A Bench-Scale Method for Evaluating the Processing Characteristics of Oilseeds for Filtration-Extraction[1]

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The use of a reliable bench-scale apparatus and procedure will reduce the cost and time required for process development by minimizing the number of the large-scale pilot plant test runs. Such an apparatus has been devised, assembled, and used for determining the extractabiilty and filterability of oilseeds prepared for solvent extraction. Similarly the apparatus also could be used for materials other than oilseeds that involve extraction and filtration operations.

The purpose of this paper is to describe the bench-scale apparatus and procedure which were used during the development of a new solvent extraction process for cottonseed (2, 8), soybeans (1, 5), and rice bran (6) and which are presently being used in the refinement of the process for these materials and in the development of the process for other oil-bearing materials.

The feasibility of the new process had been demonstrated originally in laboratory-scale tests. It was then necessary to determine the optimum material preparation conditions, not only for the initial oilseed (cottonseed) used in the process but also for other oil-bearing materials, which, due to the different inherent characteristics, required different preparation conditions. Operations such as rolling of the meats, conditioning and cooking of the rolled or flaked meats, and crisping of the cooked material were involved in the preparation phase. These operations embodied many interrelated variables, such as method of rolling and crisping, moisture during rolling, cooking and crisping, and time of cooking. Moreover several additional variables in the extraction and filtration phases of the process also required evaluation; for example, slurrying time and temperature, solvent to meats ratio, type of filter medium, cake thickness, and number and temperature of washes.

It was apparent that the time and expense to conduct full-scale pilot-plant runs to determine the optimum conditions of all the variables for the many oil-bearing materials would be excessive. Consequently the bench-scale apparatus described herein was devised, and the method of using the apparatus was correlated with the operation of the pilot-plant slurry mixer (extractor) and the 3-foot horizontal rotary vacuum filter. Information obtained by use of the bench-scale technique served as a guide for the larger scale pilot-plant runs. Thus the number of pilot-plant runs that were required to a) confirm or check preliminary data, b) obtain engineering data on continuous operations, and c) provide larger quantities of products for evaluation purposes was appreciably reduced. Savings were thereby effected in the procurement and processing of large quantities of materials and in many hours of research effort.

Bench-Scale Equipment and Procedure

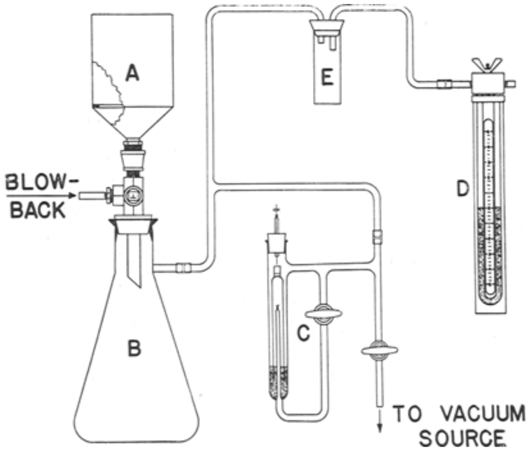

FIG. 1. Test unit.

During the start-up operations of the first commercial filtration-extraction plant, the bench-scale apparatus was used to evaluate the prepared material before extraction. This permitted necessary changes in preparation conditions to be made before large amounts of material were processed in the solvent plant. Time and cost of attaining efficient commercial operation were minimized.

The bench-scale technique as it was specifically applied to the evaluation of the filtration-extraction process variables will be described. A brief description of the process is first given in order to depict the similarity of operations between the bench and pilot-plant-scale tests.

