PRODUCTION OF CASTOR OIL FOR MEDICAL USES

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Abstract

The present investigation is concerned with the production of castor Oil for medical uses by cold press of castor beans at room temperature. The effects of process (cold press) variables, which are: applied pressure and pressing time were studied. At 38°C with applied pressure 140.51 kg/cm^2 and pressing time of 70 minutes, the maximum efficiency was found to be 0.798.

The study included the following cases:

1. Studying the effect of temperature on the press efficiency and quality, the range of temperature was 38-80°C and both the pressure and the time were fixed at 140.51 kg/cm^2 and 5 minutes respectively. It was noticed that increasing of temperature led to:
   A. Increase in the press efficiency.
   B. Increase in the acid value of produced oil.
   C. Increase in the darkness of oil color.
2. The effect of seeds cooking on press efficiency was studied and was found that the cooking increased the press efficiency.
3. Pressure - volume relation for each process (cold press, hot press and pressing of cooked seed) was found, and these relations show the behavior of specific volume of solid content (cm^2/g) or bulk density (g/cm^3) against applied pressure.
4. The pressed cake, which contains (17% oil) was subjected to solvent extraction with n-hexaneuder the operating conditions: time of extraction (10-90 minutes), temperature of extraction (30-60°C) and solvent to feed ratio (1:1 to 3.5:1). At this process the oil content was reduced to 1%.
5. Bleaching of the oil with natural earth to improve the color performed the purification of castor oil.

Introduction

Castor oil is derived from the seeds of the plant Ricinus communis; the castor seed has a high oil content usually in the range 45-50%, and also contains a toxic material; the yield of castor oil from castor bean is 45%. The conventional means of obtaining oil from oilseeds can be applied to obtain castor oil from the bean: expression in hydraulic presses or expellers and/or extraction with various solvents. Medical uses are satisfied by the "U.S. No. 1 Grade,"[1] obtained by the first cold pressing of the beans, which provides practically colorless oil. Industrial uses are satisfied by "U.S. Grad No. 3," obtained by further pressing or solvent extraction, which produces more or less colored oil. Table (1) shows the characteristics of the two grades as commercially available[2].

Table (1) Specifications of Grades of Castor Oil

Property	No.1	No.3
Sp. gr. 15.5/15.5°C	0.961-0.963	0.957-0.963
Color (Gardner)	3 (max)	7 (max)
Acid value	3 (max)	10 (max)
Iodine value	82-88	08-88
Saponification value	179-185	177-182
Unsaponifiable matater %	0.5(max)	1.0(max)

Gurnham and Masson [3] explained expression theory based upon equilibrium conditions of expression, i.e., the conditions after a constant pressure have been maintained until no further flow occurs. A logical first step in the development of expression theory is the formulation of a relation of between the volume of expressible material and the pressure upon it. For Gurnham studied series of fibrous materials, the following relation is found valid for most cases:

$$\log P = a + b/v \text{ or } \log P = a + b * D \quad \ldots\ldots\ldots\ldots 1$$

It is apparent that, if this hypothesis has validity, a plot of log P against (I/V) for any experiment should result in a straight line.

Koo and Coworkers [4, 5] studied the expression of castor oil over range of pressure (1000-4000 psi), temperature (20-80°C), pressing times (1-5hr), using laboratory cage press one liter capacity, they developed a formula:

$$E = 9.21 \times 0^{-3} vP^{6} v\theta / v^{v^{1/6}} \quad \ldots\ldots\ldots\ldots 2$$

They defined efficiency of extraction (expression) as the ratio of oil yield to oil content of the seed, on the same basis (either wet or dry). It can be written as:

$$E = (W / Wo)_w = (W / Wo)_d \quad \text{.....3}$$

Experimental Work

Pressing (Expression)

Raw materials

Domestic castor beans, Ricinus Communis., of the family Eurphorbiaceae from, origin: west Iraq (Ramadi), are used as a feed stock for pressing step, the seeds oil content is in the range 48-50% and moisture content is approximately 6%. The two values are determined according to AOCS Method Ae 3-52 and AOCS Method 2-52[6] respectively.

Experimental Equipment

1- Test cylinder (cage press) of diameter 83mm, stainless steel 316, is used to expell oil from seeds, and it can handle 500g of castor seeds. The cylinder is perforated and contains several hundred holes [380holes], with diameter 2mm.

