

# Jaocs News Feature

## Further developments in crude oil processing

K. KLEIN and L.S. CRAUER, De Laval Separator Co.,  
Poughkeepsie, New York 12602

*The processing conditions for obtaining high oil quality and yield in continuous caustic refining of the following crude oils are given: tallow, palm, fish, rapeseed, and sunflower. pH Monitoring of reaction mixture assures proper caustic dosage. pH Set point range for various crudes is noted.*

### INTRODUCTION

Regardless of availability of crude oil, proper processing conditions in caustic refining of each type of crude will be an important factor in determining the price and profitability of the finished oil product. Much data have been published on the proper procedures to use in handling large volumes of crude oils, such as soya and cotton. It is the small volumes of crude oils, i.e. tallow, sunflower, palm, which will be the subject of this discussion.

All types of crude oil can be refined in the continuous hermetic system, as shown in the typical flow sheet of Figure 1. This system can be designed with reagent additive systems and a degree of mixing flexibility to meet

any specific processing requirement.

### TALLOW

Tallow is a solid animal fat, extracted from the tissues and fatty deposits of sheep and beef. The fat source in the tissue and type of rendering method affects the rendered tallow quality and classification, that is edible or inedible.

Frequently, edible tallows are of sufficient purity, as a result of the type of rendering system utilized, to be used directly in food products. However, if the edible tallow has a free fatty acid (FFA) range 0.3-1% or contains trace quantities of collagens and proteinaceous matter, caustic refining is required to ensure acceptable stability.

Inedible tallows generally are high in FFA content (3-8%), are off-grade in color, and contain considerable soluble and insoluble impurities. To meet specifications for industrial uses, this type tallow is refined partially to reduce the FFA, to improve color, and to remove the extraneous matter.

Experimentation has shown that

frequently the best processing conditions for both classifications of tallow for high yield and quality are: preheating of the crude tallow 180-200 F and mixing intimately at low speed with theoretical quantity to 0.05% excess of the proper caustic treat, followed immediately by centrifugation. Under these conditions, refined oil yields may be in the range as noted in Table I.

These data show the highest yields are obtained with use of 24-30°Be' caustic treatment. The amount of excess is critical and must be closely controlled. If the amount of caustic used increases from 0.03 to 0.1% excess, there is a noticeable increase in neutral oil loss. Tallow readily saponifies with any excess of caustic present. As saponification occurs, more neutral oil also becomes entrained in the emulsified soap phase.

### PALM

The increased world supply and competitive pricing of palm oil has made this crude oil import more readily available to the domestic market. The crude varies in FFA content and color, depending upon source, harvesting methods, and shipping facilities. Malaysian palm oil averages 3-5% FFA, African palm oils frequently are 8-10% FFA.

In continuous caustic refining of this crude palm oil in a hermetic system, these conditions have produced good refined oil yields and quality: preheat crude to 130-160 F; neutralize with just sufficient quantity of caustic and mix to complete the chemical reaction; heat to 180-190 F to floc the soap phase; and immediately centrifuge.

The °Be' of the caustic used is dependent upon crude palm oil source, ultimate use, and color requirements. With 3-4% FFA crudes, 11-12°Be' caustic frequently gives the most favorable results. Refining factor is 1.6 or less. With oxidized color and high FFA, 24-30°Be' caustic is used. To

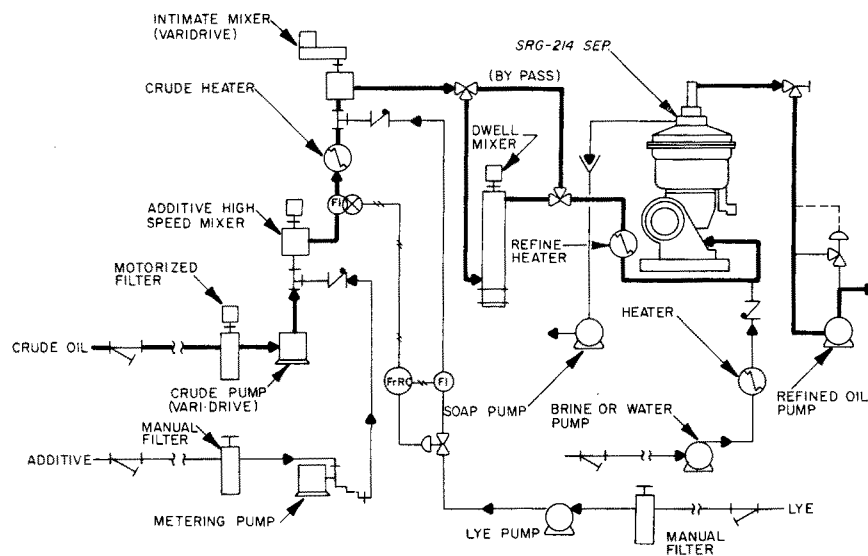


FIG. 1. Continuous caustic refining of crude oils.