Filtration-Extraction Process

Filtration-extraction (4) is a continuous process for the direct solvent-extraction of oil-bearing materials. It derives its name from the fact that it is designed around the unit operation of filtration and has as its major equipment unit a continuous horizontal rotary vacuum filter. It differs from conventional solvent-extraction systems (3) in that the prepared oil-bearing material is contacted with solvent in a mixing vessel for a period of time sufficient to dissolve the oil, and then a standard rotary horizontal vacuum filter is employed to separate and to wash out the concentrated miscella from the residual meal. The process can conveniently be divided into three phases, the preparation phase in which the vegetable oil-bearing material is made ready for extraction, the extraction phase in which the oil is separated from the meal, and the product-recovery phase wherein solvent is removed from the oil and the meal for reuse. The general pilot-plant process for cottonseed, for example, consists of delintering, hulling, cracking, flaking, cooking, and crisping. Upon discharge from the cooker the material is made to undergo a uniform temperature and moisture loss which causes the par-

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ticles to become relatively incompressible. The cooked and crisped material is contacted with hexane miscella, and the resulting slurry fed to a 3-foot diameter filter. The slurry is filtered and the cake countercurrently washed twice with progressively weaker miscellas and finally with hexane. The bench-scale test unit proved to be a valuable tool in evaluating many of the previously mentioned process variables with respect to filtration.

Figure 1 is a sketch of the bench-scale test unit. The specially constructed stainless steel filter funnel (A), 5¼ in. in diameter, rests in a 4,000-ml. suction flask (B). A modified (7) Cartesian manostat (C) is used for vacuum control. The exact reduced pressure desired is obtained by placing the rubber disc on the wire (upper, left). Proper functioning is indicated by a continuous rapid vertical modulation of the floating bell. After proper adjustment has been made, the pressure differential can be controlled for an indefinite period of time with no drifting of reduced pressure within the system. The manometer (D) and the mercury trap (E) complete the assembly. The three-way valve under the filter funnel can be placed to subject the funnel's contents to either blow-back gas or vacuum. Around the inner circumference of the filter funnel (cutaway section) is a ring on which a neoprene gasket and a filter medium insert rests. Filter screen inserts eliminated the need for several funnels and facilitated screen cleaning. Filter funnel inserts of 40, 60, 80, 100, and 150 square weave mesh and 24 × 110 Dutch weave have been used with the assembly. Inserts of any type or mesh of metallic filter medium can be easily fabricated by soldering a small circular piece of the screen to a ring similar to the one built into the filter funnel.

The bench-scale method had to simulate as nearly as possible the conditions to which the prepared material would be subjected on the 3-ft. pilot-plant filter. In the assembly a provision was made for blowing a gas up through the slurry to simulate the action of the blow-back system of the pilot-plant filter, the purpose of which is to reincorporate with the oncoming slurry any material which may not have been removed by the marc (solvent-wet, extracted meal) discharge scroll. While under the influence of the blow-back gas the solids of the slurry are kept in suspension, and it is this action which is simulated in the bench-scale procedure. As with the pilot-plant filter, slurry filtration and cake washing and draining are conducted on a horizontal plane and the pressure differential is maintained by vacuum.

The conditions for the standard test procedure used to compare filtration and extraction characteristics of differently prepared oilseed materials are as follows: cake thickness of 2 in., 60 x 60 mesh filter medium insert, slurrying time of 30 min., slurry and wash liquid temperature of 135-140° F., three washes, a solvent-to-meats ratio based on the amount of hexane required to yield a full miscella containing between 25 and 30% oil, a pressure differential of 4 in. of mercury, and a 10-second cake-drain period after the final wash. Most of these conditions have been so selected because they represent an average of what has been obtained in actual pilot-plant work. Once a satisfactory solvent ratio is established for a particular oil-bearing material, all other preparations of that material are evaluated using that solvent ratio. The 2-in. cake thickness was selected because it represented the optimum thickness with respect to capacity and extractability for most of the oil-bearing materials processed on the pilot-plant filter.

