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The Physical Refining Process

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ABSTRACT

This paper deals with influences and optimizing of changing process conditions for physical refining of palm oil. These process variables are temperature, pressure, residence time, fluid flow and stripping steam to oil ratio. These parameters influence not only finished oil quality, oil yield, energy consumption and running costs, but also content and yield of natural stabilizers like tocopherols or color compounds like carotenes, and last, but not least, environmental load of waste water and exhaust air as studied under industrial plant conditions. With the right pretreatment process physical refining of palm oil is not only much more economical than chemical refining in connection with stripping steam deodorization, but also causes much less pollution by waste water and exhaust air.

Under all these aspects the performance of continuously operated industrial plants now in use for physical refining of palm oil is being examined. Because of the water solubility of the low-boiling thermal degradation products, the effluents of nearly all installations must be specially treated to fulfill today's legal requirements on BOD and on COD as well as on oil and grease content. The only exception is a new counter-current two-step film type physical refining process in connection with a combined sophisticated steam ejector vacuum and two-step exhaust air washing system, with which, without any air pollution, COD values of <50 for waste water are possible.

For best oil quality deacidification should be done with pressure drop of less than 1 torr at 2 to 3 torr tap pressure at 260 C working temperature with residence times of 10 min and counter-current exchange efficiency of 6 to 8 theoretical plates.

PHYSICAL REFINING-DISTILLATION PROCESS

Physical refining of vegetable oils is a distillation process (1-3) in which, under low absolute pressures of 2 to 10 torr and high temperatures of 240 to 270, the accompanying lower boiling compounds are distilled off from the fatty acid triglycerides—the main components of the oils—by using unsaturated open steam as the effective stripping agent (1). The byproducts consist mainly of free fatty acids and such degradation products as hydrocarbons, methylketones and aldehydes. The temperature-pressure relationship for oils and some of the main byproducts are shown in Figure 1. According to our comparative evaluation of these data the mixture stearic acid-tristearin can be considered a good model for calculating necessary separation power in physical refining and distillative deodorization of edible oils (1).

SPECIAL SEPARATION CONDITIONS OF PHYSICAL REFINING FOR PALM OIL

Palm oil and other vegetable oils contain, beside fatty acid triglycerides, a large number of other constituents in low concentrations, some only in ppm or even ppb. Among these are free fatty acids as main components in concentrations up to several percent. In much smaller contents are pigments, stabilizers, various flavor and odor compounds, as well as products formed by autoxidative and/or heat decomposition of peroxides, pigments and natural stabilizer compounds. According to newer published results Malaysian crude palm oil on average can be characterized by the data in Table I (4-7).

In physical refining not only are the mentioned undesired low-boiling byproducts like free fatty acids, aldehydes, ketones and hydrocarbons stripped off by open steam, but parts of higher-boiling special stabilizer compounds like tocopherols and sterols are carried over into the distillate phase. In such mixtures tocopherol compounds with molecular weights of only 421 will act against triglycerides of C_{16}/C_{18}-fatty acids with molecular weights of 800 to 850, causing separation factors α between 50 and 200. By the necessary stripping steam amount of normally 1.5 to 2.5% used in this process at temperatures of above 250 C and pressures between 2 and 8 torr, not only the low boilers but also such stabilizer compounds will go more or less into the distillate.

INFLUENCE OF METALS IN RELATION TO WORKING CONDITIONS ON NATURAL STABILIZER CONTENT (8-23)

According to the data in Table II (4), the tocopherol loss in installations for physical refining can be up to 44.5%, while in steam deodorization after chemical refining these losses are below 10%. The reason for the different behavior in processing when using practically the same technical installations has to be seen in decisive lower values for temperature, residence time and open stripping steam amount. In nearly all physical refining installations the product hold-up times at temperatures above 250 C and pressures between 2 and 8 torr with open steam amounts of 2 to 3% will normally be kept in the range of 2 to 3 hours. For steam deodorization in these installations at the same temperature and pressure working conditions, residence times of only 1 to 2 hours are needed, with open steam consumptions of 1 to 2% at lower temperature profiles.

These results are in full agreement with our open observations (24) that the tocopherol loss can be markedly diminished by using a counter-current film process. The installation is working at pressures of 2 torr, temperatures not higher than 260 C with residence times above 230 C of below 20 min including heating and cooling periods and open steam amounts of less than 0.6% of oil throughput. The layout has to be made so that under working conditions the pressure drop between top and bottom of the two or three film columns is below 1 torr. Because of the high

