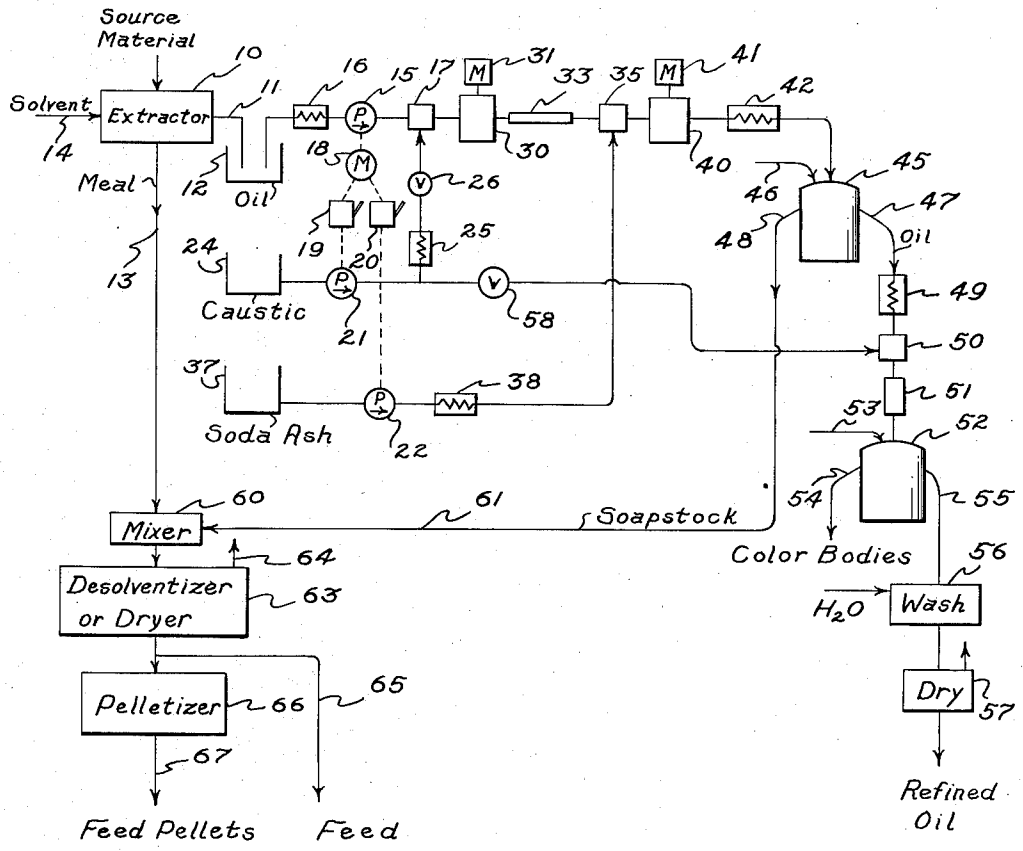
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PROCESS OF PRODUCING FEED PRODUCTS

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PROCESS OF PRODUCING FEED PRODUCTS

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My invention relates to a process for producing feed products, particularly animal feed products, from beans, seeds or other oil-bearing vegetable material.

Such source material is conventionally processed by pressing, solvent action or both to extract crude glyceride oil therefrom, leaving a residual meal which is widely used as an animal feed or an additive thereto. Particularly if the oil is extracted by solvent extraction, the meal contains very little residual oil and it is desirable that its fat content be increased by adding thereto oils or fats from other cheap sources thereof. Much of the oil is commercially refined by mixing aqueous caustic soda therewith in sufficient stoichiometric excess to react with the free fatty acids of the oil and produce soaps which appear as a part of the soapstick separated from the oil by gravitational or centrifugal action.

It has been proposed to return such separated caustic soapstock to the extracted meal to supply the fat deficiency thereof. However, the resulting feeds leave much to be desired as concerns nutritive and growth factors. One reason for this is that the caustic alkali reacts with or degrades the gums of the oil, which appear in the soapstock as constituents of little or no value because the otherwise valuable gums have been degraded or destroyed by the caustic alkali, making them relatively ineffectual in promoting the desired growth of the animal. Such caustic-degraded gums are not soluble in petroleum ether.

