✿Field Evaluation of Extraction Performance

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ABSTRACT

This paper presents 5 methods to evaluate the performance of percolation-type extractors during actual operation by determining the sources of residual oil left in the extracted meal. To determine residual oil left due to poor "flake extractability," the operator may use either or both the "shaker" and "stain and soak" methods. The "shaker" method first determines the maximum oil that can be removed from the flake and compares this result with the actual amount extracted. The "stain and soak" method involves the microscopic examination of the flakes before extraction. Three methods are described to determine the residual oil due to poor drainage in the extractor. The first involves removing the undrained oil in the laboratory by washing with additional hexane and then comparing the residual oil in the meal with a sample that has not been washed. The second is a colorimetric determination of the final-stage miscella and comparison against known standards, and, finally, a method is presented using a comparison between the design conditions of the extractor and the actual operating conditions to infer the residual oil left due to poor drainage. A combination of any or all of these methods provides a useful means for a field evaluation of the performance of a percolation-type extractor.

INTRODUCTION

Solvent extraction, as it is practiced in the fats and oils industry today, is concerned primarily with the recovery of oil from oilseeds. The heart of the process is the extractor which provides sufficient contact time between the specially prepared flakes and the solvent to extract the oil. The various types of extractors have been reviewed in a previous article (1). By far the most common type, however, is the percolation extractor where the liquid solvent is pumped over a bed of flakes, percolates down through the bed and leaves the bed at the bottom through a perforated plate, mesh screen or wedge wire screen. The successful operation of this type of extractor is, therefore, dependent on 2 primary factors—the ability of the solvent to extract the oil and the ability of the solvent-oil mixture, called miscella, to drain through the bed.

This paper discusses methods for evaluating the performance of this type of percolation extractor. In most instances, the extraction of soybean flakes with hexane is considered, but similar procedures could be developed for any oilseed/solvent system.

MECHANICS OF EXTRACTION

A schematic representation of the mechanics of extraction, as carried out in a percolation extractor, is shown in Figure 1. To extract the oil from a flake, 4 distinct steps must occur: (a) the solvent must contact the surface of the flake; (b) the solvent must diffuse into the flake and dissolve the oil; (c) the mixture of oil and solvent must then diffuse back to the surface and finally (d) the mixture must drain away from the flake. Examination of each of these 4 steps is accomplished easily in the laboratory (2). However, for evaluating an extractor as it operates in a commercial installation, quicker and simpler methods must be developed so that the operator can determine the level of performance at any given time. The following sections will summarize several methods that have been developed for this purpose and have proven useful in actual installations. Methods will be presented to evaluate the performance both from examining the prepared flake, in this case soybean, and also from evaluating the actual extractor operation.
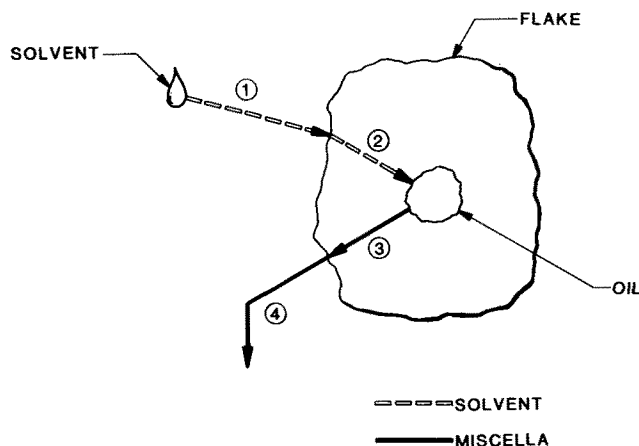

FIG. 1. Mechanism of solvent extraction.

