time, hr	% yield of oil (w/w) at different particle sizes (µm)								
	Extracting solvent, n-hexane			Extracting solvent, chloroform			Extracting solvent, ethanol		
	Particle size			Particle size			Particle size		
	123, µm	150, µm	180, µm	123, µm	150, µm	180, µm	123, µm	150, µm	180, µm
1	3.08	2.59	2.67	2.83	2.22	1.97	2.63	2.17	1.11
2	4.47	3.27	3.13	3.24	2.79	2.19	3.28	2.56	1.42
3	5.00	3.93	3.67	3.77	3.17	2.43	3.80	3.03	1.84
4	5.00	4.74	3.94	4.50	3.58	2.85	3.80	3.38	2.21
5	5.00	5.00	4.14	4.50	4.07	3.36	3.80	3.80	2.67
6	5.00	5.00	4.67	4.50	4.50	3.81	3.80	3.80	3.09
7	5.00	5.00	5.00	4.50	4.50	4.17	3.80	3.80	3.46
8	5.00	5.00	5.00	4.50	4.50	4.50	3.80	3.80	3.80
Mean ± SD	4.69±0.68	4.32±0.95	4.03±0.85	4.04±0.68	3.67±0.88	3.16±0.94	3.58±0.43	3.29±0.64	2.45±0.97

Table 4. Physico-chemical characteristics of corn oil extracted at optimized condition

Parameters	Values
Sp. gravity at 20°C	0.920
Refractive index at 15°C	1.471
Smoke point, °C	225
FFA %	1.6
Saponification value	73
Unsaponifiable matter, %	0.90
Iodine value	115.5
Peroxide value m.eq/Kg	0.6

Table 5. Lipid compositions of corn oil extracted at optimized conditions

Extracting solvents	Total Lipid (%)	Neutral Lipid (%)	Glycolipid (%)	Phospholipid (%)
n-hexane	5.00	86.31	9.97	3.71
Chloroform	4.5	86.98	9.69	3.32
Ethanol	3.8	87.06	9.39	3.54
Mean ± SD	4.43±0.60	86.78±0.41	9.68±0.29	3.52±0.19

Table 6. Neutral lipid compositions of corn oil extracted at optimized conditions

Name of the solvents	Neutral lipids (wt %)				
	Hydrocarbon & Sterol Esters	Triglycerides	Fatty Acids	Free Sterols	Partial Glycerides
n-hexane	0.6	85.10	12.0	0.6	1.7
Chloroform	0.6	86.00	11.5	0.6	1.3
Ethanol	0.6	85.90	11.3	0.7	1.5
Mean ± SD	0.60±0.00	85.67±0.49	11.60±0.36	0.63±0.06	1.50±0.20

Table 7. Fatty acid compositions of corn oil (weight %) extracted at optimized conditions

Name of the solvents	Fatty Acids (weight in percent)					
	$C_{14:0}$	$C_{16:0}$	$C_{18:0}$	$C_{18:1}$	$C_{18:2}$	$C_{18:3}$
n-hexane	0.9	2.3	9.2	37.4	49.5	trace
Chloroform	0.8	2.5	9.6	37.7	49.1	trace
Ethanol	0.6	2.4	9.8	37.1	49.8	trace
Mean ± SD	0.77±0.15	2.40±0.10	9.53±0.30	37.40±0.30	49.47±0.35	

CONCLUSION

From the study, it may be concluded that the extraction of oil from dried ground corn by solvent extraction method, n-hexane is more effective than those of other two solvents chloroform and ethanol. Although the efficiency of chloroform is almost similar to n-hexane but it should be avoided because of its hepatoxic action. On the other hand, the yield of oil extracted by ethanol is much lower than that of n-hexane. So n-hexane is the best among the three solvents for the oil extraction from whole dried ground corn.

ACKNOWLEDGEMENT

The authors express their gratitude to Dr. Md. Zahurul Haque, Director, (in charge), BCSIR Laboratories, Rajshahi for providing all research facilities.

REFERENCES

AOCS (1980) Official and Tentative Methods of American Oil Chem. Soc. 3rd. Edition, Vol.-1 USA.

Association of Official Analytical Chemists (1984) Fat (Crude) or Ether Extraction Animal Feed. Official Methods of Analysis, 14th, Edition, AOAC, Washington, DC, Method 920, 39 C.

Bennetzen JL, Hake S (2009) Handbook of Maize Genetics and Genomics, New York, NY: Springer, New York, 812.

Chen LF, Hoff JE (1987) Grain Extracting Milling. US. Patent 4, 716, 218.

Chien JT, Hoff JE, Chen LF (1988) Simultaneous dehydration of 95% ethanol and extraction of crude oil from dried ground corn. *Cereal Chem*. 65, 484-486.

Chien JT, Hoff JE, Lee MJ, Lin HM, Chen YJ, Chen LF (1990) Oil extraction of dried corn with ethanol. *Chem. Eng. J.* 43, B103-B113.

Christie WW (1982) Lipid Analysis. Pergamon Press, Oxford, England.

Food and Agriculture Statistics. UN (2011).

Food and Agriculture Statistics. UN (2012).

Hirsch J, Ahrens EH (1958) Column Chromatography. *J. Biol.Chem*, 233, 311.

Hojilla-Evangelista MP, Myers DJ, Johnson LA (1992) Characterization of protein extracted from flaked, defatted, whole corn, by the sequential extraction process. *J. AM. Oil Chem. Soc.* 69, 199-204.

IARC (1979) Monographs on the carcinogenesis risk of chemicals to humans. International Agency for Research Cancer, WHO, Lyon, France, 20, 401.

ICOMR (1971) A manual of laboratory techniques Indian council for medical research national institute of nutrition, India, 2-6.

IUPAC (1967) Standard Method for the Analysis of Oils, Fats and derivatives. 6th. Edition, pergamon press, Oxford, 56.

Kwiatkowski JR, Cheryan M (2002) Extraction of oil from ground corn using ethanol. *JAOCS*, 79, 8.

Mowlah (1990) A Handbook on Edible oils and Fats. 1st. Edition, Published by the University of Dhaka, Dhaka-1000, Bangladesh.

Reiners RA (1978) Corn Oil, Product of the Corn Refining Industry in Food, Seminar Proc., Corn Refiners Association Washington DC, 18.

Swern D (1964) Bailey's Industrial Oil and Fat Products. Third edition, Inter Science Publishers, Wiley and Sons Inc Philadelphia, USA.

Uddin MM, Rahman MS, Ahmed GM, Hossain MA, Samad A (2007) Variation in lipid content and glyceride compositions of four different verities of corn oil. *Ban. J. Sci. Ind. Res.* 42, 223-228.

Willium RM, Smith LM (1964) *J. Lipid Res*, 5, 600.

Woods CD (1907) Food Value of Corn and Corn Products. US Dept. Agric. Farmers Bull. 298pp.