It has also been proposed to employ soda ash as the alkali, the gums in the separated soapstock being then not substantially alkali-degraded. Nevertheless, it is known that soda ash refining does not usually reduce the color or other impurities of the oil to a sufficient extent, requiring a subsequent refining with caustic to produce a high quality marketable oil. This, together with the large excesses of soda ash and the new equipment costs involved, makes this process not commercially attractive to many refiners.

It has now been found that refining by the joint use of caustic and soda ash can produce unexpected benefits if the caustic is employed in carefully limited amount. If the two alkalis are employed sequentially, the resulting soapstock makes an excellent feed additive. The gums therein are intact in the sense of being substantially free of alkali degradation.

It is an object of the invention to provide a novel process of producing feeds by refining a crude glyceride oil by the joint use of caustic and soda ash, separating the resulting soapstock and adding same to a feed product, typically to the meal remaining after the oil has been extracted from the source material by any conventional method.

A further object lies in the refining of the oil by careful control of the amount of caustic employed. In this respect, it is an object of the invention to limit the caustic to an amount about 50–85% of that which would be necessary to neutralize all of the free fatty acids. It is a further object of the invention to add soda ash to the oil-caustic mixture while containing residual fatty acids.

A further object of the invention is to provide a process for refining the crude oil to produce a soapstock which contains both caustic and soda ash soaps along with the gums of the oil in substantially undecomposed state. A further object is to produce a soapstock of this type the gums of which are substantially completely soluble in petroleum ether. Another object is to control the

steps of the process to produce a soapstock which separates cleanly from the oil, the entire process minimizing refining losses and being well balanced in the economic value and usefulness of the various products produced.

Further objects and advantages of the invention will be evident to those skilled in the art from the herein exemplified embodiments of the invention.

The drawing is a pipe-line diagram of one type of apparatus capable of carrying out the processes of the invention.

The source material, typically seeds or beans suitably ground or flaked and sometimes precooked, are delivered to an extractor **10**. Here oil is extracted from the source material by pressing, solvent action or both, the oil being delivered through a pipe **11** to a tank **12** for temporary storage, the extractor producing a meal advancing along a path or line **13**. A pipe **14** may supply an oil solvent to the extractor **10**, this solvent being either removed ahead of the tank **12** or remaining wholly or partially associated with the oil throughout the subsequent steps in the process if it is desired to practice the process in the miscella stage. In the latter event, the tank **12** will be made pressure tight or may be eliminated.

The crude oil may be withdrawn from the tank **12** by a pump **15**, being heated if desired in a heat exchanger **16** and being delivered as a continuous stream to a flow mixer **17**. The pump **15** is a part of a proportioning system, diagrammatically shown as driven by a motor **18** which, through speed changers **19** and **20**, drives pumps **21** and **22** at speeds proportional to the pump **15**.

The pump **21** withdraws an aqueous caustic solution from a tank **24** and delivers a stream thereof through a heat exchanger **25** and a valve **26** to the flow mixer **17** where the caustic and the oil are initially mixed. The flow mixer may be merely a pipe junction if desired. The mixture is sent immediately to a pipe-line mixer **30** including a suitable rotary agitator driven by a motor **31** to effect a very thorough and uniform mixing of the oil and caustic in a very short period of time. Due to the fact that the amount of caustic proportioned by the pump **21** is insufficient to neutralize the free fatty acids of the oil, the mixture discharged from the mixer **30** will contain caustic soaps, residual fatty acids and gums substantially free of caustic degradation. The time in the mixer **30** is a matter of seconds and it is often desirable to allow for a brief hold-up period beyond the mixer to condition the mixture before the soda ash solution is added. For this purpose, the mixture may flow through a larger pipe section **33**, typically a five-inch pipe several feet in length, before reaching a flow mixer **35**.