2- Hydraulic press: This equipment is connected to the cylinder to transform pressure of hydraulic fluid to the test cylinder. Hydraulic press consists of hydraulic cylinder fixed in a steel construction; this cylinder contains a ram of diameter 11cm moves downward by pumping hydraulic fluid through cylinder with hydraulic pump operated by hand. The pressure can be released by relief valve. The press is provided with pressure gauge to measure the pressure in the cylinder, range of pressure of the gauge is 0-250 kg/cm^2.

3- Electronic balance is used to weigh the expressed oil with the time. Type OHAUS model GT 8000, made in U.S.A.

4- Digital thermometer.

5- Oven used to preheat seeds and cooking them. Type Gallen Kamp temperature range 20-200°C.

6- Depth vernier to measure the height of cake in the test cylinder.

Figure (1) shows the schematic diagram for the expression unit.

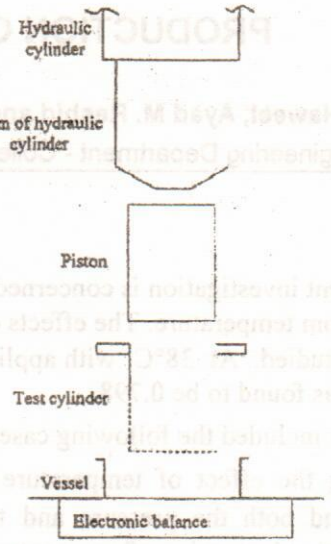

Fig. (1) Schematic Diagram for the Expression Unit

Procedure

1. 500g of cleaned seeds are charged to the test cylinder.
2. The test cylinder is connected with hydraulic press. The pressure is transformed from hydraulic press to cylinder test by the piston.
3. The pressure is established in test cylinder by the operation of hydraulic pump. The pressure should be applied gradually and the pressure range is 17-140.5kg/cm^2.
4. The seeds pressed at different pressures, in each pressure, the time and the amount of the expressed oil are recorded. The pressing time is usually in the range 1-70 minutes.
5. The hydraulic pump is operated from time to time in order to maintain the required pressure constant.
6. New seeds are used in each applied pressure.
7. For hot press process the seeds are heated in the oven to the desired temperature and fed directly to the test cylinder, the applied pressure and pressing time are kept constant (140.51 kg/cm^2 and 5 minute) for different temperatures 38, 54, 65, 70, and 80°C.
8. In the case of pressing the cooked seeds, the seeds are cooked in the oven at 100°C for 1 hour, then they are pressed at room temperature for various pressures (17-140.5kg/cm^2) and different times 1-70) minutes.

Solvent Extraction

Raw Materials

1. The press cake from expression stage is the main feedstock to solvent extraction. The analysis of press cake indicated that this cake contains 17% oil and 8% moisture. These values are determined according to AOCS Methods Bd 3-52[6] respectively.
2. Commercial n-hexane is used as a solvent to extract the remaining oil from the press cake.

Equipment

1. Extraction Vessel: A 1000 ml round flask, fitted with two side necks connected to the condenser and solvent charger to maintain the proper solvent to feed ratio. Figure (2) shows the batch extraction unit.
2. Water bath is used to maintain the required temperature.
3. Thermometer is used to measure the temperature of extraction.
4. Stirrer: A motor-driven hinged propeller type stirrer fitted with a packing gland (the stirrer type: Comtex, maximum speed of rotation is 1600 rpm).
5. Energy transformer: Type Vario, model R 52-260H is used to give the desired speed of impeller (720rpm).
6. Buchner funnel to separate the miscell from the meal.
7. Vacuum pump is used in filtration step, type Edwards model Es 50, maximum delivery 15 psi.
8. Distillation unit is used to separate the solvent from miscella, consists of flask (500 ml) with two necks for insertion of the thermometer and connecting the condenser. The required heat is supplied by a mantel heater.
9. Grinder is used to reduce the size of press cake to desired size not less than 50% passing through 300 mesh. The grinder type Retsch model BBIA.

