Direct Solvent-Extraction of Castor Beans Yields High Grade Oil[1]

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CASTOR OIL is a very important raw material for many of our essential civilian and military needs. The oil and the chemical intermediates prepared from it are used in the production of such industrial products as protective coatings, synthetic textiles, plasticizers, jet engine lubricants, hydraulic fluids, soaps and detergents, resins, waxes, cosmetics, and a variety of valuable derived products (1, 2).

The United States depends almost entirely on foreign supply for this strategic oil. However recent research developments by the United States Department of Agriculture, in cooperation with experiment stations and several industrial firms, have resulted in new high-yielding varieties of castor beans, and machines to harvest them (1, 2). These improvements have made production of this crop particularly attractive to farmers as a replacement for crops which are in surplus supply or on which there are acreage restrictions. The U. S. production for 1958 is estimated at about 30,000 short tons, which is about 2.5 times the 1957 crop and 13 times that for 1956 (2). This volume of castor beans should be sufficient to satisfy about one-fifth of our domestic requirements of castor oil.

Castor beans are conventionally processed into oil and meal by single- or double-pressing, or by pressing followed by batch solvent-extraction. These methods produce two or more grades of oil, and usually only the first pressing yields No. 1 Grade Oil. As far as is known, there are no commercial processes in use today for solvent-extracting castor beans without prepressing.

A newly introduced solvent-extraction process, Filtration-Extraction, has been employed commercially directly to extract cottonseed, soybeans, rice bran, and sunflower seeds (3, 14). It has also been employed successfully on a bench- or pilot-plant scale to peanuts (4), flaxseed (5), rice bran (6), sesame (7), and to milo germ, safflower, and press-cakes. The over-all process is similar for all oleaginous crops with only minor modifications, depending on differences in the inherent characteristics of each specific material. It comprises the steps of comminution, mild moist cooking, crisping by evaporative cooling, slurrying or soaking in solvent to dissolve the oil, separation of the oil miscella by filtration and washing, and final desolventization of the oil and meal products. It derives its name from the fact that the central unit is a continuous horizontal rotary vacuum filter.

The purpose of this investigation was to apply filtration-extraction on a bench scale to the processing of castor beans. Criteria used was the same as those for evaluating other oil-bearing materials except that it was essential that the crude oil product, after bleaching, meet all chemical and physical requirements for No. 1 grade oil as defined by Federal Specifications. Data are presented to show that either decorticated or whole beans (containing all of the seed coat) can be efficiently processed on a bench-scale to yield a defatted meal of satisfactorily low residual lipids content, and a single crude oil product which, after bleaching, is of No. 1 grade and color.

Raw Materials and Equipment

The castor beans used in this investigation were obtained from commercial processors and were representative of current oil mill receipts. Of the five lots, four were domestically produced and one was of Brazilian origin. No information was obtainable as to varieties. Two, D and E, were whole beans (hulled only), and three A, B, and C, had been partially decorticated by the processors. The respective contents of seed coat along with other data are given in Table I. All of the lots had been thoroughly cleaned, and all contained a nominally small percentage of cracked and coat-free kernels. Each lot was thoroughly mixed to insure uniformity and was stored under refrigeration at 40°F.

Decortication of the whole beans was done with 12-in.-diameter single-pass corrugated rolls and an 18-in.-wide aspirated shaker screen.

TABLE I
Characteristics of Five Lots of Castor Beans

Lot No.	A	B	C	D	E
Domestic/foreign	D	F	D	D	D
Decorticated	Yes	Yes	Yes	No	No
Coat, %[a]	5.0	8.0	16.0	25.4	24.2
Wt. per 100 seeds, g.	......	......	......	30.3	30.9
Moisture, %	4.4	4.8	4.4	4.8	6.8

[a] Includes minor percentages of hulls.

Size-reduction of whole beans prior to flaking was done with a laboratory-size Russwin food chopper equipped with a 12-tooth blade.

For flaking, a set of 12-in.-diameter single-pass smooth rolls rotating at 200 r.p.m. was used.