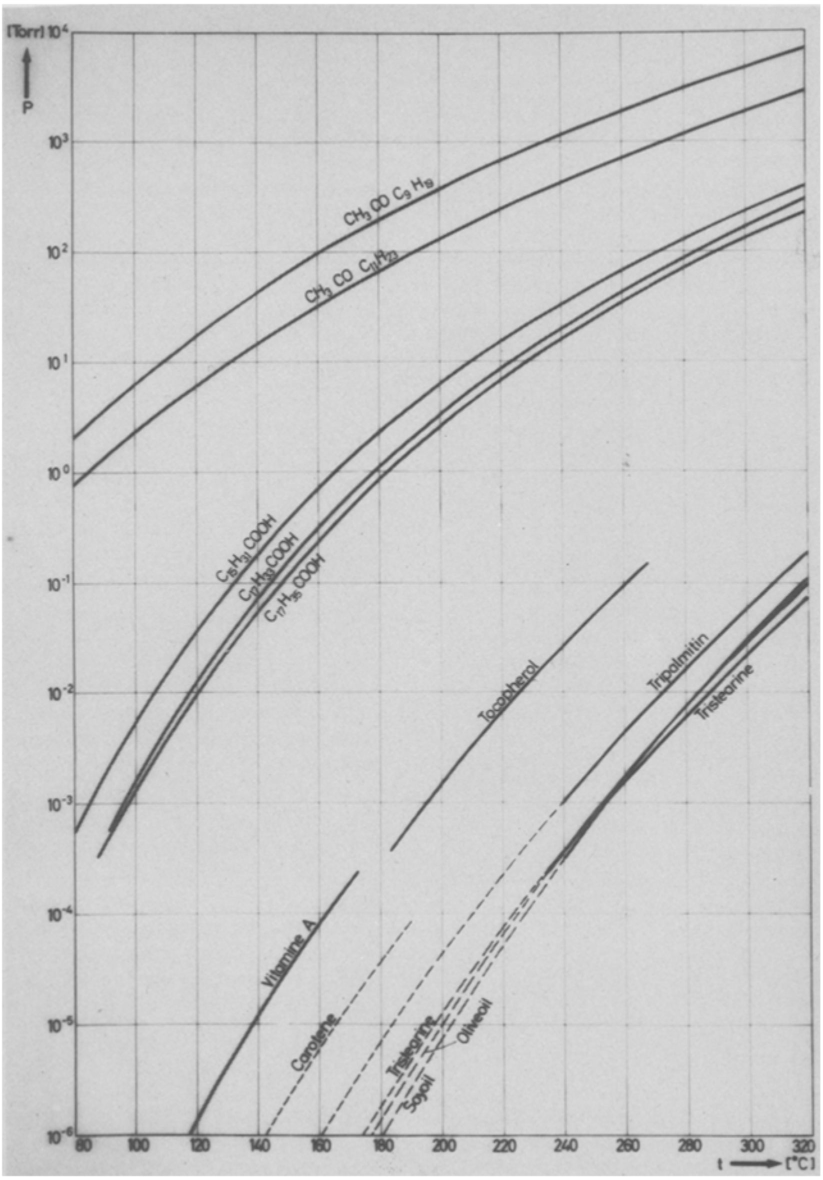

FIG. 1. Relationship between vapor pressure in torr as ordinate and temperature in C as abscissa for vegetable oils, triglycerides and some of the typical accompanying substances.

TABLE I

Characteristics of Malaysian Crude Palm Oil

Identity	Dimension	Mean value
FFA	Weight-%	3.15
Carotene	mg/kg	600.00
Tocopherols	mg/kg	800.00
Sterols	mg/kg	500.00
Iron	mg/kg	4
Copper	mg/kg	0.04
Moisture	Weight-%	0.20
Impurities	Weight-%	0.05
Relative density	50 C/H_2O, 25 C	0.8927
Refractive index	$n_D{}^{50}$	1.4553
Saponification value	mg-KOH/g oil	195.7
Unsaponifiable content	Weight-%	0.51
Iodine value	(Wijs)	52.9
Slip point	°C	34.2
Anisid. value	(E_{350})	4.6
Peroxide value	m-eq/kg	3.9

heat sensitivity of tocopherol and similar compounds the entrance temperature of the used high-temperature heating medium is not allowed to exceed 270 C. My first countercurrent single film column system has been in its pilot phase since 1981 at Walter Rau in Hilter/Germany (27,28). Even at this system's much higher values for temperature, pressure, hold-up time and open steam conditions, the finished palm oil has 1.5 to 2 times higher chemical stability than chemically deacidified and deodorized palm oil as shown by the Schaal oven test (Fig. 2).

As the oil stability and its tocopherol content will be strongly influenced by the iron content of the raw oil, iron concentration, which is almost completely derived from wear in rotating machinery in the oil mill, should be as low as possible. Today, according to measurements by PORIM, this value will be in the range of 5 mg/kg. Much lower concentrations, down to 1 mg/kg, are possible when stainless steel is used at specific areas in the process. As the iron content, which is formed by wear, is partly particular, it can be reduced by filtration or by the use of magnetic pipeline inserts. When this has not been done, the iron will be soluted by the free fatty acid content of the oil. In this ionized form iron becomes more reactive in destroying tocopherol compounds, as can be seen by the data in Table III (4). Therefore, when using the physical refining process in combination with degumming and bleaching (29-31), it is necessary that not only the free fatty acid content of finished oils leaving the plants is far below 0.05%, but also that the iron content of the incoming degummed and bleached raw oils should be below 1 mg/kg.

TABLE II

Tocopherol Loss in Process Steps of Chemical and Physical Refining

In chemical refining

	Crude	After neutralization and bleaching		After deodorization	
Inst.	mg/kg	mg/kg	loss, %	mg/kg	loss, %
A	796	744	6.5	673	9.5
B	906	698	23.0	652	6.6

In physical refining

	Crude	After degumming and bleaching		After physical refining	
Inst.	mg/kg	mg/kg	loss, %	mg/kg	loss, %
C	841	841	0.6	554	34.1
D	1103	861	22.0	478	44.5
E	811	727	10.4	548	24.6
F	892	838	6.1	503	40.0

INFLUENCE OF TEMPERATURE AND RESIDENCE TIME ON NATURAL COLOR COMPOUNDS LIKE CAROTENES (32-35)

Another byproduct, the carotenes, which are responsible for the deep orange red color of crude palm oil, is even more heat sensitive than tocopherol. According to time and temperature dependence of carotene stability (shown in Fig. 3) which were measured by M. Loncin (32) under physical refining conditions, all carotenes will be thermally destroyed. Their cracking products are comparable to free fatty acids as low boilers. The only problem for their full separation is that all cracking particles have the opportunity to come quickly to the liquid surface during steam stripping.

TABLE III

Influence of Iron Content on Tocopherol and Carotene Losses in Crude Palm Oil during Storage

Storage condition		PV	Tocopherol		Carotene	
		mg/kg	mg/kg	loss, %	mg/kg	loss, %
Fresh	A	1.2	761	0	635	0
	B	1.5	742	0	638	0
20 days at 60 C	A	22.3	605	20.5	544	14.3
	B	36.8	17	97.7	85	86.7
26 days at at 60 C	A	33.1	476	37.5	416	34.5
	B	44.5	10	98.7	19	97.0