A suitable soda ash solution is withdrawn from a tank **37** by the pump **22** and may be adjusted in temperature by passage through a heat exchanger **38** before being delivered to the flow mixer **35**. Again, the resulting mixture is immediately delivered to a pipe-line mixer **40** driven by a motor **41** to mix the soda ash solution therewith thoroughly and in a short interval of time. The stream of the resulting mixture may be adjusted in temperature by flow through a heat exchange device **42** before being delivered to the separation equipment. At this stage of the process, the oil stream contains caustic soaps, soda ash soaps, and gums free of alkali degradation, plus water which has been added as a part of the caustic and soda ash solutions. In addition, the stream may contain the excess soda ash and part or all of the oil solvent if the refining is to take place in the miscella. The soapstock separable from the stream will include the aforesaid soaps and gums, together with a minimum amount of entrained oil.

While the soapstock separation can be effected batchwise and by gravitational methods, I prefer to employ continuous centrifugal separation effected in a centrifuge **45**

which may be of the closed or hermetic type, particularly if some of the oil solvent is present in the entering stream. Water or aqueous solutions of separation-promoting agents, typically salt solutions, can be proportioned into the centrifuge 45 through the pipe 46 to facilitate the separation of the soapstock from the oil but are usually not needed. The oil and soapstock respectively discharge through spouts 47 and 48 of the centrifuge.

The separated oil may or may not be in a marketable condition at the time of discharge from the centrifuge 45. Usually it is desirable to rerefine this oil to remove additional color bodies and other impurities. This can be done in accordance with the process of Clayton Re. 23,680, using a concentrated caustic solution. Thus, the oil may be adjusted in temperature by passage through a heat exchanger 49 and may be mixed with a concentrated caustic solution in mixers 50 and 51 before being delivered to a centrifuge 52. Water or aqueous diluent is added to the entering mixture or to the interior of the centrifuge through a line 53. In this way the centrifuge 52 discharges an aqueous material containing the color bodies through a spout 54 and a purified oil through a spout 55, the purified oil being washed and dried in suitable washing equipment 56 and drying equipment 57 to produce the desired refined oil. If, as is desirable, the caustic solution in the tank 24 is relatively concentrated, this same caustic solution can be used in the rerefining steps, being proportioned either by the pump 21, as shown, or by a separate pump. The drawing shows a portion of the caustic solution from the pump 21 flowing through a valve 58 to the mixer 50.

The meal in the line 13 is delivered to a mixer 60 which also receives all or a portion of the soapstock from the spout 48 through a line 61. The resulting mixture advances to a desolventizer or dryer 63 wherein heat is applied to remove water vapors and vapors of residual solvent, indicated by the arrow 64. In some instances, the mixer 60 and the desolventizer or dryer 63 can be unified. The presence of solvent facilitates the uniform mixing of the soapstock in the meal. In addition, the heat in the desolventizer can be employed to detoxify gossypol present in most cottonseed meals or soapstocks. Necessary detoxifying temperatures are less when applied to the mixture than detoxifying temperatures applied separately to the meal and the soapstock. The resulting soapstock-fortified meal may be discharged through a line 65 as the feed product or it may be sent to pelletizing equipment 66 for formation of feed pellets discharging at 67.

Usually all the separated soapstock can be advantageously returned to the meal but lesser amounts can be employed if desired. Amounts from a fraction of a percent up to 5 percent are usually employed. The temperature in the dryer 63 is usually in the range of 180 to 250° F., the higher temperatures in this range effecting substantial detoxification of gossypol-containing soapstocks or meals or both.

