

• Technical

Treatment of Soybean Oil Soapstock to Reduce Pollution¹

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ABSTRACT

Often soapstock is acidulated to convert this byproduct of vegetable oil refining to a salable commodity. The acidic waste water from this treatment constitutes a significant part of the environmental pollution from refineries. A process of neutralizing and drying was investigated as a nonpolluting method for upgrading soybean oil soapstock. Neutralization with sulfuric acid was conducted in a ribbon blender. The neutral soapstock was dried to ca. 4% moisture, either batchwise in a natural circulation evaporator or continuously in a scraped film evaporator. The product is liquid while hot but solidifies to a waxy solid when cooled to room temperature. When added to a standard broiler ration, the feed efficiency and rate of gain of chickens equaled that obtained with a commercial feed fat added at the same level. The soapstock products fed, which contained 200-300 ppm xanthophyll, gave significantly better shank pigmentation than the commercial fat, which contained 3 ppm.

INTRODUCTION

Disposition of soapstock from alkali refining of soybean oil and other vegetable oils has become a problem with many refineries because of increased restrictions on environmental pollution. Acidulation of soapstock, widely used to convert it to salable fatty acids, produces a substantial amount of acidic wastewater containing biodegradable material (1). Disposal of the waste is increasingly difficult and costly.

Various other methods of handling soapstock are used or have been proposed. Addition of soapstock directly to oilseed meal is practical under certain conditions. However the difficulty of handling wet soapstock, preventing microbial spoilage and avoiding shipping costs often precludes such disposition. National Research Council specifications require that soapstock be neutralized before it is added to a commercial feed (2). A process of drum drying soapstock and addition of the dry material to poultry feed has been described (3). The dried material presents a dust problem in handling and feed mixing. It is hygroscopic and cakes when stored. Conversion of acidulated soapstock to fatty acid methyl esters is another method of producing a salable product, but again acidulation introduces a disposal problem (4).

In this report a process is described for treating soapstock to produce a desirable and salable product without creating either water or atmospheric pollutants. A cost estimate is given to indicate the economics of the proposed process whereby soapstock is neutralized and then dried by vacuum evaporation. Neutralization converts soaps to fatty acids; the dried product becomes liquid when warmed to ca. 45 C and remains a waxy solid at room temperature. The only effluent from the process is evaporated water.

Although neutral dried soapstock (NDSS) might be a source of industrial fatty acids by splitting the glycerides it contains by continuous high-temperature hydrolysis, the main value of NDSS would be as a source of energy and xanthophyll in poultry rations.

Analytical Methods

Moisture in NDSS was determined by drying a sample to constant weight in a rotating flask evaporator (Rinco) while heating the flask in a water bath at 90-95 C and an absolute pressure of 0.5 mm mercury. Free fatty acids were determined by AOCS Official Method Ac 5-41 and unsaponifiable material by Method Da 10-42.

Total fatty acids (free and combined) were determined by the gas liquid chromatography (GLC) method of Black et al. (5).

Sodium was determined by an atomic adsorption method similar to that described by List et al. (6). An oil soluble sodium standard was obtained from the National Bureau of Standards. The instrument was a Techtron AA 120 equipped with a sodium lamp.

Carotene and xanthophyll were determined by Official AOAC Method 39.019-31.022.

Phosphorous was determined by the method of Truog and Meyer (7).

Fatty acid composition was determined by preparing methyl esters by the AOCS Tentative Method Ce 2-66 (1969 Revision) and analyzing by GLC, AOCS Tentative Method Ce 1-62 (1970 Revision).