Cooking was conducted in a 15-lb.-capacity vapor-type, bottom-discharge, steam-jacketed Loomis mixer (13), of all-steel construction. Some of the cooks were made in a 1-lb.-capacity stainless steel vessel (8), equipped with vertical agitator, and similar in design to a single stack of a standard five-high oil mill cooker.

The 5¼-in.-diameter (500-g. capacity) bench-scale filter test unit previously described by Graci *et al.* (9), or a 1¾-in. diameter (120-g. capacity) unit was used to evaluate the filtration-extraction characteristics of the cooked materials.

Experimental

Forty-two experimental bench-scale runs were conducted on the different lots of castor beans to establish

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satisfactory processing conditions for preparation and conditioning of the materials, and for their extraction.

Preparation variables investigated were: degree of decortication of the beans; method of comminution; particle-size reduction prior to cooking; temperature range, time cycle, and moisture levels during the cooking operation; addition of sodium hydroxide in the cooking operation for reducing acid value of the oil and improving its color; and re-rolling *versus* not re-rolling prior to extraction.

Extraction variables included time duration of slurrying, solvent-to-meats ratio, and heptane *versus* hexane as extraction solvents.

The above factors were evaluated against extractibility measured by percentage content of residual lipids in extracted meal; mass velocity (rate of filtration of liquids through the filter bed, expressed as pounds per hour per square foot of filter-screen area); and against quality of oil produced as determined by physical and chemical analysis. The ranges of the variables investigated were based on past experience in processing a wide variety of oil-bearing materials.

Described below are the detailed operational procedures found adequate in this study. In Table II are given the processing conditions (with exceptions noted) which were applied to each of the five lots of partially decorticated and undecorticated beans in a series of 11 typical runs. Differences in the experiments are as follows. Four were on whole beans, and seven on decorticated beans. Experiments 5 through 11, on beans D and E, were designed to show the effect of unremoved seed coat on processability, extraction, and oil quality. Numbers 8 and 10 were simple extractions of flaked raw beans D and E to produce virgin oils for quality comparison. Experiments 2, 3, 5, and 6, on beans B and D, were made to show the effects of alkali addition during cooking.

I. *Preparation.* Preparation of the beans for extraction included the steps of decortication (where required), size reduction, cooking, crisping, and rerolling.

Decortication. Satisfactory decortication of the whole beans was accomplished by passing them once through the cracking rolls set at a clearance of 0.16 in. The procedure was effective in breaking off the coat shells from about 80% of the beans, with practically no shattering of the meats. The separated coat particles were then removed by aspiration on the shaker screen and contained no detectable fragments of entrained meats. The above steps were employed to decorticate whole beans D and E to a coat content of about 5% for use in Experiments 7, 8, and 11.

Size Reduction. The procedure found best for size reduction of decorticated beans (A, B, and C) was to pass them once through the smooth rolls set at about .04–.06-in. clearance and then to repass them at a rolls clearance of .003–.004 in., which yielded flake particles in the thickness range of .010–.014 in. The whole beans (D, E) were size-reduced by passing them once through the laboratory-size food chopper, followed by passing once through the smooth rolls set at .003–.004-in. clearance.

Cooking and Crisping. In the cooking of flaked castor beans for filtration-extraction, as with other oleaginous materials, the cooker vessel was operated in a manner which simulated the operation of a conventional stack-type of cooker, *i.e.*, the cooking cycle was carried out in the following successive stages: material preheating, moisture addition, cooking, and partial removal of moisture or drying.

In the first stage the material was preheated in the mixer to 170°F. in a period of about 8 min. Next the required amount of water was injected over a period of about 4 min., during which the temperature was elevated to 212–215°F. During the next stage these temperature and moisture levels were maintained under mild reflux conditions for about 12 min. In the final stage the charge was dried uniformly to about 7–8% moisture content over a period of about 12 min., by allowing the required amount of water to evaporate into a condenser and to collect in a calibrated receiver. Total cooking cycle was about 36–40 min. The hot, moist material was then discharged from the cooker and was cooled and crisped by shaking it through a screen of ¼-in. hardware cloth.

