

in the solvent-extracted peanut meal or cottonseed meal is very high in free fatty acids.

### Summary

The nonprotein material in protein prepared by the usual methods from solvent-extracted peanut meal has been investigated. It consists of sugars, nonprotein nitrogenous substances, lipids, and ash. The sugars are practically all reducing sugars. The nonprotein nitrogenous material is amino acid in nature with some peptide linkages. Its nitrogen distribution does not differ greatly from that reported for peanut protein. The chief component of the lipid material is fatty acids possibly including some of the chain length longer than  $C_{18}$ . There are also present some phosphatides. Protein prepared similarly from cottonseed meal contains similar nonprotein materials.

The sugars, amino acid material, and lipids are removed to a considerable extent by washing the protein with alcohol in the wet curd state. This removal is more efficient if the curds have not been coalesced by dewatering treatment.

Washing with alcohol makes drying of the protein easier and seems to remove some color, but reduces solubility to some extent. Ash content is slightly lowered by washing with alcohol and phosphorus content is not appreciably changed. Since washing with alcohol removes nonnitrogenous material such as lipids and sugar, the nitrogen content of protein preparations is increased by such treatment.

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## A Study of the Caustic Refining of Vegetable Oils

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CRUDE vegetable oils contain free fatty acids, phosphatides, color bodies, and other colloidal substances. The usual refining procedure is to mix the crude oil with a caustic soda solution which results in the formation of soapstock, usually in a finely divided condition, apply heat to the oil-soapstock mixture which causes the agglomeration of the soapstock, termed the "break," and then separate the soapstock from the refined oil. The refined oil thus obtained contains varying amounts of soapstock in suspension and moisture in solution. To remove this residual soapstock the refined oil is washed one or more times with hot water, with the separation of the water as far as possible between washings by centrifuging, and the ultimate drying of the oil usually under a vacuum. Instead of washing the oil, the suspended soapstock can be agglomerated with heat in settling tanks and the thus partially clarified oil pumped through filter presses containing spent fuller's earth.

Until about 12 years ago the mixing and heating were accomplished in tanks equipped with paddle agitators and steam coils or jackets. This process is still used with some oils that are not satisfactorily

refined using the present-day continuous process of refining which has largely supplanted the kettle process. In the present continuous process of refining the caustic solution is injected into a flowing stream of the oil in increments by means of a proportioning device termed a proportionometer which regulates the flow of the caustic solution in proportion to the oil flow. The resultant oil-caustic mixture is caused to flow first through a chamber containing rapidly revolving paddles, which mixes the oil and caustic solution, then through the tubes of a hot-water-jacketed heat exchanger where the mixture is heated to obtain the "break," with the temperature reached being usually about 140°F., and finally to a centrifuge where the soapstock is separated from the refined oil. This process is termed the Clayton continuous process (1).

In the kettle and the Clayton processes the oil and caustic solution are mixed as liquids. Recently, a new continuous process of refining has been developed which mixes the oil and caustic solution in the form of mists, collecting the mist-mixture on the side of a steam-jacketed tank down which the mixture flows in liquid form during which time the mixture is heated

to obtain a "break," and then separating the soapstock from the oil by means of a centrifuge(2).

In refining vegetable oils using liquid-mixing, empirical procedures have through the years been developed without an understanding of the mechanism which necessitates these procedures. Some of these procedures are:

1. The oil must be mixed with caustic solution in the cold, usually a temperature of 70° to 80°F., for an appreciable time.

2. The concentration of the caustic solution used is increased with an increase in the free acid content of the oil.

3. The caustic used beyond that necessary to neutralize the free acid must be increased with an increase in the free acid content of the oil.

The details covered by these procedures are different for various kinds and types of oil. The purpose of this paper is to show that these variations in procedure are mainly due to the kind and amount of phosphatides present in the oil and to the fact that the oil and caustic solution are mixed as liquids. In developing this viewpoint, this theory will be applied to the refining of fast- and slow-breaking hydraulic cottonseed oil, expeller cottonseed oil, soya bean oil, de-gummed soya bean oil, and re-refining. Consideration will first be given to liquid-mixing refining as applied to cottonseed oil using the cup refining test of the A.O.C.S. to illustrate the various points.