SOURCES OF RESIDUAL OIL

The object of the extraction process is to reduce oil content in the flake to the lowest possible level with a minimum use of solvent. There are 2 sources for the residual oil left in the flake after the extraction process is completed. First is "unextracted oil" or that oil which has not been extracted from the flake by undergoing steps 1 and 2 in Figure 1, and the second is "surface oil" or that oil that has completed steps 1, 2 and 3 but not 4, due to poor drainage characteristics of the extractor bed. To reduce "unextracted oil" to a minimum, the flake must be prepared to rupture all the oil glands and provide the maximum contact area between the solvent and the oil, whereas to reduce the "surface oil" to a minimum, the flake must be prepared to provide a bed that allows unhindered drainage. Preparing the flake for minimum "unextracted oil" will result in high "surface oil" and vice versa, i.e., to provide the maximum gland rupture and contact area between the solvent and oil, it would be advisable actually to grind the flake to the smallest size possible, but this would result in a tightly packed bed that would not drain easily. Similarly, to give maximum drainage and, therefore, minimum "surface oil," a coarse flake would be best, but this would reduce gland rupture and the contact area of solvent and oil. The modern extraction plant is designed to strike the proper balance between the flake thickness desired for most rapid extraction and that required for good drainage.

EVALUATION OF FLAKE EXTRACTABILITY

The property of the flake which measures the amount of oil that is being extracted, compared to the maximum amount that could be extracted if surface factors were not considered, is called "flake extractability." It can be measured by both quantitative and qualitative techniques.

The first method, called the "shaker" method, uses an apparatus, shown in Figure 2, that has been used previously in extraction studies (3). It consists of a cylinder, charged with a weighed sample of the flakes to be studied, hexane and a quantity of small ball bearings. The cylinder is then capped tightly and shaken vigorously in a small shaker. The ball bearings grind the flakes to a fine particle size to allow the maximum contact area between the flakes and solvent. The cylinder is then removed from the shaker, chilled to
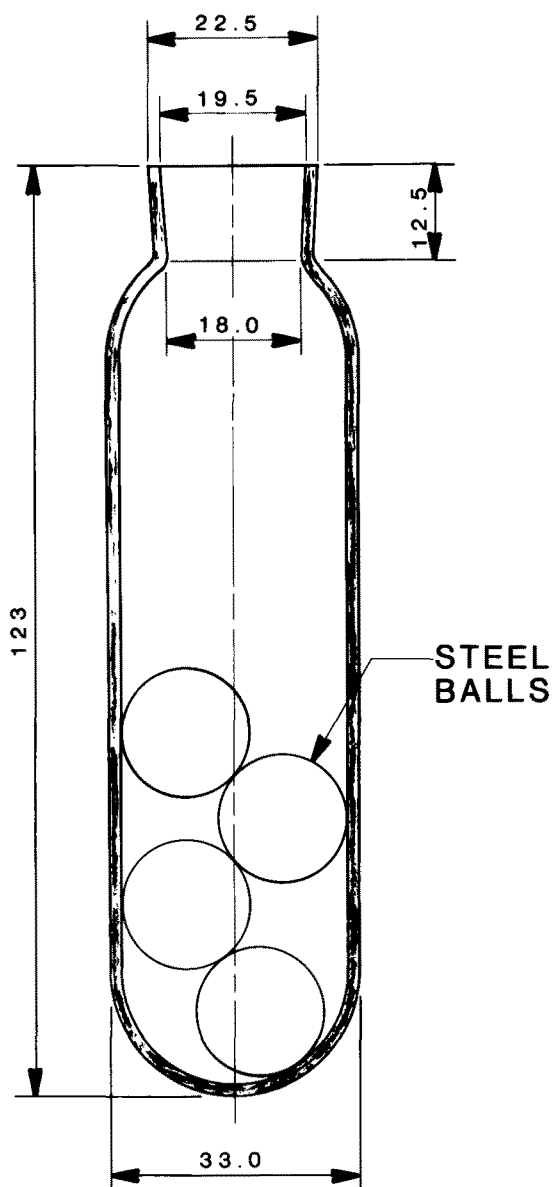
(ALL DIMENSIONS ARE IN MILLIMETERS)

FIG. 2. Stainless steel extraction tube for shaker method, Ref. Applequist, L.A., Further Studies on a Multisequential Method for Determination of Oil Content in Oilseeds, JAOCS 44 (1967).

prevent loss of hexane by evaporation, and filtered. A hexane wash of the ground flakes on the filter paper is used to eliminate effects of "surface oil" on the results. A second flake sample of identical weight is then charged into the cylinder, but without the ball bearings. An identical amount of hexane as used in the first test is added and the tube is again shaken, chilled, filtered and washed. Since there are no ball bearings present during the shaking, the flakes remain basically intact. Comparison of the oil content in the miscella or the residual oil in the desolventized flakes of this second sample against the first is then a measure of the "flake extractability," since the first sample represents the maximum extractability that is possible.