In two of the cooks, Experiments 3 and 6, an amount of NaOH equivalent to 1% of the weight of the charge was added to the water used for adjusting the moisture content.

Re-rolling. The crisped material was passed once through the smooth rolls set at .003–.004-in. clearance.

II. *Extraction.* The bench-scale test unit used to determine the extraction characteristics of each of the prepared materials is specifically designed to simulate closely the operating steps and conditions for slurrying, vacuum filtration, countercurrent washing blow-back, and vacuum drainage to which the prepared material would be subjected on a pilot-plant or commercial-size of rotary, horizontal, vacuum filter. It has been previously pointed out (11) that results obtained with the unit with a wide variety of oil-bearing materials have been correlated closely with pilot-plant-scale performance, and in the case of cottonseed (3) and soybeans (11), have been readily translatable to commercial scale.

Conditions employed for the extraction of cooked and crisped castor bean flakes comprised the following: solvent-to-meats ratio of 2 to 1 by weight, sufficient to yield a miscella of about 25% oil concentration; slurrying time, 45 min.; three washes; extraction temperature (slurrying and the washes) slightly below the solvent boiling-point; cake thickness, 1½–2 in.; filter screen, 80 × 80 mesh; vacuum 2–5 in. of mercury. The solvents were commercial hexane and commercial heptane.

For Experiments 8 and 10, in which the flaked meats were not cooked, the bench-scale unit was used strictly to extract the oil for comparison with that produced from cooked meats.

The solvent-damp extracted meals from all of the experiments were desolventized by heating in an enclosed vessel under slight vacuum (extreme precaution was exercised in the handling of solvent-extracted castor pomace because of its highly allergenic properties). The solvent-free meals were analyzed by the A.O.C.S. Official and Tentative Methods for moisture, residual lipids, and for pH values, and some were analyzed for protein content (Nx 6.25) and for protein solubility in sodium hydroxide at pH 8.5. In four of the experiments residual solvent content of the solvent-damp meals was determined.

The crude oils were recovered from their respective miscella under controlled conditions of temperature and vacuum. The miscella from each experiment was partially concentrated by evaporation at not more than 180°F. and under 25 in. of mercury vacuum. It was then filtered and was further desolventized with sparging steam at 180°F. for 30 min. under 29.7 in. of mercury vacuum. To insure thorough clarification the oils were refiltered with diatomaceous earth. Color determinations of the crude and the bleached oils were made, using a recently standardized set of Gardner 1953 color standards.

The clarified oils were bleached at 200°F. under 25 in. of mercury vacuum by stirring for 30 min. with a mixture of 2% Filtrol and 0.5% of Darco S-51 carbon, with final filtration through close-textured filter paper.

The final bleached oils were assayed by the laboratories of two industrial castor oil firms for hydroxyl and iodine values, viscosity, refractive index, and Gardner color, in accordance with the A.O.C.S. Official and Tentative Methods. Grading was in accordance with Federal Specifications JJJ-C-86 for Castor Oil, Technical.

Results and Discussion

In Table II are given the operating conditions, filtration-extraction data, and analyses of the meal and oil products for the 11 experiments reported on the five lots of beans. The decorticated beans varied in seed coat content between 5 and 16%.

The results show that castor beans, whether whole or decorticated, can be flaked, cooked, crisped, and re-rolled, as described herein, to yield materials which can be efficiently extracted at sufficiently high filtration rates to acceptably low residual lipid contents, and oil products of high quality.

Decortication. The procedure employed herein to decorticate whole beans proved very satisfactory for reducing the coat content from about 25% to about 5%. Practically no shattering of the whole meats occurred in the cracking rolls, and the aspirated coat fraction contained no detectable fragments of entrained meats. Moreover it was observed that the only particles of coat remaining in the aspirated meats fraction were those still attached to whole meats; and since they were cracked and loosely attached, it was apparent that most could be separated readily by slight impact (with a machine such as an Entoleter), followed by aspiration. Thus the method shows promise of being able to achieve almost complete decortication of castor beans with a minimum loss of fine meats in the removed coat stream. The above features coupled with the advantages of simplicity, the use of standard equipment units, and the high capacities attainable should warrant consideration as to its commercial feasibility.

