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Vegetable Oil Refining by the Modified Soda Ash Process

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THE MODIFIED soda ash process for the refining of vegetable oils differs from the original 1945 version of the Clayton soda ash process in that neither the water nor the carbon dioxide, which results from the neutralization of the crude oils with soda ash, is removed from the oil by a dehydrator, and consequently no rehydration of a mixture containing dry soda ash and soap is necessary. Eliminating these two steps materially reduces the operating costs and the initial equipment costs for new soda ash plants.

This development was made possible by recognizing that the troublesome carbon dioxide released when soda ash neutralizes the free fatty acid of a crude oil can be kept under control by either solubilizing the gas under pressure or by providing sufficient excess of the soda ash reagent to combine chemically with the carbon dioxide to make sodium bicarbonate. Under these conditions neither emulsions nor floating soap cause difficulty in the primary separation stage. Since operating with an excess of soda ash requires no additional equipment, six refiners in the United States who own and have operated the Clayton soda ash system had converted to this new method of operating without the dehydrator by the fall of 1954.

Development work based on this new method of refining has continued since these plants were converted, with progress considerably accelerated by the commissioning of the Sharples Research Refinery in Philadelphia. We are now able to release definitive instructions for operating these plants without adding expensive new equipment and without using large excess quantities of soda ash. In addition to the loss savings possible, the results have an important bearing on cost of operation and on utilization of the soapstock, which make the process quite attractive to those operating continuous caustic refineries.

An important development to come out of the recent work is the use of a small gas vent tank. This unit can be constructed by the maintenance department of any refinery and consists of a closed tank approximately 12 in. in diameter. Oil is introduced into the side of the tank, as illustrated in Figure 1, and withdrawn from the bottom. Any gas existing in the mixture of oil and reagent rises to the top of the tank and is vented by means of a standard gas vent valve. Such a valve will permit the escape of air and carbon dioxide but will automatically close when the gas is eliminated and oil rises to the valve position. Pump pressure, as developed by the usual crude oil pump and as required for the process, is transmitted undiminished across the vent tank. Figure 2 shows this tank as installed in conjunction with the balance of the process equipment.

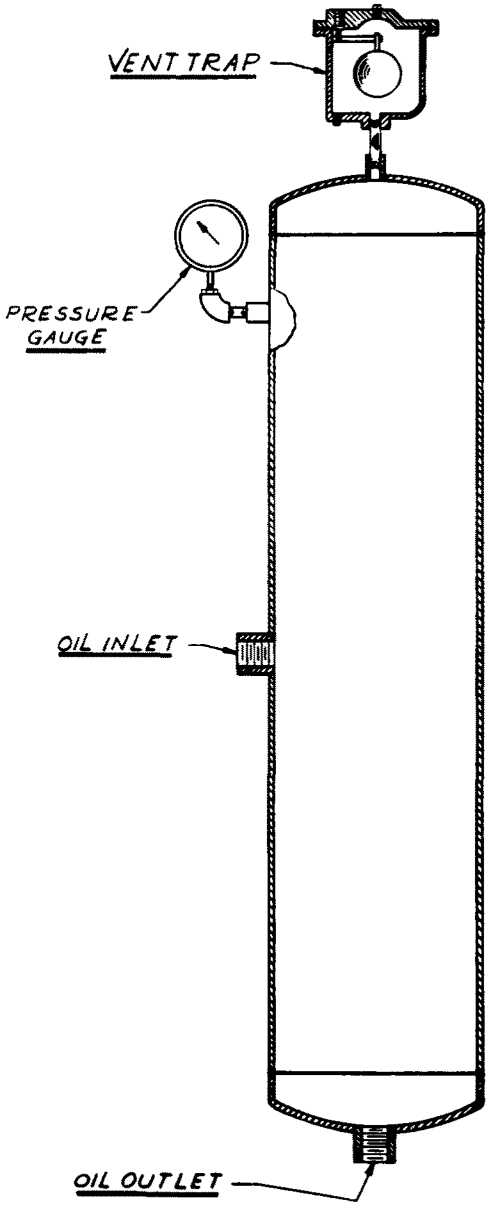
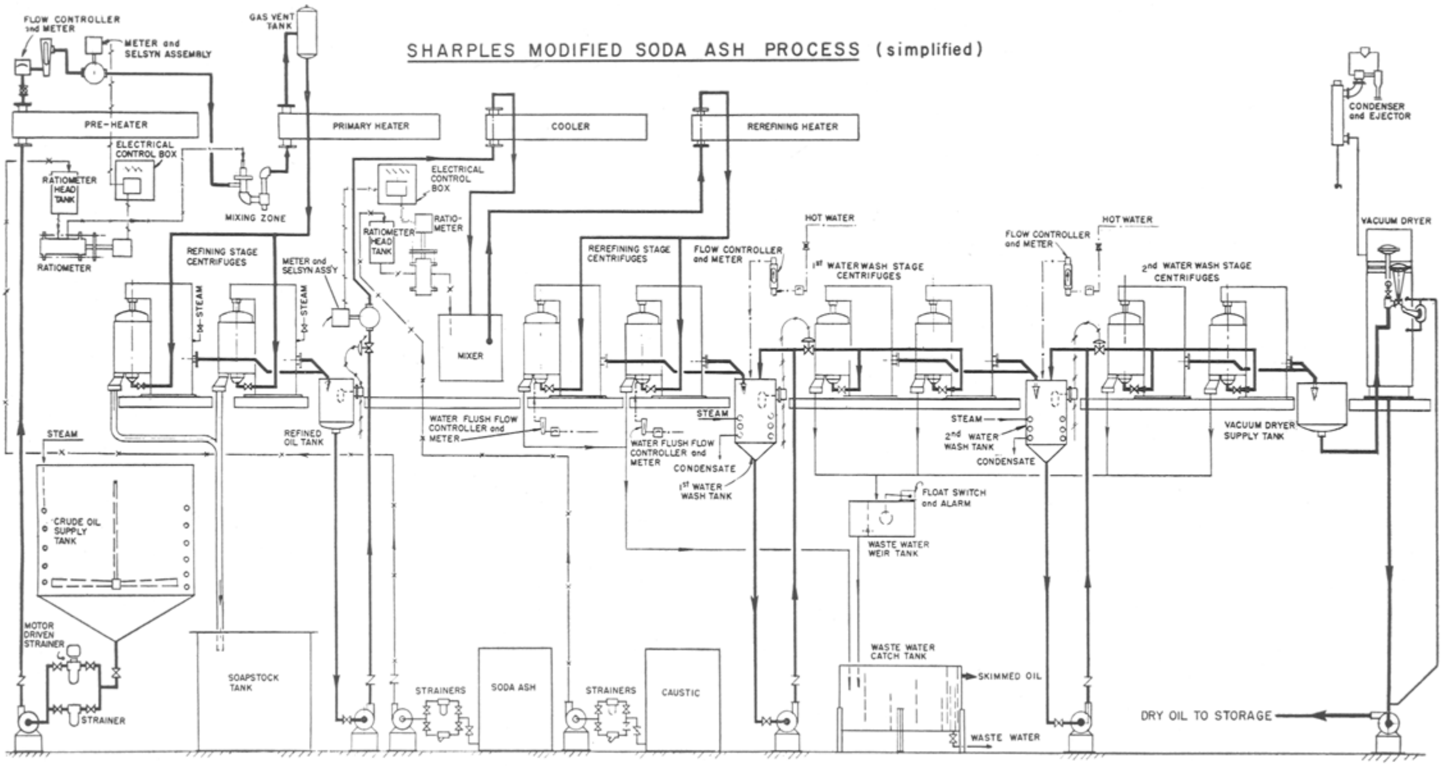