#### Mixing Time in Cold

When the caustic solution is mixed with the oil, there is an immediate neutralization of the free acids which results in the formation of finely divided soap in which is included the phosphatides, some of the color bodies, colloidal matter, excess caustic solution, and neutral oil. This mass is termed soapstock. If this soapstock is immediately separated from the oil, the neutralized oil is dark in color and still contains highly dispersed colloidal matter. To obtain a light-colored refined oil and remove the colloidal matter the finely divided soapstock must be mixed with the oil. In the case of fast-breaking type cottonseed oil this time is 15 minutes and is 45 minutes for the slow-breaking type. Likewise, the cold mixing time of expeller type cottonseed oil is 45 minutes. No explanation has heretofore been advanced as to the mechanism of the action which results in the reduction in color of the oil during this period of mixing nor of the mechanism which requires these various mixing times.

#### Concentration of Caustic Solution

Usually two concentrations of caustic solution are allowed for refining a given oil. The concentrations given are dependent upon the free acid content of the oil and the kind and type of oil; e.g., a greater concentration of caustic solution is used for refining expeller cottonseed oil than for hydraulically produced cottonseed oil for the same free acid. The color of the refined oil obtained and the per cent of refining loss are affected by the concentration. The mechanism which results in variations in refining loss by reason of differences in the concentration of the caustic solution used has been heretofore unexplained.

#### Excess Caustic

With an increase in the free acid of an oil a greater excess of caustic is required to obtain the lowest color

in the refined oil with the lowest refining loss. The excess caustic used varies with the kind and type of oil; e.g., a greater excess caustic is required for expeller cottonseed oil than for hydraulic cottonseed oil; and a smaller excess caustic is used for refining soya bean oil than is used for cottonseed oil. The reason for the necessity of these variations in the excess caustic required has heretofore not been known.

Experience has shown that a high-colored refined oil and a high refining loss are obtained when:

1. The time of mixing of the oil-soapstock mixture in the cold is too short or too long.

2. The temperature of the oil at the time the oil and caustic solution are mixed is too high.

3. The concentration of the caustic solution is too great.

4. The excess caustic used is insufficient.

5. The time of heating the oil-soapstock mixture to obtain the "break" is too great.

6. The concentration of the caustic solution is too weak.

All of the hitherto unexplainable results obtained in the refining of vegetable oils are understandable by the application of the following theoretical concepts:

1. The color particles and colloidal substances present in crude vegetable oils are in colloidal suspension by reason of positive electrical charges.

2. The soap formed in the refining process is negatively electrically charged and these negative charges neutralize the positive charges on the color particles and colloidal substances in suspension in the oil which results in their being included in the soapstock.

3. When these positive charges are neutralized, the electrically neutral color particles and colloidal matter are soluble in the oil, the rate of solution and amount dissolved being dependent upon pH, temperature, time, agitation, and material.

The theory that the color in crude cottonseed oil and some of the color in refined cottonseed oil carries positive electrical charges is supported by the results of the following tests:

Crude cottonseed oil was allowed to flow in an atmosphere of nitrogen in a flat trough under an aluminum window placed in the wall of a Crookes tube at the rate of two drops per second for 11,445 seconds. A potential of 90 kilovolts was applied to the Crookes tube thus causing Lenard rays to be emitted through the window. Since Lenard rays consist of negatively charged particles, the treatment of the oil with these rays should neutralize positive charges in the oil resulting in an oil which would refine to a lower color. The following are the refining results on the original and treated oils:

	Free Acid	Loss	Color 35.0 Y
Treated.....	0.8%	6.0%	4.8 r
Untreated.....	0.8%	6.4%	4.9 r

The soapstock obtained from the treated oil was firmer than that from the untreated oil.