TABLE I  
Tallow Refined Oil Yields

Type	NaOH ° Be'	Percent FFA		Percent refining loss	Refining factor <sup>a</sup>
		Crude	Refined		
Edible	24	0.8	0.03	0.9	1.17
Inedible	18	3.0	1.5	2.2	1.51
	20	3.0	1.5	2.1	1.43
	30	6.0	1.5	5.5	1.22
	30	4.3	0.5	4.7	1.24
	30	3.0	1.5	1.9	1.27

<sup>a</sup>Refining factor: ratio of % refining loss/% free fatty acid (FFA) removed.

minimize emulsification with these high strength caustics, 10% salt solution is added at the inlet to the centrifuge. For example, on a crude with high FFA, refining factor is 2.1 with use of 24° Be' caustic. Addition of 3% of a 10% salt solution just prior to centrifugation reduced the refining factor to 1.7.

### FISH OIL

As a direct result of pollution abatement, large schools of fish again are returning to the Gulf and East Coast waters of the U.S., thus increasing the availability of fish oils.

Typical refining factors or the ratio of refining loss/FFA of the crude as oleic are 1.6 or less. It has been found that, with the use of lower strength caustic, that is less than 20° Be', there is a tendency to make 3 and 4 phase soaps. This increases emulsification tendencies and also yields refined oils of high residual soap content.

### RAPSEED

Rapeseed is grown extensively at northern latitudes around the world as a seed crop for oil and meal source. Typical Canadian rapeseed will analyze: 0.7-0.8% FFA, 0.6-0.9% phos-

TABLE II  
Fish: Significant Loss Data

Type	H <sub>3</sub> PO <sub>4</sub> dosage ppm	NaOH ° Be'	Percent FFA		Percent refining loss	Refining factor <sup>a</sup>
			Crude	Refined		
Anchovy	500	24	1.6	0.03	1.9	1.2
Menhaden	500	24	1.8	0.03	2.8	1.5

<sup>a</sup>Ratio refining loss/free fatty acid (FFA) crude as oleic.

TABLE III  
Sunflower Refining Processing Conditions

Pretreat		Deg F		Percent refining loss	Wax vacuum dried oil, ppm
Agent	ppm	Mix	Centrifuge		
None		75	140	3.0	390
H <sub>3</sub> PO <sub>4</sub>	500	75	75	2.7	140
(NaPO <sub>3</sub> ) <sub>6</sub>	500	75	75	2.7	10-100

Due to the pressing operation, crude fish oils containing both soluble and insoluble matter and up to 2% FFA content. To obtain an acceptable finished oil product, these crude fish oils require pretreatment, prior to the refining stage, to ensure removal of the fish solubles.

Processing conditions have been found to be as follows: (A) preheat the crude oil to 160 F; (B) treat with 500-1000 ppm phosphoric acid, giving intimate contact to ensure good dispersion; (C) caustic refine with 20-24° Be' caustic, and (D) heat to 180 F, prior to centrifugation.

Table II gives significant loss data.

phatides, and 1.3-1.6% theoretical loss. To obtain high yields and oil quality in caustic refining of these Canadian crudes, it is advantageous to pretreat with phosphoric acid to break the phosphatidic linkage and ensure complete removal in the refining stage.

Typical processing conditions for such crude rapeseed oils are: (A) pretreatment with 500-1000 ppm phosphoric acid; (B) caustic treating with 0.1-0.15% excess as 16° Be'; (C) mixing, including intimate contacting plus hold time to complete the chemical reaction with the phosphatides; and (D) heating to 165-170 F to coagulate the soap, prior to centrifugation.

Typical neutral oil loss in the soap phase varies from 0.4-0.6% over the theoretical loss value. If the crude oil has a theoretical loss of 1.4%, the plant refining loss may be in range of 2% or less, with a finished oil of less than 50 ppm soap content. If this same crude rapeseed oil is not given the phosphoric acid pretreat, the refining loss may exceed 2% value. Both the soap and phosphorus content of the refined oil also may exceed the acceptable edible oil limits.

### SUNFLOWER

Since the commercial introduction of the high oil content sunflower seed in the U.S. in 1967, the annual crop acreage has increased many fold, especially in the Minnesota and Dakota areas (1). When the agronomists resolve the problems of insect infestation, plant disease, maturation time, and harvesting techniques, the acreage and availability will increase even more dramatically.