FIG. 1. Deaeration tank.

Process Equipment

Since much of the data presented in this paper is derived from the Sharples Research Refinery, and since the complete control and system of cross checks possible in this refinery make it ideal for definitive tests to compare any process or equipment variations, this grouping of components will be outlined.



Oil is brought in from agitated storage tanks, which make available 30,000 lbs. of thoroughly blended crude oil for comparative tests. Each test is based on a calibrated run of approximately 4,000 lbs., which is accurately weighed in a standard-sized scale tank. It might be noted at this point that it took two weeks of circulating refined oil in this refinery before we were satisfied that blow-down and drain lines were adequate for overall loss measurements accurate to a plant loss of 0.1%.

The crude oil is preheated in the scale tank to any desired temperature, and soda ash is proportioned by means of the Sharples Ratiometer. The soda ash solution is prepared in accurate scale tanks in such a way that an overall weight check on reagent consumption during any run acts as a check on the setting and operation of the proportioning device, and this becomes a determination of the validity of the experiment.

For contacting the crude oil with the proportioned soda ash reagent, the Clayton mixing zone usually supplied with the original soda ash process is satisfactory. This consists of a series of small annular passages designed into a pipe unit to produce shear-force mixing by pressure drop.

After mixing, the oil-soap mixture is heated in a fintube heater and sent through the gas vent tank described above. From the vent tank it is separated in a Sharples No. 26 Super Centrifuge, where the highest available centrifugal force is applied to squeeze oil from the soapstock.

From this point on, the process is the same as the original soda ash process. The oil is cooled in a fintube unit, caustic is proportioned and checked by the same type of equipment and in the same way as described for the soda ash treat, the caustic and oil are contacted in either a Sharples low speed mixer of standard design or by means of a high-shear mixer, the temperature is raised as required for separation, and a Sharples No. 26 Super Centrifuge rerefiner with water flush produces a refined oil of satisfactory color. In this refinery, double water-wash and vacuum-drying always follow any refining test in order that the final scale measurements will reflect actual commercial operation without the doubt introduced when the moisture and soap determinations, required to adjust the weight figures, are not done. The washed and dried oil is weighed in a separate, refined oil scale tank, which is checked against the crude oil scale at regular intervals.

Process Details

The crude oil is preheated to about 100°F. No harm is done by going as high as 130°F., but it is well to stay below this temperature in order not to damage the color when refining cottonseed oil.