Size Reduction. The use of cracking rolls and slow-speed, grinding mills preparatory to or for replacement of flaking rolls did not prove practical for decorticated beans because of excessive buttering. Whole beans however, because of their high coat content, can be found in a slow-speed mill or similar comminuting equipment to produce a material of satisfactory nonoily consistency that can be efficiently handled by flaking rolls.

In the flaking of raw beans it is pointed out that while the thickness recommended is in the range .010–.012 in., thicknesses of 0.014 in. and higher yielded final extracted meals having lipid contents as low as 0.6%. Since optimum flake thickness appears to vary from bean to bean, it would be to the interest of the oil mill operator, from the standpoints of rolls capacity and power requirements, to operate his rolling equipment at maximum clearances that would be consistent with acceptable extraction.

Cooking and Crisping. The cooking operation serves two principal functions. First, the action of heat and moisture renders the oil more easily removable in the subsequent extraction-phase of the process. Secondly, under the proper combined conditions of moisture, temperature, and time, the fine particles agglomerate into larger ones. These effects, combined with partial evaporative cooling of the hot cooked meats, yields a crisp material which is characteristically granular and relatively incompressible and, even after re-

TABLE II

Bench-Scale Filtration-Extraction Data on Decorticated and Undecorticated Castor Beans [a]

Beans, Lot No.	A	B		C	D				E		
Beans, Coat, %	5.0	8.0	8.0	16.0	25.4	25.4	5.0	5.0	24.2	24.2	5.3
Experiment No.	1	2	3	4	5	6	7	8	9	10	11
Cooking:											
Yes/no	Yes	Yes	Yes	Yes	Yes	Yes	Yes	No	Yes	No	Yes
Max. moisture, %	12.0	12.0	16.0	12.0	12.0	15.0	12.0	....	12.0	....	12.0
NaOH added, %[b]	0	0	1.0	0	0	1.0	0	0	0	0	0
Filtration-extraction:											
Solvent	Hept.	Hex.	Hex.	Hex.	Hex.	Hex.	Hex.	Hex.	Hex.	Hex.	Hex.
Solvent-to-meats ratio	2.0	2.0	2.0	2.0	2.0	2.0	2.0	3.5	2.0	3.5	2.0
Slurrying, min	30	60	60	45	45	45	45	90	45	90	45
Slurrying, temp. °F	180	135	140	150	145	145	150	150	145	150	150
Vacuum, in. Hg	4	5	4	5	2	4	4	15	4	12	1
Marc, % solvent	....	40	....	....	37	....	48	....	38	....	....
Mass velocity, lbs./sq.ft./hr	2800	2400	4800	3700	4900	2800	2700	30	4800	35	1800
Extracted meal, desolventized:											
Lipids, %	1.5	1.0	0.8	3.7	1.0	0.6	1.5	....	1.0	0.5	1.8
Moisture, %	8.8	....	....	....	9.7	....	10.5	....	10.3	....	11.1
pH	6.1	....	9.5	6.5	6.8	9.1	6.1	....	....	7.2	7.1
Extracted oil, desolventized:											
Crude, Gardner color	5−	5+	5+	4	3	5−	3−	3	3	2	2
Bleached, analysis:											
Gardner color	2+	2+	2+	1+	1+	2−	1+	2−	1+	1+	1+
Acid value	1.4	4.3	2.1	1.1	0.9	1.1	0.7	2.0	0.8	2.3	0.5
Visc. GH at 25°C	T+¼	....	....	....	U	....	U+¼	....	U	....	U−¼
Refr. Index at 25°C	1.4761	1.4770	1.4770	1.4770	1.4770	....	1.4770	1.4771	1.4771	1.4771	1.4770
Iodine value	85.0	85.9	84.9	84.8	84.8	84.6	85.3	85.5	84.8	86.0	84.7
Hydroxyl value	171.3	157.5	158.0	158.0	161.4	161.0	161.9	160.0	161.2	160.0	160.7

[a] Additional operational data included under Experimental.
[b] Percentage by weight of cooked meats.

rolling, possesses properties favorable for rapid miscella filtration and efficient extraction of the oil.

