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OIL EXTRACTION PROCESS

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This invention relates to an improved process for the recovery of oil from oil bearing seeds, nuts and the like, and more particularly those with a high oil content, e. g., over 25%.

Oil extraction by the use of solvents, such as hexane, from materials whose oil content is high, such as peanuts, sesame, flax seed, cotton seed, babassu nuts, and the like, meets with certain fundamental difficulties, all of which have not been successfully overcome, heretofore, by the research and practical development in this art.

In the first place initial high oil content of itself inhibits reduction of the oil content to the desirable low value by solvent extraction. In other words, as the original oil content increases, it becomes more difficult to reduce it to a desirable low value by a solvent extraction process.

Further, the ever present "fines" present a problem which is especially serious in the case of these high oil content materials. Such fines not only interfere with drainage of the miscella from the solid residue and entail increased cost for separation therefrom, but also collect in and clog the hexane recovery condensers, reducing efficiency and necessitating more frequent cleansing.

Much of the difficulty heretofore experienced in the practice of oil recovery by volatile solvents has been due more to those preliminary or preparation steps which precede solvent extraction than to the solvent extraction step or method itself. Mechanical treatment of the seed preparatory to solvent extraction has increased rather than diminished the tendency to produce fines, and thus has led in the direction of "fines" production and removal rather than toward "fines" elimination or prevention. This in part arises from the nature and quantity of the solid, and more especially the native protein content of the material being processed.

Efficient solvent extraction requires thorough penetration by the solvent of the seed particles, which accordingly should be small, with a thickness of the order of .015 inch, at least in one dimension. Thin flat flakes of that thickness are ideal for the purpose. Some of the low oil content materials, such as soy beans, have been satisfactorily extracted by flaking the particles, mechanically screening out the fines and then solvent extracting. But the higher oil content ma-

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terials before referred to do not lend themselves to such treatment, because flakes thereof are fragile, not only increasing the "fines" necessary to remove by screening, but actually producing "fines" by the agitation of screen separation, and further "fines" beyond that during conveyance to and transit through the solvent extraction column.

As a consequence, the use of flakes, in the solvent extraction of high oil content material, has been succeeded by resort to comminution of the seeds, or the seed mass, into granular particles of small size, to secure thorough penetration. But such granulation, of itself, is directly responsible for further increase in fines.

My invention is designed to overcome the objections to such prior practices by an improved process which takes advantage of the fact that some fractional proportions of the native protein and carbohydrates in these high oil content materials are water soluble, and that such water soluble constituents form gels under certain conditions or factors of temperature, moisture time, and accompanying oil content. By so regulating or controlling these four factors, and with appropriate preliminary steps or treatment, it becomes possible to recover oil efficiently and economically by solvent extraction of originally high oil content material. This is accomplished as the result of the ability to adhere to the practice of finally solvent extracting the material in flake form, rather than in fine granules, but without unreasonable production of fines or unusual or costly problems in the removal or recovery thereof.

As a first step, the high oil content seed, nuts or other material, is mechanically pressed to reduce its oil content. This may be accomplished in any suitable manner, either intermittently in batches, or continuously, such as in a pressure producing screw press, from which the oil is drained and collected, while the cake emerges in fairly large fragments broken from a sheet that may be up to one-fourth inches or more in thickness. These fragments are broken up or comminuted still further, in a suitable grinding or crushing mill, so that the particles of press cake may be readily handled in the conditioning and flaking steps described below. A typical screen

analysis of the comminuted press cake is as follows:

Screen	Weight, grams	Percent
On 0.5.....	20	9.0
On .065.....	112	51.1
Through .065 on .0328.....	52	23.8
Through .0328 on .0195.....	25	11.4
Through .0195 on .0138.....	15	6.8
Through .0138 on .0116.....	4	1.8
Through .0116.....	11	5.0

It will be observed from the relative coarseness of this comminuted press cake that fine grinding of the press cake with its inherent power costs are obviated. In fact, if the pressure producing screw press is adjusted so that a press cake of approximately $\frac{1}{8}$ " or less in thickness is produced no subsequent comminution of the cake is necessary.