One of the most important features of the present invention is the limitation in amount of caustic soda mixed with the crude oil. In normal processes in which sufficient caustic soda is employed to react with all of the free fatty acids, the excesses over stoichiometric amounts result in an attack on the gums or phosphatides, changing them from a beneficial petroleum-ether-soluble form into a degraded form of substantially less value not substantially soluble in petroleum ether. On the other hand, it is a feature of the invention that the amount of caustic should be less than that required to neutralize the free fatty acids, the alkali being then largely selective in its action and being quickly expended in neutralizing a corresponding amount of the free fatty acids, with little or no decomposition of the gums and little or no reaction with the oil, particularly if the mixing is rapid and uniform. Non-uniform mixing tends to form local pockets of strong caustic that can saponify neutral oil or attack the gums. However, by use of the flow mixer 17 and the pipe line mixer 30, the relatively strong but limited amount of caustic is mixed very thoroughly and rapidly with the oil. It is desirable that the mixing at this stage be completed in as short a time as possible. Best results have been obtained by mixing with the oil an amount of caustic equal to about 50–85% of that amount that would be necessary to neutralize the free fatty acids of the oil. The remaining or residual fatty acids are then neutralized by the later-added soda ash solution, with many attendant advantages.

The caustic soda is preferably added in aqueous solution. Best results have been obtained by employing a relatively concentrated solution but solutions ranging from 12° Bé. to as high as 50° Bé. can be employed. The lower concentrations are best for oils low in free fatty acids, e.g., less than 1%, while the higher concentrations are best for the higher free fatty acid oils, e.g., 5% or greater. The preferred range of concentrations is about 20–36° Bé.

The soda ash is likewise preferably added in the form of an aqueous solution. Solutions ranging from about 10° Bé. to saturation can be employed, the range of about 10–22° Bé. being preferred with 20° Bé. solution being satisfactory in most instances.

Sufficient soda ash solution should be added to complete the reaction of the free fatty acids. In addition, if the caustic deficiency has been such as to produce acid soaps, the amount of soda ash should be sufficient to react therewith. Such acid soaps are much more soluble in the oil than normal soap but are converted into the latter by the soda ash employed. The actual amount of soda ash employed will be in excess of that stoichiometrically equivalent to the residual fatty acids. Preferably the excess should be sufficient to prevent the evolution of carbon dioxide in or ahead of the centrifuge 45.

It is difficult to set forth precisely the exact amount of soda ash solution that will be best in all processes, particularly as there are factors other than residual free fatty acids, suppression of carbon dioxide, etc. that enter the picture. I have found that best results follow from the use of an amount of soda ash calculated on the following basis:

$$\text{Percent dry soda ash} = 0.075\text{–}0.15 \times \text{Wesson Loss of crude oil.}$$

This formula gives good results if, as is contemplated, the amount of caustic soda is in the range of about 50–85% of that required to neutralize the free fatty acids, irrespective of the exact amount employed within that range. The Wesson Loss is a well known property of glyceride oils, being expressed in a percentage figure and being determined by the method described on pages 400–402 of "Vegetable Fats and Oils" by G. S. Jamieson, published in 1932 by The Chemical Catalog Company, New York, N.Y. If this numerical figure is multiplied by 0.1, the result will be the desirable amount of dry soda ash to be employed on most oils. For oils high in gum content and low in free fatty acid content, the multiplication factor may be between 0.1 and 0.15, often at or near the upper value of this range. For oils of very high Wesson Loss the multiplication factor may be as low as 0.075.

An example in which the formula is applied is as follows:

A crude soya oil containing 0.5% free fatty acids and 1.5% gums and having a Wesson Loss of 2.0% can be refined in accordance with the invention by adding 0.15% of a 30° Bé. (24%) solution of caustic soda, representing 50% of the amount required to neutralize the free fatty acids. The amount of soda ash to add, using a multiplication factor of .1 in the above equation, is .20% dry soda ash. This corresponds to 1.33% of a 20° Bé. soda ash solution. The soapstock from this refining will contain about 30.8% moisture and 20.0% free oil, dry basis.

In another example, a crude cottonseed oil containing 5.0% free fatty acids and 1.5% gums with a Wesson

Loss of 6.5%, is refined by adding 2.5% of 30° Bé. caustic soda solution, representing 85% of the amount required to neutralize the free fatty acids. Using the multiplication factor of 0.1 in the above equation, the dry soda ash equals .65%. If a 20° Bé. solution is to be employed, 4.3% thereof would be the calculated amount. The soapstock from this process will contain about 35% moisture and about 20% free oil, dry basis.