Accurate proportioning is quite important since small dosages are used. Any device used should be accurate in the range of 1 to 5% treats. Soda ash reagent is made up to 22° Bé. There is some evidence that 26° Bé. is better. The former is recommended because of the possibility of supersaturation in the reagent solution during cold seasons.

Except for oils of very low FFA, such as degummed soybean, the total treat used is based on sufficient reagent to supply 2.5 to 3 times the quantity of soda ash required to neutralize the free fatty acid of the

TABLE I

Oil	% FFA	Chromatographic loss (%)	Plant loss (%)	
			Mod. soda ash	Caustic
Cotton	0.8	4.65	5.51	6.44
Soybean	0.6	3.01	3.70	4.04
Cotton	1.4	3.42	4.47	5.13
Soybean	0.7	4.31	4.72	5.19
Cotton	0.9	2.65	3.64	4.63
Soybean	0.5	1.89	1.99	......
Deg. soybean	0.3	0.60	0.93	......
Linseed	1.3	2.52	3.14	......

TABLE II

Refinery	Oil	No. of tank cars	% FFA	Cup loss (%)	Plant loss (%)		Caustic
					Mod. soda ash	Full soda ash	
A	Corn	1	2.0	7.2	4.28	....	5.90
A	Corn	1	1.2	5.0	2.85	....	3.23
B	Soybean	150	....	....	3.18	3.72	....
C	Linseed	15	1.2	....	2.46	....	....
D	Soybean	2	0.4	3.4	2.13	....	....
D	Deg. soybean	10	0.7	1.9	0.72	....	....
D	Mixed soybean	13	....	2.2	1.30	....	....
D	Deg. soybean	13	....	3.6	2.63	....	....

Refinery	Oil	No. of tank cars	% FFA	Cup refining		Modified soda ash refining	
				% Loss	Bleach	% Loss	Bleach
E	Cotton	17	1.4	8.0	2.5	4.68	2.4
E	Cotton	8	....	6.7	3.4	4.69	2.7
E	Cotton	2	....	7.5	3.2	4.50	2.8

oil, using the assumption that all the sodium of the soda ash is available for neutralization. On oils of low FFA we, at present, use a minimum treat of 1% of 22° soda ash.

After mixing in a mixing zone, the oil is heated to at least 180°F., and here again proper control of the temperature is important. A reasonable temperature range would be between 180 and 200°. The heated oil is then degassed in the gas vent tank and centrifuged.

The cooler should cool the primary refined oil to about 100°F. and is followed with a decolorizing caustic treat, which will depend to a large extent on the crude oil. Soya oils can be lightly washed with 8 to 12° Bé. caustic, prime cotton oils can be rerefined with low treats of 20° Bé. caustic, and dark cotton oils may require up to 3% of 26° or 32° Bé. caustic adequately to reduce color. It is important to note that, in the absence of soap from primary neutralization, excellent colors may be obtained from dark oils without seriously affecting the overall loss results. Generally the rerefining centrifuges will require between 20 and 30% water flush to assist in the discharge of the traces of soap removed.

By means of the low primary treat, the refining soapstock will be above 35% TFA, usually falling between 37 and 40%. This materially reduces the problems of acidulation and even produces a salable material as is. The most important trend of recent times in this regard is the work being done through the efforts of Refining Unincorporated and B. H. Thurman to prove beyond question that this particular type of soda ash soapstock, having a low excess of free carbonate or bicarbonate, is both palatable and nutritional as an additive to cattle feed. Preliminary reports from the Chicago stockyards and several state universities are very encouraging, based on tests made from materials supplied in quantity from the Sharples Refinery.

Data

It is readily recognized that data based on laboratory analysis or on an arbitrary selection of cup loss values could lead to doubt. For this reason we have chosen to relate each run to the Wesson loss or the chromatographic loss of each crude and to associate most modified soda ash runs with a run of equal length, using the same crude supply but operating as a caustic refinery. In each case we have taken pains to carry out the caustic run according to the best known techniques available to our experienced refiners, and in some cases this attention to detail has led to a better caustic refining than might be expected from day to day plant operation.

Table I shows the results of these comparisons.

On a basis involving much less control, this process has been operated successfully by a number of commercial refiners. Table II shows the data available to us for general publication from these installations.

Both of these tables show a superiority of ½ to 1% in refining loss for the soda ash process over the caustic process. Refinery E reported in Table II shows a reduction in bleach color of cotton oil below the bleach color established in the settlement cup refining, and this advantage has been maintained over a period of at least one year. During this period savings over cup refining losses have averaged about 30%.

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Surface-Tension Determinations with a Precision Micro Capillary Rise Apparatus[1]

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THE MORE COMMONLY used procedures for measuring surface tension, such as the ring detachment method (13, 9), the drop weight method (14, 15), the bubble pressure method (24), the hanging drop method (10), and the classical capillary rise method (15, 21, 22), are either very time-consuming, employ a complicated apparatus, require a large sample, do not give absolute values, and thus require correction factors or are extremely tedious techniques. Since the author desired a quick, micro method for determining surface tensions, he tried Ferguson's method (5) with a series of methyl esters of normal saturated fat acids. Although the results (20) were encouraging, they showed that Ferguson's method needed refine-

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