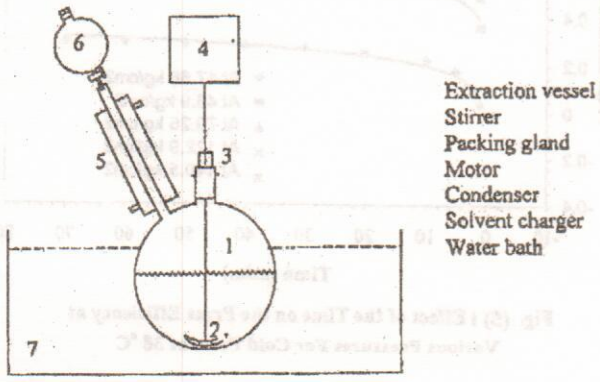

Fig. (2) Extraction Experimental Unit

Procedure

i- The cake from expression stage is fed to the grinder machine to reduce its size.

ii- The condition of extraction process are: -

1. The solvent to feed ratio in the range 1:1 to 1:3.5 by weight.
2. The slurring time in the range 10-90minutes.
3. The temperature of extraction in the range 30-60°C.
4. miscella is separated from the meal by filtration using Bunchner funnel under vacuum about 5inch mercury.
5. n-hexane is recovered from miscella by distillation unit, the temperature of distillation is 70°C.

The Refining Process

Medicinal castor oil is refined by bleaching to remove colored particles using clay earth.

Materials

1. Castor oil produced by cold press at 140.51 kg/cm^2 has a color in Lovibond scale about (1red and 5 yellow), and acid value about 1.9.
2. Adsorbent (bentonite) domestic production. Its properties are given in Table (2).

Table (2) Typical Properties of Bleaching Earth

Property	Value
1. pH at 5% Solution	9.4
2. Seive test	
on 125 mesh	7.2%
on 75 mesh	36.8%
through	13.6%
3. Bleaching test with Sunflower oil	
Before bleaching, color of the oil (lovibond, 1 inch cell)	18 red, 70 yellow
After bleaching, color of the oil (lovibond, 1 inch cell)	7.3 red, 70 yellow

Equipment

1. Bleaching vessel: A 500 ml round bottom flask, fitted with three side necks.
2. Thermometer is used to measure the temperature of bleaching.
3. Vacuum pump is used to evacuate the bleaching vessel to desired pressure.
4. Magnetic stirrer and thermostat hot plate: Type Gallen Kamp is used to heat the bleaching vessel.
5. Lovibond color meter type (AOCS tintometer) is used to compare the oil color with the standard Glasses Lovibond.

The schematic diagram of the refining unit is shown in Figure (3).

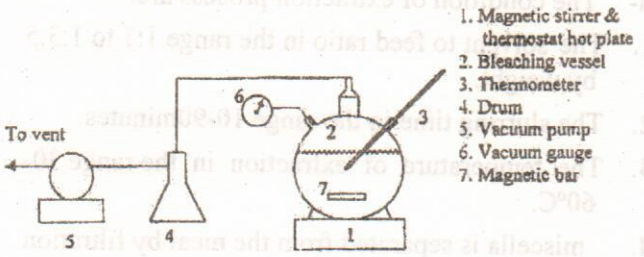

Fig. (3) Refining Unit

Procedure

1. 100 g of castor oil is fed to the bleaching vessel; and an amount of clay earth in the range 0.5-2.5% by weight is added to the oil.
2. The mixture is heated to a temperature in the range 90-120°C.
3. After the desired temperature is reached the vessel is evacuated to 26inch mercury.
4. The process continues for 30 minutes, and then the vacuum is broken up.
5. The clay earth is separated from the oil using Buncher funnel.
6. The color of bleaching oil is measured by Lovibond colormeter to evaluate the refining step.
7. Finally the acid value is measured after refining.

Result and Discussion

Expression

Effect of the Pressure on Press Efficiency

Figure (4) shows the effect of pressure on press efficiency. For condition of cold press at 38 °C with period of time 70 minutes, the increasing of pressure from 17.56 to 140.51kg/cm^2 led to increase the press efficiency from 0.296 to 0.798 respectively.

Increasing hydraulic pressure on the cage press above 140.51 kg/cm^2 had no advantage to be gained, a similar trend was noted by Caster [7] who reported that increasing of pressure from 105 to 175 kg/cm^2 increased the extraction of oil from cottonseed by only 1%. The increasing of pressure to about 240 kg/cm^2 in the pressing of castor bean, the efficiency increased by only 0.5% compared with the efficiency that be gained at 140.51 kg/cm^2.

