Vol. 33

Use of the Multistage Countercurrent Contactor For Degumming and Refining of Vegetable Oils¹

T^{HE REMOVAL OF GUMS} (phosphatides) and certain other nontriglyceride impurities from vegetable seed oils has been practiced for many years in the edible and inedible oil industries. The process generally employed consists of mixing water with the oil (for degumming) or caustic soda (for refining) and then separating the added phase centrifugally. Further steps in the refining process are water-washing to remove dissolved soap, vacuum-drying, and bleaching.

This paper describes the use of a centrifugal countercurrent multistage extractor, which carries out the degumming or refining process with lower centrifugal force than is used in conventional centrifuges and yet gives more complete removal of gums or soaps, with lower initial outlay and lower cost for operation and maintenance. Data are given for installations handling edible and inedible vegetable oils.

The good results are attributable to radical differences between the multistage contactor and the usual centrifugal oil refining equipment. These are made clear by the following brief excursion into the theory of centrifugal separation.

Theory of Centrifugal Separation (2)

A particle in a flowing stream of liquid, acted on by an external force, will be removed from the stream if the velocity, V_c (determined by the throughput and diameter of the conducting passage), is sufficiently low for the settling velocity, V_s , to bring the particle through the settling distance, S, before it has been swept on out in passing through the travel distance, Y (Figure 1).

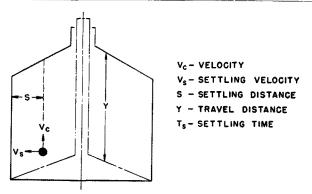


FIG. 1. Theory of centrifugal separation. General design hollow bowl centrifuge.

The limit for settling out such a particle is that its settling time, T_s , through the settling distance, S, shall be just equal to, or less than, the time, T_e , required to move through the travel distance, Y.

$$T_s = \frac{S}{V_s} = T_c = \frac{Y}{V_c}$$

Limiting V_c , and simplifying, we obtain the equation:

$$\mathbf{V}_{c} = \frac{\mathbf{Y}}{\mathbf{S}} \mathbf{V}_{s}$$

Therefore clarifying throughput capacity is directly proportional to the travel distance, Y, and inversely to the settling distance, S.

Centrifuge Design

Hollow bowl centrifuges were the first type developed. They are relatively simple in design and, when built in long tubular form of small diameter, allow the use of high centrifugal force with reasonable ratio of travel to settling distance (Figure 2). Centrifuges of this type have however the following inherent limitations:

1. The settling distance must be nearly equal to the radius of the tubular bowl.

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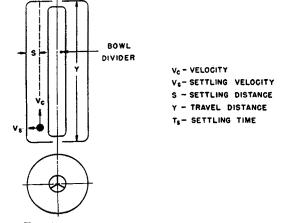


FIG. 2. General design of tubular centrifuge.

- 2. The liquid, entering at the center, is at first exposed to only a small percentage of the centrifugal force available at the periphery of the bowl.
- 3. Any sediment collecting on the inside of the bowl decreases settling distance and reduces the maximum centrifugal force on the liquid. It reduces holdup, which, with constant liquid velocity, also reduces the time of exposure to centrifugal force.
- 4. Extremely high centrifugal force is necessary to maintain efficiency of separation.
- 5. As single machines of high capacity are not feasible, multiple installations are necessary.
- 6. The necessity of operating at tremendous speed results in high maintenance costs.

In another widely used type of centrifuge the separating chamber is a bowl containing cone-shaped discs with thin spacers which produce very shallow settling spaces. The liquid is subjected to long travel with guided flow and moves with a moderate velocity. Centrifuges with this type of bowl also have several disadvantages. The bowl is heavy, has an objectionable number of internal parts to be handled and cleaned, is expensive, and has high maintenance cost. Like the tubular form of machine, the capacity of a single unit is limited, making multiple installations necessary.