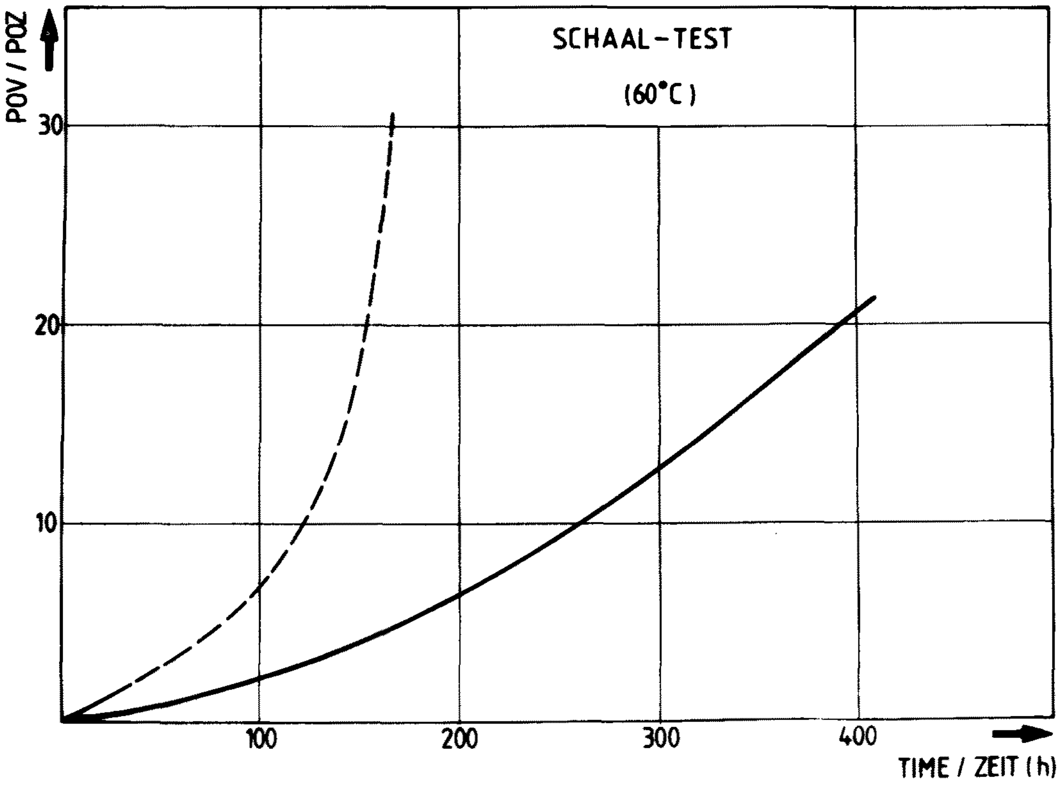

FIG. 2. Relation between peroxide value as ordinate and time in hr as abscissa at 60 C (Schaal Test) for finished oils physically refined in a one-step film installation (full-line) and chemically refined with further distillative deodorization (dotted line).

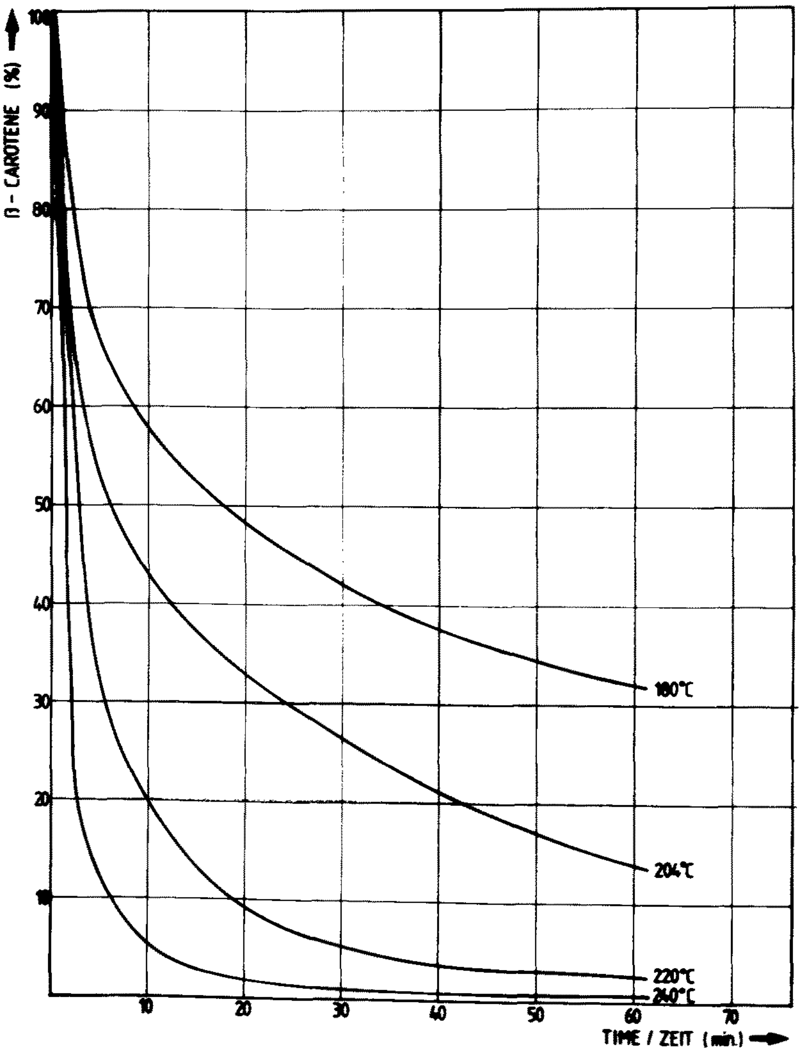

FIG. 3. Relation between residual β-carotene content in % as ordinate and reaction time in minutes as abscissa with the temperature in C as parameter, according to M. Loncin.

According to our observations these conditions are ideally realized in counter-current film installations, as shown in Figure 4, working at low pressures and pressure drops with totally external heat (24-26).

DIFFERENT WORKING PRINCIPLES OF INDUSTRIAL INSTALLATIONS FOR PHYSICAL REFINING AND DISTILLATIVE DEODORIZATION

In principle it is true for all installations (Fig. 5) that the lower the working temperature and pressure can be kept, the more successful will be the deacidification. For large-scale industrial equipment the pressure is always between 1 and 10 torr, whereas the actual deacidification process will mostly be effected in the range of 4 torr in order to optimize the operating costs. However, this low pressure will only be fully utilized for the separation of undesired oil accompanying substances when during the whole contact time with the ascending steam, mostly in the form of bubbles, the oil is kept practically at the same boiling pressure and temperature in this steam stripping treatment.