The steps that the operator performs in evaluating a material preparation are as follows. The slurry and wash liquids are heated for 30 min. to a maximum temperature of 140° F. Oil-free hexane is used for the slurrying operation and for the three washes, except where residual lipids in meal is to be determined. In this case miscellas of 10, 5, and 1% oil concentration are used for slurrying and for the 1st and 2nd washes, respectively. Oil-free hexane is always used for the final wash. With the manostat set, the manometer indicating 4 in. of mercury vacuum and air blowing up through the filter funnel, the slurry is poured into the funnel. Meanwhile the system is maintained under vacuum up to the 3-way valve on the spout of the funnel. The three-way valve is then turned, cutting off the blow-back gas and subjecting the slurry to vacuum. Simultaneously with the turning of the valve a stop watch is started. The time required for the slurry liquid to disappear from the surface of the filter cake is recorded as the slurry filtration time. The three wash liquids are then charged to the funnel, one behind the other, as the liquid level drops from view, and the respective time periods are recorded for each wash filtration. A 10-second cake-drain period under vacuum is allowed after the third wash. It will be noted that there is very little time lapse, if any, between washes. This is equivalent to operating the pilot-plant filter at near flooding conditions.

After the 10-second drain period the solvent-damp filter cake is analyzed for its solvent content. The combined slurry and wash filtrates weights and the total filtration cycle time are used to calculate the over-all mass velocity. Residual lipides and moisture content determinations are made on the desolventized filter cake.

If it is desired to get an indication of the amount of fines to be expected in the full miscella of a pilot-plant run, a sample of the material is slurried and filtered, using the bench equipment and a fines determination of the slurry filtrate made. The quantity of fines present is usually reported as a weight percentage of the filtrate or as a weight percentage of the feed material.

The mass velocities of the slurry filtration and of each of the wash filtrations can be determined separately by timing and weighing each filtrate. With this information the percentage of the available annular filtering area of a continuous pilot-plant or commercial filter that should be devoted to each filtration step can be readily calculated. Thus the location of the "bridges" and partitions in the filter valve, which controls the separation of filtrates, may be pre-determined. The size of a filter for certain capacity ranges also can be determined from the mass velocity data.

In using the bench-scale test equipment to determine optimum filtration conditions, a single material preparation is used, and slurry time, extraction temperature, filter medium, cake thickness, solvent ratio, or vacuum, etc., may be varied. By plotting the particular variable with respect to mass velocity, residual lipides, or any other factor, the effect of that variable can be ascertained.

Results and Discussion

Table I is a tabulation of three bench-scale experiments. Preparation conditions together with the resulting mass velocities and residual lipids are shown. Experiments 1 and 2 were conducted, using cottonseed received from Lubbock, Tex. Experiment No. 3 was made using cottonseed from Greenwood, Miss. The differences in the preparation conditions between experiments 1 and 2 resulted in a 2-fold change in mass velocity and a 3-fold change in residual lipids content. Experiment 3 is an example of a preparation condition which yielded both a reasonably high mass velocity and a reasonably low residual lipids content.

TABLE I

Bench-Scale Experiments

	Experiment No.		
	1	2	3
Rolling			
Space between Rolls 1 and 2, in.	0.025	0.025	0.017
Space between Rolls 2 and 3, in.	0.016	0.016	0.010
Space between Rolls 3 and 4, in.	0.008	0.008	0.005
Space between Rolls 4 and 5, in.	0.003	0.003	0.000
Rolling rate, lbs/hr.	480	480	480
Flake Moisture, %	7.0	6.9	11.1
Cooking			
Retention time, min.	48	48	48
Temperature Range, °F.	176-234	156-250	197-230
Highest Moisture Reached, %	25.0	14.5	24.7
Moisture at discharge, %	13.2	7.4	13.1
Crisping			
Moisture after crisping, %	6.7	6.7	10.5
Oil content, %	32.08	33.09	26.00
Extraction			
Solvent/Meats ratio, lb/lb.	1.2/1.0	1.2/1.0	1.2/1.0
Extraction temperature, °F.	135	140	140
Slurry time, min.	30	30	30
Cake thickness, in.	2.0	1.9	2.0
Vacuum, in. Hg.	4	4	4
Mass velocity, lb/hr/sq. ft.	2956	1500	2249
Meal (Air Desolventized)			
Moisture, %	8.70	7.30	9.00
Oil, %	1.78	0.58	0.99

