Laboratory Investigations on the Extraction of Oil from Vegetable Oil-Cakes with Ethanol[1]

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VEGETABLE OIL EXTRACTION from oleaginous materials with ethanol presents some difficulties. The main hurdles are pressure extraction and dilution of the solvent. However the disadvantages are counterbalanced by the better quality of oil and partial elimination of desolventization of the miscella. Not much work is reported on the problem of alcohol extraction of oils. Sato, Inaba, and Kitagawa (11) employed a specially devised circulating extraction system, and from their diagram it appears that the apparatus is not suitable for extraction under pressure. Castor seeds were extracted in a battery of extractors with industrial alcohol by Chatterjee (3) and Chatterjee and Saxena (4). Swisher and Fiero (15) obtained maximum efficiency in extracting castor oil with ethanol. Soybean oil was extracted with ethanol by Sato and Ito (12), Shoji Igarashi (14), and Beckel *et al.* (1, 2). Raghunath Rao (5, 6, 7) used absolute alcohol near its boiling point for the extraction of peanut oil and cottonseed kernels (8). Satyan and Rao (13) extracted cottonseed oil with ethanol and found that the oil was superior in color and refining properties to both expressed oil and petrol-extracted oil. A process was also described (9) for the extraction of oils by using ethanol as the solvent. In the present work a batch extraction-apparatus of 1-kg. capacity was designed and fabricated. The effect of solvent concentration, moisture content, and particle size of the cake on the quality of oil and meal is investigated.

Description and Operation of the Laboratory Solvent-Extraction Unit

The extraction apparatus (10) consisted essentially of five sections (Figure 1): extractor, cooler, separator, pre-heater, and the meal-desolventizing section equipped with a coil condenser and a solvent receiver. The extractor was made of ¼ in. mild steel sheet and consisted of a dished bottom and a cylindrical shell of 6 in. in diam. A ⅛ in. thick perforated plate served as a support for the oil-cake during extraction. The weighed amount of oil-cake (1 kg. on air-dried basis) was charged to the electrically heated extractor, and preheated solvent (3½ liters at 76°C.) was added to the cake near the extraction temperature (77°C.). By closing the extractor top with a flange and the valves V_2, V_4, V_7, V_9 and by opening the rest of the valves, the miscella passed through the cooler and then to the separator, where at 25 to 30°C. most of the extracted oil separated. The lean miscella entered the preheater and then to the extractor at 65.5°C. The recirculation was achieved by natural convection currents. The rate of solvent recirculation was about 1.0 c.c. per second. The pump was

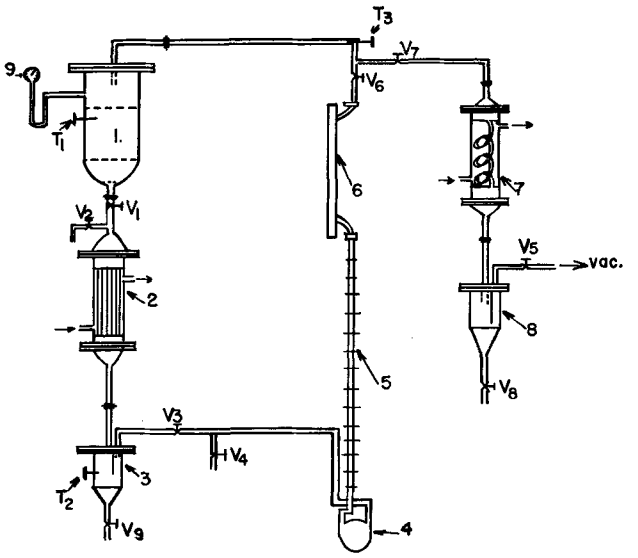

FIG. 1. Laboratory solvent-extraction unit. (1) Extractor, (2) Cooler, (3) Separator, (4) Pump, (5) Pre-heater, (6) Sight-glass, (7) Condenser, (8) Receiver, (9) Pressure and vacuum gauge.

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TABLE I
Extraction of Mowrah Cake

