Internal Standard

An internal standard is obviously desirable as an alternative, or back-up, to quantification by standard additions, especially when an integration system is available. A method using n-decane as internal standard was supplied by Shell Research, Amsterdam, The Netherlands. Table II shows that n-decane could be used, but it has a long retention time (5.49 times that of hexane). Under our experimental conditions, octane (relative retention time 1.90) may be more suitable as an internal standard. The addition of an internal standard would involve similar precautions and difficulties as those discussed above (see Preparation of Standards). Because of limited funding, this aspect was not considered in detail.

Other Uses

To extend this technique to other liquid foodstuffs and to oil-soluble materials, such as lecithin, may be possible. In particular, solvents used to extract oleoresins, e.g., 1,2-dichlorethane, ethyl acetate and acetone, could be determined by a similar approach.

Scope of the Technique

While this paper has studied low concentrations of light petroleum, i.e., less than 0.5 mg kg^{-1}, other workers (16, 17) have used a similar experimental procedure to determine the much higher levels found in crude oil. Italian workers (16,17) claim a linear response of 5-20,000 mg kg^{-1}. The combination of our results with these indicates that a linear range is probably 0.05-20,000 mg kg^{-1} for determining residual solvents by the technique of direct injection.

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♣A Comparative Study of Batch and Continuous Refining of Cottonseed Oil in the Sudan

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ABSTRACT

This study is concerned with a comparison of some technical aspects regarding batch vs continuous refining (centrifugal alkali refining) of crude cottonseed oil. Implication of processing modes of operation were examined in light of their effect on the following performance criteria: (a) percentage of refining loss as a function of the initial crude-oil free fatty acid (FFA) content; (b) refined oil color as a function of initial crude-oil FFA; (c) caustic soda consumption as a function of initial crude-oil FFA; (d) bleachability characteristics of refined oil. The study shows, in quantitative terms, that continuous refining of cottonseed oil is more efficient in each of these performance criteria, particularly the percentage of refining loss.

INTRODUCTION

In recent years a trend has been growing toward replacing the traditional batch edible-oil refining techniques by what was believed to be superior continuous refining methods. Although the continuous methods proved to be more efficient on a commercial basis, little attention was paid toward quantifying this superiority (1-3).

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Kuroda et al. (4) reported satisfactory results for the commercial testing of a direct-fired, semicontinuous deodorizer (capacity 18 tons of oil per day), using refined cottonseed oil, for a period of more than 300 hr, including 200 hr of continuous operation. On the basis of laboratory tests, a technological scheme for the continuous adsorption purification of cottonseed oil was reported (5).

Figure 1 shows an outline of the 2 main processing schemes generally employed to produce edible oil: conventional (chemical) refining and physical refining.

In this study, a comparative analysis of batch and continuous conventional (chemical) oil processing operational plants is undertaken. The units, whose performance is investigated, are located at the Bittar Oil and Soap Factory in Khartoum North, Sudan. The emphasis of the study is on the heart of the processing scheme, the refining, washing and bleaching stages.

Alkali refining is practiced as a purifying treatment designed to remove free fatty acids (FFA), phosphatides, gums, coloring matter, insoluble matter and miscellaneous unsaponifiable materials. These impurities may be present in true solution or may be present as a colloidal suspension.
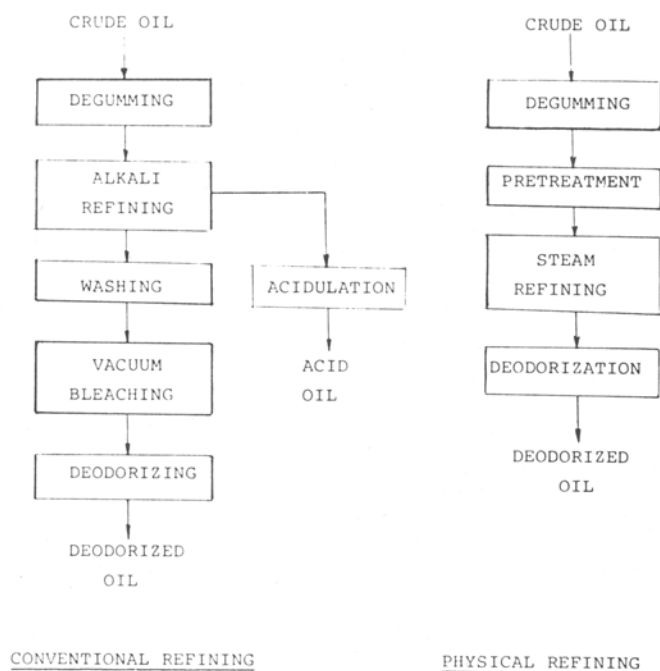
FIG. 1.

The primary objective of the bleaching operation is to remove oxidation products, pigments and entrained soaps. Although bleaching generally improves oil color, initial and aged flavor and oxidative stability, other, less obvious effects, have been reported in the literature (6,7).