The second method, called "stain and soak," provides a qualitative, microscopic technique to evaluate "flake extractability." The method discussed here, however, differs from a standard-type histological preparation of tissue in that it is a short-cut method that gives qualitative results in

a short period of time. Comparison of flakes produced under different plant conditions, coupled with the existing knowledge of what constitutes an "extractable" flake, can then be used to evaluate extraction performance.

The standard method of preparing the flakes for microscopic examination usually includes 7 steps: (1) fixation: the treating of the flake with chemicals to prevent degradation during the remaining steps of the procedure, (2) dehydration: removing chemically all water from the sample to make it firm, (3) infiltration: replacement of the chemical dehydrant from step 2 with an inert paraffin material, (4) casting: the sample which is encasted in pure paraffin after step 3 is cast into a mold and allowed to cool, (5) sectioning: several possible sections of the flake sample are cut from the paraffin mold, (6) slide preparation: the best sections from step 5 are attached to standard slide plates and (7) staining: the slides are dipped into a fat-seeking staining solution to accentuate the oil and protein parts of the sample.

Unfortunately, this complete procedure often takes several days to prepare a sample and, thus, would not be useful in evaluating the operation of an extractor. The "stain and soak" method discussed here consists of only the dehydration and staining steps which are then followed by a microscopic examination of the sample. Samples were first dehydrated chemically, by soaking, and then a fat-seeking stain, dissolved in a solvent, was applied. After drying, slides were prepared and photographs of these slides taken through a microscope.

In developing this method for soybean flakes, it was necessary to examine various staining and soaking times to develop slides that could be used to infer extractability. As shown in Figure 3, increasing the stain time tends to darken the sample and develop a more pronounced difference between the oil and protein portions. This is particularly noticeable on the thinner flakes. Increasing the soak time for a given staining time tends to lighten the sample.

There are several ways that flakes prepared with this "stain and soak" method can be used to help determine flake extractability. First, under high magnification, the degree in which the oil is dispersed across the surface of the flake can be examined. As shown in Figure 4, although there are a few areas of high oil concentration, as evidenced by the dark spots, the oil is generally well dispersed across the surface when compared with the flake in Figure 5 where a larger number of dark high oil content areas are evident. Since the rate and efficiency of the extraction are dependent on contact between the solvent and the oil, the greater the total flake surface covered by the oil, resulting
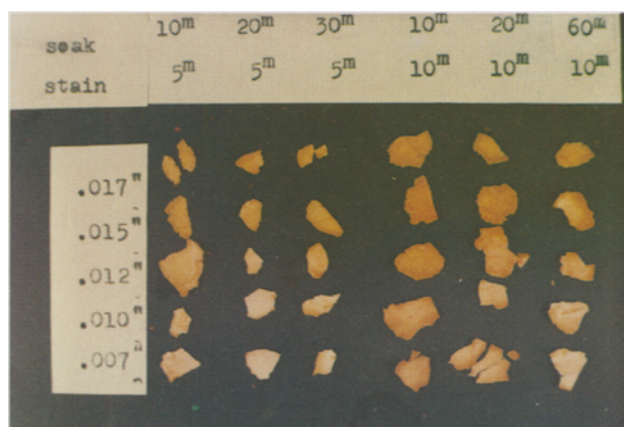

