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Determination of Moisture, Oil, and Free Fatty Acids in Castor Beans

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IMPORTED castor beans have normally been purchased without reference to variations in oil, moisture and free fatty acids. Consequently official methods for trading and processing purposes have not been developed and standardized. The Commodity Credit Corporation has undertaken a domestic production and procurement program for castor beans in order to meet the critical shortage of castor oil. In connection with this program, standardized analytical methods are required to provide a basis for marketing since the oil content of the beans may vary, due to a number of causes, including varietal and environmental effects and inclusion of immature and broken beans as a result of mechanical harvesting and hulling.

Castor beans have several characteristics which must be considered in selecting suitable methods for analyzing them. The seed coat is tough and fibrous, and the oil is easily expressed so that the preparation of the beans for analysis requires considerable care. In addition, castor oil is unique among vegetable oils in respect to its solubility characteristics. It dissolves in its own volume of petroleum ether and is soluble in alcohols, chloroform, and carbon tetrachloride. Since castor beans are so different from other oil-bearing seed, several individuals who have had considerable experience in analyzing them were kind enough to supply the details of the procedures used by their organizations (2, 3, 8). Based on these procedures a systematic investigation of the determination of moisture, oil, and free fatty acids in castor beans was undertaken.

Samples and Their Preparation

A sample of castor beans of the Conner variety which was representative of regular commercial shipments was obtained from the Production and Marketing Administration. The field analysis supplied with the sample was as follows: foreign material 1.5%, unhulled beans 1.0%, cracked and broken beans 8.8%. The average weight per 100 beans was 32.41 g. In accordance with the recommendations of others (3, 8) the beans were hand-picked to remove foreign matter and were ground in a food chopper equipped with a 12-tooth blade as specified in A.O.C.S. Official Method Aa 6-38 (1). The use of this mill gave satisfactory preparation of the sample for analysis when the beans were fed slowly so as to avoid expression of oil. Attempts to prepare the sample by use of the Wiley mill[2] or the Henry nut slicer[2] were unsuccessful. The best procedure found for mixing the ground sample was to roll it gently on a glazed paper or in a large glass jar.

A sample of commercial castor pomace was obtained through the courtesy of the Baker Castor Oil Company. It was ground through a Wiley mill equipped with a 1-mm. screen.

No attempt was made to condition either the beans or the pomace to moisture equilibrium with the atmosphere prior to grinding. Moisture values on the whole and ground beans as determined by the Karl Fischer method were essentially the same, while the higher value on the ground as compared to the unground pomace indicated an increase in moisture on grinding. The application of the Karl Fischer method to castor beans is not considered practical as a standardized method because of the difficulty of obtaining complete extraction of water with methanol and avoiding interference from atmospheric moisture.

Moisture and Volatile Matter

Curves for the loss in weight on drying *vs.* time were prepared from data obtained on the ground beans and from ground and unground pomace by heating 5-g. samples contained in official A.O.C.S. moisture dishes at 101°C. and 130°C. in a forced-draft oven. Similar curves were prepared from data obtained using 50-g. samples of whole beans contained in metal dishes, 3¾ in. in diameter and 1 in. deep, and having tight fit-

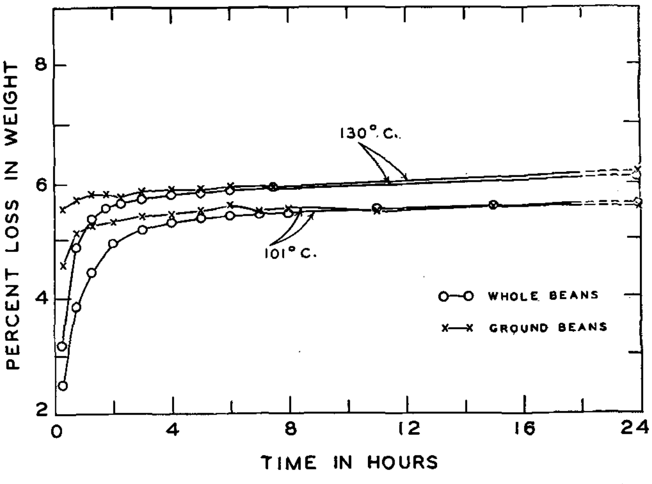

FIG. 1. Loss in weight on oven-drying whole and ground castor beans.

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[2] Mention of the names of firms or trade products does not imply that they are endorsed or recommended by the U. S. Department of Agriculture over other firms or similar products not mentioned.

ting covers. The oven was equipped with a torsion balance sensitive to 5 mg. and a mechanism by which the samples could be weighed at selected time intervals without removing them from the oven. Handling of the samples and adjustment of the oven were exactly as described previously for other investigations on the determination of moisture and volatile matter (4, 5, 7).