Process Considerations

Figure 2 shows the flow diagram of the batch process used in this study. The batch refinery is a 45 T/D unit in actual commercial operation, stationed on 2 floors with the oil storage tanks, pumps and the bottom portions of the neutralizing, settling and washing vessels all housed on the ground floor. The baffled refining vessel (neutralizer) is made of mild steel and has a diameter of 2.8 m and an overall capacity of 23 tons of crude oil. It is equipped with a paddle mixer driven by a 15 ph induction motor running at the relatively low speed of 975 rpm. The actual operational speeds required for neutralization crude oil (30-50 rpm) are obtained by coupling the motor to a Craft variable-speed gear box. The heating coils of the neutralizing vessel are connected to a low-pressure steam supply (4-5 kg/cm^2). The vessel is equipped with a caustic soda dosing system in the form of a perforated tube running around the inner upper periphery of the vessel, which is connected through a cock valve to the caustic soda overhead feed tanks. Figure 3 shows a drawing of the crude-oil neutralization vessel.

The water-washing kettle is made of mild steel and has a capacity of 15 tons. The vessel is baffled and equipped with a stirrer of the paddle type that rotates at a constant speed of 35 rpm. Perforated circular pipes at the top of the vessel facilitate the dosing of hot water or caustic soda into the neutral oil.

We found that a batch of 15 tons of refined oil (with an average FFA content of 2.5%) would require 3.5-4.0 hr for a clear separation. The mannual separation of the neutral oil represents one of the drawbacks of the batch technique because the skill and experience of the operator in timing the end of the separation of the neutral oil from the soap stock has a strong bearing on the overall efficiency of the process. This is because any oil left on top of the soapstock layer represents an additional loss to the neutral oil trapped in the soapstock layer itself. The degree of firmness of the
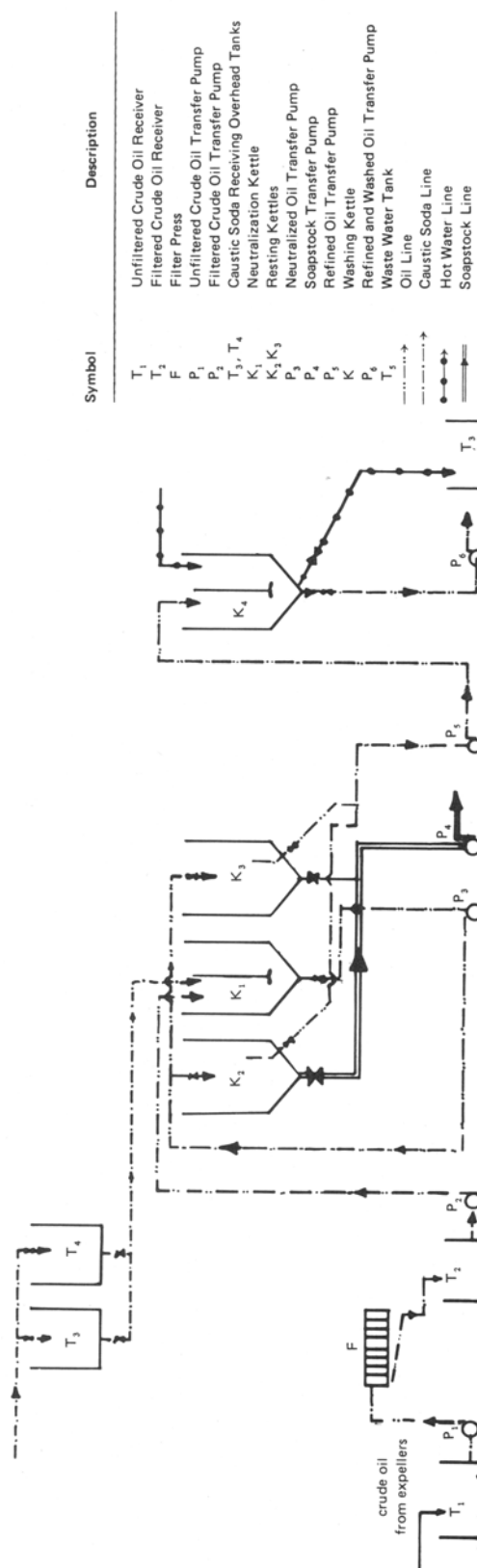

soapstock layer was found to have an inverse relationship to the amount of trapped oil.

The manual decanting of the bottom washing residue from the washing-kettle layer introduces another possibility for neutral oil loss.

