

Some workers have attempted to correlate growth-depressing action of heated oils to polymer formation, but as no reliable method is available for determining the percentage of polymers in heated oils, this relationship is difficult to determine. It can be assumed that corn oil which had the highest percentage of polyunsaturated fatty acids would give the highest percentage of thermal polymers, but whether the polymers formed during thermal-oxidation are strictly thermal in nature has not been shown. Butter oil contains a low percentage of polyunsaturated fatty acid and would contain therefore a low percentage of the type of fatty acids which form polymers easily. If polymers and polymeric type products are responsible for the growth depression, the present results would be expected.

An attempt was made to correlate changes in constants, such as iodine value, acid value, and peroxide value to growth depression. The absolute decrease in iodine values was related to the growth-depressing action of the oil, but whether the growth depression is due to compounds formed by the reactions which led to the decreased iodine value is not known. However percentage losses in iodine value were about equal for all of the oils and peroxide values and acid values did not seem to be related to growth depression. Thermally oxidized butter oil, which had no apparent growth-depressing action, had the highest acid value and as high a peroxide value as the thermally oxidized corn oil.

The recovery of animals which had been changed from a thermally oxidized corn oil diet to a fresh corn oil diet would seem to indicate that the thermally oxidized oil did not cause permanent metabolic damage. The animals rapidly regained a normal appearance, had no diarrhea, and exhibited normal growth. As adequate vitamins were provided in both diets, it would appear that this recovery was due to the removal of a growth-depressing product from the diet and that this product was responsible, either directly or indirectly, for the symptoms noted in the animals. Whether the growth-depressing action of thermally oxidized corn oil was due to a simple irritation effect which caused the severe diarrhea and lowered food intake, or to destruction of some vitamin or enzyme factor is not known. The rapidity at which the symptoms appeared would seem to indicate that a simple vitamin deficiency was not responsible. Studies with enzyme systems and oxidized oil indicated that these oils did produce

materials which were inhibitory to respiratory enzyme systems in liver tissue, and it was possible that similar inhibitory products were produced in the thermally oxidized oils (1, 10). The rapid recovery obtained when the animals were changed to a fresh corn oil diet would indicate that there was no destruction of enzyme-producing sites.

It is possible that the entire effect was due to an irritation of the intestinal tract, causing diarrhea and a disruption of normal metabolism. However, on diets containing a lower percentage of heated oils, other workers have found that even when diarrhea was not present, there was still some growth-depressing effect (8). It would therefore appear that the growth-depressing action of thermally oxidized corn oil was due to other factors than the irritation or diarrhea effect.

Summary

Thermal oxidation of corn oil under laboratory conditions, at 200°C., led to the formation of an oil exhibiting definite growth-depressing action under both *ad libitum* and paired feeding conditions. Under similar conditions margarine base stock gave only slight growth depression, and none was noted with butter oil. The effect was not a permanent one as animals that were changed to a normal diet quickly recovered and grew to maturity. It appeared that the products formed during the thermal treatment were related to the unsaturated or polyunsaturated portions of the oil. The growth-depressing effect appeared to be multiple in nature, it had an irritant or diarrhea effect and possibly an enzyme-inhibiting or vitamin-destroying effect.

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An Evaluation of Methods for Production of C. P. Glycerine¹

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GLYCERINE IS PRODUCED by a variety of methods, the more important of which are as follows: the hydrolysis or saponification of fats and oils for the production of soap with a yield of glycerine as a by-product and the chlorination of allyl alcohol and allyl chloride to produce synthetic glycerine. The glycerine produced by these processes may be evaporated to crude glycerine and further distilled under

vacuum to yield pure glycerine. The over-all recovery of C. P. glycerine by this method varies from 90 to 96%.

The purification of sweetwaters and crude glycerine, without distillation, has been reported as early as 1928. In 1951 Stromquist and Reents (1) published an extensive study on glycerine production entitled "C. P. Glycerol by Ion Exchange." As a result of these studies U. S. Patent No. 2,615,924 (1952) (2), "Method of Purifying Glycerine," was issued to

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	SWEETWATER	SOAP LYE CRUDE
GLYCEROL	96.8%	99.02
% RESIDUE	0.0012	0.0010
% ASH	0.003	0.0004
% CHLORIDES (NACL)	0.0001	0.00016
F.A. & ESTERS	0.09 CC.	0.08 CC.
DU PONT TEST	EQUAL OR BETTER	EQUAL OR BETTER

Fig. 1. Partial analyses of two purified samples.

Reents. In most cases the glycerine produced, as was outlined in this paper, will meet C. P. specifications. It has been established that certain non-ionized impurities, such as polyglycerides and allyl chlorides, are not removed by ion exchange. Therefore the production of C. P. glycerine from synthetic crude glycerine by ion exchange presents problems concerning the non-ionized impurities.

A continuous investigation is being carried out on ways and means of bettering product quality and of decreasing operating costs. As described by Wheaton and Bauman (3), by using the ion exclusion (4), it is possible to separate sodium chloride from glycerine. The investigation was carried out on a pilot-plant scale and was reported by Prielipp and Keller (5). It was reported that C. P. glycerine could be produced for 1¢ per pound by using the combined processes of ion exclusion and ion exchange. The cost figure is based upon the treatment of soap lye crude glycerine. To produce C. P. glycerine from soap lye crude glycerine by the use of straight ion exchange would cost 2.8¢ per pound. Similarly the combined processes can be used to produce C. P. glycerine from synthetic material, either diluted or concentrated. The separation of glycerine from its dissolved salts by use of the ion exclusion process is described in detail in the aforementioned paper entitled "Purification of Crude Glycerin by Ion Exclusion."

