# Miscella Refining

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## ABSTRACT

Miscella refining can be practiced as a batch process or, preferably, as a continuous process with oil concentrations through the range of 30-70% by wt of oil. Miscella refining is preferably practiced at the oilseed solvent extraction plant for the economic reason of single solvent recovery system. Three immediate benefits are lower refining loss, lighter colored refined oil, and elimination of water washing. Various types of chemical conditioning, mechanical conditioning, and combinations of both are discussed for miscella refining certain oils. Blends of compatible crude oils can be advantageously miscella refined and, if desired, winterized or hydrogenated to produce oils with unique properties.

Of the currently important fats and oils of commerce, cottonseed, safflower, sunflower, sesame, soybean, palm, coconut, and tallow have been successfully miscella refined (1-3). This type of refining should be done at the solvent extraction plant (4) as soon as possible (5) and preferably within ca. 6 hr after the oil is extracted from the source material.

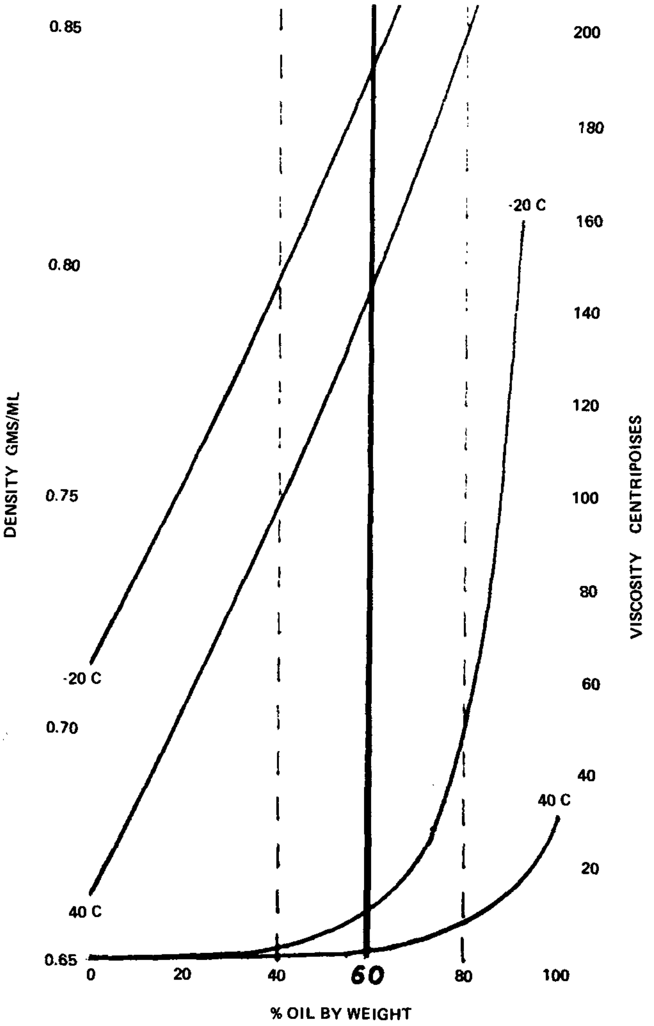

FIG. 1. Density and viscosity curves for cottonseed oil miscella.

The justifications for miscella refining compared to conventional crude oil-caustic soda refining are (a) lower refining loss, (b) lighter colored oil without bleaching, and (c) elimination of water washing the refined oil or miscella (6-8).

Miscella refining is a simple process. Miscella from the extraction plant is adjusted to the desired concentration. This can be done by (a) evaporating part of the solvent from a direct extraction operation, or (b) combining prepressed or purchased crude oil with miscella from the extractor. Two volumes of solvent to one volume of oil results in miscella ca. 40% oil by wt. Equal volumes of solvent and oil result in miscella ca. 58% oil by wt. Most miscella processing is done in the 40-58% wt range. Figure 1 shows the densities and viscosities of cottonseed oil miscella at 40 C, which is approximately the temperature during refining, and at -20 C, the temperature for winterizing cottonseed oil miscella. The shaded area on the graph indicates the concentration range in which most miscella processing is done.

The dilution effect of an inert solvent (hexane) has little or no bearing on the time required for caustic soda to react with and neutralize free fatty acids in crude miscella (9). Hydrolysis of phosphatides and pigments in crude oil miscella requires either intense mixing with caustic soda in an homogenizer for ca. 15 sec (10) or the addition of chemical additives (11-13) to the crude miscella followed by a short mixing period prior to the addition of caustic soda. Gum conditioning additives, when used, are added in amounts varying from 50 to 1,500 ppm depending on refining characteristics of the crude miscella. Chemicals that have been used as crude oil conditioners are numerous and varied and include nonionic surfactants (14), mineral acids (15), mineral acid salts, organic acids (16), oxidizing agents including hydrogen peroxide, and alcohols (17).