Experimental evidence in the literature indicates that in addition to physical adsorption, which is usually associated
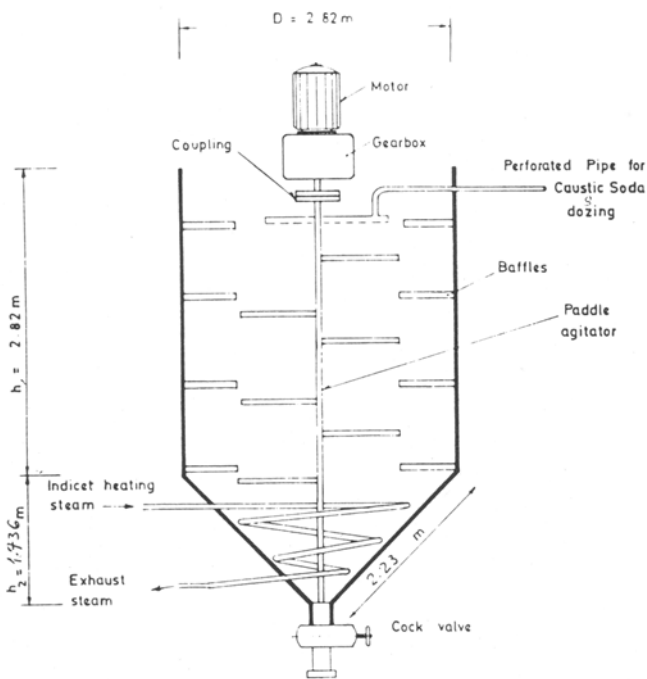
K.M. WAGIALLA, M.E. ABDUL RAHMAN AND E.A. ELAWAD

FIG. 3. Crude oil neutralization vessel.

with pigment removal, processes involving chemisorption and subsequent chemical reaction proceeds on the surface of the activated clay (8) in the bleaching process. Reports have been made that one of the primary results of the bleaching process was the removal of the peroxides and traces of soap and pigments. An analysis of activated earth types, properties and mode of operation has been presented in the literature (8).

Figure 4 shows the flow diagram of the continuous process. The plant is composed of the following sections: the crude oil handling section; the refining section; the re-refining section; the first wash section; the second wash section; the vacuum drying section.

The automatic coninuous refining units (varying in capacity from 20 to 60 T/D), were manufactured by messrs. Sharpless (Pennwatt) of England. The units had been in operation for 3 years when this study was undertaken.

Crude oil was filtered through either filter presses or a superdecanter so that the percentage of solids was as low as 0.05-0.1%. It was normally decanted into 2 15-ton, stirred, mild-steel vessels.

In the refining section, a relatively strong caustic soda solution (26 Be) was dosed via a 450 kg/hr, 30 m-head centrifugal pump directly coupled to a 2 hp high-speed motor. The mix of oil and suspended soapstock particles leaving the first refining mixer was normally fed to a batch of 3 refining centrifuges. These were of the As-16 Super Centrifuge type, spinning at 15,000 rpm and driven by 2 induction motors.

In the vacuum section, the vacuum-producing device consisted of a barometric condenser and a 2-stage jet ejector assembly. Steam at a pressure of 7 kg/cm^2, flowing at the rate of 50 kg/hr, was used to supply the first and second ejectors. The unit was designed to remove 20 kg/hr of water vapor and 2 kg/hr of air, producing a vacuum of 26″-28″ Hg. The resultant dried oil leaving the vacuum drier usually contained less than 0.05% H_2O and had a temperature of 75 C.

The continuous operation has the starting operational advantage over the batch process. When automatic operation begins, the caustic-soda flow rate is automatically
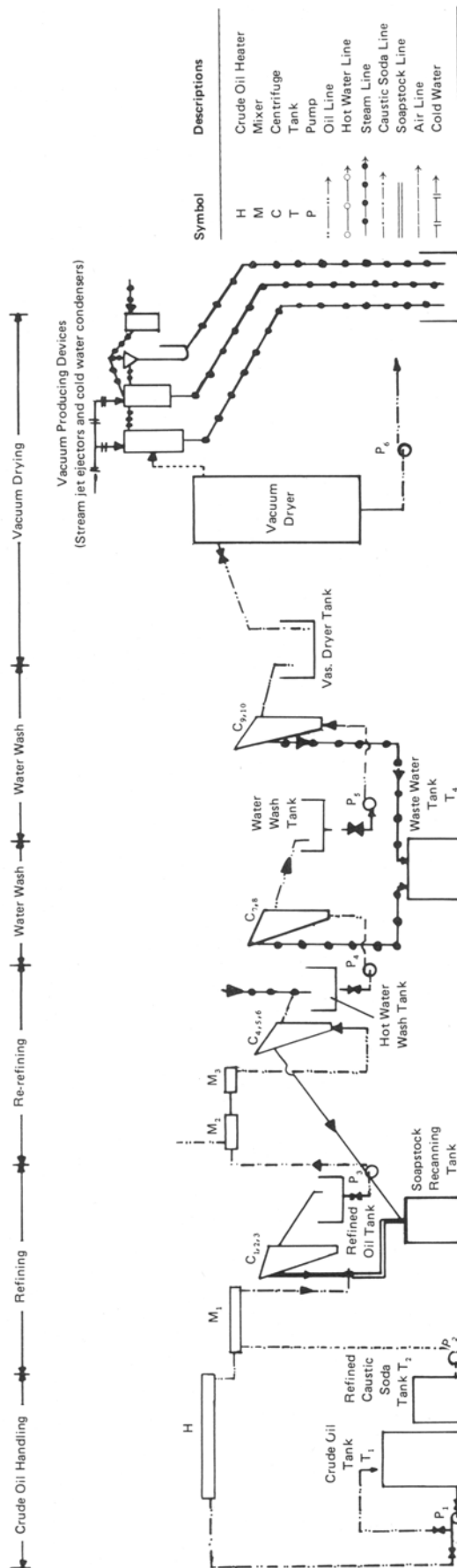