From an economical standpoint, when the crude or sweetwater glycerine to be treated contains over 1.5% dissolved ionized materials, it is recommended that the combined processes of ion exclusion and ion exchange be investigated. Similarly, when dissolved ionized materials are less than 1.5% straight ion exchange is applicable.

Some crude glycerines and sweetwaters to be treated by the combined processes of ion exclusion and ion exchange are given below.

1. Soap lye crude glycerine containing 10% sodium chloride, 80% glycerine.
2. Saponification crude glycerine containing 1% to 4% ash, 85% glycerine. Although some saponification crude glycerine may be low in ash, 1%, the combined processes are recommended because of the large amount of color bodies present. Most of the color will be excluded during the ion exclusion step.
3. Synthetic crude glycerine containing 8% to 10% sodium chloride, 80% glycerine.
4. Soap lye containing 5% to 15% sodium chloride, 3% to 8% glycerine.
5. Twitchell sweetwater containing 2% sulfuric acid, 10% glycerine.
6. Synthetic glycerine solutions containing 10% to 18% sodium chloride, 15% to 30% glycerine.

7. Waste streams, high in ash, containing 5% to 10% ash, 3% to 5% glycerine. For example, a waste glycerine stream containing 4% glycerine and 8% sodium sulfate has been treated by ion exclusion. A separation of 98% of the sodium sulfate from the glycerine was achieved. The product concentration was 3.0% glycerine.

Some glycerine sweetwaters to be treated by straight ion exchange are Emery-Colgate process sweetwater containing 0.2% to 0.5% ash, 8% to 15% glycerine; and waste streams, low in ash, containing 0.2% to 0.5% ash, 3% to 5% glycerine.

By using the ion exclusion process, the ion exchange process, or the combined processes to produce C. P. glycerine from the above mentioned crude or sweetwater glycerines, the entire yield of glycerine is obtained as a high-quality product. The glycerine produced will have an analysis comparable to that given in Figure 1.

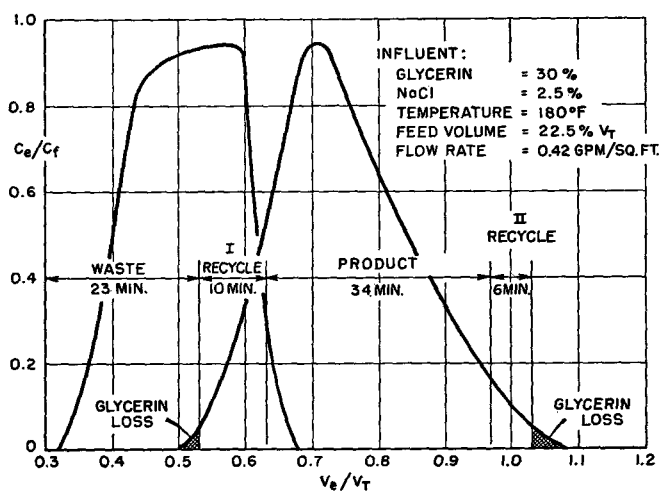


Fig. 2. Elution curve and time cycle.

The operation of an ion exclusion column will be completely automatic with simple adjustments. The effluent from the column can be followed by recording conductivity and density instruments. A typical elution curve-and-time cycle is shown in Figure 2. It should be pointed out that a large amount of the color bodies are excluded to the waste or ionized material. This color exclusion is attributed to the large ionic size of the color bodies, which will not penetrate the resin bead during the ion exclusion process.

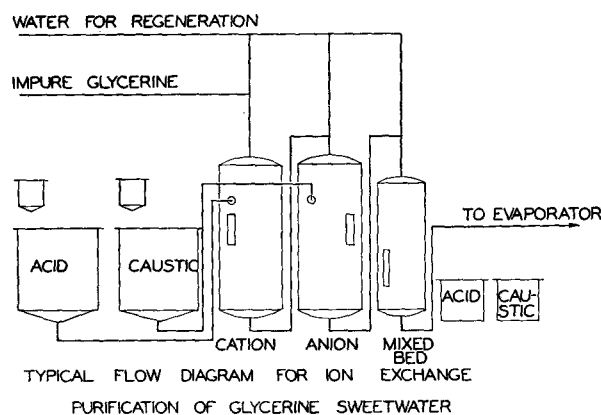


Fig. 3.

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.

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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).

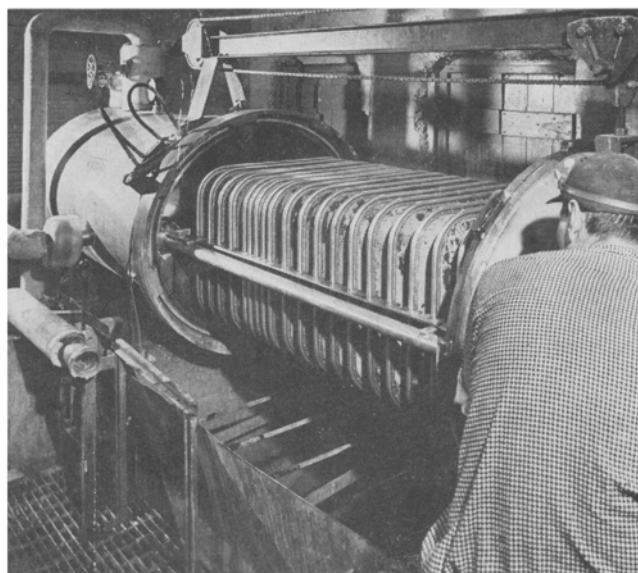


FIG. 1. Niagara H style pressure leaf filter with retractable leaf carriage.

The filter leaves (see Figure 2) consisted of two layers of a 24- x 110-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