The curves for the loss in weight during oven-drying for ground and unground castor beans for temperatures of 101°C. and 130°C. are shown graphically in Figure 1. Similar curves for castor pomace are plotted in Figure 2. The individual points shown are averages of duplicate analyses agreeing within 0.1%. Although the ground samples show considerably higher initial losses at both temperatures, the approach to constant weight occurs in whole beans almost as rapidly as in the ground sample. As in the case of soybeans (6) and sesame (7) moisture apparently cannot be removed completely at 101°C. in the forced-draft oven even with prolonged heating since the values for both ground and unground castor beans and pomace at 130°C. are 0.4-0.5% higher than those obtained at 101°C.

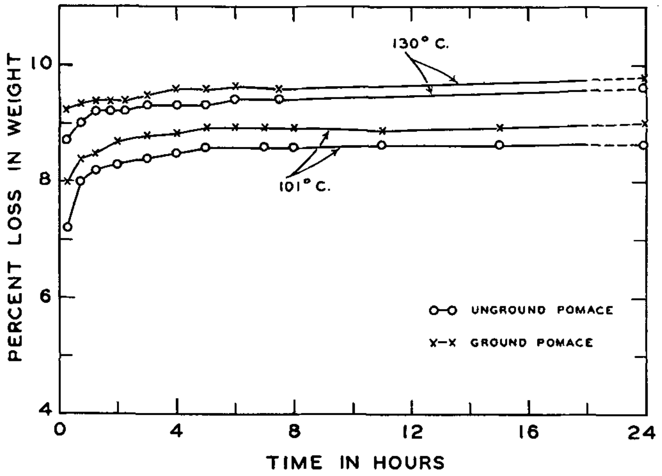

FIG. 2. Loss in weight on oven-drying castor pomace.

The data indicate that the most satisfactory value for moisture and volatile matter is obtained by heating 50-g. samples of whole beans for 4 hours or 5-g. samples of ground beans or pomace for 2 hours at 130°C. in a forced draft oven.

Determination of Oil

Carbon tetrachloride was selected as solvent for the extraction of oil from castor beans based on the following considerations. It readily extracts the oil from castor beans giving a clear, light-colored extract while alcohols give cloudy dark extracts containing nonoil material. Yields of oil on extraction have been found (8) to be the same as with chloroform or anhydrous ethyl ether and higher than with petroleum ether. Carbon tetrachloride is readily available as a pure solvent and offers no fire or explosion hazard. Further it has been used satisfactorily by others (2, 3).

To establish conditions required for the removal of solvent after the extraction, 2.5-g. samples of U.S.P. castor oil were weighed in tared 100-ml. extraction flasks, 50 ml. of carbon tetrachloride added, and the flasks heated for varying periods of time on the steam bath or in the forced draft oven at 101° or 130°C., after which the flasks were again weighed. The last portion of the solvent is difficult to remove and cannot be detected by odor. No detectable odor of carbon tetrachloride could be observed in samples of oil containing as much as 50 mg. of residual solvent. When heated on the steam bath, approximately 24 hours were required for complete removal of the solvent. While castor oil samples could be heated for 24 hours at 101°C. without change in weight, heating for even 2 hours at 130°C. resulted in some loss in weight, indicating a slight decomposition of the oil. It would therefore seem undesirable to heat the extracts at temperatures much above 101°C.

When the flasks were heated for an hour on the steam bath after boiling ceased and then for 2 additional hours in the forced draft oven at 101°C., complete removal of solvent was achieved. These conditions were found to be adequate for the removal of solvent from extracted oil in most cases. However since several extracted oil samples were found to contain residual solvent after this treatment, it was found necessary to continue the heating in the forced draft oven until samples showed no further loss in weight.

Preliminary experiments on the extraction of 5-g. samples of ground castor beans indicated the necessity of regrinding the sample after the initial extraction in order to obtain complete removal of the oil by subsequent extraction. It was found necessary to use 0.5 g. of 60- to 80-mesh sand to obtain adequate regrinding with a mortar and pestle. Regrinding through a Wiley intermediate mill[2] equipped with the 20-mesh screen did not reduce the sample sufficiently to allow complete extraction of the oil in a reasonable time.

To establish the time required for complete extraction of the oil, 5-g. samples of the ground castor beans were extracted for 2 hours in a Butt extractor as specified in A.O.C.S. Official Method Aa 4-38 (1) except that carbon tetrachloride was used as solvent. The partially extracted sample was removed from the extractor and transferred to a porcelain mortar having an internal diameter of 4 in. After the addition of 0.5 g. of 60- to 80-mesh sand the sample was reground with approximately 200 strokes of the pestle during a period of about 2 minutes. The reground material was quantitatively returned to the original filter paper with the aid of a camel's hair brush, rewrapped, and replaced in the extractor. The extraction with carbon tetrachloride was continued for selected time intervals of from 2 to 22 hours. The re-

TABLE 1

Yield of Oil from Castor Beans by Varying Extraction Time

Total extraction time	Yield of oil[a]	
	Without regrind	Reground with sand[b]
hours	%	%
2	45.43	....
3	45.44	....
4	46.17	47.58
5	....	47.55
6	....	47.63
8	47.46	47.76
16	46.93	47.87
22	46.96	47.48

[a] Averages of closely agreeing replicates.