FIG. 4. Flow chart for continuous vegetable refining.

adjusted (using a PID controller) according to an optimum caustic-to-oil flow ratio. This controlled delivery (via a 450 kg/hr, 30 m-head centrifugal pump, directly coupled to a 2 hp motor) is of particular significance because feedstock uniformity is essential for steady state and efficient centrifugal separation. The result is an improved rate of fatty acid elimination without excessive loss of oil from saponification. The FFA reacts with the alkali to form oil-insoluble soaps. This resulting suspension of soapstock is then continuously separated from the neutral oil in a set of 3 centrifuges operating in series.

A temperature of 65-70 C has been recommended as the optimum temperature for the neutralization of cottonseed oil (9).

The re-refining operation has a 2-fold function; to bring about a further contact of a relatively weak caustic lye with the refined oil for removal of any traces of unreacted FFA and to enhance the oil color. The refined oil is mixed with hot, soft water and then centrifugally separated to remove residual soap. The water-washed refined oil, containing traces of moisture, is passed through the continuous vacuum dryer and is then ready for bleaching.

In the continuous process, centrifugal force is used for separation in contrast to batch operation where the less efficient and time-consuming gravity-settling process is relied on.

EXPERIMENTAL RESULTS

The experimental work was based on the performence of 2 refineries operating commercially. The results are presented in the light of the 4 most important criteria that either affect the quality of the finished oil or the general economy of the process.

Relationship of Percentage of Refining Loss as a Function of the Crude Oil Percentage of FFA

The FFA content of the crude cottonseed oil belonging to 2 different seasons was determined. In the first season (1972-73), the batch process was in current use, whereas, in the following season (1973-74), the continuous process was introduced. The FFA content was determined using the AOCS method of analysis (10).

The percentage of refining loss was determined accord-

ing to the following relationship: % Ref. loss=

$$\frac{(\text{mass of crude oil processed} - \text{mass of refined oil yield})}{\text{mass of crude oil processed}} \times 100$$

The results of the percentage of refining loss data and their relationship to the percentage of FFA content is shown graphically in Figure 5 and 6. As this relationship is expected to be linear (10); the best straight line correlating the data was obtained using the method of least squares.

The practical significance of such plots is that once the FFA content is determined experimentally, the expected refining loss can be estimated graphically.

Relationship of the Refined Oil Color as a Function of the Initial Crude Oil FFA Content

The color of refined cottonseed oil varies directly with the FFA content of the crude oil. This is caused by the production of oil-soluble pigments as a result of oil hydrolysis in the seed.

Determination of the refined oil color was made by the AOCS methods, using the standard colormetric method, which employs the Lovibond Tintometer with a 4¼" cell to get the required match between the standard and the sample cells.

The percentage of FFA vs color ratings are plotted in Figure 7 and 8. The best straight-line fit was established (using the method of least squares) as this relationship is expected to be linear (11). Such plots enable the processor to predict the color rating of the refined oil once the FFA characteristics of the crude oil are known.

Caustic Soda Comsumption as a Function of the Initial Crude FFA Content

This performance criteria is of special significance because it influences the overall production cost. The actual monthly consumption of caustic soda and the average monthly crude oil's FFA were determined for each process.

The theoretical requirements, hence the percentage of excess caustic soda used, was then calculated. Table I and Figure 9 show these experimental results in tabular and graphical form for the batch process. The correspinding results for the continuous case are shown in Table II and Figure 10.
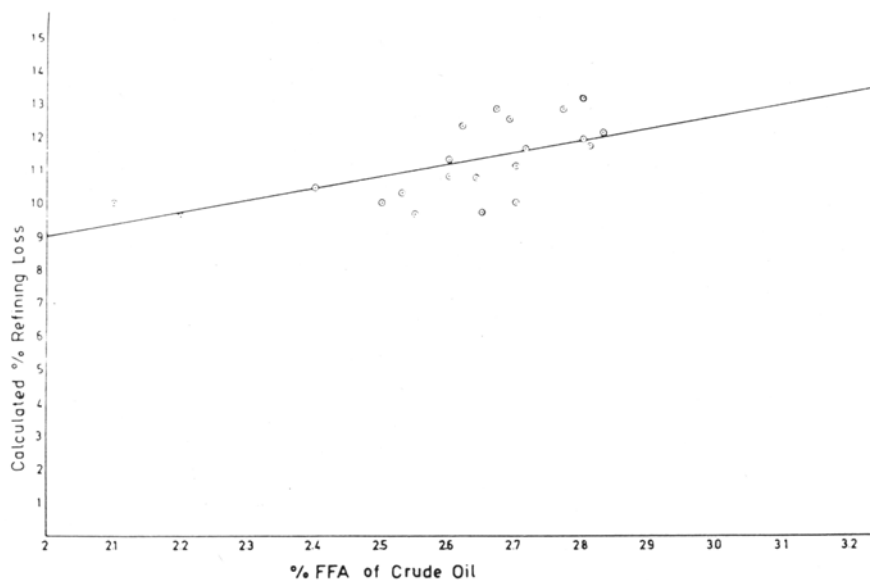