Multistage Countercurrent Contactors

Countercurrent centrifugal contactors, specially designed to handle fatty oils and known as Duozons, make the most effective use of centrifugal force of any machine yet devised. The settling distance, S, is very small compared with the travel distance, Y. Of particular importance is the fact that the direction of travel is separated by 180° from the direction of settling, in contrast with the conventional centrifuge where Y and S are separated by 90° . The travel distance Y may be increased, keeping the same S distance, by increasing the

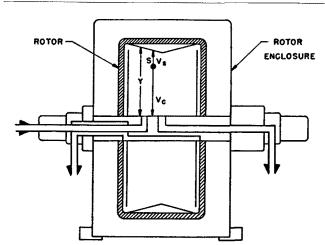


FIG. 3. General design of Podbielniak Duozon.

diameter of the rotor within the limitations of allowable r.p.m. and retention time (Figure 3).

The Duozon consists essentially of a horizontally positioned, totally enclosed rotor, mounted on a shaft with heavy duty ball bearings, force-feed lubricated. It is constructed of stainless steel with heavy welded steel base and rotor cover. Inside the rotor are many contacting elements which provide for intimate mixing coupled with low liquid velocity and controlled settling rates. The many contacting elements provide several times as much coalescing surface per unit volume processed as do conventional centrifuges.

The heavy liquid and light liquid are kept separate by pressure balanced mechanical seals, two on each side of the rotor. These seals are designed for pressures up to 700 p.s.i. and temperatures of 300° F.

Design factors have been worked out which allow retention of the advantageous characteristics of centrifugal contactor: high ratio of holding time with low flow rate, high ratio of travel time to settling time, and effective operation with relatively low gravities by machines of any practicable size.

The operation of the machine when used as a countercurrent liquid contactor (Figure 4) is as follows. The light liquid

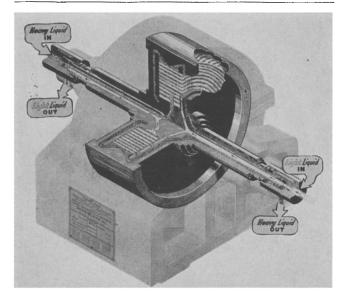


FIG. 4. Flow of fluids in the Duozon multistage contactor.

enters through the shaft and is directed to the outer periphery of the rotor. The heavy liquid entering through the shaft at the opposite side is directed to the center of the rotor. Centrifugal force moves the heavy liquid outward, and it displaces the light liquid toward the center of the rotor. The contacting elements are so designed that passage through the orifices provides multistage mixing and separation.

When a semi-solid, such as soapstock, is processed, it coalesces on the inner surfaces of the contacting elements and as it increases in amount, moves to the outer periphery of the rotor, where it deposits on the V-shaped annulus, which directs it to the spill-over sides of the rotor, whence it moves to the shaft and is discharged through a seal-shaft annulus.

The light liquid moves through a best state transmission of the shaft and is discharged. A back pressure regulator automatically maintains a pressure on the light liquid attempting to leave. The effect of this pressure is to control the position of the interface (it is comparable to the ring dam in an ordinary centrifuge) separating light and heavy liquid, *e.g.*, oil and soapstock, in the rotor. The position of the interface can be varied at will while the machine is operating.

Degumming and Refining Soybean Oil

The A. E. Staley Manufacturing Company in Decatur, Ill., has made extensive use of the Duozon in degumming soybean oil. A pilot plant extractor was used in development work. The machine had a rotor 1 in. wide and 25 in. in diameter and was designed for a throughput of 1 gal. per minute (500 lbs. per hour). A series of tests was made in which the water-oil ratio, temperature, back-pressure adjustment, and speed were varied in order to determine optimum operating conditions.

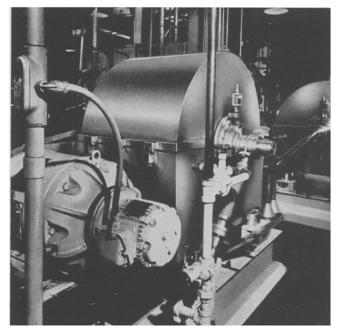


FIG. 5. Installation of Duozons and water-wash Hydrazons at the A. E. Staley plant.