[b] Samples were extracted 2 hours, reground, and extraction continued to give the total extraction time specified.

sults obtained with and without regrinding are given in Table I. They indicate that for the determination of oil in castor beans a 5-g. sample of ground beans should be extracted for 2 hours as specified in the A.O.C.S. Official Method Aa 4-38, using carbon tetrachloride as solvent, reground with 60- to 80-mesh sand as described above, and re-extracted for 2 additional hours.

Similar data for the extraction of 5-g. samples of castor pomace are shown in Table II. These results indicate that a regrind is unnecessary and that satisfactory extraction of the oil can be obtained in 4 hours.

TABLE II

Yield of Oil from Castor Pomace by Varying Extraction Time

Total extraction time	Yield of oil[a]	
	Without regrind	Reground with sand[b]
hours	%	%
2	2.34	......
3	2.38	......
4	2.42	2.46
8	2.42	2.39
16	2.44	2.54
22	2.41	2.53

[a] Averages of closely agreeing replicates.

[b] Samples were extracted 2 hours, reground, and extraction continued to give the total extraction time specified.

Free Fatty Acids

Methyl alcohol, isopropyl alcohol, and carbon tetrachloride were investigated as possible solvents for the extraction of oil from castor beans for the determination of free fatty acids. To a series of weighed samples of U.S.P. castor oil of known free fatty acid content were added 100 ml. of methyl alcohol, isopropyl alcohol, or carbon tetrachloride. After removal of the solvent by evaporation, the samples were titrated for free fatty acid with ca. 0.25 *N* sodium hydroxide solution, accurately standardized, using phenolphthalein as the indicator as directed in A.O.C.S. Official Method Aa 6-38 (1). No correction for acidity was found necessary for any of the solvents used.

Preliminary experiments showed that a percolation extraction procedure similar to the A.O.C.S. Official Method Aa 6-38 for cottonseed (1) was impractical since the percolation rate was extremely slow with all three solvents.

Several other procedures using each of the solvents were also tried, including refluxing the ground sample with the solvent, maceration of whole beans with solvent in a Waring blendor,[2] and agitation of the ground sample with the solvent at room temperature. Each of these procedures was found to extract over 60% of the oil in the sample, and values for free fatty acids obtained on all of the extracted oils, including those obtained in the quantitative method, were in close agreement, varying from 0.65 to 0.73%. However both carbon tetrachloride and isopropyl alcohol were difficult to remove completely from the amount of oil required for the determination. Even when a vacuum was applied to the flask, long periods of heating were required to remove the last traces of these solvents. On the other hand, methyl alcohol was readily removed under reduced pressure by heating on the steam bath. It is therefore recommended as the best of the solvents.

In view of the above, the following procedure is considered to be the most satisfactory for the determination of free fatty acids in castor beans. Suspend approximately 20 g. of the ground beans in 100 ml. of methyl alcohol at room temperature with occasional agitation for 30 minutes. Filter by means of either a Buchner funnel or folded filter paper. Remove solvent by heating under reduced pressure on a steam bath, being careful to remove the last traces of solvent. Weigh a sample of the oil into a flask or bottle, being careful to avoid transferring the nonoil material which adheres to the bottom of the original flask, and titrate as directed in A.O.C.S. Official Method Aa 6-38 (1) with 0.25 *N* sodium hydroxide to a persistent pink color with phenolphthalein used as the indicator.

Caution: Castor beans contain a protease to which some individuals are allergic. It is recommended that workers wear rubber gloves when preparing and handling the analytical samples and that they avoid inhaling any of the dust arising from such samples.

Summary

Castor beans may be satisfactorily prepared for analysis by grinding in a food chopper equipped with a 12-tooth blade.

For the determination of moisture and volatile matter a 5-g. sample of ground beans or pomace or a 50-g. sample of whole beans should be dried for 2 and 4 hours, respectively, at 130° C. in a forced-draft oven.

For the determination of oil in castor beans a 5-g. sample of ground beans should be extracted as directed by the A.O.C.S. Official Method Aa 4-38, using carbon tetrachloride for 2 hours, reground with 0.5 g. of 60- to 80-mesh sand, and subsequently extracted for 2 additional hours. For castor pomace similar extraction for 4 hours without regrinding is satisfactory.

The free fatty acid content of castor beans can be determined by extracting the ground beans at room temperature with methyl alcohol and titrating the extracted oil as directed in A.O.C.S. Official Method Aa 6-38 after removal of solvent by heating under reduced pressure.

Acknowledgments

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