FIG. 5. Relationship between the percentage of FFA content of crude oil and refining loss for batch processing, 1973 season (straight line fixed by least-squares method).

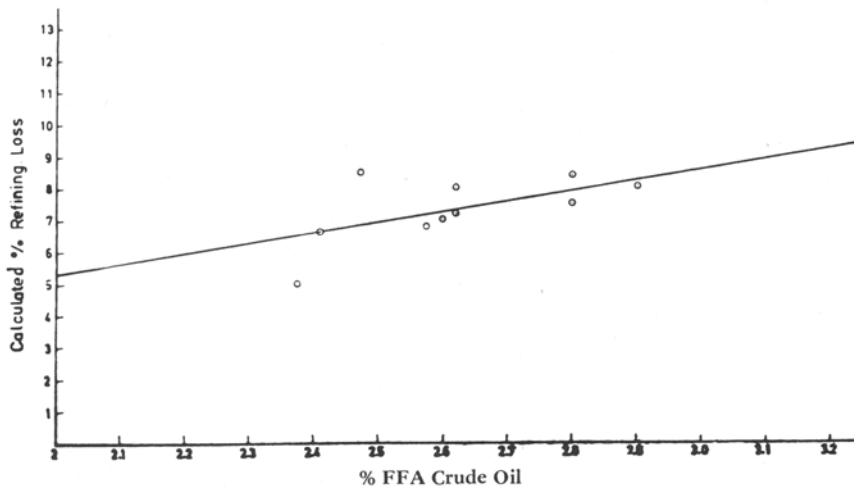K.M. WAGIALLA, M.E. ABDUL RAHMAN AND E.A. ELAWAD

FIG. 6. Relationship between percentage of FFA of crude oil and corresponding refining loss for continuous system, 1974 season (straight line fixed by least-squares method).

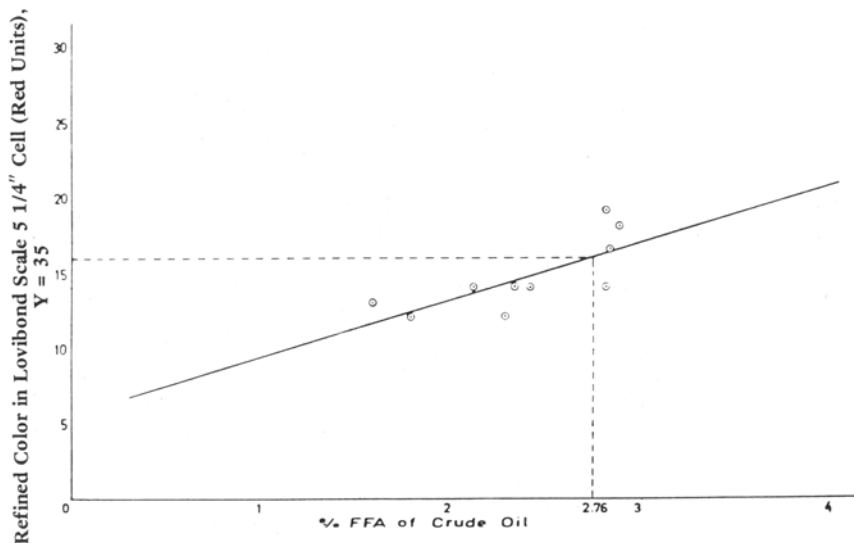**FIG. 7. Percentage of FFA of crude oil vs refined color, March 1976 batch system (straight line fixed with least-squares method).**

