

Once the crude and sweetwater glycerines have been excluded, further treatment will be carried out by the ion exchange process, consisting of three columns. These three columns are cation reactor, anion reactor, and mixed-bed. The flow diagram illustrating this process is shown in Figure 3.

In conclusion, it should be stated that the over-all loss of glycerine for the combined processes of ion exclusion and ion exchange is less than 1.5%. The entire product is of extra quality C. P. glycerine. Color and light stability of the excluded and exchanged glycerine is unsurpassed. With these points in mind the combined ion exclusion and ion exchange process appears to present a decided economic and end-product advantage over other methods.

Summary

This paper reviews, describes, and evaluates the processes for the production of C. P. glycerine, using ion exchange resins. By using the ion exclusion-ion exchange processes or the combined process to produce C. P. glycerine from all types of crude glycerines or sweetwaters, the entire yield of glycerine meets and/or exceeds U. S. P. specifications.

REFERENCES

1. Stromquist, D. M., and Reents, A. C., *Ind. and Eng. Chem.*, **43**, 1065 (1951).
2. U. S. Patent No. 2,615,924 (1952).
3. Wheaton, R. M., and Bauman, W. C., *Ind. and Eng. Chem.*, **45**, 228 (1953).
4. U. S. Patent No. 2,684,331 (1952).
5. Prielipp, Glenn E., and Keller, Harold W., Fall Meeting, Am. Oil Chemists' Soc., Philadelphia, Pa., Oct. 10-12, 1955.

Leaf Filter for Foots Removal from Crude Oil Reduces Labor and Eliminates Filter Cloth and Paper

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AS PRODUCED by mechanical screw presses or Expellers, almost all vegetable oils contain a certain amount of foots which must be removed to reduce the refining loss of the oil and make it more saleable. Normally the separation of the foots from the oil is accomplished by pressure filtration or centrifugation, and the former method is the most common. Conventional plate and frame or recessed plate presses have been used in the past, and a great many mills still use this type of filtration equipment.

In recent years a great deal has been done to improve existing filter designs to reduce operating and maintenance costs. Emphasis has been on savings in labor and on filter media costs, ease of cleaning, and over-all improved filtration efficiency. The pressure leaf filter is not by any means a new design, but its general application to the various filtration operations in the oil and fats industry has been relatively recent. Some of the first leaf type of filters used in this field were installed on the removal of nickel catalyst from hydrogenated products for the removal of bleaching clay from oils, fatty acids, and tallow, and for the filtration of fines from miscella.

Leaf filters designed specifically for ease of dry cake discharge were used successfully for the removal of foots from crude linseed and copra oils in 1952. Production units in operation since that time have demonstrated the adaptability of the leaf type filter for screw press oil and proved the fact that woven wire cloth filter media were adequate to remove all of the foots from the oil, thus eliminating the need for the usual filter cloths and papers.

In order to ascertain the filtration characteristics of crude cottonseed oil from mechanical screw presses or Expellers, a production-sized, horizontal style, vertical leaf filter was installed on a test basis at the West Memphis, Ark., Cottonseed Oil Mill of Perkins Oil Company, Memphis, Tenn.

Test Filter

This filter consisted of a horizontal cylindrical steel pressure tank in which a series of vertical filter elements were supported on a retractable carriage (see Figure 1).

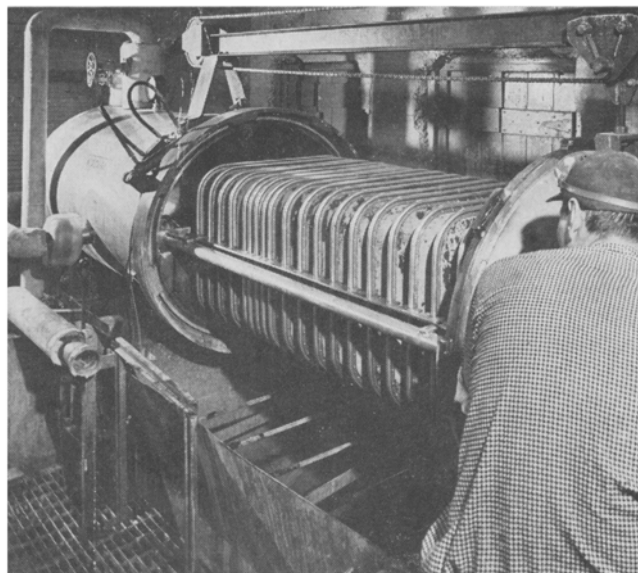


FIG. 1. Niagara H style pressure leaf filter with retractable leaf carriage.

The filter leaves (see Figure 2) consisted of two layers of a 24-x110-mesh Dutch weave wire cloth separated by a 4-mesh drainage member. A tubular binding holds this "sandwich" together, and a nozzle is welded at the bottom to allow passage of the filtered oil out of the leaf and into the pipe manifold through which it is discharged from the filter. Each element is separate and removable by means of an "O" ring gasket, which makes a seal between the leaf nozzle and the discharge manifold (see Figure 3). The leaves on the test filter were spaced on 4-in. centers.