	5–10 Mesh Cake				10–25 Mesh Cake				−25 Mesh Cake			
	Extn. with 98.9% eth.		Extn. with 95.5% eth.		Extn. with 98.9% eth.		Extn. with 95.5% eth.		Extn. with 98.9% eth.		Extn. with 95.5% eth.	
	Air-dried	Oven-dried	Air-dried	Oven-dried	Air-dried	Oven-dried	Air-dried	Oven-dried	Air-dried	Oven-dried	Air-dried	Oven-dried
1. M and V of cake, MFB (%)	9.6	0.1	9.6	0.34	6.19	0.08	6.19	0.98	5.14	0.24	5.14	0.52
2. Oil content of cake, MFB, %	10.05	10.05	10.05	10.05	8.38	8.38	8.38	8.38	12.16	12.16	12.16	12.16
3. F.F.A. of oil in cake, %	7.05	7.05	7.05	7.05	7.46	7.46	7.46	7.46	11.32	11.32	11.32	11.32
4. Extn. time, minutes	125	125	80	125	125	125	125	125	125	125	125	125
5. Extn. temp., °C	80	80	100	100	80	80	100	100	80	80	100	100
6. Extn. press., p.s.i.g.	3	3	20	20	3	3	20	20	3	3	20	20
7. Ethanol concn. after extn. (wt. %)	95.2	98.5	93.2	94.5	94.4	98.6	94.1	95.0	94.8	98.4	94.2	95.4
8. Oil content of meal, MFB (%)	2.26	3.93	5.75	4.89	1.73	2.25	3.95	3.48	6.41	3.76	1.60	1.27
9. F.F.A. of oil in meal, (%)	4.19	2.77	3.94	2.20	1.68	4.80	2.17	1.70	1.45	2.13	5.74	3.53
10. Color extd. oil (¼ in. cell Lovibond)	21.2Y	6.1Y	2.5Y	18.0Y	38.0Y	22.0Y	2.7Y	2.2Y	7.2Y	22.0Y	10.1Y	9.2Y
	4.7R	0.7R	0.1R	1.8R	8.0R	7.8R	0.1R	0.3R	0.7R	5.2R	1.2R	1.1R
11. R.I. of oil at 25°C	1.4703	1.4713	1.4713	1.4708	1.4703	1.4728	1.4718	1.4733	1.4718	1.4703	1.4708	1.4703
12. F.F.A. of extd. oil, %	5.61	1.30	1.75	0.53	11.29	3.17	1.26	1.87	3.89	2.14	1.37	1.42
13. Sap. value of extd. oil	198	200	200	204	196	197	201	215	210	204	196	189
14. I.V. of extd. oil	63.2	61.7	61.3	56.3	55.0	52.0	64.4	50.8	64.2	59.4	60.7	61.8

TABLE II

Extraction of Safflower Cake

	5–10 mesh		10–25 mesh		–25 mesh	
	98.6% eth.	95.6% eth.	98.6% eth.	95.6% eth.	98.6% eth.	95.6% eth.
M and V of cake, MFB %	1.04	1.49	0.52	1.19	3.63	1.40
Oil content of cake, MFB %	6.96	6.96	7.98	7.98	10.73	10.73
F.F.A. of oil in cake, %	3.32	3.32	27.40	27.40	44.20	44.20
Extn. temp., °C.	80.00	90.00	80.00	90.00	80.00	90.00
Concn. of eth. after extn., wt. %	97.00	94.50	98.20	95.00	96.30	94.10
Extn. press., p.s.i.g.	4	10	3	10	4	10
Oil content of meal, %	3.36	2.17	1.05	0.75	1.98	0.68
F.F.A. of oil in meal, %	7.72	5.32	3.38	6.04	4.20	7.46
Color of extd. oil (¼ in. cell, Lovibond)	3.8Y	3.1Y	5.2Y	2.2Y	24.0Y	Oil trapped
	0.8R	0.6R	1.0R	0.4R	6.3R	with the
R.I. of oil at 25°C.	1.4793	1.4808	1.4793	1.4803	1.4798	pptd. non-
F.F.A. of extd. oil, %	0.65	1.06	1.34	1.32	5.22	oily sub-
Sap. value of extd. oil	205	198	200	195	200	stances.
I.V. of extd. oil	137	137	135	141	134	

TABLE III

Extraction of Peanut Cake

	5–10 mesh		10–25 mesh		–25 mesh	
	98.6% eth.	95.6% eth.	98.6% eth.	95.6% eth.	98.6% eth.	95.6% eth.
M and V of cake, MFB %	0.43	0.85	0.92	1.36	0.79	1.11
Oil content of cake, MFB %	6.85	6.85	7.60	7.60	9.59	9.59
F.F.A. of oil in cake, %	1.70	1.70	1.77	1.77	4.48	4.48
Extn. temp., °C.	80	90	80	90	80	90
Extn. press., p.s.i.g.	4	10	4	10	4	10
Concn. of eth. after extn., wt. %	98.40	95.00	97.80	94.50	98.20	95.30
Oil content of meal, %	1.53	1.38	1.07	0.82	2.40	1.35
F.F.A. of oil in meal, %	2.28	4.97	2.40	4.42	2.86	2.84
Color of extd. oil (¼ in. cell, Lovibond)	2.0Y	3.3Y	1.8Y	3.8Y	5.0Y	2.1Y
	0.3R	0.8R	0.2R	1.0R	1.2R	0.3R
R.I. of oil at 25°C.	1.4721	1.4745	1.4741	1.4741	1.4741	1.4741
F.F.A. of extd. oil, %	0.16	0.15	0.30	0.46	0.90	0.28
Sap. value of extd. oil	194	194	194	197	194	197
I.V. of extd. oil	92	92	92	89	86	91