Refined cottonseed oil was treated in a similar manner for 17,815 seconds. If the negative charges of the Lenard rays neutralized positive charges on color particles in the oil, then the color of the oil should be

increased because the tinctorial power of the color particles in solution is greater than that in the charged condition. The following are the results obtained:

	Free Acid	Color 35.0 Y
Treated.....	0.040%	4.8 r
Untreated.....	0.025%	4.5 r

The color change in the case of the crude oil is within the limits of experimental error and therefore is inconclusive. The results in the case of the refined oil lend support to the theory.

### Cottonseed Oil

When a caustic solution is mixed with cottonseed oil, the color removal is dependent upon the negative charges on the soap formed contacting the positive charges on the color particles. Since the caustic combines immediately with the free acids in the oil with the formation of soapstock particles of appreciable size, the charged surface of the soap particles is greatly reduced, so mixing of the oil and soapstock particles must be continued in order to bring fresh oil in contact with the surface of the soapstock particles. During this mixing saponification of some of the oil in contact with the caustic solution on the surface of the soapstock particles occurs with the formation of additional negative charges which are available for neutralizing the positive charges on the suspensoids present in the oil. As the concentration of the caustic on the surface of the soapstock particles is reduced, caustic from the interior of the particles diffuses to the surface, thus gradually reducing the caustic concentration (4). If the concentration of the original caustic solution is too great or the starting temperature of the oil too high, the consumption of caustic will be so rapid that an insufficient amount of positive charges on the color particles will be neutralized before the caustic is exhausted, thus resulting in obtaining a dark-colored refined oil. If the concentration of the original caustic solution is too low, the rate of saponification of neutral oil will be slow, thus retarding the formation of sufficient negative charges to obtain refined oil of a satisfactory color. Also the rate of diffusion of the caustic from the interior of the soapstock particles to the surface will be slower than if a more concentrated solution of caustic had been used originally, thus further contributing to a slow rate of saponification of neutral oil. These factors result in the refined oil being high in color.

The electrically neutral color particles and colloidal matter are slightly soluble in the oil. The partition of these materials between the oil and soapstock is partially governed by the pH of the soapstock; the higher the pH of the soapstock the less will be the amount of these materials dissolved in the oil in a given time, other conditions being the same. When sodium carbonate or lime solutions are used to refine an oil the color of the refined oil is extremely high. This high color is due to the low pH of these solutions and to the fact that these reagents cannot saponify neutral oil so as to produce the additional negative charges required to neutralize the positive charges on the suspensoids in the oil. The electrically neutral color bodies dissolved in the oil will yield a dark-colored oil while the dissolved electrically neutral colloidal matter will stabilize a water-in-oil emulsion in which

the soapstock is the water phase. The amount of this colloidal matter dissolved in the oil is also governed by the length of time of contact of the soapstock with the oil and the amount of agitation. This emulsion prevents the proper separation of the soapstock and oil from each other, thereby increasing the refining loss.

The oil in soapstock is present as a water-in-oil emulsion stabilized by the electrically neutral colloidal matter dissolved in the oil, as an oil-in-water emulsion stabilized by the lecithin present in the oil and as occluded oil. That the soap formed from the free acids is not the stabilizing agent is apparent when the fact is considered that the concentration of the original caustic solution is so high that the solution of the soap in the water of the caustic solution is prevented by the salting-out effect. At the end of the refining operation when the concentration of the caustic has been reduced, some oil-in-water emulsion may be formed that is stabilized by the soap. This idea that the lecithin is the stabilizing agent is supported by the low oil content of the soapstock from re-refining. Thus soapstock contains a multiple emulsion because the oil occurs both as an oil-in-water emulsion and as a water-in-oil emulsion (3). The oil in soapstock, when the refining is properly conducted, is mainly present as an oil-in-water emulsion. If too strong a concentration of caustic solution is used in refining or if the oil-soapstock mixture is heated rapidly so that considerable excess caustic remains at the end of the refining operation, the soapstock will contain occluded oil because the soapstock particles will not fuse together because of the salting-out effect of the strong caustic solution. This brings up the subject of the conditions that accompany a satisfactory "break."