Maximum allowable oil acidity to give refined oil color that lies within the allowable margin specified by Sudanese standards, 1976.
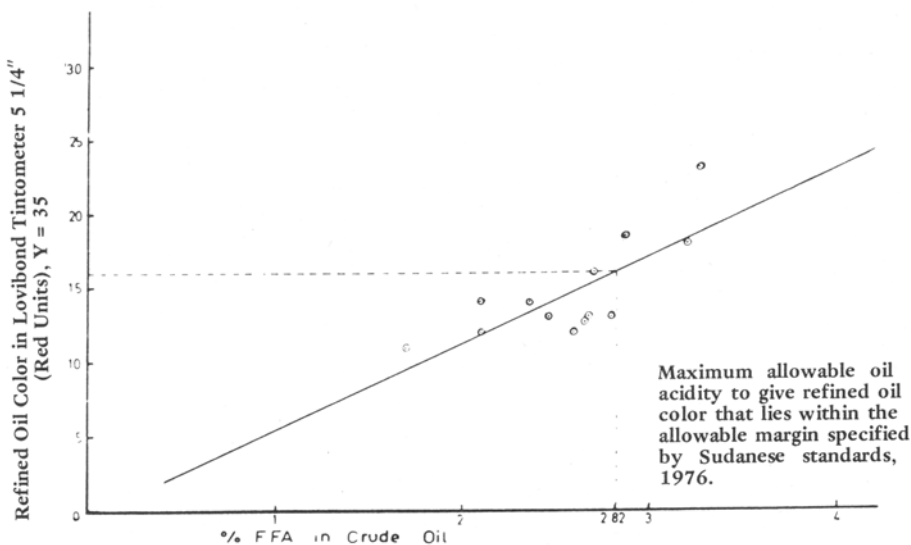
FIG. 8. Percentage of crude oil vs resulting refined oil color, March 1974 continuous system (straight line fixed with least-squares method).

TABLE I

Caustic Soda Consumption for Batch System (1975-1976 Season)

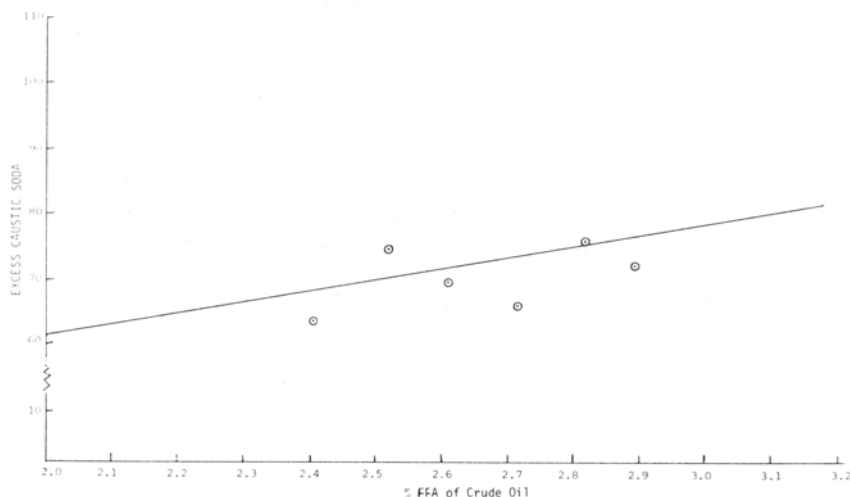| Month	Tonnage of crude being refined	Average percentage of FFA thoughout month	Resulting FFA tonnage	Theoretical requirements of caustic soda (pure) needed for neutralization of FFA (tons)	Actual consumption of FFA (tons)	Excess of NaOH over theoretical used requirements (tons)	Excess (%)
Sept.	380	2.52	9.576	1.358	2.380	1.022	75.26
Oct.	575	2.61	15.007	2.129	3.570	1.441	67.68
Nov.	660	2.7143	17.914	2.541	4.165	1.624	63.91
Dec.	624	2.400	16.848	2.390	4.529	1.554	65.0
Jan.	514	2.889	14.849	2.106	3.570	1.464	69.52
Feb.	564.5	3.024	17.070	2.421	4.359	1.938	80.05
March	599	2.825	16.922	2.400	4.165	1.765	73.54

FIG. 9. Dependence of excess caustic soda consumption over theoretical demand on the percentage of FFA of crude oil (batch system 1975-76).

TABLE II

Caustic Soda Consumption for Continuous System Operation (1974 Season)

Month	Tonnage of crude being refined	Average percentage of FFA thoughout month	Resulting tonnage of FFA	Theoretical requirements of NaOH (pure) to neutralize FFA as oleic acid (tons)	Actual consumption of NaOH (tons)	Excess caustic soda used over theoretical needs (tons)	Excess (%)
Mar.	793	2.561	20.309	2.881	3.982	1.101	38.2
Apr.	660	3.346	22.084	3.132	5.355	2.223	70.98
May	690	2.935	20.252	2.783	4.165	1.382	49.66
Jun.	665	2.850	18.953	2.668	4.120	1.432	53.27
July	645	2.950	19.028	2.707	4.200	1.493	55.15
Aug.	705	2.889	20.367	2.889	4.160	1.271	43.994
Sept.	792.5	2.63	20.843	2.596	4.200	1.244	42.084
Oct.	652	2.41	15.713	2.229	2.975	0.746	33.47
Nov.	679	2.376	16.561	2.288	2.983	0.695	30.38
Dec.	495	2.472	12.236	1.736	2.310	0.574	33.06
Jan.	686	2.370	16.258	2.306	4.170	1.864	80.839

Bleachability Characteristics of Refined Oil

Because of the significance attached to the bleaching stage as a color-enhancement process, the extent to which refined cottonseed is bleachable when treated with standard fuller's earth is important to know. This is particularly so as bleaching test results do not figure in the trading rules of crude oils.