It is noted that the residual lipids content in Experiment 1 is relatively high (1.78%). Here the bench-scale unit would be indispensable for determining the extent to which the slurrying time and/or the solvent to meats ratio, for example, should be increased to reduce the residual lipids content to 1.0% or lower.

The bench-scale equipment has been used to determine the pilot-plant filter pan speed. Most of the pilot-plant runs have been made at a feed rate of either 5 or 7.5 lbs. of prepared material per minute and with a 2-in. filter cake thickness. In the evaluation of certain variables however changes were made in the preparation conditions, which resulted in the formation of a filter cake either thicker or thinner than 2 in. The filter pan speed can be set to yield the 2-in. cake if the amount of material required is known. The bench test, for example, showed that 530 g. of a material were required to yield a 2-in. cake. This material, when processed on the pilot-plant filter at 7.5 #/min. required a filter pan speed setting of 0.250 r.p.m. A second preparation required only 480 g. to form a 2-in. cake. The correct filter pan speed setting at 7.5 #/min. for this second preparation would be $\frac{530}{480}$ times the previous setting, or 0.275 r.p.m. By knowing, as nearly as possible, the equipment speed settings, the whole system can be brought to equilibrium much sooner. This in turn reduces the time required to make a pilot-plant run.

The bench-scale technique can be used to evaluate a preparation variable as illustrated in the following example. Cooked cottonseed flakes were subjected to four different grades of crisping. Crispness, or incompressibility, is that characteristic which the cooked meats acquire by undergoing a uniform loss in moisture under proper conditions. A sample of the cooked meats was taken as the material discharged from the pilot-plant cooker and was allowed to cool in a closed container. This represented no crisping. The second sample was allowed to cool in the open air on a tray for approximately 20 min. The third and fourth samples were heated in a forced draft tray oven for 10 and 30 min., respectively, further to crisp the material. Each of the four treated samples was then filtration-extracted, using the standard procedure, to determine mass velocity. Mass velocities of approximately 1,400, 2,100, 4,200, and 5,000 lbs. per hr. per square foot were obtained for samples 1 to 4, respectively, showing the extent to which the mass velocities increased as the degree of crispness increased.

A correlation which has been established between the bench- and the pilot-plant-scale equipment is that a cottonseed preparation must have a mass velocity of about 2,000 lbs. per hr. per square foot or higher as measured by the bench equipment to insure trouble-free operation of the 3-ft. filter when operating at a capacity of nine tons of cottonseed per day, a solvent to meats ratio of 1.2 to 1, 3 washes, and a 2-in. cake. Limited work with high oil content materials suggests that as the solvent to meats ratio is increased, the mass velocity must also increase if plant capacity is to remain unchanged. Sesame seed, for example, if extracted at a 1.7 to 1 solvent ratio, would require a bench-scale mass velocity somewhat higher than the 2,000 for cottonseed. Thus it is possible to predict with a fair degree of accuracy the pilot-plant processibility of a particular material preparation. Several man-days of research effort are saved when the bench-scale test equipment indicates that a particular preparation does not have the necessary filtration-extraction characteristics to warrant a pilot-plant run.

Summary

A simple laboratory testing apparatus and its use have been described. With relatively few pounds of material it is possible to evaluate many preparation and filtration variables and to predict with a fair degree of accuracy the behavior of a continuous horizontal vacuum filter. It is felt that such a unit will be invaluable for use in commercial plants, especially during the start-up operations of a new plant. The bench-scale unit should also find application in connection with other processes employing the unit operations of extraction and filtration.

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