Continuous Soapstock Acidulation[1]

D. B. TODD and J. E. MORREN, Podbielniak, Inc., Franklin Park, Illinois

Abstract

A newly developed process is described for the continuous acidulation of soapstocks derived from alkali refining of vegetable oils. Results are presented for cottonseed, soybean, coconut and corn oil based soapstocks. The acidulation is accomplished by continuously reacting hot soapstock with H_2SO_4 and separating and countercurrently water washing in a Podbielniak centrifugal contactor. This process offers savings in labor, H_2SO_4 and steam over conventional batch acidulations, with improved recovery of fat. Typical performance data indicate around 98% recovery of the available TFA, acidulated greases containing less than 0.01% mineral acid, and acid water containing ca. 1% mineral acid.

Continuous Soapstock Acidulation

Most soapstock is acidulated with H_2SO_4 to hydrolyze the soap and release the fatty acid as an insoluble lighter layer. A large percentage of these recovered fatty acids, also known as acidulated greases or black acids, are now being used as a source of fat in feeds. The acidulated grease, in addition to being in a more acceptable form chemically, is also more acceptable physically, since it contains more than 90% TFA and can be transported more economically than the original dilute soapstock, which may have as little as 30% TFA. The acidulated grease also is not plagued with the fermentation problems associated with raw soapstock.

In the batch acidulation process, soapstock and an excess of H_2SO_4 are simultaneously pumped into a stirred tank, forming an emulsion of fatty material and dilute H_2SO_4. The emulsion is brought to boiling for 4–6 hr by continuous addition of live steam. Because of the exothermic reaction, it is usually desirable either to add the acid dilute or to add concd acid to cold soapstock to avoid violent local boiling, foaming and spewing which occurs if acid is added to hot soapstock. The emulsion is usually allowed to settle overnight. The aqueous phase containing the dissolved salts can be neutralized and sewered. Usually there persists an intermediate phase—insoluble material which retains some absorbed oil so that the resulting mixture is of intermediate density—which may be recycled for reprocessing. The top black acid layer contains 90–95% TFA. Before being discharged it may be boiled with water to wash out some of the residual mineral acid. The batch process is cumbersome, but for the small scale refiner it may be expedient without being inordinately inconvenient (1).

Various improvements in the acidulation process have been suggested, such as that in the batch process for a small capacity mill which can be obtained by automating the sequence of steps (2). Several years ago, a competitive process for soapstock purification and continuous acidulation was described (3) for plants processing from 300–900 lb of fatty acid/hr, but this process has failed to offer enough economic incentive to justify wide acceptance. The

(Continued on page 178A)