FIG. 3. Stained and soaked soybean flakes: effect of staining and soaking time and flake thickness.
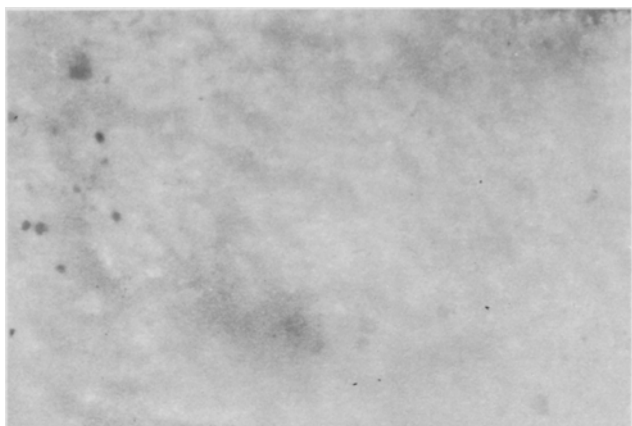
FIG. 4. Magnification of stained soybean flake—good flake extractability.
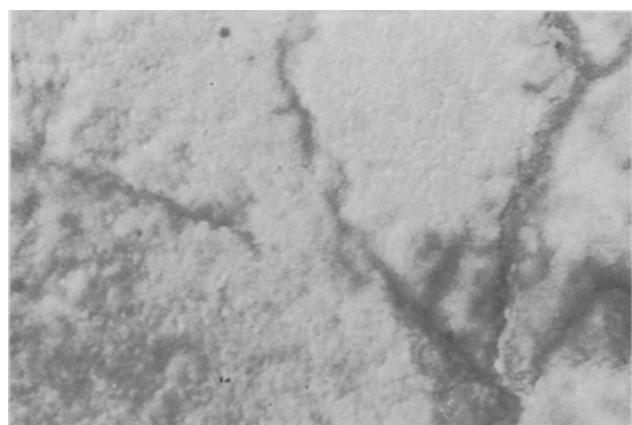

FIG. 5. Magnification of stained soybean flake—poor flake extractability.

FIG. 6. Stained and soaked soybean flakes: effect of moisture content and flake thickness.

tions of oil in specific areas compared to the lighter flakes in which the oil is more dispersed. Also, for a given flake thickness, the oil appears to be more dispersed at a moisture content of 9.2% than at the higher moisture contents.

It is obvious that the "stain and soak" method, unlike the shaker method described previously, is strictly a qualitative approach. However, when sufficient background with the method is obtained on a given operation, it can be very useful in discovering changes in the operation that may effect extraction efficiency, and when combined with the quantitative data obtained from the shaker method, it can be a useful tool for elevating and improving the overall solvent plant performance.

Finally, microscopic examination of flake samples can also be useful in measuring soybean "flake extractability" as it pertains to operation of the flaking rolls. It is often assumed that, once the flaking roll differential is set, flakes of uniform thickness are being produced. As shown in Figure 7, however, the scratching of a roll surface by a stone or other object can produce a "rippled" flake. A cross-sectional view of this type of flake (Fig. 8) shows clearly its varying thickness; part of the flake is at the optimum thickness for extraction whereas other parts are not, thus reducing the flake's overall extractability.

EVALUATION OF SURFACE OIL

The second source of residual oil is "surface oil," which is oil left due to poor drainage in the extractor. It can easily be evaluated by 3 methods: flake washing, final stage mis-
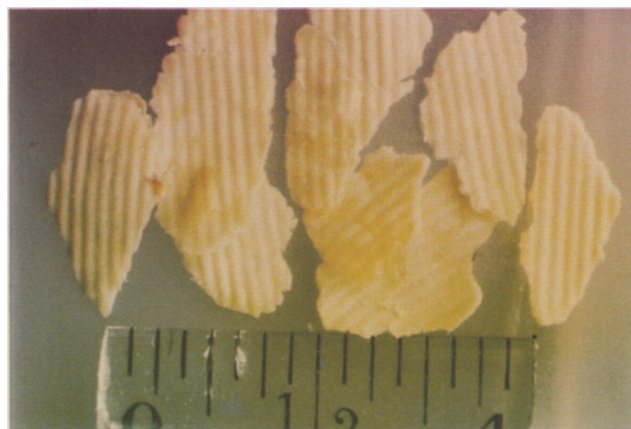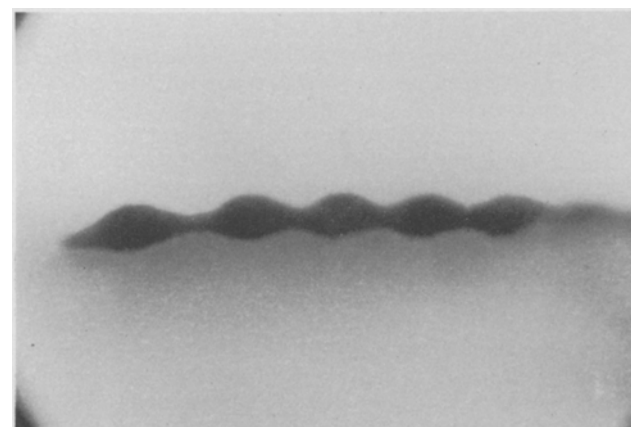

FIG. 7. "Rippled" flakes.