Homogenizers with a shear mixing intensity sufficient to homogenize milk have been found satisfactory to hydrolize phosphatides and pigments with caustic soda during miscella refining (18). Two basic in-line homogenizers in successful commercial miscella refining service are (a) high pressure piston pumps with homogenizing valves (Fig. 2) and (b) jet-edge liquid processors (Fig. 3), or "whistles."

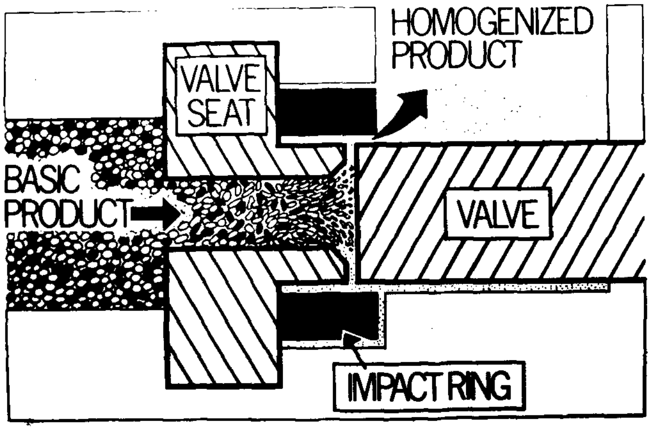

FIG. 2. High pressure piston pump with homogenizing valve.

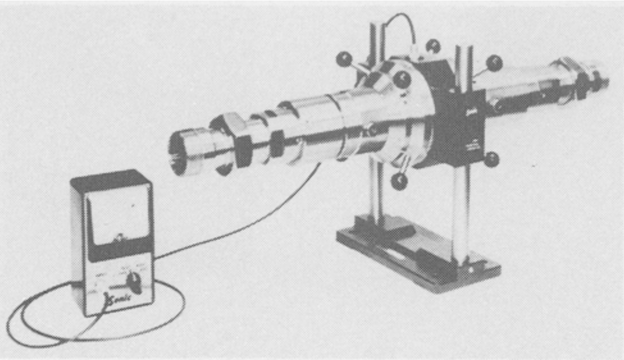

FIG. 3. Jet-edge liquid processor.

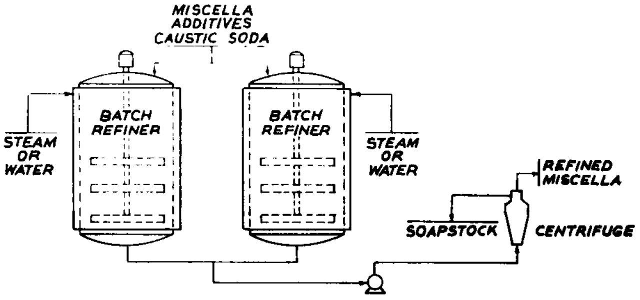

FIG. 4. Batch miscella refining.

Properly conditioned and refined miscella results in a two phase mixture which is then passed through a centrifugal separator in which the soapstock (heavy phase) is separated from the refined miscella (light phase). The latter is continuously filtered through a precoated pressure leaf filter. The precoat is usually diatomaceous earth but can be a mixture of bleaching clay and diatomaceous earth. The refined and filtered miscella can be stripped of hexane to produce a neutral yellow oil, or it can be further processed as miscella. This option will be discussed in greater detail later.

In small solvent extraction plants or where feed stocks change frequently, batch miscella refining vessels can be advantageous. (Fig. 4). These are usually totally enclosed tanks of ca. 5,000 liter capacity equipped with coils for rapid heating and cooling. Variable speed stirrers for agitating the contents of the vessels are also essential. Two of these tanks make batch-continuous operation possible. One tank is charged with ca. 4,000 liters of crude miscella. Concentration is adjusted as desired, and a conditioning agent is added in the proper amount while the miscella is continuously agitated (19). After 2-5 min, caustic soda is added in an amount equal to ca. 0.3% excess. When the soapstock begins to break free from the miscella, agitation is increased, and heat is added until the soapstock melts. Then the miscella is cooled as rapidly as possible to ca. 45 C. This batch is then started through a centrifugal separator. Concurrently, the other refining kettle is filled, conditioned, refined, and started through the centrifuge when the first vessel is empty. Alternating this procedure between two tanks makes batch-continuous miscella refining possible.

In continuous solvent extraction plants, continuous miscella refining is usually the most efficient and preferred procedure. Surge tanks used to check miscella concentration and clarity prior to conditioning and refining are desirable (Fig. 5). Two of these tanks, each with capacity to hold a minimum of 2 hr plant production of miscella, make precise concentration control and continuous operation possible. Filtered miscella from plant production is flowed into a tank which is continuously agitated with a high volume, low head pump circulating crude miscella from the cone bottom to near the top of the tank. As the tank is filled to ca. 80% of capacity, a final concentration adjustment is made. Conditioning chemicals can be added to the crude miscella if desired and the miscella started through the refining process.

