

Continuous Miscella Soapstock Acidulation Process¹

L.S. CRAUER, The De Laval Separator Company,
Poughkeepsie, New York

ABSTRACT

A process is described for the continuous splitting of miscella soapstock in a pressurized two-stage centrifugal system. Data collected from such a process, functioning at specified operational conditions, has yielded a recovery of 92% or more of the fatty acid from cottonseed miscella soap as a quality acid oil, analyzing 94% or better TFA content. The hexane in the soap feed is recovered for reuse from the vacuum stripper.

Solvent processing in the seed industry has been under continuous development for the past 30 years. Although the first patents for miscella processing of vegetable oils made their appearance in the 1940's, the first commercial miscella refining systems did not appear until the late 1950's. In the past 10 years the number of plants continuously miscella-refining vegetable oils at the mill has steadily increased. This has been the result of technological and economic developments, larger mills, year-round operation, change in transportation and distribution patterns, the advent of more sophisticated equipment and controls, the demand for the best oil quality, and reduced processing costs.

In most of these solvent plants, the soapstock from the miscella refinery area is pumped directly to the desolventizer-toaster in the extraction area. This addition of soapstock to the meal increases oil and weight content, makes the meal less dusty, easier to handle and pelletize (1). The hexane that is entrained with the oil in the miscella soapstock is recovered in the extractor's solvent recovery system for reuse.

With the tremendous growth of the meat industry in the past decade, meal formulation requirements have changed and become very specific as to type and percentage of ingredient in a particular blend, and are built for a special animal diet. These marketing trends have had a definite influence on the seed mill's meal production and quality control. Also, during this same period, fatty acid demand and utilization has increased. The progressive mill operator has seen a byproduct of enhanced value in the acid oil from miscella soapstock. The process for continuous miscella soapstock acidulation was developed to fulfill these needs in the changing times.

Miscella soapstock cannot be easily or completely desolventized, and it cannot be processed in regular conventional batch processes or the usual continuous acidulation system, due to the presence of the hexane in the soapstock. The quantity of hexane in the miscella soapstock may vary from as little as 1% to as much as 8%, depending upon the processing conditions and operational

control in the refinery. This hexane is entrained with the neutral oil in the soapstock. As the soapstock is heated gradually from usual centrifuge discharge temperature range (130-135 F) to 140 F or higher, there is excessive foaming. With the addition of heat and dispersion in order to liberate the hexane, the tiny phosphatidic particles (1-3 microns in size) that surround the soap micelle act as a thin protective stable film. This foam persists at the alkaline pH ranges. However, as soon as this soapstock is treated with acid to a pH of 6 or less, all foaming subsides. Under proper processing conditions, the soapstock can be readily split to recover the fatty acids in the miscella soapstock as an acid oil. The flow chart for such a process appears in Figure 1.

In this process the soapstock from the hermetic separator in the refinery is fed into a soap funnel, along with the flow of cold dilution water from the extraction plant area. The water passes through a flow meter to control the ratio of water to soap in the range of 1.8-3 parts water to 1 part soap. The combined flow passes through the feed pump for the process, to a soap mixer, to blend the dilute soap to uniform consistency. Passage through a soap heater brings the dilute soap to a temperature range of 110-120 F. Sulfuric acid is added to the dilute soap and the two streams contacted in an intimate, slow speed mixer to bring the reaction mixture to a pH range of 2.0-2.3 at the pH cell. The volume of concentrated sulfuric acid addition by the metering pump from the acid supply source is controlled automatically by the pH recorder-controller. In the area of sulfuric acid addition to the system, there is special metallurgy utilized, in order to minimize the corrosion from acid contacting and heat of reaction.

The reaction mixture at temperature range of 130-140 F passes to the first hermetic centrifuge, where a clear acid oil miscella is separated as the light phase from the heavy phase. This second phase is a phosphatidic acid oil-water emulsion, which passes to a reaction vessel, equipped with a steam sparge. Here the mixture is heated to 180-200 F and then pumped to a second hermetic centrifuge. In this separator, an acid oil miscella is separated from the acid water phase, which is sent to the waste treatment area.

The acid oil miscella from both of the hermetic separators is fed to a receiver tank, then pumped through a spiral heat exchanger to bring it to an elevated temperature for desolventizing in the flash evaporator stripper. The

TABLE II

Chemical Analysis of Cottonseed Acid Oil

Component	Per cent
Total fatty acid	94-97
Oxy fatty acid	0.84-3.2
Free fatty acid	55-67
Moisture	0.2-2.0
Acetone insolubles	0.5-1.0

TABLE I

Cottonseed Miscella Soapstock

Component	Per cent
Moisture	42-50
Hexane	3-8
Total fatty Acid	30-41
Oxy fatty acid	0.3-1.8
NaOH	5-7.5
Phosphatides	5-7

TABLE III

Chemical Analysis of Acid Water

Component	Per cent
Total solids	7.1-7.8
TFA	0.3-0.6
Ash	2.3-4.6
Organic matter	3.2-5.1
BOD, ppm	11,400-13,200