The addition of soda ash to the mixture of oil, caustic soaps, residual free fatty acids and gums or phosphatidic materials results in a number of advantages. The soda ash not only completes the reaction with the free fatty acids but makes the gums and soapstock more insoluble in the oil, thereby making it possible to effect a more complete, efficient and effective refining of the oil. If any acid soaps have formed, these are neutralized and made insoluble in the oil. If any localized concentration of the caustic exists, resulting from imperfect mixing, this is quickly dissipated and the localized causticity reduced by the soda ash solution, which also acts as a buffer to prevent any substantial attack by the caustic on the gums or phosphatides so that the latter remain intact and in a form soluble in petroleum ether. Addition of the soda ash solution also assists in breaking the emulsion so that the separated soapstock has a minimum content of occluded oil, thus increasing the oil yield from the complete process and the economic value thereof. Further, the addition of the soda ash solution after the caustic gives a soapstock which is readily separable and pumpable, having a desirable moisture content, e.g. about 25–45% usually about 30–35%. Finally, the soda ash soaps and the carbonated gums, present in the soapstock along with the caustic soaps, appear to augment to an unexpected degree the growth and nutritive value of meals or feeds to which they are added.

The temperature in the various portions of the process is not particularly critical. Best centrifugal separation in the centrifuge **45** is obtained if the entering mixture is about 190–200° F., although temperatures of 160–210° F. can be employed. The oil from the extractor **10** is usually cooled to about 80–100° F. the additional heat can be supplied partly or entirely by the heat exchanger **42** just ahead of the centrifuge **45**. Sometimes, however, a portion of the heat may be supplied in the heat exchanger **16**. In addition, the caustic solution and the soda ash solution can be heated in their respective heat exchangers **25** and **38**.

The oil issuing from the spout **47** of the centrifuge **45** will usually contain less than 0.10% free fatty acids while the soapstock issuing from the spout **48** will contain about 15 to 22% free oil, usually about 20% thereof, dry basis. If the oil is to be rerefined, this may be done by mixing therewith a relatively strong caustic solution of about 20–50° Bé. before or while the oil is at a temperature of about 80–100° F., preferably, and the mixture subsequently heated to 120–180° F. before centrifuging. To permit separation into the two phases previously mentioned, the resulting mixture is diluted by water or other aqueous diluting agent added through pipe **53** to the mixture or to the interior of the centrifuge. The amount of the diluting agent will desirably be such as to reduce the concentration of the excess caustic solution to 10° Bé. or less. The amount of strong caustic solution added through the mixer **50** will be substantially in excess of the amount required to react with any residual impurities in the oil and will usually be 0.5–3.0% of the caustic solution.

The following examples will illustrate typical applications of the process in practical operation.

Example I

As an example of a process employing caustic soda solution in amount about 70% of that required to neutralize the free fatty acids, a crude cottonseed oil having 2.7% free fatty acids, a Cup Loss (by the Official Method, American Oil Chemists' Society) of 9.7% and a Wesson or Absolute Loss of 4.5% was refined with 1.12% of 30° Bé. caustic soda solution proportioned in flow into the crude oil at a temperature of 90° F. The solution was very rapidly and intimately mixed, using a pipe-line mixer **30** with its agitator rotating at a speed of 3600 r.p.m. After a residence time in flow of 20–30 seconds in the larger pipe section **33**, 2.6% of 22° Bé. soda ash solution (17.5% Na_2CO_3) was proportioned into the mixture, the resulting mixture being quickly mixed in a similar pipe-line mixer and then heated to 190°–200° F. in the heat exchanger **42**. The resulting centrifuged oil was rerefined with 1.25% of 30° Bé. caustic soda, water-washed and vacuum-dried. The refining loss was 5.63%, which loss was 42.0% lower than the official Cup Loss. The entire soapstock, separated in the centrifuge **45**, is desirably returned to the meal ahead of the desolventizer or drier **63**. The amount of soapstock in the meal was about 2.75% by weight.