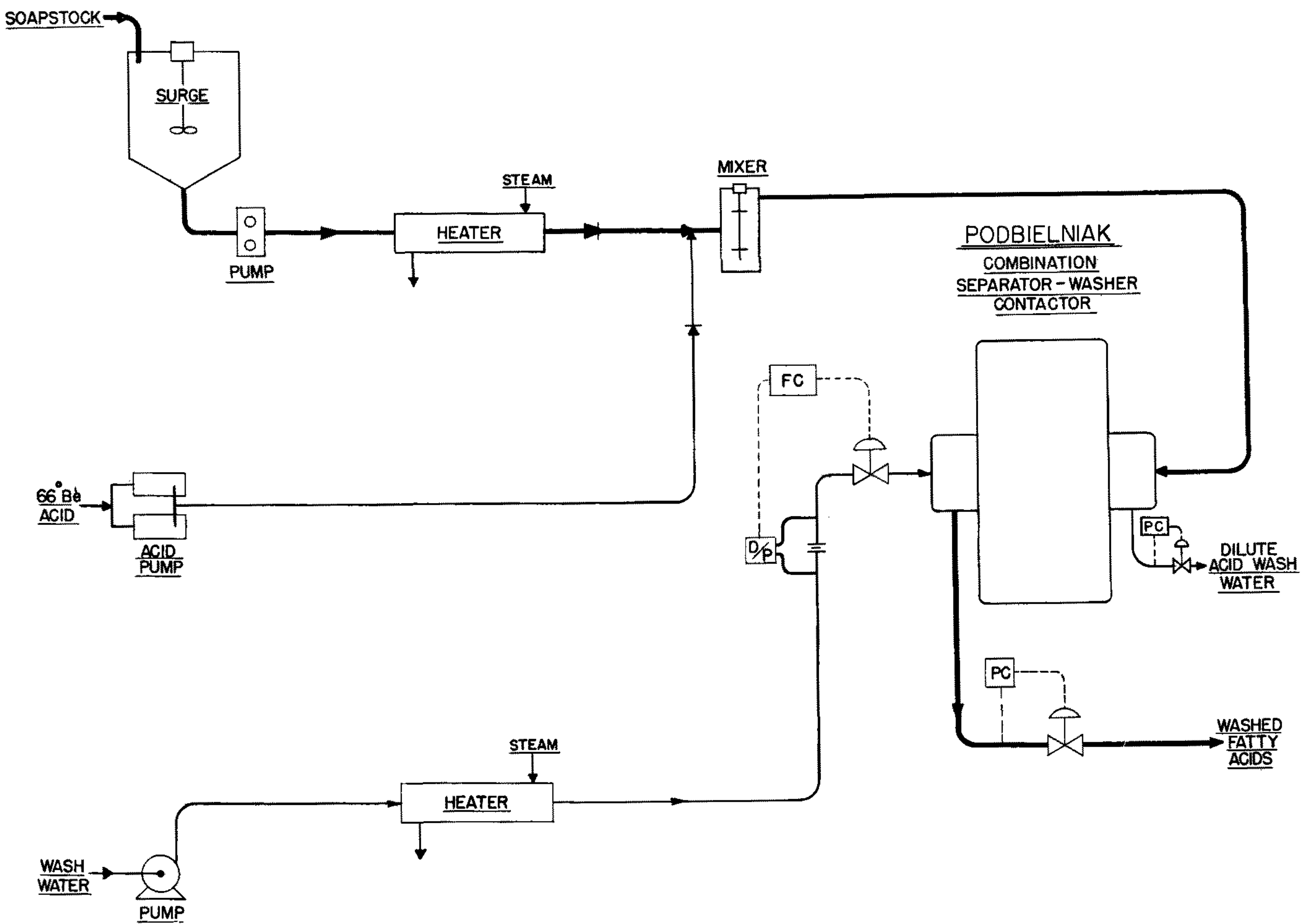

Fig. 1. Process flowsheet for continuous acidulation.

[1] Presented at the AOCS Meeting, New Orleans, 1964.

Continuous Soapstock . . .

(Continued from page 172A)

TABLE I

Continuous Soapstock Acidulation Data

Soapstock	Feed	Acidulated grease			Acid water		TFA
	TFA	TFA	M&V	MA	TFA	MA	Recovery
	%	%	%	%	%	%	%
Coconut	10.7	96.5	1.8	0.003	0.2	0.5	98
Corn oil	12.6	93.4	1.8		0.06	1.75	99
Cottonseed	20.0	92.2	2.0	0.0024	0.03	1.5	99
Soybean	9.7	94.1			0.6	0.7	94
Soybean	34.8	92.2			0.74	0.9	98

TFA = % Total fatty acid, AOCS Method G-3-53.
M&V = % Moisture and volatiles, AOCS Method Ca 2a-45.
MA = % Mineral acid, AOCS Method F 7-44.

obvious goals of a continuous process are to circumvent the shortcomings of the batch process, to increase recovery of the available fat and to provide enough savings in operating cost to justify the increased cost for continuous processing equipment. Another requirement is the flexibility to accommodate the diversity of soapstocks arising not only from the differences in the precursor vegetable oils but also from the differences in the extraction and refining methods employed (4).

In the process we are describing here the first goal achieved was the elimination of having to add the H_2SO_4 to cold soapstock, so that advantage could be taken of the heat content of the soapstock leaving the refining stage. By adding H_2SO_4 continuously to hot soapstock, the splitting can be carried to completion in a much shorter time and an additional saving in space and labor can be achieved. Since no open boiling is required, heat losses are minimized and greater heat economy is provided. Also with the completely enclosed system, there are no obnoxious fumes liberated and the dangers associated with large quantity of reacting H_2SO_4 are avoided.

The second goal was immediate separation of the emulsion so storage would not be tied up for 16 or more hr which is normally required for a batch to break and phase separate. By adaptation of a Podbielniak Centrifugal Contractor, similar to those already in service in vegetable oil degumming, refining and water washing, the premixed hot soapstock and concd H_2SO_4 are separated, and the acidulated grease is continuously washed with warm water. Figure 1 depicts a schematic representation of the process.