Sunflower oil is a high quality oil, used in cooking and salad oils, and also may be used in shortenings and margarines.

Today, the crude sunflower oil that is domestically available contains a small wax fraction from a trace to 1.0%, phosphatides (0.4-1%), and FFA (to 1.5%). The amount of wax esters present varies as a result of the dehulling operation. This wax fraction is not removed completely by conventional refining methods. As a result, there is a cloud in the chilled finished oil.

A study of this problem has shown that the amount of residual wax esters in the vacuum dried oil can be reduced by changing the refining processing conditions (Table III).

Pretreatment of the crude sunflower oil with either phosphoric acid or a phosphate salt is advantageous. Sodium pyrophosphate or hexametaphosphate appears preferable to the phosphoric acid. Dosage is in range of 500 ppm.

Temperature is 70-80 F for mixing of the pretreat agent, caustic addition, and centrifugation. If the temperature of separation is increased, the amount of wax esters increases in the refined oil.

With these process parameters as described, a typical crude sunflower oil of 1.2% FFA and 2.1% theoretical loss was refined with plant loss of 2.7%.

The phosphate salt pretreatment and low temperature processing reduced the wax fraction analytically to 10-100 ppm.

In the conventional cold test, this oil is clear with only a trace of wax fraction evident.

TABLE IV

pH of Reaction Mixture  
to Refining Separator

Type of crude oil	pH Set point
Cotton	10.8
Corn	10.6
Soya (nondegummed)	10.5-10.8
Soya (degummed)	10.3-10.4
Peanut	10.4-10.6
Palm	10.2-10.4
Palm kernel	9.8
Coconut	9.6- 9.8
Tallow	9.8-10.0

The pretreatment also helped to increase yields.

### PH MONITORING

pH Monitoring of the reaction mixture in refining is a processing aid (2), which ensures proper caustic treat and minimizes excess neutral oil entrainment, saponification, and emulsification.

Each oil and process have a specific pH of reaction mixture, which gives the best refined oil quality to meet finished product requirements. This pH reading is made on the reaction mixture when it is being fed to the refining separator to ensure that the chemical reaction is complete.

The pH may be monitored manually by the plant operator or automatically. A small sample of the reaction mixture is collected, diluted with 2 parts water, and the pH read. pH Set points in crude oil refining have been observed and appear in Table IV. These data cover the most common oils processed in the U.S. today. It should be noted that most oleic oils, containing phosphatides, are in the pH range of 10.4-10.8. Lauric oils are in range pH 9.6-9.8.

At any plant location, the pH set point may be established for optimal refining conditions on any type crude. By constant monitoring to this value, over treatment and under refining can be minimized.

### REFERENCES

1. Robertson, J.A., JAOCS 49:230 (1972).
2. Crauer, L.S., "Special Techniques to Optimize Vegetable Oil Refining," ISF Congress, Goteborg, Sweden, June 1972.

[Received August 26, 1973]

Looking for a job? Try  
the Classified Ads

## Michael Hein named vice president of Capital City Products division



The appointment of Michael Hein as vice president of the Chemical Specialties Division was announced by Richard E. Helland, president of Capital City Products Company, Division of Stokely-Van Camp, Inc.

Hein assumed responsibility for all marketing, sales, and processing arrangements in April. A graduate of the Technical University in Graz, Austria, and The Ohio State University, he joined the Company in 1957 as a chemical engineer. He has since served as refinery superintendent; manager, New Products Division; sales manager, Sterotex and Chemical Divisions; sales manager, Industrial Products Division; and, most recently, as vice president of production development.

He is a member of the American Oil Chemists' Society, the American Chemical Society, the Fatty Acid Producers Council, and the Institute of Food Technologists. ■

## Brandau named associate director, technical services

Robert P. Brandau has been named associate director, technical services, for the Chemical Specialties Division of Henkel, Inc., in Hoboken, N.J. He will be responsible for the activities of the company's recently expanded service laboratories concerned with chem-

ical specialties for the cosmetic, detergent, plastics, and paint industries.

Brandau joined Henkel's cosmetic technical service staff in 1964. He received degrees from the University of Chicago. ■

# BOMBS and PRESSURE VESSELS

Parr offers a wide selection of bombs and pressure vessels for applying heat, pressure and agitation to any chemical reaction or physical test system. These are made in several different styles and in all principal corrosion resistant alloys. Capacities range from 15 to 2000 ml. with working pressure limits of 1000, 3000, 7500 psig. and higher. Valves, thermowells, dip tubes and other attachments can be provided.

*Ask for our  
Pressure Vessel Catalog.*



**PARR INSTRUMENT COMPANY**  
211 Fifty-Third St. Moline, Ill. 61265  
Telephone (309) 762-7716