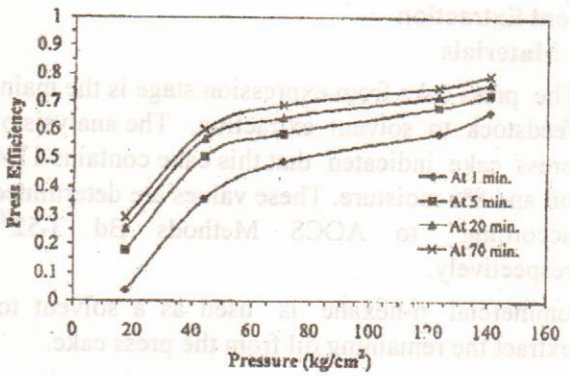

Fig. (4) Effect of Pressure on the Press Efficiency at Various Time For Cold Press at 38 °C

Effect of the Pressing Time on Press Efficiency

Figure (5) shows the effect of pressing time on press efficiency for cold press at different applied pressures 17.56, 43.9, 70.25, 122.9 and 140.5 kg/cm^2. The total increasing in press efficiency in the period between the first minute and 70th minute proportion inversely with applied pressure, the total increasing is 0.258, 0.242, 0.20, 0.16 and 0.13 respectively, while the amount of the extracted oil in the first minute expressed as fraction of total oil extracted in 70 minutes is 12.8, 60, 71, 78 and 83% respectively. These two observations indicated that the effect of time decreases with increasing applied pressure. For higher pressure such as 140.5 kg/cm^2, it may be seen that after a pressing time of 45 minutes, there is a little advantage gained by further pressing, only 0. 6% is extracted after 70 minute.

Hickok [8] recommended that the most economical pressing time of expression of cotton seed is between 45 and 60 minutes because he noticed that only 0.1% oil is extracted after the first hour. In the present investigation the economical time of expression of castor beans is 70 minutes.

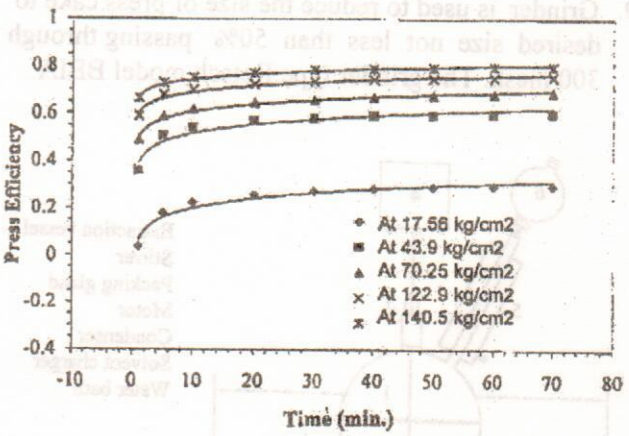

Fig. (5) Effect of the Time on the Press Efficiency at Various Pressures For Cold Press at 38 °C

Effect of Temperature on Press Efficiency and Oil Quality

Figure (6) shows the effect of temperature on the efficiency of extraction. At a selected pressure about 140.25 kg/cm^2 and time of pressing about five minutes, the increasing of temperature of pressing causes decreasing of the viscosity of oil during pressing of seeds, this increases the efficiency by a large amount. The press efficiency for cold press at this pressure and time about 5 minutes is approximately 0.73 while for hot press at 80°C is 0.852, the efficiency increased by 17% for hot pressing over cold press.

Table (3) shows the effect of temperature on the oil quality. Cold press yields oil, which has light color. The value of color of non-refined oil in Lovibond color scale for 1inch cell is 1 red and 5 yellow; the corresponding value in Gardner color scale is 5. The hot press caused darkening of oil, for example, oil produced by hot press at 80°C has value of color in Lovibond color scale 5 red and 50 yellow and the corresponding value in Gardner color scale is 13. The higher temperature of pressing dissolves more colored particles with extracted oil, more concentration of colored particles will complicate refining process.

Another effect of temperature is the increasing of acid value of extracted oil; the acid value for cold press is 2 while for pressing at 80°C is 4.8. High quality castor oil having lower acid value, for example the acid value of medical castor oil is 2.

Extraction at higher temperature causes disintegration of triglycerides of castor oil produced free fatty acid, which is mainly carboxylic acid. Oils having a high acid value required process tends to reduce free fatty acid of such oils, called deacidfication or neutralization, and this additional cost makes the hot press less economic than cold press and on other hand the lower oil acid value produced by cold press which satisfied the specification of medical quality made cold press more suitable than hot press. The oil from cold press is usually grade No.1 while that from hot press is grade No.3 and the analysis of the two oils is given in Table (4), oil of grade No.1 satisfies medical specifications while oil of grade No.3 is satisfied industrial specifications.