The press is so adjusted as to pressure, and is so operated, as to reduce the oil content to a reasonably low value, but not too low. In a broad sense oil content should be reduced to a value approximating one-fourth, by weight, of the total quantity of protein present in the mass. Otherwise, the flakes later formed do not have the required ductility and tenacity. Resulting oil content of the high oil content materials here considered should be within the range of 8 to 18% of the total weight, and preferably from 10 to 12%, for most high oil containing materials. Lower oil content produces fragments, and later, flakes, which are hard, more or less inflexible, and friable, with excessive tendency to produce increasing quantities of fines as the oil content goes down in proportion to the native protein. Approximately one part of oil to four parts of total protein, in the solid cake, are proportions which produce tough, tenacious flakes capable of withstanding the rigors of screening, conveyance to extracting tower, and the motion, agitation and friction of solvent extraction without over-production of fines. Higher oil content, above the ranges referred to, is objectionable, not only because, inherently, such content of itself tends to inhibit economical and efficient oil extraction, but also because when the oil content is high, other steps of the preparatory treatment do not result in the desirable native protein conversion or gelatinization, thereby yielding a mealy product which produces excessive fines.

Moisture content is important. This should be within the range of 10 to 20%, at least at the time of flaking, if not earlier. Therefore, the moisture content is adjusted, prior to flaking, in any suitable manner. If the moisture content is low it may be increased by adding water, which is converted to vapour as the temperature rises, or by introducing steam into the mass during its treatment. This may occur before or after expressing the oil, but always prior to flaking.

If the moisture content before flaking is less than 10% the resultant flakes are friable and yield excessive fines during conveyance to and through the extraction tower. A moisture content of the press cake prior to flaking higher than 20%, e. g. 21 to 25% does not inhibit flake formation and has no deleterious effect upon the toughness of the flakes. However, the excessive water in a flake containing high moisture must be removed before solvent extraction in order to obtain efficient extraction of the oil. The addition of more than 20% moisture to the press

cake prior to flaking, therefore, is not harmful to flake formation, but it does introduce a succeeding drying step that otherwise would be unnecessary.

Also, prior to flaking the material is heated to a temperature of from 145° F. to 200° F., the temperature varying between those limits with the nature of the particular material, and usually in proportion to its content of native protein or the ratio of protein to oil content. The greater the proportion of protein, the lower should be the temperature at the time of flaking, because gradual rise in temperature for the time necessary to bring it up to 150° F. or 200° F., or whatever is desirable, when the oil content has been reduced to low value, insures the formation of the gels referred to, and enables the cake fragments to be flaked and subsequently solvent extracted, without undue fines.

The necessary rise in temperature may be produced in any suitable manner.

If the pressing of oil is not conducted so that the press cake is immediately ready for flaking, the temperature and moisture of the press cake both generally must be increased for proper conditioning prior to flaking. This is most conveniently and economically accomplished by mixing the ground press cake with live steam. Many experiments have indicated that the raising of the moisture content of the ground press cake to 11 to 12% with live steam results in a temperature of the press cake in the range of 175° F.

If desired, the seed mass to be treated may be preliminarily heated, and its moisture content may be adjusted to the appropriate level, in a suitable steam heated tank or vessel from which it is conveyed or discharged, promptly, into the continuous press. Under those conditions the press may be run at high capacity, with but little temperature rise from the friction of screw press oil expulsion. Or, when low capacity is suitable or desirable, the material may be fed cold to the press and temperature rise may occur therein. Moisture may be adjusted at any time or stage.

It has been found that the moisture content of the press cake is directly proportional to the moisture content of the seed mass following very nearly the equation: % moisture in seed mass times 1.24+0.6 equals the moisture in the press cake. Therefore, the moisture in the seed mass may be conveniently adjusted to give the moisture in the press cake desired or required for direct flaking of the press cake.

In either case, as before stated, the press cake is broken or crushed into fragments of less than one-half inch in diameter. These are passed through standard flaking rolls to crush them to thin flat leaves or flakes with a thickness of .005 to .02 inch, but far beyond that in superficial area.