By varying the operating conditions using the same oil, concentration of caustic and amount of caustic solution, the "break" can occur at any temperature from 100°F. to 180°F. The "break" occurs by reason of the concentration of the caustic on the surface of the soapstock particles being below the salting-out concentration. Thus with oils having a high free acid content, a "break" occurs rapidly because a considerable number of soapstock particles have the concentration of the caustic reduced by the free acids, permitting agglomeration. By stirring the oil-soapstock mixture of a low free acid oil for a long time at a temperature of 100°F., the oil will "break." By heating the mixture rapidly, the "break" can be caused to occur at 180°F. These statements do not mean that the refining losses will be satisfactory at the different temperatures.

The above statements have been given regarding the liquid-mixing refining of cottonseed oil in general. The theories and facts stated will be applied to specific types of cottonseed oil, to soya bean oil, and to a comparison of the kettle process, the Clayton process, Chisholm process, and the mist-mixing process of refining for the purpose of explaining the reasons for the different methods used in refining various kinds and types of oil and the difference in results obtained by the various refining processes.

### Fast-breaking Cottonseed Oil

When fast-breaking cottonseed oil is refined, the soapstock obtained contains about 18% to 20% oil which is lower than that resulting from refining the

slow-breaking type of oil. This lower content of oil is due to the smaller amount of lecithin in the fast-breaking type oil. When the fast-breaking type oil and caustic solution are mixed, the smaller amount of oil-in-water emulsion formed, because of the lower content of lecithin, permits a more rapid diffusion of the caustic from the interior of the soapstock particles to the surface so that the positive charges on the color particles are rapidly neutralized by the negative charges formed on the surface of the soapstock particles by the saponification of oil, thus rapidly refining the oil. For this reason, the mixing time at the cold temperature need be only fifteen minutes. Likewise, the rapid consumption of caustic permits the soapstock particles to fuse together so that a satisfactory "break" is obtained. A fast-breaking type oil having the same free acid content as a slow-breaking oil will have a materially lower refining loss.

#### Slow-breaking Cottonseed Oil

When slow-breaking cottonseed oil is mixed with the caustic solution, a larger amount of an oil-in-water emulsion is formed than is the case in the refining of fast-breaking oil because of the presence of a greater amount of lecithin in the former. This emulsion retards the diffusion of the caustic to the surface of the soapstock particles and therefore a longer mixing in the cold is required to obtain the desired color and "break." The cold-mixing time of this type of oil is 45 minutes. If the cold-mixing time for fast-breaking oil were used with slow-breaking oil, the resultant soapstock would contain a large amount of occluded oil because the soapstock particles would not fuse together on account of the high concentration of the caustic whose consumption had been retarded by the reduction in the rate of diffusion of the caustic to the surface of the soapstock particles by the oil-in-water emulsion. While there would be some increase in color of the refined oil obtained by this reduced cold-mixing time with slow-breaking oil, the principal effect would be to increase the refining loss.

#### Expeller Cottonseed Oil

When expeller cottonseed oil and the caustic solution are mixed, an immediate "break" occurs, the soapstock being present in large flakes. The excess caustic is contained in these flakes. Because of the small surface of these flakes the oil-soapstock mixture must be mixed for 45 minutes in the cold in order to effect the necessary saponification of oil to obtain sufficient negative charges to neutralize the positive charges on the suspensoids in the oil and to reduce the caustic concentration to the point where the soapstock flakes will fuse together.

#### Re-refining

In re-refining vegetable oil the absence of lecithin prevents the formation of any appreciable quantity of an oil-in-water emulsion. Therefore in order to obtain sufficient saponification of oil so as to obtain the necessary negative charges to neutralize the positive charges on the remaining color suspensoids present in the oil, a high concentration caustic solution is used on the order of 20° Bé. The soapstock formed does not agglomerate because such a large amount of caustic solution is used (usually about 2%) that the caustic is not consumed sufficiently to permit the fusion of the soap particles. As a result, the soapstock consists of a bottom layer of a strong caustic

solution red in color and an upper layer of unagglomerated soap. Because of the absence of lecithin, the color reduction may be a few points only of red. The refining loss is usually from 1½% to 2%.