The batch deacidizer shown as A in Figure 5 operates by adding roughly half of the liquid level height to the working pressure, as the steam bubbles rising in and penetrating the liquid are in mean subjected to this pressure. In B, with forced circulation by the principle of gaslift pumps, all oil particles will be dispersed superfinely when discharging out of the pump due to impinging effect of the live steam. Therefore, there are ideal conditions at this discharge point for the adjustment of the vapor-liquid-phase-equilibrium to the prevailing pressure and temperature conditions and steam feed rate. Results depend not only on the equilibrium relations but also on the circulation flow which depends in turn on the amount of liquid and shape of the vessel. In these plants the amount of fatty acid carried over per kg of steam used decreases with the falling fatty acid concentration during deacidification. In this regard continuously operating multistage counterflow plants of low pressure drop to C and E, offering considerable advantages as the concentration of fatty acid and other byproducts in the upgoing vapor stream depend only on the constant and higher concentration of these components in the feed.

If a tray column of type C with bubble caps or valves is used (36-38), then the oil flowing out of the bottom is not under the top pressure but under a pressure which will be increased by the pressure drop of the trays. With the four to five trays usually required this means a doubling to tripling of the working pressure. The live steam consumption required for a sufficient deodorizing then results from the

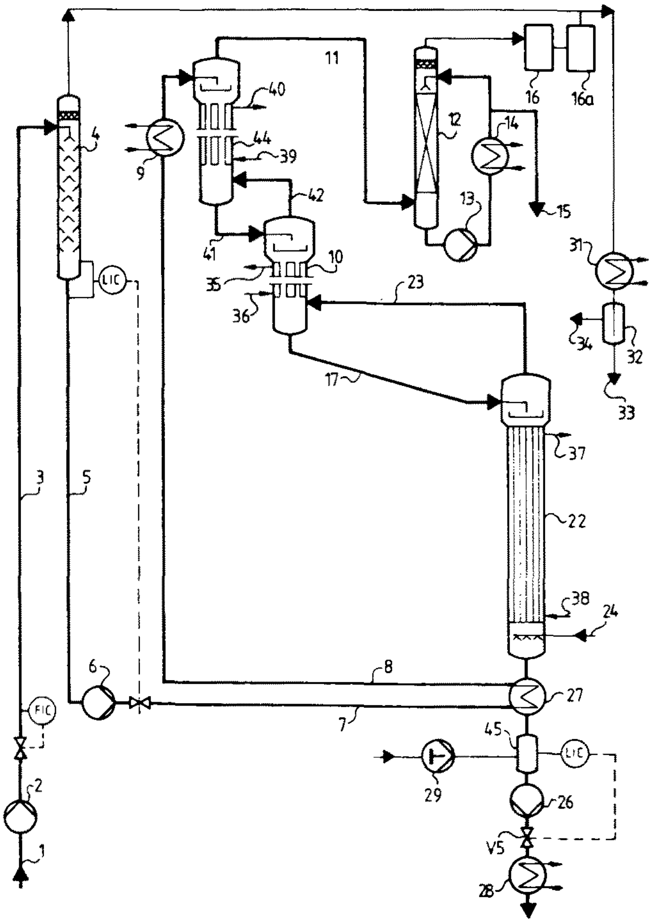

FIG. 4. Flowsheet of ATT-two step counter-current film process in which both exchange stages together with the spray condenser section are combined in one column.

higher pressure at the respective bottom working temperature. An example is shown in Figure 6, the Wurster and Sanger installation (36).

Today, most industrial deacidizers are operating according to the principle shown as D of Figure 5. All these quite different constructions operating both semi- and fully-continuously and, to A and B, discontinuously, are more or less one-stage plants. In all these cases the live steam used is only equilibrating once with the oil of a certain fatty acid concentration. As long as the same final acid content is aimed at, all plants of this type in principle require the same quantity of live steam for comparable pressure and temperature conditions, irrespective of their method of operation. The processes followed one after another with one-stage discontinuous batch apparatus are done simultaneously with fully-continuous equipment. It must be arranged by means of appropriate constructive measures that the back-mixing is as low as possible. With this goal, a long way for the oil would be of advantage. It is of no interest for the deacidizing effect how to obtain this long way, as working pressure and temperature will not be influenced. Therefore, there are big differences regarding the outer appearance of plants of this type. Examples of this principle are given in Figures 7-10 showing the installations of Gianazza (39), DeSmet (40), Alfa-Laval (41) and Kirchfeld (42).

Under most adiabatic conditions the ideal counter-flow exchange as multistage process can be materialized in a trickling or film-type column as seen in E of Figure 5. Hereby, the steam entering into the bottom gets fully saturated with fatty acids according to the feed concentration and temperature, when passing through the deacidifer with the liquid pouring down (1). Such counter-current two- or three-step film installations were shown in Figure 4. The real reaction time between ascending vapor stream and falling oil film is, according to the data of Table IV, less than 15 sec with Reynolds' numbers in the film between 223 and 277 and pressure drops between top and bottom of < torr.

