to higher filtration pressures, resulting in a 30–50% increase in throughput per unit area if the filtration is continued to a pressure of 100 p.s.i.g. Thus a total capacity of 80–110 lbs. of filtered oil per sq. ft. could be obtained at an average rate of approximately 70 lbs. of oil/hr./sq. ft. In practice the actual filtration rate would be considerably lower than this since a production filter would be sized to accommodate a specified throughput of oil per cycle, and the area requirement would be dictated by the amount of cake to be accumulated on the leaves. If a filtration pressure of 100 p.s.i.g. were used, the filter leaves would be placed on 5-in. centers to allow a greater cake thickness.

It is felt that these filtration data are valid for any crude cottonseed oil from mechanical screw presses or Expellers, which has been pretreated to remove the bulk of the coarse foots. Differences in filtration rate and total through-put can be expected because of variations in cooker operation and other process variables. These factors can be taken into account in the initial sizing of a production unit.

Conclusions

A consideration of the economics of installing a leaf type of filter for crude oil in a new mill or as a replacement for existing filtration equipment must take into account the following advantages which a pressure leaf filter offers:

1. Entire filter operation can be handled by a single operator with no assistance required for cleaning;
2. complete elimination of cost of filter cloths and Viskon papers normally used to dress filter presses;
3. elimination of storage, handling, cutting, etc., of cloths and papers;
4. completely enclosed, leak-free filter installation;
5. drier filter cakes with less air-blowing time and lower air consumption;
6. a permanent filter medium (woven wire cloth) of stainless steel which cannot corrode, or rupture during operation, thereby causing passage of solids into filtered oil;
7. high degree of solids removal, resulting in low refining losses;
8. a cleaner filter station;
9. less strenuous labor; and
10. economical construction in carbon steel.

Acknowledgment

The author wishes to express sincere gratitude to Allen Smith of Perkins Oil Company, Memphis, Tenn., for his fine cooperation and assistance in conducting the test work and in collecting the experimental data.

[Received March 12, 1956]

Checking Alkali Delivery of a Proportioner in Vegetable Oil Refining

M. MATTIKOW and S. M. EDMONDS, Refining Unincorporated and
The City College of New York, New York, New York

The advent of continuous processes has brought with it almost complete mechanization. In going from batch to continuous processes, means had to be provided for proportioning or metering the component streams to keep them in the desired ratio.

The first successful continuous process for refining fatty oils stressed the importance of and necessity for proportioning (1). More recently two-stage refining processes such as the soda ash process (2) and others that require the addition of specific, often critical, amounts of strong caustic soda solution, just enough to neutralize in some instances, (3) have made reliable proportioning an important factor for obtaining the maximum yields of refined oil very low in residual impurities. It is also apparent that the use of more reagent than required means not only a waste of chemical but may, in instances in which strong caustic soda solutions are added, result in increased loss of oil through saponification and emulsification.

Irrespective of the merits of the proportioning device employed, it is sound practice to be able to check its performance independently and so have data on the actual delivery of reagent. There is the possibility, borne out many times by the experience of one of the writers, that the setting of the proportioning device does not always reflect the amount of reagent because of some mechanical defect or failure or a moving part sticking.

The purpose of this paper is to describe a simple, rapid method of determining how much reagent the proportioner is delivering, *i.e.*, how much reagent there is in the mixture of oil and reagent, a sample of which is withdrawn from the refining unit. The information, available in less than 10 min., can be used to make such changes in the setting of the proportioner as the analysis dictates. The refiner thus is not completely dependent on the outward signs and indicators of the proportioning devices that may not be delivering according to the setting and calibration.

In continuous refining of fatty oils, now very widely practised, periodic checking of the proportioner feeding alkali is desirable and a simple rapid, routine method of analysis is needed. The alkali is usually fed at a predetermined rate along with the oil so as to maintain the desired constant proportion of the two. The flows of oil and alkali solution are mixed, and the mixture is subsequently separated centrifugally.

Since neutralization of free fatty acid by the alkali is practically instantaneous but not necessarily complete in the flowing stream, the behavior of the proportioning equipment can only be followed by a determination of both free and combined alkali in the mixture before centrifugation. A method of analysis developed in these laboratories some years ago depends upon a direct titration of both soap and alkali with standard sulphuric acid, using methyl orange as indicator. Results are satisfactory, but end-point detection is difficult, especially for highly colored oils. Another method developed here depends upon the determination of water content of the oil-soapstock mixture before centrifugation by a toluene distillation following the Dean-Stark technique. A knowledge of the water content, *i.e.,* the concentration of the alkali in the reagent permits the calculation of the proportioning of alkali since water is introduced only as the known percentage of alkali solution. It is further assumed that the crude oil is normal in respect to moisture content, which is usually about 0.1%. Though reliable, the method is necessarily slow since the distillation requires about two to three hours. So long a delay between sampling and the results of analysis in the check of a flowing system is of course objectionable and impractical because the information, when made available, is ''obsolete'' and may no longer be applicable.