At flow rates up to 3.5 gal. per minute and water rates previously used the products obtained were better than those from the existing centrifuge installation. The lecithin obtained, as shown in Table I, had about 70% acetone-insoluble (dry basis), and the degummed oil had as low as 0.11% Gardner break. At a throughput of 2 g.p.m. results superior to previous centrifuge operations were obtained with a water treat considerably below the normal figure of 1.6%.

TABLE I Duozon Soybean Oil Degumming Analysis of Wet Gum

Oil Rate 1.0% Tre		1.2% Treat	1.4% Treat	1.6% Treat	
g.p.m.	H ₂ O AI(DB)	H ₂ O AI(DB)	H ₂ O AI(DB)	H2O AI(DB)	
1.0	% % 24.0 71.8	% % 30.6 69.8	% % 34.2 68.6	% % 32.0 69.9	
1.5 2.0	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	$\begin{array}{cccc} 27.4 & 69.7 \\ 23.5 & 70.7 \end{array}$	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	
2.4	21.4 71.8 Gardn	22.4 69.8 er Break on Des		100.0 0012	
	GBb	GB	GB GB		
%		%	%	%	
1.0	.16	.19	.12	.12	
$\frac{1.5}{2.0}$.15 .30	.13 .11	.17 .10	.12 .13	
2.4	.25	.15	.12	.11	

Acetone Insoluble (Dry Basis).
^b Gardner Break.

At the conclusion of pilot plant testing in July, 1953, Staley engineers reported that "the tests indicate that the Duozon can be used quite successfully in the degumming of soybean oil." In an economic survey which they submitted to the executive committee of this company, the following advantages of use of the Duozon over previous operations were emphasized: higher throughput with given capital investment; gums of higher acctone-insoluble value; more nearly continuous operation, without periodic shut-downs for cleaning; lower operating and maintenance costs; more automatic operation, requiring less attention from the operator.

On the basis of the extensive test work the Staley company purchased in 1953 a production size unit for water degumming of soybean oil. It had a rotor 15 in, wide and 36 in. in diameter. The rated capacity was 10 tank cars per day of crude throughput. It was installed with automatic control equipment.

A careful evaluation was made of this machine's performance on water degumming of soybean oil. One sustained run of four months' duration was made without interruption for maintenance or cleaning. At the same time development work was carried out on a unique degumming process with the pilot plant extractor. On the basis of the production experience and the pilot plant experience, one additional Duozon and two water-washing multistage centrifugals (Hydrazons) were purchased. In February, 1956, these machines were placed on stream, water degumming and water washing soybean oil directly into an oil suitable for bleaching and deodorizing without caustic refining. The physical chemistry of the process was described in the paper Hayes and Wolff presented at the A.O.C.S. Meeting in Houston, Tex., April 24, 1956. Plant operational details will be the subject of a later paper. The plant installation is shown in Figure 5.

Other Applications

Caustic refining of extracted degummed soybean oil and of crude soybean oil and cottonseed oil with the Duozon has been carried out by another refiner. While a complete report will not be available until a later date, preliminary results are presented in Table II. The caustic refining of degummed soybean oil shows a 50% saving over cup loss. Caustic refining of crude cottonseed oil gives a 24% saving and of crude soybean oil a saving of 48%.

TABLE II Caustic Refining of Cottonseed and Soybean Oil Properties of Original Oils

	Crude Oils		Degummed Oils	
	Cottonseed	Soybean	Soybean	Soybean
FFA, %	1.0	0.50	0.18	0.18
Color, Lovibond Concentration	50/5.0	55/5.5	50/5.0	50/5.0
caustic solution, %	11.5	8	10.5	12.0
Pro	operties of R	efined Oils		
FFA, %	.04	.03	.02	.02
Color, Lovibond	20/3.5	32/3.8	35/3.5	35/3.3
Soap, p.p.m	450	900	600	500
$H_2O,\%$.06	.19	.12	.16
Loss, %	4.5	2.6	.87	.89
Cup Loss, %	5.9		1.89	1.8
Wesson Loss, %	2.5		.6	.6

Similar refining tests are under way in four other refineries on the following oils: crude peanut, crude linseed, crude coconut, lard, and tallow.