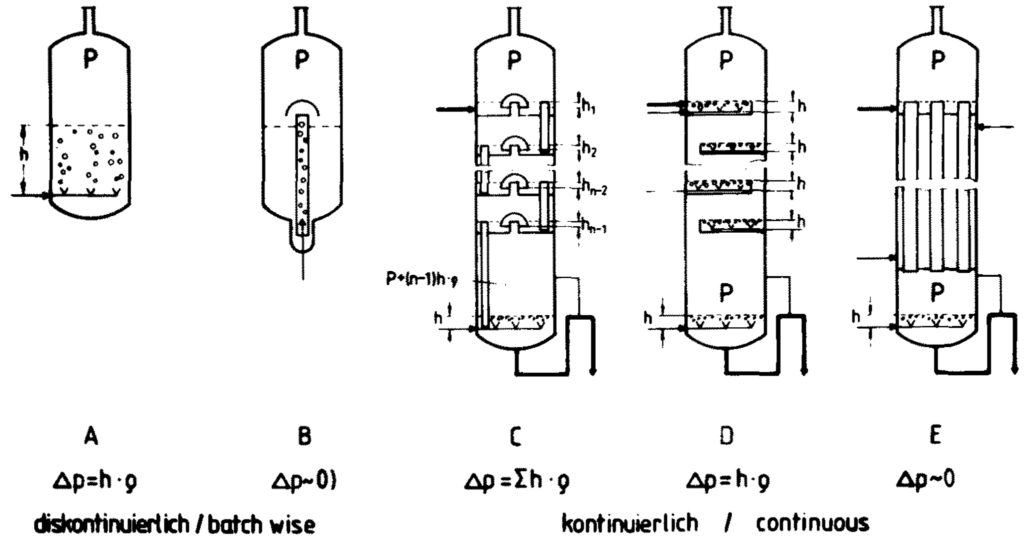

FIG. 5. Schematic representation of the different possibilities for refining with regard to the pressure drop for the stripping steam used. A, simple batch installation; B, batch installation with mammoth pump; C, continuously working bubble tray column; D, continuously working column with overflow plates, and E, continuously working counter-current film type column of low pressure drop.

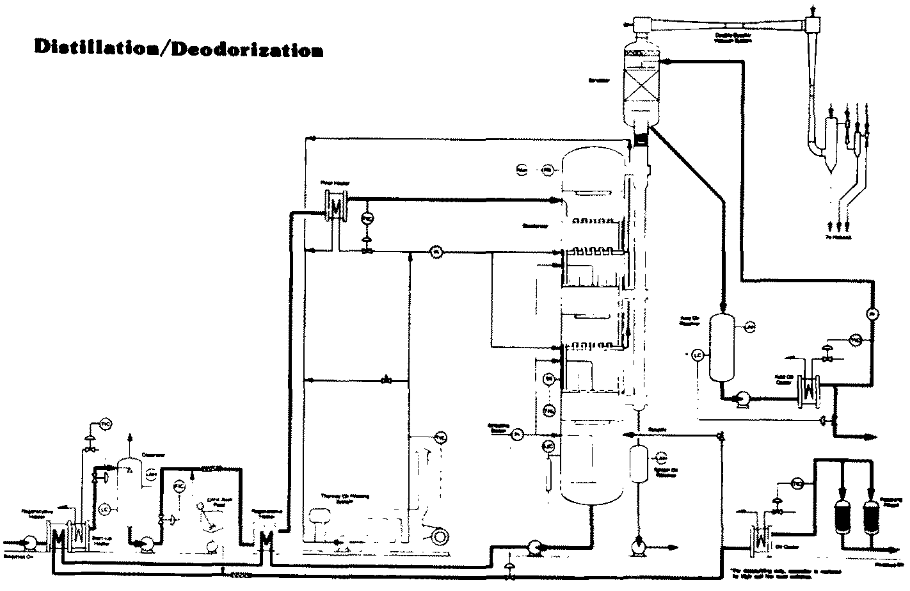

FIG. 6. Schematic diagram of the main parts of a Wurster and Sanger installation.

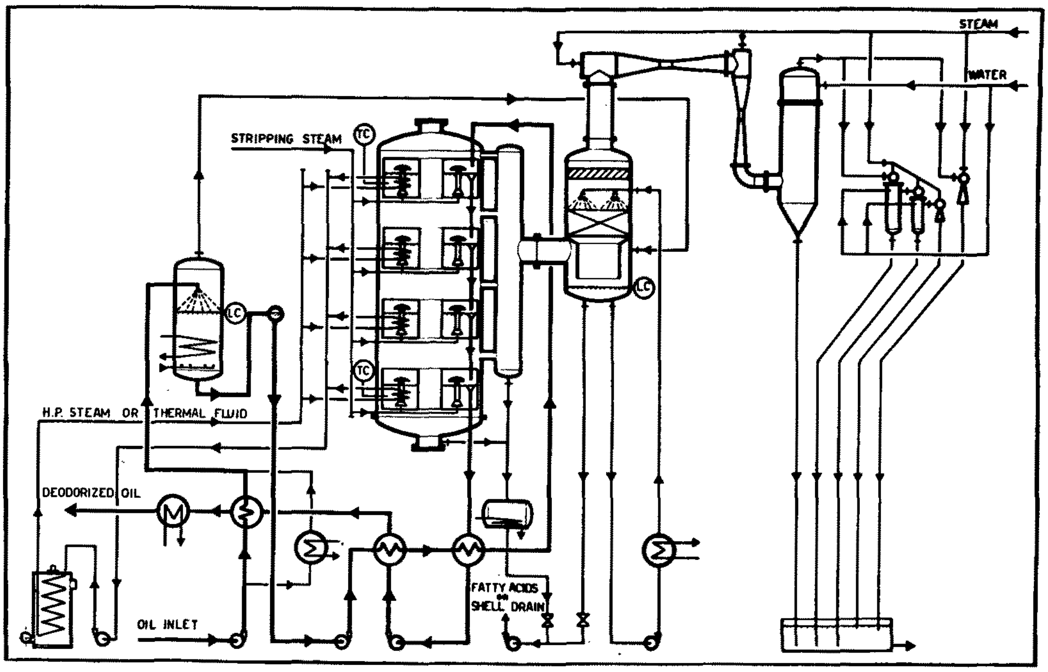

FIG. 7. Flowsheet of a Gianazza-Dovert-T-Phy installation.