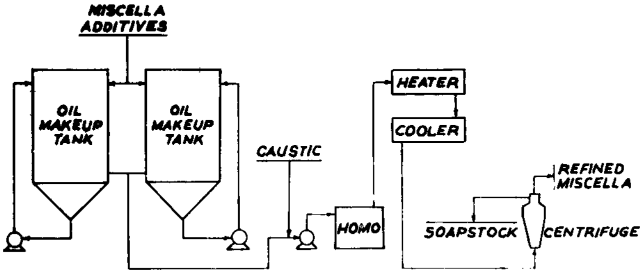

FIG. 5. Continuous miscella refining.

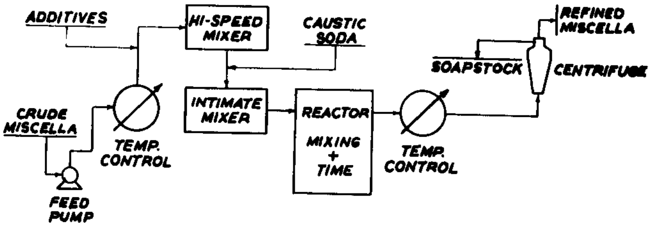

FIG. 6. Continuous miscella refining using additives.

If a homogenizer is used, caustic soda in the range of 7-26 to 26° Be′ is metered into the suction side of the refinery pump in sufficient amount to provide ca. 0.3% excess. This mixture is immediately mixed in a homogenizer, heated to ca. 65 C to melt the soapstock, cooled to ca. 45 C, and centrifugally separated.

If conditioning chemicals are used to hydrolize the phosphatides and pigments prior to caustic refining (Fig. 6), these can be added batchwise to the crude miscella surge tanks, or they can be continuously metered into the crude miscella stream in the continuous refining process followed by mixing of proper duration and intensity (20). After conditioning, the crude miscella is treated with caustic soda to neutralize free fatty acids and remove impurities. Caustic concentration may vary from 12 to 30° Be′ with 18-26° Be′ the preferred range. Amount of excess treat over theoretical requirement is in the range of 0.25-0.60% for crude cottonseed, soybean, and rice bran oil. Tallow can be satisfactorily refined with 0.1% excess over theoretical requirements (21). After the caustic addition, the miscella is agitated in variable speed mixers at sufficient intensity and time to precipitate the impurities in the crude miscella. This chemically conditioned and caustic refined miscella is then centrifugally separated.

In both of the systems described there must be temperature control and mixing for several minutes after the addition of caustic soda to melt and condition the soapstock for centrifugal separation. The quality and characteristics of the refined miscella from both processes are quite similar. Either product can be stripped of hexane to yield a high quality, light colored neutral yellow oil, or, as stated earlier in this paper, can be further processed as miscella.

By chilling refined miscella (22), the more saturated fats and waxes, if present, can be fractionally crystallized out of the miscella solution (23) and centrifugally separated from unsaturated fats followed by solvent recovery from each phase (24).

Diametrically, oil in soap free, refined miscella can be continuously hydrogenated to a precisely predetermined degree of saturation by heating in a temperature controlled, agitated pressure vessel with continuous metered addition of a proper catalyst and hydrogen. The catalyst is continuously removed by filtration or centrifugal separation from the hydrogenated miscella and can be reused.

A study of polymorphic and morphological behavior of certain fats and oils (25) indicates that those with similar fatty acid composition and similar crystallographic behavior can be blended to result in compatible mixtures. Fats and oils having these characteristics in common will produce products which will remain stable indefinitely without subsequent separation of the components. This is especially true when fats are blended and hydrogenated to produce shortenings. For example, cottonseed oil, palm oil, and tallow share these compatible characteristics. Cottonseed oil is not compatible with soybean oil, lard, or mixtures of these products.

Interesting new fats with unusual properties can easily be produced by combining crude palm oil with crude cottonseed oil miscella in various proportions and refining the mixture in accordance with the procedures described in this paper. The blended refined miscella can be fractionally crystallized by controlled chilling to produce products with unique properties. The same is true of tallow-cottonseed oil miscella blends. The number and variability of products can be manipulated and modified substantially with compatible multifat systems which are miscella refined and then fractionally crystallized at various temperatures (26).

The trend in this industry is toward larger solvent extraction plants and toward processing fats and oils to consumer products at the oilseed extraction plant. Continuous miscella refining, hydrogenation, and fractional crystallization (27) offer great opportunities to increase the number and physical variability of fats available (28). Blending these fats in various combinations can result in new "tailor made" fats for specific uses. Finally, solvent fractional crystallization of single or compatible blends of fats followed by centrifugal separation in a solvent extraction plant may be more economical than hydrogenation for producing certain fats with specific properties.

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