#### Soya Bean Oil

While crude soya bean oil contains a larger amount of phosphatides than crude cottonseed oil, the refining loss is less for soya bean oil than for cottonseed oil having the same amount of free acid. This result is due in part to the low oil content of the soya bean oil soapstock, which is only about 4%. This is contrary to the results which would be expected, based on the effect on the refining loss in the case of fast-breaking and slow-breaking cottonseed oil and caused by an increase in the amount of phosphatides in the oil. This low oil content of the soya bean oil soapstock can be attributed to the composition of the phosphatides present in the oil.

The phosphatides in soya bean oil consist of about 40% lecithin and 60% cephalin. Cephalin is not an emulsifying agent under the refining conditions as is lecithin. When the soya bean oil is mixed in the cold with the caustic solution, the cephalin coats the particles of soapstock, thus greatly retarding the formation of an oil-in-water emulsion stabilized by lecithin, and at the same time the cephalin reduces the rate of diffusion of the caustic from the interior of the soapstock particles to the surface. Because of this interference with diffusion, the oil-soapstock mixture has to be mixed 90 minutes in the cold in order to reduce the caustic concentration to the point where the soapstock particles will fuse together and the positive electrical charges on the color particles and colloidal matter will have been neutralized.

#### De-gummed Soya Bean Oil

When crude soya bean oil is mixed with water, a major portion of the phosphatides is precipitated and can readily be separated from the oil. This de-gummed oil is difficult to refine, using liquid-mixing. When de-gummed oil is refined, it behaves in a manner similar to refined cottonseed oil when it is re-refined. The principal difference is the immediate formation of soap from the free acid in the soya bean oil. The de-gummed oil contains positively charged color particles and colloidal matter whose charges have to be neutralized by the negative charges on the soap formed mainly from the saponification of neutral oil during the time of mixing in the cold. By the reduction of the phosphatide content in the de-gumming operation, sufficient emulsification to incorporate the caustic solution in the soapstock does not occur. As a result, the formation of a two-layer soapstock is not infrequent.

#### Kettle and Clayton Processes

In the kettle and Clayton processes the oil and caustic solution are mixed as liquids. All of the above statements regarding refining in the liquid state are applicable to these processes. However, the more vigorous and rapid mixing in the Clayton process, as well as the more rapid heating, accentuates undesirable features of liquid-mixing refining.

In the Clayton process because of the turbulence in the mixing operation and the time required for the oil-soapstock mixture to pass through the mixing chamber and heat exchanger, some electrically neutral colloidal matter contained in the soapstock is

dissolved in the oil. As previously stated, this electrically neutral colloidal material stabilizes a water-in-oil emulsion in which the soapstock constitutes the water phase. In the centrifuge which separates the refined oil from the soapstock the mass of the soapstock is readily separated from the oil. However, smaller pieces of soapstock remain suspended in the oil as a water-in-oil emulsion. This soapstock has to be removed by one or more washings with hot water, with centrifuging after each washing to separate the water. After the final washing and centrifuging, the oil is still saturated with water which is removed by vacuum drying. The soap contained in the water evaporated from the oil becomes dry and then dissolves in the oil.

Because of the relatively rapid heating of the oil-soapstock mixture in the Clayton process and the relatively rapid separation of the soapstock from the oil as compared with the kettle process, the caustic concentration in the soapstock is not reduced sufficiently to permit the fusion of the soapstock particles that is obtained in the kettle process. As a result, the soapstock from the centrifuge contains considerable unconsumed caustic as well as oil occluded by the soapstock by reason of the non-fusion of the soapstock particles caused by this unconsumed caustic. This excess caustic is gradually consumed by saponifying the oil in the soapstock, thus producing a fictitious appearance of efficiency when only the oil content of the soapstock is considered. The method of heating the oil-soapstock mixture produces variations in the apparent oil content of the soapstock.