ANALYTICAL TREATMENT OF PHYSICAL REFINING (1,43,44)

The two extreme cases of the so-called discontinuous single-stage operation and the fully-continuous multistage counter-current process have been subjected to an analytical treatment. The result is shown in the following equations:

$$S_{con} = (z_2 - z_1)\,\frac{M_{H_2O}}{M_O}\cdot A\left[\frac{100}{z_2} - B\right]$$

$$S_{dis} = \frac{M_{H_2O}}{M_O}\cdot A\left[230{,}26 \log\frac{z_2}{z_1} - B\cdot(z_2 - z_1)\right]$$

$$\frac{S_{con}}{S_{dis}} = \frac{\frac{100}{z_2} - B}{\frac{230{,}26}{z_2 - z_1}\log\frac{z_2}{z_1} - B}$$

$$A = \frac{P_G - P_O}{P_{FA}} \qquad B = 1 - \frac{P_G - P_{FA}}{P_G - P_O}\cdot\frac{M_O}{M_{FA}}$$

S denotes the steam consumption for deacidizing 100 kg of oil, z_2 and z_1 the weight percent of fatty acid in incoming and out-flowing oil, M_{H_2O}, M_{FA} and M_O the molecular weights of water, fatty acid (FA) and oil (O) and finally P_G, P_{FA} and P_O the total, fatty acid and oil pressure at a

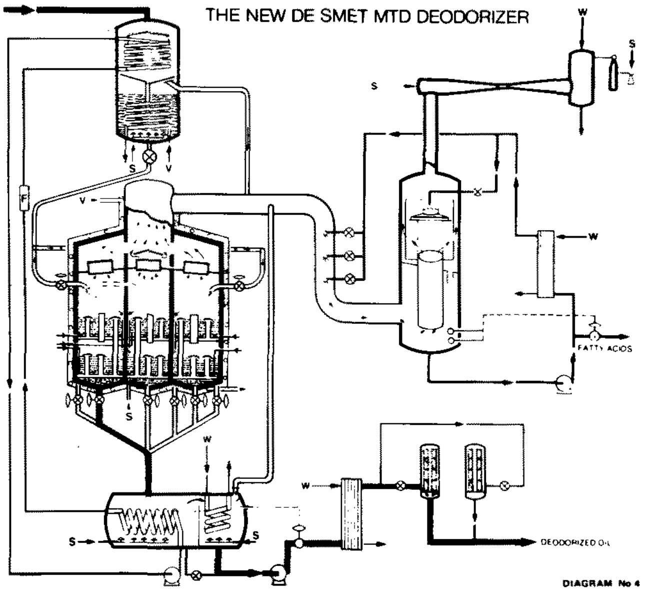

FIG. 8. Flowsheet of a DeSmet installation for steam refining and deodorizing.

TABLE IV

Flowing Data in Counter Current Film Deacidifying of 10,000 kg/hr Palm Oil with 5% FFA at 4 torr and 255/265 C

Pipe dimensions		Reynolds' numbers		Film thickness	Film res. time	Δp
ϕ,mm	length, m	vapor	oil film	mm	sec.	torr
2-film step installation						
108	4	1096	223	0.35	7.8	0.06
33	8	326	227	0.38	13.6	0.59
3-film step installation						
120	2	1508	223	0.35	3.9	0.02
84	3	656	255	0.37	5.4	0.03
33	7	327	277	0.38	11.9	0.56

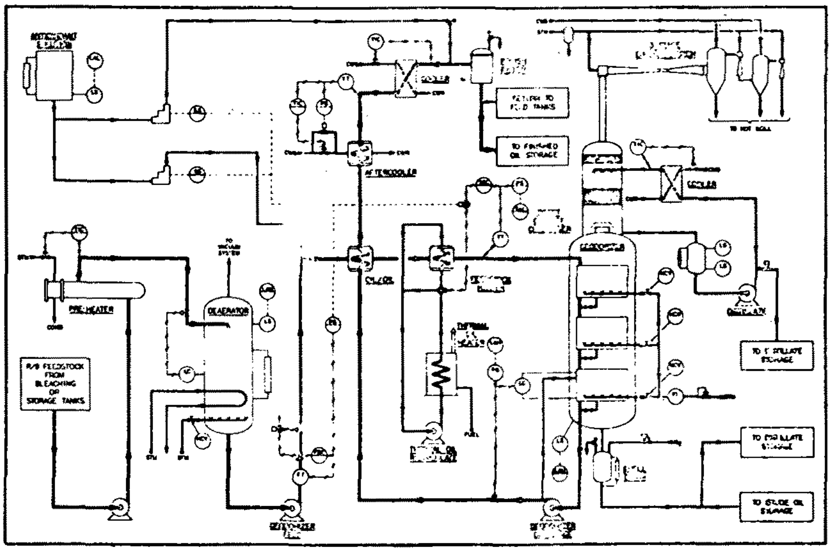

FIG. 9. Flowsheet of a Sullivan-Alfa Laval installation.

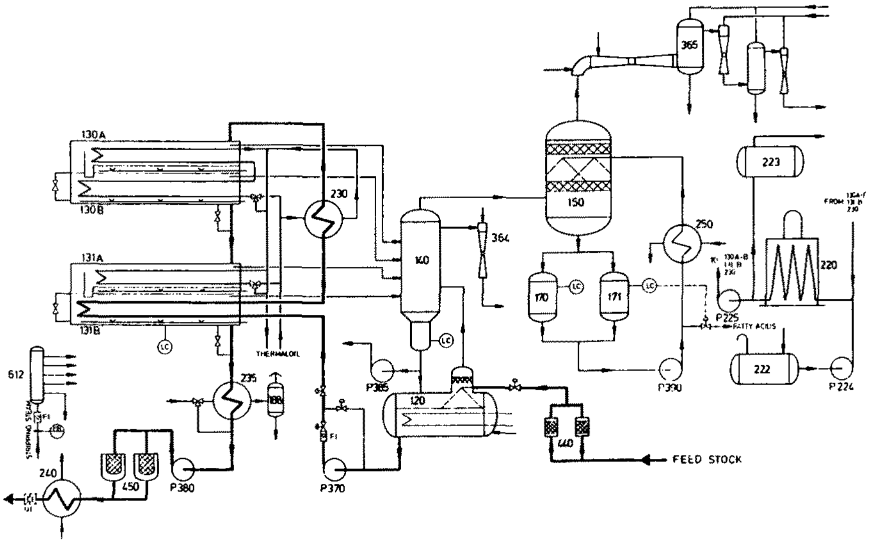

FIG. 10. Flowsheet of a Kirchfeld installation.

working temperature considered. For the calculation of the single-stage plants to A, B and D the vapor-liquid-phase-equilibrium-diagram of the selected working temperature solely is decisive. For continuous deacidification of C and E a multistage counter-flow process is required.