Encouraging preliminary reports have been received from these operations. One refiner has reported that, in degumming, the Duozon showed a savings over parallel operations, using high speed centrifuges of 0.8 lb. oil per 100 lbs. processed, and in caustic refining a saving of as much as 1.9 lbs, oil per 100 lbs. processed.

Figure 6 presents a typical flow sheet for the continuous caustic soda refining of vegetable oils with the Duozon. The combined oil and caustic solution are automatically metered to the primary machine where the soapstock is separated from the refined oil. The oil is then water-washed in a second unit where after six to eight stages of extraction, it contains not over 10 parts per million of soap. The wash water has a maximum total fatty acid content of 0.60%.

The plant is furnished as a completely engineered unit, fabricated largely of stainless steel. The system is air-tight with no open tanks, which allows the refiner to keep the process areas clean and free from oxidized oil.

A recently installed Duozon has operated very satisfactorily in an inedible oil plant. Its ability to operate continuously under adverse conditions was notable. The refinery was processing solvent-extracted oil containing a large proportion of fines. During a one-week test period the high speed centrifuges. running parallel to the Duozon had to be stopped and cleaned 22 times, after every four hours of operation. The Duozon operated without a single stop, and it is estimated that operation could have been continued indefinitely.

Conclusions

Duozon centrifugal contactors seem to have the following advantages in the refining of glyceride oils: decreased losses and increased yields of products; large capacity in a single machine resulting in low first cost, reduced requirement for floor space, and simple flow and installation; because of comparatively low speed, low maintenance cost and low power

REFINED OIL TO DUOZON DUOZON VACUUM DRYER GRUDE REFINED WASH OIL OIL WATER SOAPSTOCK WASTE WATER PRESSURE MIXER OPERATED PUMP BY-PASS VALVE CAUSTIC

FIG. 6. Continuous caustic refining of vegetable oils with countercurrent centrifugal contactor.

requirement; practically automatic operation; continuous operation unhampered by particles of meal in the charge; and an entirely enclosed system, allowing no oxidation of hot oil.

Acknowledgments

Gratefully acknowledged are the contributions made by Noel Myers, senior chemical engineer, A. E. Staley Manufacturing Company, Decatur, Ill., and others who submitted data on plant operations but who prefer to remain anonymous at present.

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On the Educational Front

Shell Companies Foundation Inc. will contribute about \$450,000 during 1956 to educational programs, according to M. E. Spaght, executive vice president of Shell Oil Company and president of the Foundation. This will include 50 graduate fellowships, 20 grants for fundamental research to 42 colleges and universities, and merit fellowship seminars at Cornell University and Stanford University for 60 high school science and mathematics teachers.

'Recent Advances in Catalytic Polymerization'' will be the subject of a special two-week summer program at Massachusetts Institute of Technology, Cambridge, Mass., July 17-27, 1956.

A five-day conference on the management of industrial research will be held June 11-15, 1956, under the auspices of Columbia University on its campus in Harriman, N. Y.

"Career Opportunities vs. the Shortage of Scientific Personnel in Industry," by Theodore S. Hodgins, is the title of the leading article in a recent issue of Apparatus Digest, published by A. Daigger and Company, Chicago, Ill. It is the first in a series of articles comprising a symposium on opportunities in industrial research which will eventually be reprinted in booklet form.

Podbielniak Institute, Chicago, Ill., has announced a twoweek course in analysis of gases and vaporizable liquids with sessions beginning on September 24, November 5, and December 3, 1956, and January 21 and February 25, 1957, respectively.

Announce New Bibliography

The National Bureau of Standards has issued as Circular 566 a 1528-page "Bibliography of Solid Adsorbents, 1943 to 1953," by Victor R. Deitz, containing about twice as many references as are found in "Bibliography of Solid Adsorbents, 1900 to 1942." Copies are available at \$8.75 each from the Government Printing Office, Washington 25, D. C.