Operating Technique

As it comes from the French screw presses, the cottonseed oil is passed over a 50-mesh vibrating screen or through a French screening tank and is then pumped to an agitated holding tank. The oil at this point contains 5-6% foots. The test filter was located to receive crude oil from this tank by means of a gear pump. The oil enters the filter through

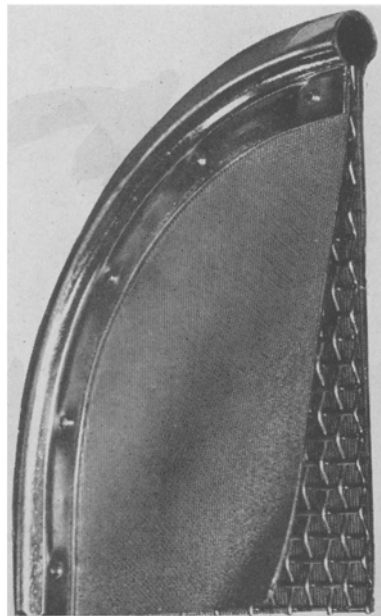


FIG. 2. Leaf construction showing woven wire cloth, drainage wire and tubular binding.

dual inlet connections at the side and when the filter has been completely vented of air, the oil is recirculated through the leaves back to the feed tank until clarity is established. The filtrate is then directed to the finished oil storage tank. This recirculation period normally requires less than one minute since a thin layer of flocs particles remains on the wire cloth after cleaning and serves as a precoat which retains the flocs in the oil as it passes through.

The end of the filtration cycle is indicated by the pressure build-up in the filter tank, and at this point the feed pump is cut off and compressed air at 40–50 p.s.i.g. is applied to the top of the tank. The unfiltered "heel" left in the tank is blown back to the feed tank to be filtered during the next cycle. Air is then passed through the cake on the leaves to reduce the residual oil content of the flocs cake.

The filter tank is then vented, and the filtrate line is disconnected by means of a quick-coupling. The cover is released hydraulically, and the carriage is

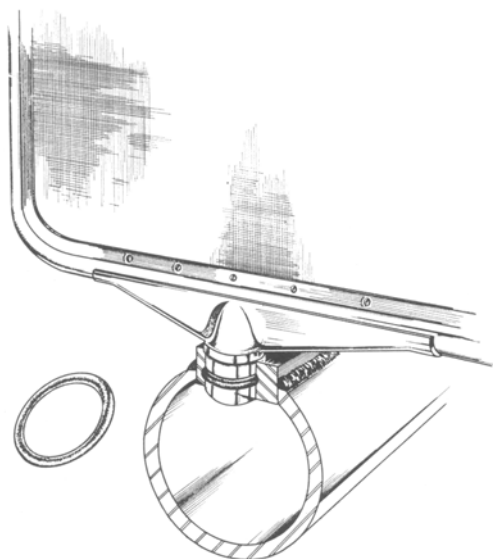


FIG. 3. Leaf nozzle construction showing O-ring gasket.

Test Results

Summary of Filtration Data

Run No.	Filtration ^a time (min.)	Average filtration rate lbs. oil/hr./sq. ft.	Capacity lbs. oil/sq. ft.	End pressure (p.s.i.g.)	Cake thickness (inches)
1	50	71.5	59.7	75	1 ¼
2	52	69.0	59.9	75	1 ¼
3	47	73.5	57.7	75	1 ¼
4 ^b	110	46.1	84.5	85	2
5 ^b	77	57.2	73.5	75	1 ½

^a Includes recirculation time.

^b French screening tank pretreatment of oil.

rolled out. The air-blown cake separates easily from the wire cloth, and cake is discharged from the leaves by tapping the side of each leaf with a rubber mallet. After cleaning, the carriage is returned to the tank, the cover resealed, and the filtrate line reconnected. The unit is then ready for the next cycle.

Clarity of Filtered Oil

Samples of filtered oil were taken immediately after the initial recirculation period, at various times during the filtration, and at the end of the run when the pressure had reached 75 p.s.i.g.

1. Sedimentation Tests: no solids settlement after two weeks of standing.

2. Gasoline Insolubles Test: 0.025%.

Residual Oil in Air Blown Cake

Representative samples of air-blown flocs cake were taken from various places on the leaves and analyzed for residual oil by solvent extraction.

Average oil content of cake

(10 min. air blow)—39%

(20 min. air blow)—35%

Downtime for Cleaning

Draining tank of unfiltered heel.....	5 min.
Air blowing.....	10 min.
Opening.....	2 min.
Cleaning.....	10 min.
Closing.....	2 min.
Refilling tank.....	5 min.
Recirculation to establish clarity.....	1 min.

Total.....35 min.

Discussion of Results

The linear relationship shown in Figure 4 between filtration pressure and filtration time indicates that the flocs cake is relatively non-compressible at a constant rate. These curves can safely be extrapolated

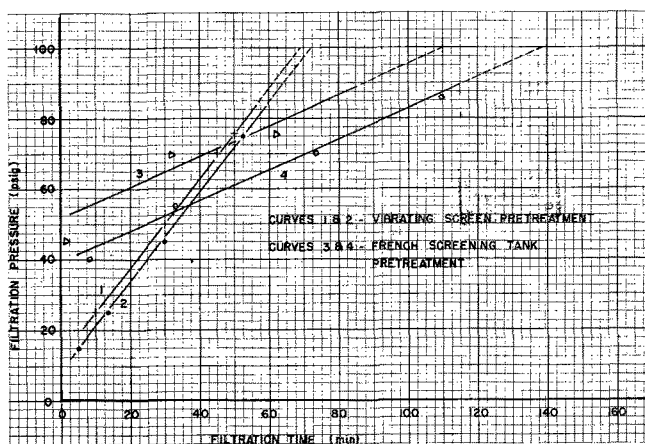


FIG. 4. Graph showing linear relationship of filtration pressure and filtration time.

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.

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Checking Alkali Delivery of a Proportioner in Vegetable Oil Refining

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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