Atmospheric bleaching was carried out at 105 C under moisture-free conditions. The same quantity of fuller's earth was used to bleach equal quantities of refined oil from the batch and continuous processes. The samples were withdrawn for analysis after the contents were agitated for 15 min. The results of these tests are shown in Tables III and IV. Reports in the literature (12) suggest that excellent

bleaching results were obtained by maintaining the temperature between 200 F and 260 F.

DISCUSSION OF EXPERIMENTAL RESULTS

Refined-oil losses may occure during neutralization for the following reasons (9): neutralization of the FFA present in the oil; saponification of neutral glycerides; neutral oil loss with soapstock; saponification of mucilaginous and colored matter.

The refining losses sustained for either mode of operation were found to be more pronounced for the batch case than for the continuous case. From the graphical presentations of the refining losses, a crude oil with an FFA level of 2.5% would be expected to sustain an average refining
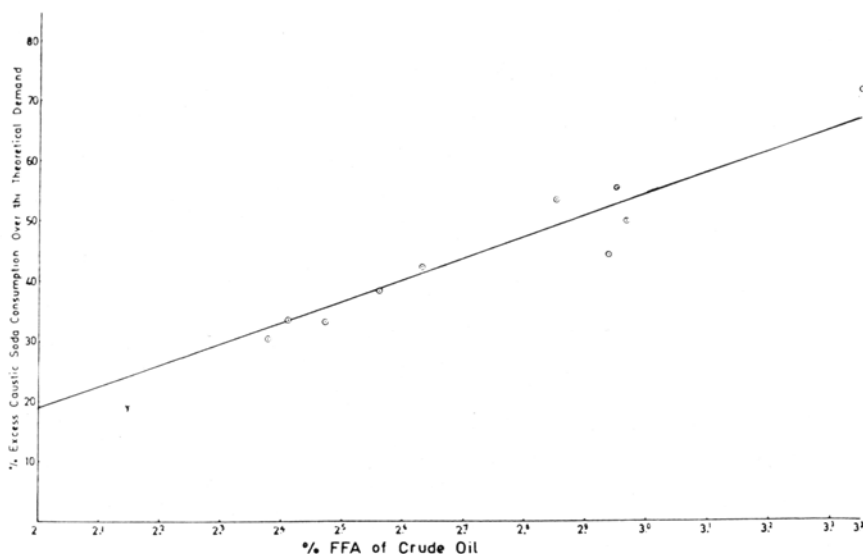
K.M. WAGIALLA, M.E. ABDUL RAHMAN AND E.A. ELAWAD

FIG. 10. Dependence of the percentage of caustic soda consumption over theoretical demand on the percentage of FFA of curde oil continuously processed in the 1974 season (straight line fixed by least-squares method).

TABLE III

Bleachability Data for Batch System

Number	FFA of crude oil (%)	Refined oil color (red units—5¼ cell)	Bleached oil color (red units—5¼ cell)	Color reduction (%)	Earth used (%)
1	2.95	12	6.5	45.8	0.5
2	2.9	15	7.0	53.3	0.5
3	1.95	15	9.5	36.67	0.5
4	1.665	13	7.5	42.31	0.5
5	1.65	11	7.5	31.82	0.5
6	2.74	13	6.16	52.62	0.5

TABLE IV

Bleachability Data for Continuous System

No	FFA of crude (%)	Refined oil color (red units—5¼ cell)	Bleached oil color (red units—cell)	Color reduction (%)	Earth used (%)
1	2.950	12	6.5	45.8	0.5
2	2.900	15	7.0	53.3	0.5
3	1.950	15	9.5	36.67	0.5
4	1.665	13	7.5	42.31	0.5
5	1.65	11	7.5	31.82	0.5
6	2.740	13	6.16	52.62	0.5
Bleachability Data for Batch System					
1	2.83	12	8	33.33	0.5
2	2.13	16	9.5	34.375	0.5
3	2.9	14.5	10.0	31.03	0.5
4	1.8	14	9.5	25.00	0.5
5	2.85	16.5	11.5	30.30	0.5

loss during batch operation of ca. 10.8%. On the other hand, continuous refining of an identical crude will result in a refining loss of 6.9%. Thus, in this particular case, continuous refining would result in a 36% saving in refined-oil loss. This outcome may be explained by the fact that a more efficient separation of refined oil from soapstock is made possible by centrifugation, because a force of separation several thousand-fold greater than gravitational force is used. Moreover, continuous centrifuges reduce the time of contact between oil and alkali to a minmum, leading to lower losses of neutral oil through saponification or occlusion in the soapstock.