EXPERIMENTAL PROCEDURES

Three lots of soapstock produced in a commercial plant by alkali-refining crude, nondegummed soybean oil were obtained at different times. Lot 1 of soapstock was converted to NDSS by placing 42.6 lb. in a 1 cu ft, stainless-steel, batch ribbon blender and mixing thoroughly. A 10 g aliquot of the blended lot was dispersed in 50 ml

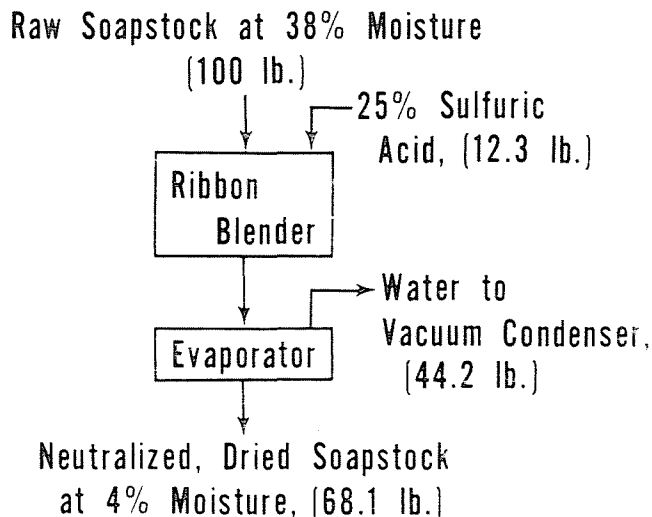


FIG. 1. Flow sheet for neutralized, dried soapstock.

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TABLE I
Partial Analysis of Neutralized, Dried Soapstock (NDSS)
and a Commercial Feed Fat

Composition	Soapstock, lot no. ^a					Commercial feed fat
	1	2	3	4	5	
Total fatty acids, %	71.0	64.0	54.0	—	—	97.3
Free fatty acids, %	34.0	28.3	27.1	—	—	41.6
Moisture, %	4.6	4.4	5.7	0.0	0.0	1.6
Unsaponifiable, %	2.0	2.0	2.1	—	—	0.046
Phosphorous, %	—	1.04	0.80	—	1.66	0.0019
Sodium, %	1.8	1.6	4.5	—	—	0.0
Carotene, $\mu\text{g/g}$	350	575	229	78.0	89.0	4.0
Xanthophyll, $\mu\text{g/g}$	202	313	231	40.0	38.0	3.0
Fatty acid composition						
C-16	—	—	—			10.1
C ₁₆	16.7	17.6	17.3			25.8
C ₁₈₋₀ , stearic	4.6	4.2	4.4			14.6
C ₁₈₋₁ , oleic	15.9	15.6	15.7			37.0
C ₁₈₋₂ , linoleic	55.7	55.7	55.4			11.2
C ₁₈₋₃ , linolenic	7.1	6.9	7.2			1.3

^aLots 1 and 2 prepared with a natural circulation evaporator and used in feeding trials. Lot 3 prepared with a scraped film evaporator. Lots 4 and 5 prepared by pilot-plant refining and laboratory-scale drying; lot 4 without and lot 5 with neutralization.

TABLE II
Growth, Feed Conversion and Shank Pigmentation Scores of
6-Week-Old Straight-Run Broilers

Treatment	Averages of two trials ^a		Shank pigmentation, Roche yolk color fan number ^c
	Weight at 6 weeks, g	Feed conversion ^b	
Broiler diet ^d	1312	1.87	4.84
NDSS diet ^e	1307	1.91	6.83 ^f

^aEighty chicks per treatment in each of the two trials.

^bFeed consumed per weight gain.

^cRoche color fan, F. Hoffman-LaRoche Co., Ltd., Basle, Switzerland.

^dDiet containing 4 % commercial feed fat.

^eDiet containing 4 % neutralized, dried soapstock (NDSS).

^fHighly significant ($p \geq 0.01$).

distilled water, and 1% sulfuric acid was slowly added from a burette with rapid stirring until a pH of 7.0 was reached that persisted for more than 1 min. From this titration the calculated amount of 25% sulfuric acid (5.25 lb.) was slowly added to the soapstock while it was being mixed in the ribbon blender. Mixing was continued for ca. 15 min after addition of the acid, and a 10 g sample dispersed in 50 ml of distilled water gave a pH of 6.9.