The over-all data on the cooking of flaked beans indicate that the particular conditions evaluated for each of the important variables are not critical and can be varied within fairly wide limits without adversely affecting processing performance. It was observed that maximum moisture content during cooking can range from 10 to 16%; final moisture content after drying, from about 5 to 10%; cooking cycle from 30 to 60 min. or longer; and temperatures can be as high as 235°F., which may be required in actual mill operation sufficiently to dry the cooked material. The use of moisture levels below about 10%, and over-drying below 4%, resulted in somewhat better extraction but reduced mass velocity significantly because of excessive disintegration of the material into fine particles during extraction.

Experiments 3 and 6 on beans B and D indicate that, with beans containing oil of high acid value, the acid value was reduced appreciably (4.3 to 2.1) by the addition of 1% of NaOH in the cooking operation whereas the reduction was negligible with beans of low acid value. In additional cooks with beans B, D, and E it was observed that the use of a lower percentage of NaOH (0.25%) not only did not reduce the oil acid values but appeared to have an unexplained effect of increasing the acid values. It is apparent from the data that the addition of as much as 1% of NaOH in cooking decorticated or undecorticated beans did not have any measurable deleterious effect upon extraction performance or on color and other quality characteristics of the oil products.

Re-rolling. The purpose of re-rolling after cooking and crisping is to insure oil extraction down to 1%. This step is usually required only when the material being extracted contains more than about 35% oil. Re-rolling was found necessary in the case of castor beans in order to flatten out small agglomerates formed in cooking and any associated pieces of curled hulls or coat which entrap particles of cooked meats. The material after re-rolling was relatively nonoily and relatively free-flowing.

In Table III is shown the effect of re-rolling after cooking, upon mass velocity and residual lipids in extracted meal, for both decorticated and undecorticated beans. The effect upon residual lipids is appreciable, and mass velocities are reduced significantly because of reduction in average particle-size.

TABLE III
Effects of Re-rolling After Cooking and Crisping

Bean, Lot No.	B		E	
Coat, %	5.0		24.2	
Re-rolling [a]	No	Yes	No	Yes
Lipids in meal, %	5.9	1.4	4.2	1.0
Mass velocity	6000+	3800	6000+	3700

[a] One pass through one-high smooth rolls set at .004-in. clearance.

Extraction. Residual lipids content in the extracted meals produced from the cooked materials (except Experiment 4) ranged between 0.6 and 1.8% and averaged 1.1%. Values for mass velocity varied between 1,800 and 4,900 and averaged 3,400, which would be considered suitably high for commercial-scale application. Moisture contents were in the high range desirable for reducing meal dustiness.

Protein contents of the meal for the whole and decorticated beans averaged about 30% and 55%, respectively. Percentage of protein solubility in NaOH at pH 8.5 averaged about 38%, which is lower than for meals produced by filtration-extraction of other materials. Protein solubility of the meals from the cooks with added NaOH averaged 17%.

Experiments 8 and 10 show that, for the extraction of uncooked flakes, a relatively high solvent ratio and a longer slurrying period are required to reduce lipids satisfactorily and that the rate of filtration is impractically low.

Hexane gave excellent results as an extraction solvent and was selected over heptane for the reasons that it dissolves the oil readily at a lower temperature, has a lower boiling point and a narrow boiling range, and can be more readily and completely removed from the oil and meal products. Other advantages are lower cost and more ready availability. However Experiment 1 shows that heptane is also suitable.

Oil Quality. Analytical data on the bleached oils indicate that all were of about equal high quality and closely comparable to typical No. 1 grade hydraulic-pressed and bleached castor oil. They were also judged to be approximately equal in quality to the bleached virgin oils extracted from the respective raw flakes. The bleached oil from bean B was evaluated by one commercial firm for the manufacture of Rilsan (Polyamide 11, Nylon type of product) by the Organico process (12) and was reported to be of satisfactory quality for this purpose. The oils have not been evaluated to establish their suitability for the manufacture of light-colored dehydrated oil and of esters, urethane, and other derivatives.