As to the refined oil color, Sudanese Standards (1976) specify the maximum acceptable refined-oil color as that color which matches the reading of the Lovibond tintometer of the combination of 35 yellow units and 16 red units using a 5¼″ cell.

Continuous operation was shown to result in a marked imporvement in refined-oil color. The experimental data indicate that a color combination of 35 yellow and 16 red units or less can be obtained from crude oil with an FFA content of 2.82% in the case of continuous operation. The same color readings cannot be obtained from a crude oil with an FFA content of even 2.76% in the case of batch

operation.

The experimental results show a considerable saving in caustic soda consumption when continuous operation is employed. The data indicate that a crude oil with 2.5% FFA would require only 36.5% excess caustic soda for continuous operation, whereas the same crude oil would require ca. 68% excess caustic soda if batch operation is used. For this particular case, a savings of ca. 84% in caustic soda consumption is realized.

The results of the bleachability tests indicate that continuously refined oil tends to acquire better bleaching properties. This result can be explained as being caused by 2 factors. First, the temperature during continuous operation is controlled in the range of 40-45 C. Thus, oil oxidation from overheating is avoided. Second, color fixation as a result of oxidation is not possible during continuous operation because the drying operation is carried out under vacuum.

Continuous operation gave significant savings in refined-oil losses and caustic-soda sonsumption as well as enhanced color and bleaching properties. The savings in refining losses were shown to be particularly significant.

Recent advances in continuous refining (13) indicate that refining losses can be markedly reduced by employing steam distillation rather than caustic refining. However, in such processes, degumming is usually included as a pretreatment step to remove phospholipids by water hydration and centrifugal separation.

From an operational point of view, the continuous process eliminates interstage storage (except surge storage). It is also readily susceptible to continuous automatic and on-line control, thus achieveing high throughputs with better product quality and at the same time reducing operational and supervisory labor, hence reducing production costs. A full description of a proposed on-line computer control system for an oil refining and hydrogenation plant has been compiled (14).

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♣Application of TLC Chemical Confirmatory Tests to Minicolumn Chromatography of Aflatoxins

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ABSTRACT

The minicolumn (MC) proposed by Holaday and Lansden was developed with standard aflatoxin solution and also with the extracts of corn, rice, wheat, cottonseed, peanut cake and black pepper; each having different levels of aflatoxins. One-half mL each of 2,4-dinitrophenylhydrazine, p-anisaldehyde, 20% H_2SO_4, 20% HCl and trifluoroacetic acid (TFA) with 25% HNO_3, which were used for confirming aflatoxins on TLC, were applied to the developed column. Among these, all the 3 acid reagents changed the blue fluorescence of aflatoxins to yellow and thus were found to be satisfactory confirmatory tests. The TFA with 25% HNO_3 had the lowest detection limit—5 ppb.

INTRODUCTION

Concern is growing about the aflatoxin contamination in foods and feeds because of their harmful biological effects and the consequent losses in agricultural trade. Thus, the routine screening of agricultural commodities, foods and feeds for aflatoxins has become very essential. Several minicolumn (MC) methods have, therefore, been developed during the last decade for avoiding lengthy, expensive thin layer chromatography (TLC) techniques (1). In these analytical methods, mistaking aflatoxins for naturally occurring blue fluorescent substances is always possible (2-4). Several chemical tests to confirm aflatoxins on TLC have been proposed in the past (5,6). Jemmali (7) made a pioneering attempt by suggesting a confirmatory test with 50% H_2SO_4 for aflatoxins in cellulose-tube MC. An attempt has been made here to introduce a chemical confirmatory test as a regular step in MC chromatography.

EXPERIMENTAL PROCEDURES

Materials

MC fabricated from a 20-cm length of 6 mm o.d. and 4 mm i.d. borosilicate wall tubing flared into a funnel top, as suggested by Holaday and Lansden (8), was used. Solvents, e.g., acetone, ethanol, methanol, benzene, hexane, acetonitrile, concentrated sulphuric acid, concentrated hydrochloric acid, trifluoroacetic acid (TFA), concentrated nitric acid and glacial acetic acid, used were of analytical reagent grade. Salt solution was prepared by dissolving 15 g sodium chloride and 15 g zinc acetate in 100 mL distilled water. p-Anisaldehyde reagent and 2,4-dinitrophenyl hydrazine reagent were prepared as described by Scott et al. (9) and Crisan (10). Aflatoxin reference standard containing 1 μg/mL was prepared in benzene/acetonitrile (98:2, v/v) as outlined in the official AOAC method for standards (11). Neutral alumina (100-200 mesh) activated by drying at 100 C for 2 hr and florisil (100-200 mesh) activated by drying at 100 C for 2 hr were used for packing the column.

Methods

The MC was packed as described by Holaday and Lansden (8) and stored overnight in a desiccator containing saturated NH_4Cl solution for equilibration with 80% RH.