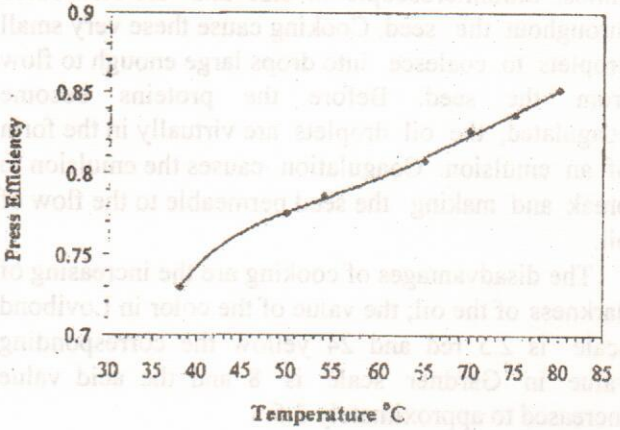

Fig. (6) Effect of Temperature On the Press Efficiency at 140.51 kg/cm^2 and 5 Minutes

Table (3) Effect of the Temperature on Press Efficiency and Oil Quality

Temperature Of Extraction °C	Efficiency	Color, Lovibond scale 1 inch cell		Gardner color scale	Acid value
		Red	Yellow		
38	0.730	1	5	5	2.00
54	0.780	2	20	6	3.60
65	0.808	3	30	9	3.83
70	0.826	4	35	11	4.60
80	0.852	5	50	13	4.80

Table (4) Analysis Of The Three Oils produced By Cold Press, Hot Press And Solvent Extraction

	Property	Method of production		
		Cold press *	Hot press at 80°C *	Solvent extraction at 60°C
1.	Acid value	1.8	5.1	4.4
2.	Hydroxyl value	158	157	156
3.	Iodine value	89.1	88.8	86.7
4.	Peroxide	1	0.7	1.9
5.	Saponification value	180	184.0	180.5
6.	Unsaponfiable matter %	0.4	0.45	0.24
7.	Degree of color in Lovibond scale 1inch cell (Bleached oils)	0.3 red 3 yellow	2.5 red 20 yellow	3 red 30 yellow
8.	Gardner color	3	6	9
9.	Specific gravity, 25°C	0.955	0.960	0.958
10.	Refractive index, 25°C	1.475	1.479	1.48

* Applied pressure is 140-51 kg/cm^2.

Effect of Cooking on Press Efficiency

Figure (7) and (8) show the effect of cooking on press efficiency. The cooking process led to increase the press efficiency by 6% compared with uncooked seeds. At pressure of 140.5kg/cm^2 and pressing time 70 minutes the press efficiency of uncooked seed is 0.798 while for cooked seed is 0.843.

The press efficiency increased after cooking because cooking facilitate separation of oil from proteinaceous materials by coagulation of the proteins, in addition to the oil droplets in the seed are almost ultramicroscopic in size and are distributed throughout the seed. Cooking cause these very small droplets to coalesce into drops large enough to flow from the seed. Before the proteins become coagulated, the oil droplets are virtually in the form of an emulsion. Coagulation causes the emulsion to break and making the seed permeable to the flow of oil.

The disadvantages of cooking are the increasing of darkness of the oil; the value of the color in Lovibond scale is 2.5 red and 24 yellow the corresponding value in Gardner scale is 8 and the acid value increased to approximately 3.5.