The neutralized soapstock was dried batchwise by drawing it into a small steam-heated, natural-circulation evaporator under a 22 in. vacuum and heating cautiously because of a tendency to foam. As evaporation proceeded, steam pressure reached 10 psi; when circulation stopped, the batch was withdrawn into a suitable container where it solidified to a waxy solid on cooling to room temperature. A flow sheet of the process and a material balance based on laboratory tests with soapstock from lot 1 are shown in Figure 1.

Lot 2 was prepared in the same manner, but lots 1 and 2 were used separately in broiler feeding studies conducted at Beltsville, Md. Lot 3, not used for feeding, was neutralized and then dried continuously by a single pass through a steam-heated, thin film (scraped film) evaporator operated under an absolute pressure of ca. 5 mm of mercury with steam at 20 psi in the jacket. Compared with the first two lots, the product was visibly darker brown, apparently as a result of the higher temperature (steam pressure) used. The relation between color and feeding value was not determined. A thin-film evaporator like that used to dry commercial lecithin should be the most satisfactory drier for NDSS because the wet material tends to foam and

because its heat sensitivity is similar to that of lecithin.

Lots 4 and 5 were prepared from soapstock produced by alkali-refining crude soybean oil on a continuous pilot plant scale. Lot 4 was dried without neutralization or other treatment and lot 5, after neutralization with dilute sulfuric acid. Both lots were vacuum-dried in a rotating flask evaporator at 90 C; both were tan in color, while lots 1, 2 and 3 were brown to dark brown.

RESULTS AND DISCUSSION

The composition of several lots of NDSS prepared in the pilot plant and laboratory is given in Table I.

Witte and Sipos (3) showed that soapstock dried directly or after addition of more alkali retained a high xanthophyll content. However when soapstock is acidulated, xanthophyll is destroyed (8,9). NDSS retains a substantial content of both carotene and xanthophyll as shown from compositions of lots 1, 2 and 3. Refining conditions used for lots 4 and 5 were possibly not optimum for removing xanthophyll and carotene from crude oil, since the material was much lower in xanthophyll than the soapstock obtained from a commercial plant and used for other tests. The similarity in the carotene and xanthophyll contents of lots 4 and 5 shows that the process of neutralizing and drying soapstock resulted in virtually no destruction of these materials.

NDSS had no signs of microbiological growth or other spoilage after several months at room temperature. Although solid at room temperature, it liquefies at ca. 40-45 C and is fluid enough at 45-50 C for easy incorporation in a feed.

Because of its phosphatide content, the yield of NDSS should be higher than for acidulated soapstock. Thus for lot 1, the maximum yield of acidulated soapstock from 100 lb. of raw soapstock would be ca. 50 lb., an amount considerably less than the 68 lb. of NDSS we obtained. The difference in yields would represent the glycerine and phosphatide moieties which would be lost in the wastewater from acidulation. They apparently constitute the major portion of the BOD content of the wastewater that contributes to environmental pollution problems.

According to a recent report (10), alkali refining does not selectively remove chlorine-containing pesticides from vegetable oils; therefore soapstock or products therefrom used in feeds should not contain significant levels of such materials. Another report confirms that it does not (Private communication, Wisconsin Alumni Research Foundation). However deodorizer distillate may contain substantial amounts of such pesticides, and it should not be added to feeds unless analysis shows it to be suitable.

Lots 1 and 2 were compared with a commercial feed fat used routinely in broiler rations (Table I). Two 6 week feeding trials were conducted with straight-run broiler chicks in which NDSS was compared with a commercial feed fat. Growth and feed efficiency were not significantly different for either diet as shown in Table II. However improvement of shank pigmentation with NDSS was highly significant. Mortality rates were the same for both treatments and both trials and were not related to the dietary regime. Detailed results of the feeding trials will be published elsewhere (11).