A method of analysis has been developed for the determination of free and combined alkali in samples of the flowing oil-alkali mixture. Depending upon a direct potentiometric titration with sulphuric acid, the procedure is rapid and accurate for oils of varying free fatty acid content.

It was found that oil-alkali samples, when diluted sufficiently with water (1:4) and well stirred, can be rapidly titrated. A pH meter and the conventional glass electrode-calomel electrode assembly are satisfactory. In these experiments a Beckman Laboratory Model and an Industrial Model pH meter and the Beckman electrodes were used.

Procedure

A sample of approximately 50 g. of the oil-alkali mixture, weighed to the nearest tenth of a gram, is collected in a previously weighed 400-ml. beaker or jar. To avoid settling problems which would arise with the additional transfer it is advisable to use the sampling container as the titration vessel. Add 200 ml. of water, introduce a motor-driven stirrer (about 1,800 r.p.m.) and the glass-calomel electrode assembly. Stir one minute and proceed to titrate with 0.5 N sulphuric acid. The rate of addition of the acid should be adjusted so that the pH drops smoothly to about 5. The acid may then be added in 0.5 ml. or smaller increments until the pH remains at 4 or below for one minute. Probably because of adsorption of soap on the precipitated gums there is a tendency for the pH to drop to about 4 and drift up again to above 5 when within a few ml. of the end-point. Any error from this source is avoided by taking the end-point after a one-minute wait. The entire titration should require no more than eight to 10 min.

TABLE I
pH Titration of Mixtures of Crude Oil and Alkali Solution.
Weight of Crude Oil—50.0 g.

No.	% F.F.A.	Ml. acid used for titration, 0.5058 N	% alkali added — 15% Na_2CO_3 sol.	% alkali found calculated as 15% Na_2CO_3 sol.
			%	%
1	1	13.0	4.6	4.6
2	1	13.4	4.6	4.8
3	1	13.5	4.6	4.8
4	2	13.4	4.6	4.8
5	3	13.4	4.6	4.8
6	4	13.4	4.6	4.8
7	1	19.0	6.9	6.8
8	2	19.6	6.9	7.0
9	3	19.6	6.9	7.0
10	4	19.4	6.9	6.9
			11% NaOH sol.	11% NaOH sol.
11	1	18.2	6.7	6.7
12	1	18.2	6.7	6.7

The method was tested, using a crude cottonseed oil containing 1% free fatty acid to which up to 6% stearic acid had been added. Oil-alkali mixtures of various proportions were analyzed, utilizing 15% soda ash, 11% sodium hydroxide, and 14% ammonia solutions. Results as shown in Table I were excellent for both soda ash and sodium hydroxide. The method is accurate and reproducible. The figures in the last two columns of the table represent the percentage of 15% soda ash or 11% sodium hydroxide in the oil-alkali mixture. Blanks on representative 50-g. samples of crude fatty oils were between 0.1 to 0.2 ml. of the standard acid. For purposes of checking the proportioner no blank correction will normally be required.

Ammonia analyses yielded erratic results. However the strong odor of ammonia from hot oil-alkali mixtures would point to serious losses of ammonia in the sampling process. Before the method could be applied satisfactorily to ammonia-oil mixtures, closed container sampling and treatment with excess acid and back titration with sodium hydroxide would be indicated.

Summary

A method has been developed for the determination of soda ash and sodium hydroxide in vegetable oil-alkali mixtures as a check on proportioning equipment feeding oil and alkali. The method depends upon the potentiometric titration in one step of free alkali and soap, using a pH meter and the glass electrode-calomel electrode assembly. The analysis is sufficiently rapid to serve as a convenient control in a continuous refining process.

REFERENCES

1. Clayton, B., Kerrick, W. B., and Stadt, H. M., U. S. 2,100,274 (1937).
2. Clayton, B., U. S. 2,249,701 (1941).
3. Clayton, B., U. S. 2,641,603 (1953).

[Received May 21, 1956]

Novel Method for Refining Soybean Oil

LESTER P. HAYES and HANS WOLFF, A. E. Staley Manufacturing Company, Decatur, Illinois

The usual processing of crude soybean oil to edible grade oil consists of degumming, alkali-refining, bleaching, winterizing, and deodorizing. Degumming by addition of a small amount of water to the crude oil has assumed greater prominence in the last decade because of increased demand for lecithin, which is obtained from the gums. Before this was the case, the bulk of crude soybean oil was alkali-refined without prior degumming. It is the object of this paper to describe the results of studies on refin-