The soapstock recovered from alkali refining is combined with any waters also employed in water washing in order to salvage the maximum recoverable fatty acids. The dilution of the soapstock also aids pumping and subsequent contacting with acid. The process works well with as little as 65% water, and with a max that becomes limited by economics rather than feasibility. The dilute soapstock is pumped from storage through a heat exchanger, where it is heated with steam to the desired prereactor temp. The hot soapstock is then metered and fed to a special Carpenter 20 Alloy steel

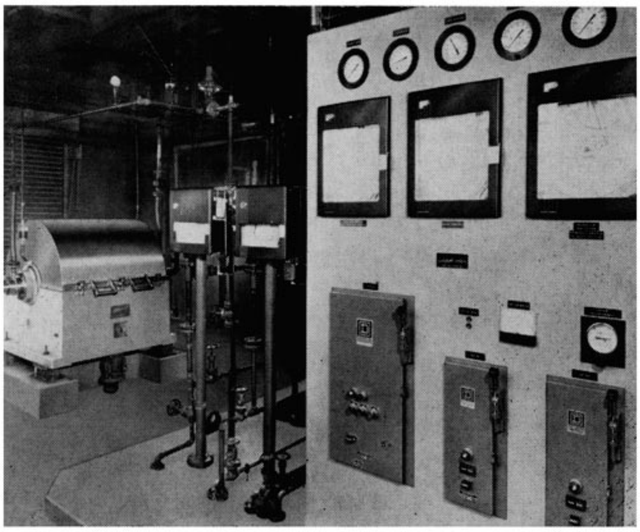

FIG. 2. Podbielniak continuous acidulation installation.

TABLE II

Waste Water Analysis, Continuous Acidulation Soybean Oil Soapstock

Dissolved solids	43,083 ppm
Suspended solids	1,255 ppm
B.O.D.	9,400 ppm
C.O.D.	16,184 ppm

reaction vessel. The acid requirement is dictated by the amt of soap to be split and the excess acidity required to overcome the excess caustic, plus enough to maintain an adequate rate of reaction. An additional temp rise occurs in the reactor, and at this high temp, reaction is fast so the splitting is accomplished with very little residence time. Since the entire process is hermetically enclosed, treating temp higher than 212F may be advantageously employed. Operating temp from 210–230F generally have been found desirable. The mixture from the reaction vessel flows continuously to the centrifugal contractor, also made of Carpenter 20 Alloy steel. The acidulated grease separates from the acid water. Water for washing is heated, metered and added to the contactor. The water flush aids considerably in removing the interfacial cuff material.

Figure 2 shows a typical plant installation. All accessory equipment, including the motor starters and instrument panel, can be located on a common skid. In this particular installation, the soapstock pump and heater were located close to the soapstock supply tank, but still controlled from the panelboard. Operation requires a min of attention unless the nature of the soapstock is varying considerably. Frequency of cleaning is dependent upon the nature of the contaminants present in the soapstock (5), and upon such factors as whether the crude oil was degummed before alkali refining.

Representative data for several soapstocks are shown in Table I. The residual moisture is a function of the amt of hydrated gums and oxygenated fatty acids present, as well as solubility. A residual moisture content of at least 1% is to be expected in the acidulated grease by reason of solubility alone (6). The low mineral acid contents are characteristic of the centrifugal separation. The analysis of the waste water stream from one of the soybean runs is shown in greater detail in Table II. The waste water amounted to ca. 17,500 lb/tank car of soybean oil at a refining loss of 3.25%.

Packaged soapstock acidulation plants have been designed for recovery of from 2000–4200 lb/hr of acidulated grease, and capacities up to 7000 lb/hr are obtainable. Typical performance data indicate ca. 98% recovery of the available TFA, acidulated greases containing less than 0.01% mineral acid and acid water containing around 1% mineral acid. The high TFA recovery is achieved by limiting the degradation of fatty acid which can occur from prolonged contacting with the H_2SO_4, and by obtaining a cleaner separation under centrifugal force, which frees most of the adsorbed oil from the interfacial layer.

REFERENCES

1. Levin, H., and J. S. Swearingen, JAOCS *30*, 85–88 (1953).
2. Krumbein, J. P., Paper presented at AOCS Meeting, New Orleans, 1964.
3. Keith, F. W., Jr., V. G. Bell and F. H. Smith, JAOCS *32*, 517–519 (1955).
4. Stansbury, M. F., V. O. Cirino and H. R. Pastor, JAOCS *34*, 539–544 (1957).
5. Keith, F. W., Jr., F. E. Blachly and F. S. Sadler, JAOCS *31*, 298 (1954).
6. Hoerr, C. W., W. O. Pool and A. W. Ralston, Oil Soap *19*, 126–128 (1942).

[Received June 2, 1964—Accepted August 13, 1964]

Infrared Spectroscopy Institute

Canisius College, Buffalo, New York, will hold its Ninth Annual Infrared Spectroscopy Institute June 28–July 2, 1965.

The Institute will feature four simultaneous seminar-type sessions on spectroscopy. The four sessions will be elementary, interpretation of organic and biomedical compounds, infrared and Raman spectra of inorganics and organometallics, and group theory considerations.

The fee is $100, including textbook and registration, and should be made with Dr. H. A. Szymanski, Canisius College, 2001 Main St., Buffalo, New York 14208.