A preliminary cost estimate has been prepared for a plant producing 20,000 lb. of NDSS daily by the process described. The production of 20,000 lb. of NDSS at 3% moisture would require processing ca. 30,500 lb. of untreated soapstock at 36.5% moisture. For a plant operating 300 days per year, 24 hr a day, the estimated fixed capital investment, as detailed in Table III, is \$150,000. This hypothetical installation is considered an adjunct to an existing soybean oil processing plant, and presumably adequate building space is available for the proposed process.

Estimated processing costs for producing NDSS are ca. 1.55 cents/lb. Cost items listed in Table IV and included in processing costs are sulfuric acid, utilities, labor and supervision (shared with existing plant), maintenance and supplies, fixed charges and general plant overhead. Not included in processing costs are cost for untreated soapstock, administrative and selling expenses, interest on investment and profit. If the soapstock has a moisture content of 50%, the cost to product NDSS with 3%

TABLE III

Estimated Fixed Capital Investment for Plant Producing 20,000 lb. Neutralized, Dry Soybean Oil Soapstock Per Day^a

Equipment delivered	Cost, \$
Evaporator, agitated, thin film, 316 SS	48,000
Pumps, metering	1000
Heat exchanger, 316 SS	600
Mixer, static, in-line	800
Pumps, miscellaneous use	1300
Tanks, process and storage	16,300
Equipment delivered, total cost	68,000
Installation, piping, wiring, etc.	60,000
Engineering fees	12,000
Contingencies	10,000
Estimated fixed capital investment	150,000

^aPlant operations: 24 hr/day, 300 days/yr.

moisture will be increased ca. 0.1 cents to 1.65 cents/lb.

In the hypothetical plant, an agitated thin-film evaporator is used to remove water from the neutralized soapstock. In actual practice, if a forced-circulation evaporator could be substituted for a thin-film unit, capital investment could probably be reduced. Since low pressure steam can be used for the evaporation step, exhaust steam, if available in a plant, could find application. In the estimate steam costs are based on the use of high pressure steam. It is conceivable, therefore, that actual plant processing costs could be made lower than the listed estimated costs by adopting certain process modifications.

In previous reports we described a water-recycle process for washing alkali-refined or hydrogenated soybean oil in which the water used to wash residual metal ions from the oil is passed through a cation exchange resin in the hydrogen form (12,13). The water is then reused. When the resin is periodically regenerated by rinsing it with acid, excess acid must be discarded. In the proposed method for treating soapstock, this excess acid plus additional strong acid could be used for the neutralization step and the water evaporated in drying the neutral soapstock could be used for rinsing regenerated resin. Such a setup would provide an integrated solution to a refinery pollution problem.

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TABLE IV

Estimated Processing Costs to Produce 20,000 lb. Neutralized, Dry Soybean Oil Soapstock Per Day^a

Cost item	Dollars per day
Raw materials	
Sulfuric acid, 98%, 1008 lb., \$0.016/lb.	16.13
Utilities	
Steam, 30,000 lb./day, \$0.75/M lb.	22.50
Water, 35,000 gal/day, \$0.15/M gal	5.26
Electricity, 960 kwh/day, \$0.015/kwh	14.40
	42.16
Labor and supervision	
Operators, 12 man hr/day, \$4.50/hr	54.00
Laboratory technician, 3 man hr/day, \$4.00/hr	12.00
Supervision, 20% of operator and technician	13.20
Overhead	15.84
	95.04
Maintenance and supplies, 6%/yr on \$150,000	30.00
Fixed charges, 13%/yr on \$150,000	65.00
General plant overhead	62.52
Estimated daily production costs, \$/day	310.85
Estimated processing costs, cents/lb.	1.55

^aPlant operations: 24 hr/day, 300 days/year.

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