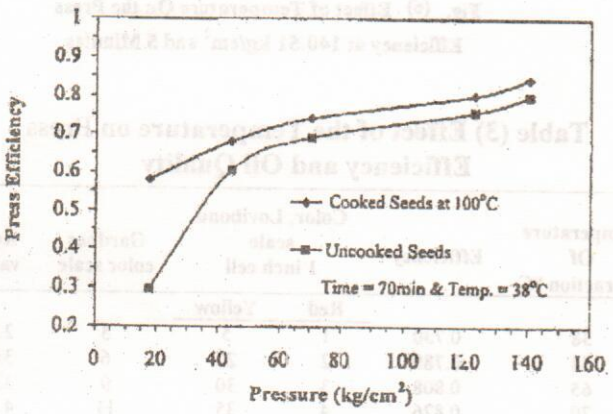

Fig. (7) Effect of Cooking on the Press Efficiency at Various Pressures

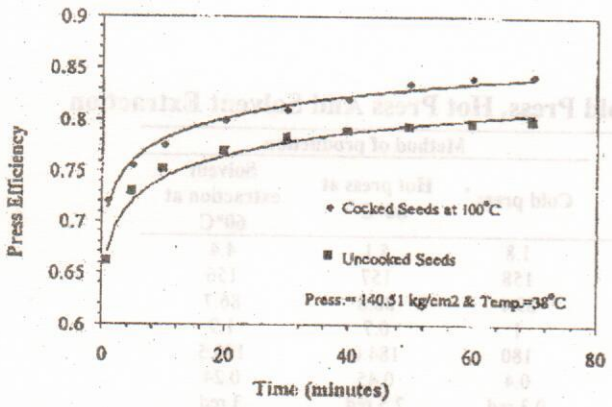

Fig. (8) Effect of the Cooking on the Press Efficiency at Various Times

Pressure-Volume Relations

Figures (9), (10) and (11) show pressure volume relations for cold press, hot press and pressing of cooked seeds respectively. Generally the specific volume based on solid volume decreases with applied pressure, while bulk density increases, after equilibrium condition equation (1) gives a close fit to observed data for experiments on cold press, hot press and pressing of cooked seeds, the expression of castor seeds is found to obey above equation similar to other materials studied by Gurnham [3].

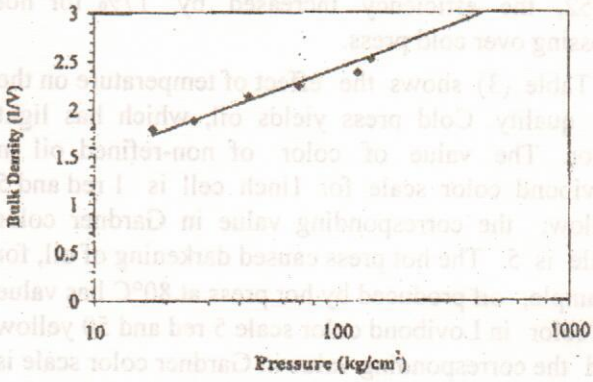

Fig. (9) Pressure-Volume Relation of Cold Press of Castor Seeds at Pressing Temperature of 38°C and Pressing Time of 70 Minutes

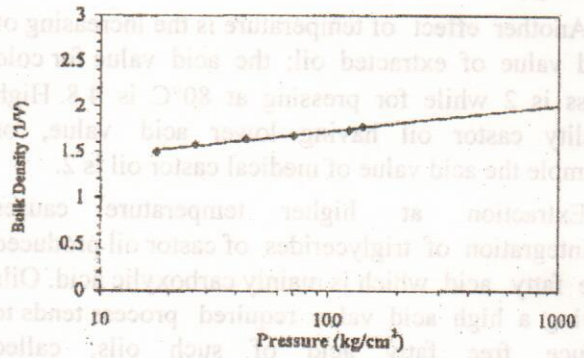

Fig. (10) Pressure-Volume Relation of Hot Press of Castor Seeds at Temperature Range 80-60°C & Pressing Time of 5 Minutes

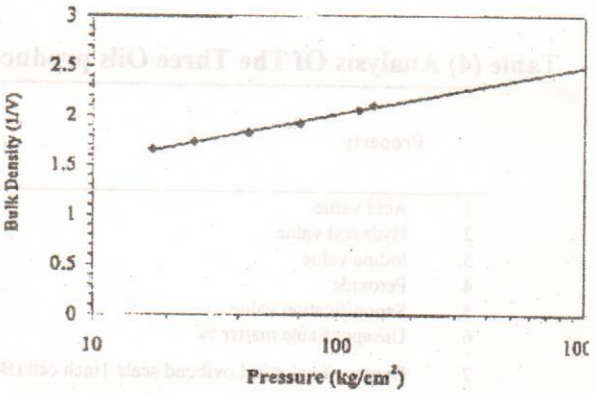

Fig. (11) Pressure-Volume Relation of Pressing of Cooked Seeds at Pressing Temperature of 38°C and Pressing Time of 70 Minutes

Solvent Extraction

The Effect of Time on Residual Oil

The effect of time on residual oil after solvent extraction at different temperatures is shown in figure (12). It is clear from this figure that residual oil decreased with increasing of the time of extraction depending mainly on specific conditions i.e. flaking size, solvent ratio and temperature of extraction. Both flaking size and solvent to feed ratio are fixed during experiment, the flacking size is not less than 50% passing through 300 mesh as recommended by Graci [9] and solvent to feed ratio is fixed at 2:1.