In the James process, which the Patent Office decided was anticipated by Clayton, introduced by The Sharples Company, the mixing chamber and the tubular heat exchanger mentioned above in the description of the Clayton process are used. In Clayton's own installations the mixing and heating occurred in a pipe shaped into cylindrical coils heated by hot water. As a result of the more rapid heating in the heat exchanger, more unconsumed caustic is present in the soapstock from this method of heating than when using the type of coil used by Clayton. The effect of this additional unconsumed caustic in the soapstock is to reduce the oil content of this soapstock on aging below that in the soapstock from Clayton's type of coil.

The presence of considerable unconsumed caustic in the soapstock is due to the short time between the addition of the caustic solution to the oil and when the oil is heated. In the kettle process the time is about 60 minutes while it is about six minutes in the Clayton process.

In the original Clayton process, using cylindrical coils to mix and heat the oil-soapstock mixture, rapid differences in the color of the refined oil occur because of the fact that the proportionometer used to feed the oil and caustic solution injects the caustic solution into the flowing stream of oil in slugs. Thus the caustic is not distributed uniformly through the oil and in the passage through the coils this non-uniformity persists, resulting in the variations in color. This variation does not occur when using the equipment of the James process as installed by The Sharples Company. The non-uniform oil-caustic solution mixture from the proportionometer is rendered uniform in the mixing chamber of The Sharples Company installations.

The essential chemical difference between the kettle process and the Clayton process is that in the kettle process the concentration of the caustic in the soapstock is reduced in the 60 minutes during the mixing and heating operation to a point where the soapstock particles will readily fuse together and separate from the oil by gravity-settling over night. In the Clayton process the mixing and heating which require only six minutes result in a soapstock made up of particles that will not fuse together immediately because of the high concentration of caustic and, therefore, centrifugal force is applied to squeeze the occluded oil from the soapstock. In both cases, the oil-in-water emulsion stabilized by the lecithin is present and in the same amount. The reduction in refining loss obtained by the Clayton process as compared with the kettle process is due to the reduction in time to mix and heat the oil-soapstock mixture to obtain the "break," thereby reducing the consumption of caustic. There is no reduction in the oil contained in the soapstock of the Clayton process as an oil-in-water emulsion as compared with the soapstock obtained in the kettle process. This loss of oil is inherent in the refining of oils containing lecithin and can be reduced only by preventing the formation of the oil-in-water emulsion.

#### Caustic-silicate Process

Chisholm (5) found that by using a sodium silicate solution in conjunction with the caustic solution he could obtain a lower refining loss on some oils. The "break" occurred more rapidly and the soapstock obtained was more fluid when hot and firmer when cold than kettle process soapstock. The refined oil obtained contained an abnormal amount of soapstock in suspension in a finely divided condition. Trouble was experienced in obtaining a desirable color of the refined oil. The mechanism of the process which resulted in effecting the reduction in refining loss was not understood. Because of the trouble in controlling the color and its limited application to some oils only, the use of the process was abandoned. Endeavors to use sodium silicate in the Clayton process have been unsuccessful.

#### Mist-mixing Process

In the mist-mixing process of refining vegetable oils the extreme fineness of the soapstock particles obtained when the oil and caustic solution are mixed in the form of mists results in the caustic diffusing to the surface of the particles at a rapid rate. Also because of the fineness of the soapstock particles, the surface area of these particles is enormous, resulting in a major portion of the positive charges on the color particles and colloidal matter in the oil being neutralized by the negative charges on the soap formed from the free acids. Thus, this process differs materially from those processes using liquid mixing where the major color reduction occurs during the long mixing in the cold in which time negative charges formed by the saponification of oil neutralize the positive charges on the suspensoids in the oil. The time required for the oil-caustic mixture to pass through the mixing zone in the mist-mixing process is about 0.01 seconds. In the Clayton process the oil-caustic mixture is in the mixing chamber about 10,000 times longer than that, about 1½ to 2 minutes.