Example II

Evidencing an operation in which the amount of caustic was 80% of the amount that would be required to neutralize the free fatty acids, a crude cottonseed oil containing 8.4% free fatty acids, Cup Loss 24.3%, Wesson Loss 10.84%, was refined with 4.0% of 30° Bé. caustic soda and 5.6% of 24° Bé. soda ash solution (19.2% Na_2CO_3) was mixed in the same manner and under the same conditions as Example I. The separated oil was rerefined with 1.5% of 30° Bé. caustic. The refining loss was 14.68% or 39.6% lower than the Cup Loss. This represents a saving of 5,772 lbs. for a tank car of 60,000 lbs. of crude oil. The soapstock was an excellent feed additive.

Various changes and modifications can be made without departing from the spirit of the invention as defined in the appended claims.

I claim as my invention:

1. A process for producing animal feed products from source materials such as vegetable seeds and beans, which process includes the steps of: processing said source material to extract therefrom a crude glyceride oil and produce a residual meal, said oil containing gums and free fatty acids; mixing with the crude oil an amount of caustic sufficient to neutralize only about 50–85% of the fatty acids of the oil thereby producing a mixture of oil containing soaps, residual fatty acids and gums; mixing with said mixture before separation of the constituents thereof an amount of soda ash in excess of that required to neutralize said residual fatty acids to produce a resulting mixture, said soda ash being added in aqueous solution, the amount of the soda ash being approximately the amount determined by the equation:

$$\text{Soda ash} = 0.075\text{–}0.15 \times \text{Wesson Loss of the oil}$$

where the amount of soda ash is expressed as percent of dry soda ash; separating from the resulting mixture a soapstock containing caustic soaps, soda ash soaps and gums free of substantial alkali degradation and substantially completely soluble in petroleum ether and mixing at least a part of said separated soapstock with said meal.

2. A process for producing an animal feed product, comprising: mixing with a crude glyceride oil containing free fatty acids and gums and of known Wesson Loss an amount of caustic soda solution sufficient to neutralize only about 50–85% of such free fatty acids to form caustic soaps therein; then mixing with the oil while said caustic soaps and gums are present an amount of soda ash determined by the equation:

$$\text{Soda ash} = 0.075\text{–}0.15 \times \text{Wesson Loss}$$

where the amount of soda ash is expressed as percent of dry soda ash; separating the resulting mixture into oil and soapstock containing caustic soaps, soda ash soaps, gums substantially free of alkali degradation and substantially completely soluble in petroleum ether, and water; and

adding to a feed product an amount of said soapstock ranging from a fraction of one percent to about five percent.

3. A process for producing from a crude glyceride oil containing gums and free fatty acids a soapstock having excellent growth and nutritive factors when added to a feed product, said oil having a known Wesson Loss, which process includes the steps of: mixing with the crude glyceride oil an amount of aqueous caustic soda solution sufficient to neutralize only about 50–85% of such free fatty acids to form caustic soaps in the oil; then mixing with the oil while said caustic soaps and gums are present an amount of soda ash determined by the equation:

$$\text{Soda ash} = 0.075\text{–}0.15 \times \text{Wesson Loss}$$

where the amount of soda ash is expressed as percent of dry soda ash, said soda ash being mixed with the oil in aqueous solution; and separating the resulting mixture into oil and soapstock containing caustic soaps, soda ash soaps, gums substantially free of caustic degradation and substantially completely soluble in petroleum ether, and water.

4. A process as defined in claim 3 in which said aqueous caustic soda solution is of a concentration about 12–50° Bé., in which the aqueous soda ash solution is of a strength ranging from about 10° Bé. to saturation, and in which sufficient water is present during the separation of the soapstock to produce a soapstock containing about 25–45% water.

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