TABLE IV

Extraction of Cottonseed Meats

	Oven-dried meats		Cooked and oven-dried meats	
	98.6% eth.	95.6% eth.	98.6% eth.	95.6% eth.
M and V of cake, MFB %	1.50	1.11	1.20	0.82
Oil content of meats, MFB %	29.66	29.66	24.77	24.77
F.F.A. of oil in meats, %	26.77	26.77	17.22	17.22
Extn. temp., °C.	80	100	80	100
Extn. press., p.s.i.g.	3	20	3	20
Concn. of eth. after extn., wt. %	98.20	94.80	98.00	95.00
Oil content of meal, %	19.82	17.86	15.83	10.36
F.F.A. of oil in meal, %	6.14	9.94	7.65	7.42
Color of extd. oil (¼ in. cell, Lovibond)	152Y	70Y	32Y	51Y
	40R	20R	11R	17R
R.I. of oil at 25°C.	1.4743	1.4743	1.4743	1.4743
F.F.A. of extd. oil, %	5.49	3.89	2.86	4.45
Sap. value of extd. oil	199	198	198	199
I.V. of extd. oil	101	96	102	100

used only to clean the apparatus. Samples of strong miscella from the extractor and the lean miscella from the separator were collected at 5, 10, 15, 20, 30, 45, 60, 80, 100, and 125 min. after the start of the extraction. At the end of the extraction the oil and the miscella were drawn out by opening V_9. The solvent was removed from the meal by desolventizing under vacuum (8 in. Hg.), by closing valves V_1, V_6, V_8 and opening V_7 and V_5. The desolventization was continued for 3 to 4 hrs. until a maximum temperature of 100°C. was attained in the extractor.

Oil-Cake Extraction

Mowrah, safflower, and peanut cakes obtained from the mechanical screw presses were crushed and separated into three particle-size ranges, 5–10, 10–25, and –25 mesh fractions (British Standard Sieve). All three sizes were dried in an air oven for 3 hrs. at 101°C., then extracted with 98.6% and 95.6% (by wt.) ethanol. Also air-dried samples of mowrah cake were extracted to investigate the effect of moisture on the products and the solvent. Experiments were carried out with dried cottonseed meats, also with cooked and dried cottonseed meats with 98.6% and 95.6% ethanol to illustrate the refining properties of ethanol. The results are presented in Tables I to IV.

Discussion

It is observed from Table I that the moisture content of the cake has a great influence on the concentration of ethanol. If moist cakes are employed, the strength is considerably reduced and the solvent needs rectification for further use. The concentration of ethanol is little affected if cakes with moisture content below 1.0% are used. For a constant period of extraction the oil content of the meal decreases with the decrease in the particle-size, also with the increase in the pressure of extraction for any given particle-size. It is curious to note that better quality of oil in respect to color and F.F.A. is obtained with 95.5% ethanol than with 98.9% ethanol though the extraction temperature is higher in the former case than in the latter.

Safflower oil extracted from 5–10 and 10–25 frac-

tion cake had a light color and very low F.F.A. It is also observed from Table II that 95.6% ethanol extraction yielded better quality oil. Because of the presence of large amounts of F.F.A. −25 mesh cake fraction yielded darker oil.

Extraction of peanut cake with ethanol resulted in oil of very good color and very low F.F.A. content (less than 1.0%). As the moisture content of the cakes was low, there was little change in the concentration of the solvent (Table III).

Dried cottonseed meats were extracted with 98.6 and 95.6% ethanol. From Table IV it is contended that the high oil content of the meal was not unexpected since the initial oil content in the meats was quite high (29.66%). However extraction with fresh solvent would reduce the oil content of the meal to the usual levels. The color of the cottonseed oil obtained by 95.6% ethanol was lighter and the F.F.A. content was lower than that of the oil extracted by 98.6% ethanol. Cooked and dried cottonseed meats extraction resulted in higher yields of much lighter oil with lower F.F.A. content even compared to hexane extracted oil. The extraction studies of high F.F.A. content meats illustrated the refining properties and advantages of ethanol as a solvent for oil extraction.

Better quality of oil obtained during extraction with 95.6% ethanol may be attributed to the increased solubility of fatty acids and nonoily substances in ethanol at higher temperatures and lower concentrations.