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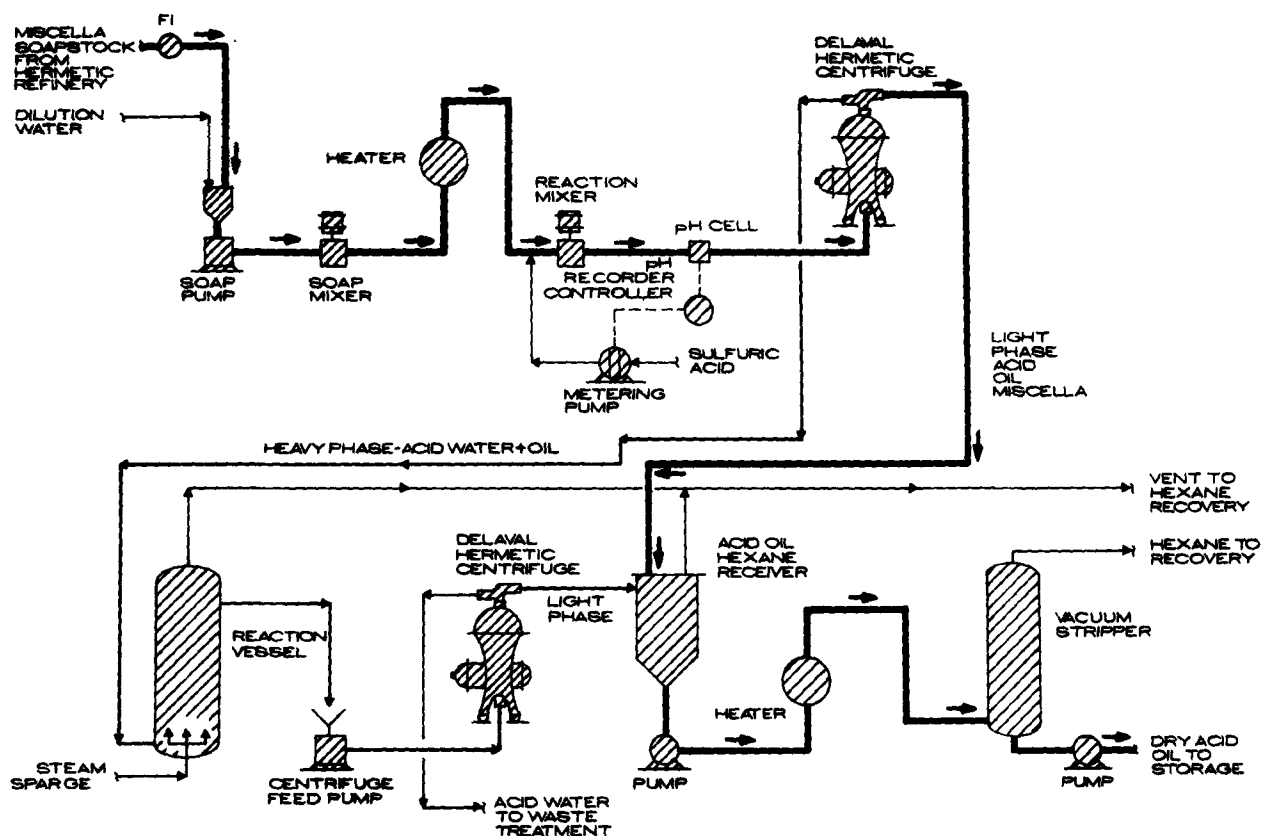


FIG. 1. Continuous miscella soapstock acidulation.

hexane that is liberated at the reaction vessel and this vacuum stripper are recovered for reuse in the solvent plant. The dry acid oil from the vacuum stripper is pumped directly to storage. In this process the dilution water for the soap may be used water from other areas in the mill. However it is important that it be free of lint and other insoluble impurities.

The source of sulfuric acid is 66 Be'. The amount required varies with the amount of caustic used in refining. Experience has shown that treat is generally 8.5-9.5% by weight of the soap feed. Acid dosage is controlled automatically at the set point of the pH recorder-controller.

The hermetic centrifuge has been specially modified for this application. Double seals with water, as barrier and coolant, are provided at the inlet and outlet to maintain cool-running sealing surfaces and to avoid solvent loss. The separator is a part of the completely closed system. Inlet pressures to these units are in the range of 60-90 psi. Back pressure on the acid oil miscella is controlled in the range of 25-50 psi to assure desired product clarity.

In the first separator, generally 70-80% of the available acid oil in the original soapstock is recovered. The remaining acid oil miscella is recovered in the second separator as a result of time and temperature in the reaction vessel and centrifugal force.

Throughput to these separators for this application is in the range of 10,000-11,000 pph which will easily handle the soap flow from a 20,000 pph miscella refinery.

Chemical analyses of typical samples of cottonseed soapstock fed to such a system appear in Table I.

The acid oil miscella as discharged from the hermetic separator has been found to analyze 5-14% hexane. At the temperature and pressure that this acid oil miscella enters the vacuum stripper, the hexane is removed readily at 25-26 in. vacuum.

Typical analyses of the dried acid oil, as pumped to storage, appear in Table II. Flash point of such an acid oil is in the range of 300 F. In the miscella system, due to the presence of hexane, the acid oil has little affinity for water and is readily separated. In the appraisal of such a system over a 24 hr period, yields have consistently averaged 92-94% recovery of TFA of the original soapstock feed in the acid oil phase.

Typical analyses of the acid water phase from such a system appear in Table III.

This process is of compact design and can be located in an area adjacent to the miscella refinery. Automatic pH control minimized the need for operator attention. All the acid oil lines in the system are steam-traced. The system is designed for cleaning-in-place, with caustic recirculated through the centrifuges and the lines as the cleaning agent. The caustic dissolves any fatty acid crystal or phosphatidic deposition. When controlled by the same operating personnel as the refinery, the acidulation system is a good monitor of refinery operation. It quickly indicates, by variation in temperature and pressure, the loss of hexane-oil ratio in the crude miscella, temperature and caustic changes. The acid oil yield is a direct measure of refinery loss.

ACKNOWLEDGMENTS

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REFERENCE

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