For example at the same temperature of extraction (60°C) the residual oil is found to be 7.10% at time 10 minutes while it is only 4.7% at 60 minutes.

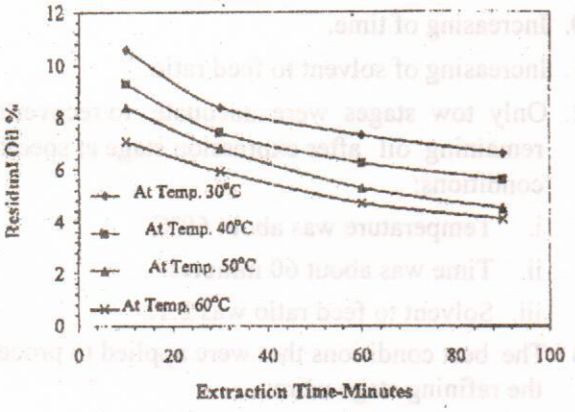

Fig. (12) Effect of Time on the Residual Oil After Solvent Extraction

Effect of Temperature on Residual Oil

Figure (13) shows the effect of temperature on the residual oil after solvent extraction. As observed from this figure the residual oil decreased with increasing temperature for condition of experiments flaking size (not less than 50% passing through 300 mesh) and solvent to feed ratio is 2:1. For example at 30°C and 60 minutes the residual oil is 7.32% while it is 4.700% at temperature 60°C. This effect is due to:

1. The direct effect of temperature on the solubility of the oil in the solvent.
2. The increasing of diffusion rate with increasing of temperature.

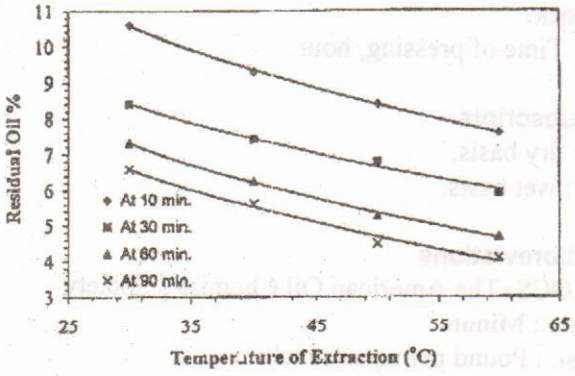

Fig. (13) Effect of Temperature on the Residual Oil After Solvent Extraction

Effect of Solvent to Feed Ratio on Residual Oil

Referring to Figure (14) and at specified condition of temperature (60°C) and time (60 minute) and flaking size (not less than 50% passing through 300 mesh), the variation of solvent to feed ratio from 1:1 to 2:1 is fruitful and the residual oil decreased from 13% to 5.2%. There is little advantage when the ratio increased to 3.5:1, for example the residual oil 4.8% for ratio about 2.5:1 while the residual oil is 4.3% for ratio about 3.5:1.

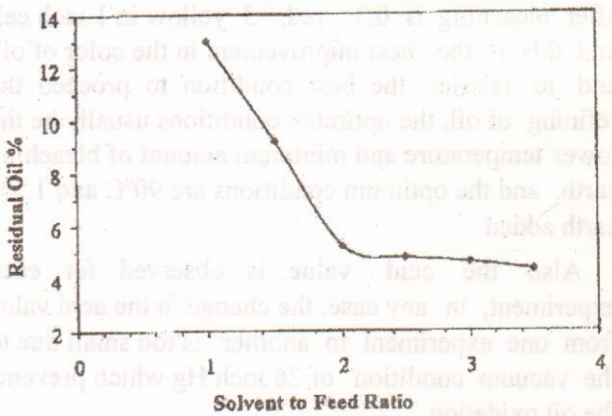

Fig. (14) Effect of Solvent to Feed Ratio on the Residual Oil After Solvent Extraction

Minimum Stages Required for Maximum Oil Extraction From Pressed Cake

The study of the effects of time, temperature and solvent ratio on the extraction of oil from the pressed cake (from expression stage) gives the following condition:

1. The best time of extraction is 60 minutes.
2. The best temperature of extraction is 60°C.
3. The best solvent to feed ratio is 2:1.