Figure 11 shows clearly the influence of the number of stages on the stripping steam requirements for deacidifying oils. This is shown by taking the fatty acid content of the incoming oil as ordinate, the stripping steam requirement as abscissa, and different plate numbers as well as temperatures as parameters. To deacidify oil from an acid content of 5% by weight to an acid content of 0.01% by weight at 10 torr and 240 C the stripping steam requirement for 100 kg of oil is 4.0 kg at five stages, 2.15 kg at 10 stages, and 1.2 kg with an infinite number of stages.

This graph also demonstrates the great influence of changes in temperature and pressure. Under operating conditions usually used in large-scale plants, with 5 torr and 240 or 260 C, the graph gives for five-scale operation 1.8 or 0.68 kg, for an infinite number of stages 0.38 or 0.12 kg, and for discontinuous single-stage operation as much as 3.7 or 1.47 kg stripping steam per 100 kg of oil. These data are maximum figures, since they refer to the separation of stearic acid from stearic acid triglycerides. In palm oil admixtures a considerable proportion of the substances which have to be separated have markedly higher vapor pressures than stearic acid.

ENERGY REQUIREMENTS

The decisive factors determining the overall energy requirements of the deacidification process are separate from the stripping steam requirement. These factors are principally the amounts of heat necessary to reach and maintain the required working temperature, to vaporize the unwanted light fractions, and to cover the heat losses thereby incurred. To heat the oil from 60 to 260 C required approximately 110,000 kcal/ton. Of this amount, it is possible using the continuous process to recover by heat exchange around 80,000 to 95,000 kcal/ton at economically viable costs by using a new special type of fully-welded plate heat exchanger (45). The remainder must be equalized by high-temperature heat on the raw oil side and by means of suitable cooling equipment on the finished oil side. To cover the heat losses and to supply the latent heat energy required for vaporizing the light fractions, a 200 ton-per-day plant would for example require an additional 6,000-10,000 kcal/ton. The total requirement for high-temperature heat thus comes to about 20,000 kcal/ton.

The assessment of performance of a deacidifying plant does not only consist of comparing the requirements per ton of such inputs as stripping steam, steam for the vacuum equipment, high-temperature heat, cooling water, and electrical energy for pumps, regulators and air-operated controls. Performance also has much to do with the quality and

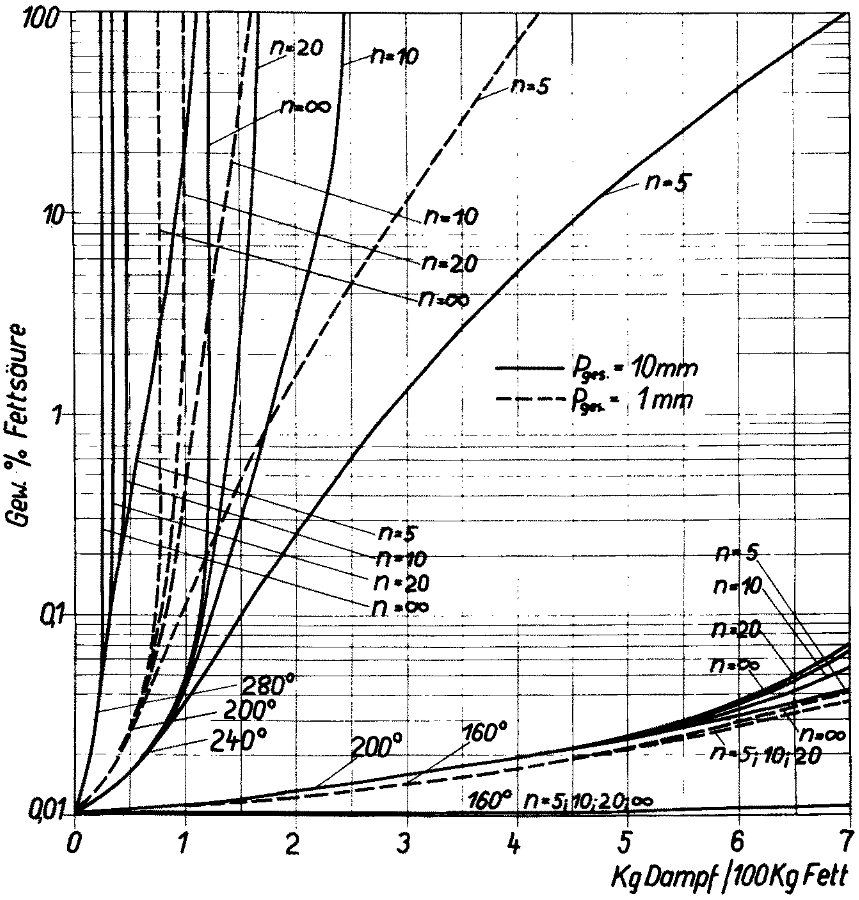

FIG. 11. Consumption of stripping steam in kg/100 kg of oil as ordinate in relation to the free fatty acid content (FFA) of the oil as: 1) full closed lines at pressures of 10 torr, temperatures of 160, 200, 240 and 280 C for exchange efficiencies of 5, 10, 20 and ∞ theoretical exchange units; and 2) dotted lines at pressures of 1 torr, temperatures of 160 and 200 C for the same exchange efficiencies as before.

yield of the products, and the degree of potential environmental problems caused by liquid and gaseous effluents and noise. In general the properties which determine oil quality are strongly dependent on the degree of thermal and oxidative stress to which the oil is subjected during its processing and storage. For comparable handling times, this stress is less with lower working temperature. For example, palm oil is generally processed currently at temperatures of about 260 C. Therefore, the temperature difference between the oil and the chosen heating medium is of special importance. To avoid any heat-induced damage to palm oil the input temperature of the heating medium used should in our experience not exceed 275 C.

The vapors of the substances removed by the stripping steam are condensed in a spray condenser column in almost every plant.