The short mixing time in the mist-mixing process, 0.01 seconds, requires the use of more concentrated

caustic solutions than are used in liquid-mixing processes. The concentration used varies with the kind and type of oil and the presence or absence of other refining agents. When refining a slow-breaking cottonseed oil, a concentration of 18% (23.9° Bé.) is used, while when sodium silicate is also added and in the cases of fast-breaking cottonseed oil, re-refining, and soya bean oil, a concentration of 26% (32.1° Bé.) is used.

The time required for the oil-soapstock mixture to flow down the steam-jacketed side of the tank surrounding the mist-mixer, during which time the mixture is heated and the "break" obtained, is about five seconds. Thus the total mixing and heating time to obtain the oil-soapstock mixture in a condition ready for centrifuging is only about 1/70th of that required for the Clayton process. The effect of this short contact time is important because a smaller amount of electrically neutral colloidal material will dissolve in the oil. This reduction in colloidal material dissolved in the oil has a two-fold effect; one on the amount of soapstock particles remaining suspended in the oil after the refining centrifuge, and the other on the quality of the refined oil.

The refined oil discharged from the refining centrifuge in the mist-mixing process is clear when hot and becomes cloudy on cooling. The moisture content of this oil is about 0.18% and the soap content is about 0.02%. This oil can be rendered ready for use by simple filtration through a press of spent fuller's earth or by running it through a clarifying centrifuge. Since the soapstock by these procedures is always wet, no soap will dissolve in the oil.

The problem of flavor reversion in refined soya bean oil has not been solved as yet. One group believes that the reversion is related to the structure of specific glycerides present in the oil. Another group thinks that the reversion is caused by the presence of colloidal matter dissolved in the oil. If this latter group is correct, then soya bean oil refined by the mist-mixing process should be more stable than that refined by liquid-mixing processes because of the rapidity of the refining which prevents major solution of colloidal matter in the oil.

Because of the small excess of caustic required and the high concentration of the caustic solution used, the soapstock obtained has a total fatty acid content of from 60% to 65%.

Since the time factor is so low, oil can be refined by the mist-mixing process starting at almost any reasonable temperature. Oils have been refined starting at 80°F. and at 160°F. The color of the refined oil obtained is good. When oils are refined by liquid-mixing processes starting at high temperatures, dark colored refined oils are obtained.

Mixtures of caustic soda and sodium silicate solutions can be successfully used with the mist-mixing process. The refining loss is further reduced by the use of sodium silicate and good colored refined oil is obtained. The reduction in refining loss is due to the action of the sodium silicate in reducing the oil-in-water emulsion in the soapstock. The mechanism of the action of sodium silicate in the refining of vegetable oils has been determined so that its use can be controlled. The use of sodium silicate in continuous refining is covered by a patent application pending.

In present day liquid-mixing processes of re-refining where a centrifuge is used to separate the oil and soapstock, the two-layer soapstock obtained results in the collection in the centrifuge bowl of a layer of soap between a layer of caustic water against the bowl wall and the refined oil being discharged. Ultimately this soap layer becomes so thick that the soap is discharged with the oil. In the mist-mixing process only about 0.2% of a 26% (32.1° Bé.) solution of caustic is used. Because of this small amount of caustic, the soapstock has only one layer so that continuous re-refining is practical. The color reduction is very much greater than can be obtained by liquid-mixing and the refining loss is about 0.5% only.

Because of the small excess of caustic used and the rapidity of the refining, the refining loss obtained with the mist-mixing process is lower than can be obtained with liquid-mixing, and the refined oil is cleaner both as regards suspended soapstock and dissolved colloidal matter.

#### Summary

In the caustic refining of vegetable oils using liquid-mixing the concentration of caustic solution required, the amount of excess caustic used, the length of time of mixing the oil-soapstock mixture in the cold, and the oil content of the soapstock are governed by the amount and kinds of phosphatides present in the oil.

In the mist-mixing process of refining vegetable oils using caustic, the mixing time is kept constant, the concentration of the caustic is kept constant for a particular kind or type of oil, the excess caustic is kept constant for a particular kind or type of oil, and the oil content of the soapstock is governed by the amount and kinds of phosphatides present in the oil, and by the use of sodium silicate.

#### REFERENCES

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