At these conditions two washes are carried out to reduce oil content to 1% in the cake after solvent extraction, the residual oil is 5.2% after first wash and 1% after second wash.

Comparison Between Cold Pressing and Solvent Extraction

The crude oil produced by cold press has an acid value less than the oil produced by solvent extraction for example in the cold press the acid value is 2. While for solvent extraction is 4.4.

The other disadvantage of solvent extraction is the increasing of darkness of color of the oil, the color degree in Lovibond scale is 4.4 red and 40 yellow in 1inch cell while for cold press is 1 red and 5 yellow. The best oil is obtained by use of hydraulic press and the obtained oil is grade No.1. Solvent extraction

method are unsuitable for the production of medicinal castor oil (10) and the produced is grade No. 3 and the analysis of the two oils is given in Table (4)

Refining (Bleaching)

The result of the refining stage is given in Table (5). Some experiments have the same improvement in the color of the bleached oil but at difference conditions of temperature and clay earth concentration, for example experiments nos. 2, 4, 5, 9, 10, and 11, the degree of color in Lovibond scale after bleaching is 0.3 red, 3 yellow in 1 inch cell and this is the best improvement in the color of oil, and to restrict the best condition to proceed the refining of oil, the optimum conditions usually be the lower temperature and minimum amount of bleaching earth, and the optimum conditions are 90°C and 1.5% earth added.

Also the acid value is observed for each experiment, in any case, the change in the acid value from one experiment to another is too small due to the vacuum condition of 26 inch Hg which prevents the oil oxidation.

Table (5) Results of Refining (Bleaching) Stage

Run No.	Temp. °C	Earth Concent. %	Color, Lovibond Scale 1 inch cell		Acid value
			Red	Yellow	2.028
1	94.39	0.792	0.5	5	2.035
2	94.39	2.207	0.3	3	2.040
3	115.39	0.792	0.5	5	2.038
4	115.60	2.207	0.3	3	2.017
5	105	1.5	0.3	3	2.0007
6	120	1.5	0.3	3	2.058
7	90	1.5	0.3	3	2.018
8	105	2.5	0.3	3	2.018
9	105	0.5	0.5	5	2.026

Conclusions

1. The oil from cold press was usually grade No.1 and satisfied medical requirement and had acid value about 2 and light color in Lovibond color scale was 1 red, 5 yellow in 1inch cell.
2. In the study of the factor affecting the efficiency of cold press, the press efficiency increased with: -
   i. Increasing of pressing time.
   ii. Increasing of applied pressure.
3. The conditions that had been recommended in expression of castor beans were pressure about 140.5kg/cm^2 and pressing time about 70 minutes, these conditions ensure a good recovery of the oil with efficiency about 0.798.
4. The increasing of applied pressure up to 240 kg/cm^2 had no effect on the press efficiency.
5. The effect of time on press efficiency decreases with applied pressure.
6. The efficiency increased with increasing of the temperature of pressing and the oil produced by hot press was grade No.3 and this oil was improper for medical requirement because its acid value was greater than 2 and had a dark color.
7. The cooking of seeds increased the efficiency of pressing by 6% compared with uncooked seeds.
8. The amount of residual oil after solvent extraction was found to be decreased with:
9. Increasing of temperature of extraction.
10. Increasing of time.
11. Increasing of solvent to feed ratio.
12. Only tow stages were adequate to recover the remaining oil after expression stage at specified conditions:
    i. Temperature was about 60°C.
    ii. Time was about 60 minutes.
    iii. Solvent to feed ratio was 2:1.
13. The best conditions that were applied to proceed the refining stage were:
    i. Time of bleaching was 30 minutes.
    ii. Temperature of bleaching was 90 °C.
    iii. Earth concentration was 1.5% by weight.

Nomenclature

a, b: Constants depending on the nature of the material and condition of expression.
D: Bulk density of solid portion of the system, g/cm^3.
E: Press efficiency.
P: Pressure applied, kg/cm^2 or psi.
V: Specific volume of system based on the solid content, cm^3/g.
W: Oil yield % on dry basis.
W_o: Oil content % of seeds on dry basis.
V: Kinamatic viscosity of oil at the press temperature, stock.
θ: Time of pressing, hour.

Subscripts

d: dry basis.
w: wet basis.

Abbreviations

AOCS: The American Oil Chemist's Society
min.: Minute
psi. : Pound per square inch.
Press: Pressure.

Sp. gr.: Specific gravity.
Temp: Temperature.

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