FIG. 8. Cross sectional area of "rippled" flakes.

in more contact area for the solvent, the better will be the extraction. Thus, the flake shown in Figure 4 can be expected to extract more easily than the one shown in Figure 5.

This "stain and soak" method can also be used to examine the effect of moisture and flake thickness on "flake extractability." As shown in Figure 6, at a given moisture level, with an increasing flake thickness, the prepared samples grow darker, indicating greater concentra-

cella color measurement and final stage miscella concentration measurement. As with the "flake extractability," it is the use of all 3 methods together which will give both the quantitative and qualitative information to evaluate the system performance properly.

Figure 9 is a schematic diagram of the "flake washing" procedure. It consists of running 2 separate tests on the solvent-wet flakes and comparing the results. Half of the sample taken is washed quickly with a large quantity of fresh hexane, the nexane is decanted and the sample allowed to air dry. This washing effectively removes any surface oil that may have been left because of poor drainage, and it is assumed that this additional short contact time with hexane will not extract any oil left in the flake. The other half of the sample is left to dry as is without additional hexane washing. The residual oil content of the washed sample is due only to the unextracted oil; the residual oil content of the unwashed sample is due to both unextracted and surface oil. The difference between the two is the surface oil. Typical limits that can be used for a soybean plant are as follows: less than 0.25% difference—excellent drainage, 0.25%-0.50%—fair drainage, greater than 0.50%—poor drainage.

"Surface oil" can also be measured by comparing the color of the final stage miscella against standard solutions. In the final extraction stage of a typical countercurrent extractor, fresh hexane is contacting the flakes after most of the oil has been extracted. In other words, the flakes should enter this final stage with little unextracted oil and, if drainage in the previous stages has been satisfactory, with little surface oil. Therefore, the color of miscella from this final stage can be used as a measure of the efficiency of the extractor operation. The use of this method is illustrated in Figure 10. First, miscella samples were prepared with oil concentrations of 0.25%, 0.50%, 1.0% and 2.0% and each sample was placed in a sealed test tube. The tubes were then mounted side by side with the lowest concentration on the left. The tube shown on the far left contains pure hexane. Samples of miscella can then be placed in another test tube of the same diameter and compared against a good light with the prepared samples. The concentration of the sample can be judged well enough by color. For a soybean plant, there should be less than ¼% by weight oil in this miscella.

The amount of surface oil may increase if an incomplete separation of oil and hexane is being made in the miscella distillation system. The recovered hexane will then contain oil when it is recycled back to the extractor, increasing the surface oil in the final extraction stage. These colormetric procedures can also be used, therefore, to examine the feed hexane; the oil content of which should be less than 0.25%.

A graph based on the material balance around the extractor provides the third method for measuring "surface oil." A series of curves for miscella concentration can be drawn based on the analysis of the feed flakes, the standard design parameters of solvent/flake ratio and the percent solvent in the extracted flakes. Figure 11 shows 3 of these curves. Although the common design parameter is the ratio of solvent to extractor feed, extraction plants typically do not attempt to measure the flow of flakes to the extractor, so the ratio chosen for this graph is the flow of solvent at any given time to the extraction plant throughput. In preparation of this graph, therefore, it is necessary to consider the average oil content of the seed and the amount of hulls and/or cleanings removed from the oilseed before the flakes are prepared. As an example, consider typical design parameters for the extraction of soybean flakes; 0.24 gpm solvent per ton per day of flakes and 33% solvent in the meal which corresponds to ca. a 1:1 ratio
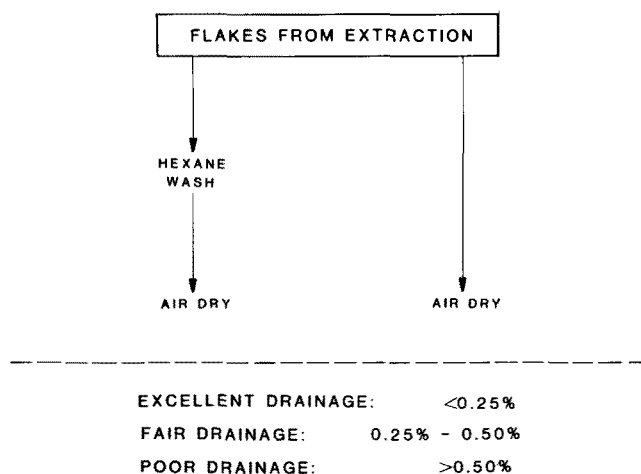