Such flakes may be produced from original high oil content material, because care has been taken to see to it that the characteristics of the fragments to be flaked are suitable to that end. In other words, by reducing the oil content, adjusting the moisture, raising to the required temperature, and subjecting to heat for the time necessary to reach such temperature, the water soluble proteins and carbohydrates have formed gels and the press cake, and the fragments thereof, are soft, porous, ductile and flexible. When these fragments are flaked their soft, ductile and flexible nature permits them to yield to the pressing action of the rolls. Upon cooling the fragments to the temperature required

for extraction by hexane (less than 135° F.) or upon subsequent heating of the flakes as necessitated by the 95% ethanol extraction process a partial denaturization of the proteins occurs and the flakes thereby become more tough and resistant to fracture without losing their porosity and flexibility.

By following the conditions of this invention for the formation of flakes from high oil content seed no excessive quantity of fines is produced and the flakes are mechanically strong and flexible enough to successfully withstand the punishment of screening, conveyance, and solvent extraction.

The flakes are passed over a rotox or other suitable screen to remove dust or fine particles, which are returned to join the stream of material supplied to the press. The screen usually is one operating by horizontal vibration alone, although this is not essential.

The elimination of fines from the solvent extraction process permitted by this invention may be illustrated by the following data. A flaxseed press cake containing about 12% oil was ground preparatory to conditioning for flaking. The ground press cake contained 7.5% fines passing a screen of 0.0138" openings. This material was then conditioned at 190° F. after increasing its moisture content to 11.3%. After flaking, the material contained only 3% fines passing a screen of 0.0138 inch openings. Weighed quantities of the flakes and of the ground, unconditioned press cake were placed in different containers with measured quantities of hexane. After violent agitation the supernatant hexane over the unconditioned press cake contained slightly more than four times the amount of fines than there were in an equal volume of supernatant hexane over the flakes.

In the extracting tower a stream of the flakes is moved in counter current relation to a stream of solvent, with separation of miscella from solids, as is usual. The miscella is then treated in the customary manner to settle or otherwise remove fines and recover the solvent.

The rate of extraction of oil from high oil seed prepared according to this invention is equivalent to if not slightly better than that from standard soybean flakes. This is due to the relatively greater porosity of the flakes produced according to this invention over that of standard soybean flakes.

By way of further illustration of the invention, but without any limitation thereon, reference is made to the following examples:

Example No. 1

Commercial whole flax seed were dried in a continuous steam heated drier to a moisture content of 5% and immediately treated in a mechanical screw press. A press cake of $\frac{3}{8}$ " thickness containing 12% oil and 6.8% moisture was obtained. This press cake was ground in a nut and burr type of grinder to pass a $\frac{1}{2}$ " screen. The ground press cake was then conditioned with live steam to a moisture content of 11.8% and a temperature of 175° F. The time of conditioning (approximately 10 minutes) was adjusted so that the ground press cake became uniformly conditioned. The conditioned press cake was then treated on flaking rolls to yield flakes of 0.008 to 0.015 inch thickness. The flakes passed over a screen having 0.0138 inch openings, yielded only 4% of fines which were returned to the feed to the screw press. The flakes thus produced were

extracted with hexane in a test solvent extraction unit to yield an extracted flake of less than 1% oil. A determination of the amount of fines produced by agitating the flakes in a measured amount of hexane demonstrated that the amount of fines produced were only 5% more than obtained from standard soybean flakes.

Example No. 2

Whole decorticated peanuts were dried in a continuous steam jacketed drier to a moisture content of 5.0%. The peanuts at a temperature of 150° F. were immediately treated in a mechanical screw press to yield a press cake containing 16% oil. The press cake was at a temperature of 165° F. After grinding the press cake in a nut and burr type of grinder the press cake was conditioned with live steam to a moisture content of 15%. At this moisture content the temperature of the press cake attained a temperature of 190° F. The conditioned press cake was then immediately flaked to sheets of .008 to .012 inch thickness and passed over a screen of .0138 inch openings. Only 2% of fines were recovered for return to the feed to the screw press. The flaked peanuts were extracted in a test solvent extraction unit with hexane to less than 1% oil content. A weighed sample of the peanut flakes agitated in a measured volume of hexane gave about one-half the fines in the supernatant liquor as standard soybean flakes similarly treated. In fact the supernatant liquor was substantially free of fines.

The invention is simple, practical, and economical, and enables high oil content material to be treated without over production of fines. Other advantages will be apparent to those skilled in the art.