Whole versus Decorticated Beans. Experiments 5 through 11 afford a good comparison of the over-all processibility of whole beans D and E *versus* that for the same beans after removal of about 80% of the seed coat. The results show that both materials yielded low residual lipids in meal at acceptably high filtration rates and that the extraction efficiency and filtration rates were somewhat higher for the whole beans. The data also indicate definitely that the presence of the entire seed coat had little or no adverse effect on the color and other quality characteristics of the crude and bleached oils. Further it is significant to note that in the entire study, in not a single instance, with either whole or partially decorticated beans, did the crude oil, after bleaching, test darker in color than No. 3 Gardner. Since color of castor oil is generally believed to be closely associated with the content of coat in the beans, that No. 1 grade oil can be produced from whole (undecorticated) beans is considered an unusual result and may be attributed to the fact that moist cooking, as employed herein, either alters the chemical composition of the pigment bodies or renders them less soluble in the solvent.

Economic advantages of by-passing the decortication step would be elimination of the decortication equipment and its operation expense, and elimination of the loss of meats (oil and nitrogen) entrained in the seed coat that is removed. Disadvantages would be reduced mill capacity on account of the larger bulk of material to process and the need for screening equipment to remove the excess hulls from the meal in order to raise its protein content.

While the development of an efficient process for continuous direct solvent-extraction of castor beans would be a significant contribution to the industry, it is important to emphasize that the problem of the toxicity and allergenic properties of castor pomace remains a serious deterrent to the rapid expansion of castor bean production in the United States. A number of research groups are at work in attempting to develop feasible methods to destroy or inactivate harmful constituents, particularly the allergen. It is the consensus among castor bean processors that the industry needs a practical method for completely detoxifying the ricin and inactivating the allergen without affecting the quality of the oil, one which will not seriously degrade the meal quality for industrial uses.

Summary and Conclusions

Data on five lots of castor beans are presented to show that the filtration-extraction process, now in commercial use for the continuous direct extraction of cottonseed, soybeans, sunflower seed, and other oil-bearing crops, can be successfully applied on a bench scale to whole or decorticated castor beans to yield a meal product with residual lipids content of 1% or less and high quality crude oil which either meets specifications for No. 1 grade oil or can be bleached to No. 1 grade. Since the color of castor oil is generally believed to be closely associated with the coat content of the beans, that No. 1 grade oil was produced from whole (undecorticated) castor beans is considered an unusual result and may be attributed to the fact that moist cooking, as employed herein, either alters the chemical composition of the pigment bodies or renders them less soluble in the solvent. The oils compare closely in color and in analysis, with currently produced commercial oils, but have not been evaluated to establish their suitability for the manufacture of light-colored dehydrated oil, esters, urethane, and other derivatives.

Conditions found acceptable for preparation are size reduction and flaking to .010–.012-in. thickness, preheating to 170°F., addition of moisture to 12–14%, cooking at 212–215°F. for 20 min., drying to a moisture content of 7–8%, crisping to a temperature of 130°F. and a final moisture content of 5–7%, and re-rolling through rolls set at .003–.004-in. clearance. Conditions suitable for extraction are slurrying for 45 min., hexane-to-meats ratio of 2 to 1, three washes, cake thickness of 2 in., and temperature of extraction of about 140°F. The above-prescribed conditions were found to be adequate within the limitations of the study and are not to be considered as optimum.

The process utilizes standard equipment units to carry out the operations of size reduction, cooking and crisping, re-rolling, extraction by slurrying and washing on a continuous horizontal rotary vacuum filter, and oil and meal desolventization.

Advantages of filtration-extraction for castor beans are that it eliminates the need for pressing prior to extraction and that the single oil product produced is of No. 1 grade. An additional feature is that the ability of the process to handle whole beans eliminates the decortication step as well as the loss of meats and oils inherent in the operation of removing the bulk of the seed coat.

Based on the close correlation obtained to date with a wide variety of vegetable oil-bearing materials between bench- and industrial-scales of operation, the results reported herein indicate that little or no difficulty should be anticipated in the commercial application of filtration-extraction to castor beans.

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