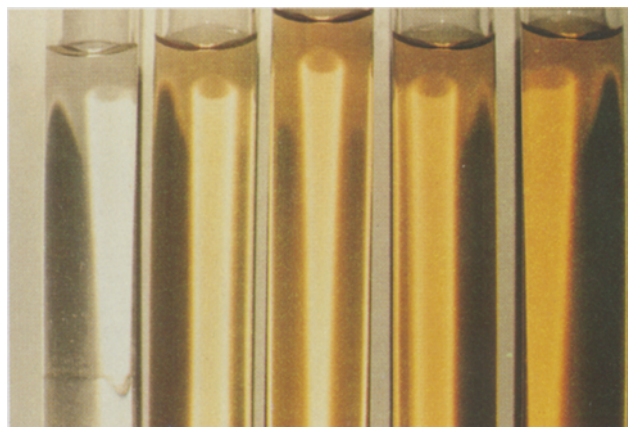FIG. 9. Evaluation of surface oil by flake washing.

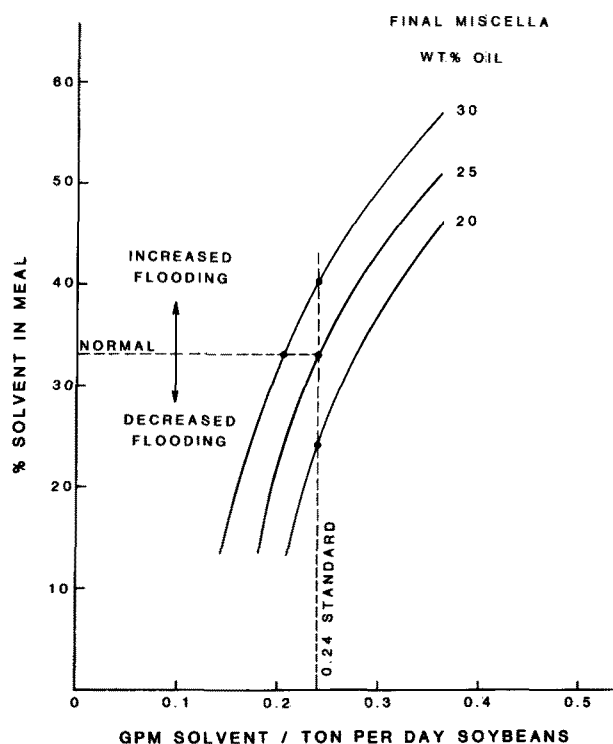FIG. 10. Color determination of miscella concentration.

FIG. 11. Material balance for soybean extraction.

by weight of solvent and flakes. By material balance this would give a miscella concentration of 24%. Should the actual miscella concentration be 30%, this would indicate that less solvent is leaving with the miscella and more with the meal, 40%. Thus, there is poor drainage since some of the solvent is being held up in the final extraction stage. This is a condition of "increased flooding" in the extractor as the compartments are "flooded" with hexane that cannot drain.

Similarly, at the same solvent to flake ratio, a 20% miscella indicates that better-than-design drainage is occurring in the final extractor stage, resulting in a diluted miscella and less solvent, 24%, in the meal. Assuming the solvent recovery system would be able to handle this miscella, this might seem to be a desirable operation; however, deviating

from the design point might be an indication of "decreased flooding" or channeling of the solvent through the bed without sufficient contact between the flakes and solvent. This would show up as high residual oil in the extracted meal.

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2. Kulkarni, B.S., E.L. D'Aquin, A.V. Graci, Measurement of Extractability of Raw and Cooked Cottonseed Flakes, JAOCS 23:12 (1955).
3. Appelquist, L.A., Further Studies on a Multisequential Method for Determination of Oil Content in Oilseeds, JAOCS 44 (1967).