What I claim is:

1. A process of extracting oil from high oil content nuts and seeds, consisting in mechanically expressing the material to reduce the oil content to a value in the order of about one-fourth the total protein content as existing at the termination of the expressing step, comminuting into fragments which pass a screen with one-half inch openings, subjecting the fragments to the action of flaking rolls to convert them into wide, thin flakes from .005 to .02 inch thick, and solvent extracting such flakes.
2. A process of extracting oil from high oil content nuts and seeds, consisting in mechanically expressing the material to reduce the oil content to a value in the order of about one-fourth the total protein content as existing at the termination of the expressing step, comminuting into fragments which pass a screen with one-half inch openings, adjusting the moisture content to from ten per cent to twenty per cent, subjecting the fragments to the action of flaking rolls to convert them into wide, thin flakes from .005 to .02 inch thick, and solvent extracting such flakes.
3. A process of extracting oil from high oil content nuts and seeds, consisting in mechanically expressing the material to reduce the oil content to a value in the order of about one-fourth the total protein content as existing at the termination of the expressing step, comminuting into fragments which pass a screen with one-half inch openings, adjusting the moisture content to from ten per cent to twenty per cent, heating the material to a temperature of from 145° F. to 200° F., subjecting the fragments while hot to the action of flaking rolls to convert them into wide,

thin flakes from .005 to .02 inch thick, and solvent extracting such flakes.

4. A process of extracting oil from high oil content nuts and seeds, consisting in mechanically expressing the material to reduce the oil content to from eight to eighteen per cent of the total weight of material as determined after the expressing step, reducing the material to fragments which will pass a screen with one-half inch openings, adjusting the moisture content to from ten to twenty per cent of such weight, heating to a temperature of from 145° F. to 200° F., thereby producing material which is tough, strong, flexible and ductile, subjecting the material to the action of flaking rolls to convert the fragments into wide, thin flakes from .005 to .02 inch in thickness, and solvent extracting such flakes.

5. In the process of extracting oil from nuts and seeds having an oil content of 25% or higher, and wherein the material is subjected to the preliminary step of expressing a substantial portion of the oil content, and to the later step of solvent extracting the residual oil, the novel steps, between the above steps, of retaining in the material after the expressing step natural oil content of about one-fourth the total protein content as existing at the termination of the expressing step, comminuting the solid press cake from the expressing step into fragments which pass a screen with one-half inch openings, and subjecting the fragments to the action of flaking rolls to convert them into wide thin flakes from .005 to .02 inch thick, whereby the aforesaid solvent extracting step can be performed with materially reduced development of fines.

6. In the process of extracting oil from nuts and seeds having an oil content of 25% or higher, and wherein the material is subjected to the preliminary step of expressing a substantial portion of the oil content, and to the later step of solvent extracting the residual oil, the novel steps, between the above steps, of retaining in the material an oil content of the order of about one-fourth the total protein content as existing at the termination of the expressing step, comminuting the solid press cake from the expressing step into fragments which pass a screen with one-fourth inch openings, adjusting the moisture content to from ten per cent to twenty per cent of the comminuted cake, and subjecting the fragments to the action of flaking rolls to con-

vert them into wide thin flakes from .005 to .02 inch thick, whereby the aforesaid solvent extraction step can be performed with materially reduced development of fines.

7. In the process of extracting oil from nuts and seeds having an oil content of 25% or higher, and wherein the material is subjected to the preliminary step of expressing a substantial portion of the oil content, and to the later step of solvent extracting the residual oil, the novel steps, between the above steps, of retaining in the material an oil content of the order of about one-fourth the total protein content as existing at the termination of the expressing step, comminuting the solid press cake from the expressing step into fragments which pass a screen with one-fourth inch openings, adjusting the moisture content to from ten per cent to twenty per cent of the comminuted cake, heating the material to a temperature of from 145° F. to 200° F., and subjecting the fragments while hot to the action of flaking rolls to convert them into wide thin flakes from .005 to .02 inch thick, whereby the aforesaid solvent extraction step can be performed with materially reduced development of fines.

8. A process as defined in claim 7 and wherein the oil content is controlled by means of the expressing step so that the oil content is from eight to eighteen per cent of the total weight of the press cake after the expressing step.

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