Rates of Extraction

The difference in the concentration of the strong and lean miscella plotted against time yielded qualitative extraction rates (10). Higher rates of extraction were observed during the extraction of air-dried mowrah cake when compared to oven-dried mowrah cake. The increased extraction rate might result from the dilution of alcohol, which contributes to the increased solubility of nonoily substances. The rates of extraction for 95.6% ethanol were greater than the rate obtained for 98.6% ethanol, independent of the particle-size of the cake being extracted. Decrease in particle-size increased the extraction rates irrespective of the cake nature.

Conclusions

1. Undue dilution of alcohol can be prevented by employing oleaginous materials of moisture content less than 1.0% for ethanol extraction.
2. The residual oil content of the meal depends on the particle size of the cake for a constant period of extraction.
3. Better quality oil in respect to color and F.F.A. is obtained with 95.6% ethanol extraction though the temperature of extraction is higher than the temperature employed with 98.6% ethanol.
4. F.F.A. of the extracted oils is low and within 1.0% for most of the oils, hence a reduction of refining loss. The color of safflower and peanut oils compares with the color of the screw-press oils.
5. In the case of cottonseed meats extraction, the cooking of meats results in a lighter color oil and increases the yield for the same period of extraction. Cottonseed extraction also illustrates the advantages of ethanol as the solvent for oil extraction.

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REFERENCES

1. Beckel, A. C., Belter, P. A., U. S. 2,469,147, May 3, 1949.
2. Beckel, A. C., Belter, P. A., and Smith, A. K., J. Am. Oil Chemists' Soc., *25*, 10–11 (1948).
3. Chatterjee, N. G., J. Indian Chem. Soc., Ind. & News Ed. (India), *2*, 171–177 (1939).
4. Chatterjee, N. G., and Saxena, R. S., *ibid.*, *3*, 23–28 (1940).
5. Raghunatha Rao, Y. K., J. Sci. and Ind. Research, India, *12A*, 373–379 (1953).
6. Raghunatha Rao, Y. K., Soap, India, *6*, No. 11, 12–18 (1953).
7. Raghunatha Rao, Y. K., J. Sci. and Ind. Research, India, *11A*, No. 9, 414–415 (1952).
8. Raghunatha Rao, Y. K., and Krishna Murty, R. G., J. Proc. Oil Technologists Assoc. India, *10*, 30–33 (1954, 1955).
9. Raghunatha Rao, Y. K., Bull. Central Food Technol. Research Inst., Mysore, India, *2*, 155–160 (1953).
10. Ramalingam, K., Ph.D. thesis, *1955*, Osmania University, Hyderabad, India.
11. Sato, M., Inaba, T., and Kitagawa, K., J. Soc. Chem. Ind., Japan, *37*, No. 11, 720–721 (1934).
12. Sato, M., and Ito, C., Brit. 336,273, June 10, 1929.
13. Satyan, B. C., Rao, Y. K. R., Bull. Central Food Technol. Research Inst., Mysore, India, *2*, 305–306 (1953).
14. Shoji Igarashi, J. Soc. Chem. Ind., Japan, *49*, 135–138 (1946).
15. Swisher, M. C., and Fiero, G. W., J. Am. Pharm. Asso., *21*, 579–582 (1932).

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ABSTRACTS . . . . R. A. REINERS, Editor

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• Fats and Oils

FORMATION OF OIL AND ITS IODINE VALUE IN THE DEVELOPING LINSEED. M. N. Hashad (Ain Shams Univ., Egypt), A. K. Ghamrawy, and S. M. El-Sherif. *Ann. Agr. Sci.* (Cairo) **1**, 37–66 (1956). Oil accumulations in the Baladi and Hindi varieties of flax seeds were slow and similar (about 0.5% daily) during the first three weeks after flowering. Subsequent rapid rates extended to the thirty-sixth and forty-second day after flowering for Baladi and Hindi, respectively, with resultant oil contents of 34.12 and 41.74%, respectively. Maximum amounts of 37.72 and 43.72% were reached after 48 and 51 days in Baladi and Hindi varieties, a period midway between flowering and ripening. The iodine value increased during the entire period but at a lower rate toward the end, the greatest rate of increase occurring after the oil content was the maximum value which suggests unsaturated acid formation in the late period. The iodine value increased during field ripening after harvesting in Hindi but not in Baladi. The crop was ready for harvesting for optimum fiber production at the maximum oil content stage, but oil quality improvement continued to the fifty-seventh day for Baladi and Hindi. (*C.A.* **52**, 18698)

THE OCCURRENCE OF RANCIDITY IN SALT HERRING. R. Marcuse. *Fette, Seifen, Anstrichmittel* **60**, 482–7 (1958). The tendency of salt herring to turn rancid during storage can be estimated by determining the oxygen uptake in the presence of oxidation