ENVIRONMENTAL PROBLEMS IN PHYSICAL REFINING

Loading of the vapors with low boilers from the oil leaving the spray condenser and sucked by the vacuum ejector corresponds to the partial pressure of the condensate at its feed temperature of 60-80 C. These vapors consist mainly of stripping steam. The loading of the plant's effluent with organic material is therefore a function of the amount of stripping steam used. Because of the extremely low open stripping steam demand of our counter-current physical refining film process, all today's legal demands on effluents and offgas with COD-values < 50 can easily be fulfilled with our new vacuum system shown in Figure 12.

As palm oil and all other vegetable oils are very sensitive to oxidation at the working temperatures necessarily used, the amount of seal area must be reduced to a minimum. In addition, at least all those flanges which are subjected to

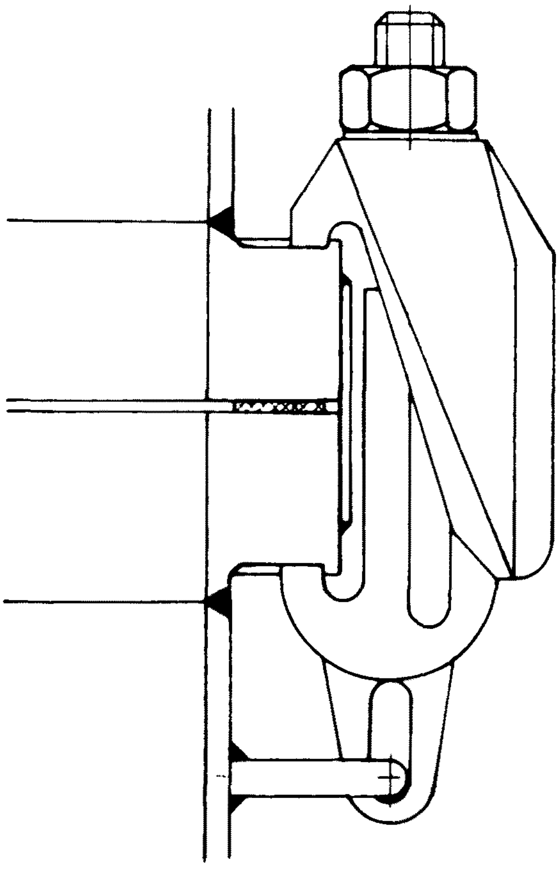

FIG. 13. Seal-welded flange joint with clamp-screws using a sheet strap.

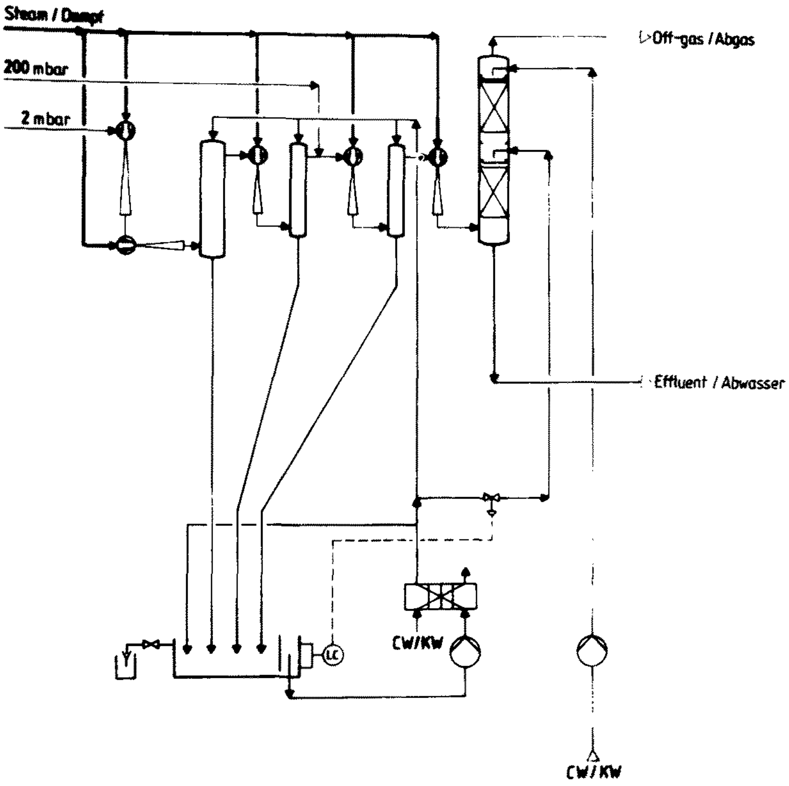

FIG. 12. ATT-vacuum system for getting at low energy consumption rate effluent with COD-values < 50.

TABLE V

Performance Data Comparison

Process	Wurster	De Smet	Alfa-Laval	Gianazza	Kirchfeld	ATT
Type to Fig. 9	C	D	D	D	D	E
Work-press. torr	3	2	5/6	2	3	2
Oil temperature C	250	250/255	275	260	260	255/265
Low press. steam kg/to	130	116	240	180	84	30
Stripping steam kg/to	10	?	30	30	13	6
High temp. heat kcal/to	25,000	?	30,000	30,000	39,000	20,000
Heat exchange kcal/to	90,000	?	77,000	80,000	68,000	98,000
Resid. time minutes	90	100	60	120	120	10

thermal stress should be designed so that they can be additionally sealed by welding. Figure 13 shows a clamp-screw design which has proved itself in many applications. A thin sheet of metal is laid around the outer seal of the flange and then welded to each half of the flange. If necessary this connection can easily be separated by using a cutting disc, and can later be reapplied.

EVALUATION OF DIFFERENT REFINING PROCESSES

Energy comparisons for the different installations discussed here are given in Table V and show that for counter-current film type systems the lowest demands for steam and high temperature heat are needed. The oil content in the distillates of installations for physical refining will normally be held between 8 and 12 weight percent. This leads to refining factors in the range of 1.1, compared to average values of 1.5% for chemical refining/deodorization plants. This means roughly a 2 weight percent higher oil yield. Because of the fully realized counterflow of the two phases under nearly ideal conditions in our film process we reach even better oil yields with oil levels in the distillate of below 5 weight percent. This corresponds to refining loss factors of 1.05%.

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