JKAU: Eng. Sci., **vol. 11 No. 2,** pp. 115-126 (1419 A.H. / 1999 A.D.)

Techno-economical Evaluation of Oil Recovery and Regeneration of Spent Bleaching Clay

YAHIA A. SALEH ALHAMED and ABDULRAHIM A. AL-ZAHRANI
Chemical & Materials Engineering Department,
King Abdulaziz University, Jeddah, Saudi Arabia

ABSTRACT. Edible-oil refining industries in Saudi Arabia have expanded considerably in the last twenty years. The major waste generated by these industries is spent bleaching clays. It is estimated that about 6000 ton of this waste is generated annually. The cost of the disposed bleaching clay along with oil it contains (20-40% of the spent clay weight) are major costs of refining. Furthermore, the disposal of spent bleaching clay in landfills can cause environmental pollution problems in addition to being a source of odors and fire hazards.

This paper describes a process for the recovery of oil from spent bleaching clay by solvent extraction followed by reactivation of the deoiled clay by acid treatment. Also, a preliminary cost estimation of the suggested process was presented for a plant which can process 4800 ton/year of spent bleaching to produce 3600 ton/year of reactivated spent bleaching and 800 ton/year of recovered oil. The study indicates that the suggested process is economically viable. The return of investment was estimated to be 25%.

1. Introduction

Edible-oils refining involves the treatment of degummed and neutralized oil with bleaching clay to eliminate dissolved coloring materials and to reduce the quantity of oxidizing matter. The oil content of the spent bleaching clays is in the range from 20 to 40 wt.%. This oil represents a large part of the cost of bleaching and it is important to be recovered[1]. The cost of the spent clay with its oil contents comprises a considerable portion of the total refining cost. The disposal of spent bleaching clays in landfills can cause environmental pollution problems in addition to being a source of odors and fire hazards. This problem can be solved by developing a process for the recovery of the oil entrained in the spent clay followed by the regeneration of the deoiled clay for the reuse. Edible-oils refining industry in Saudi Arabia can benefit from such a process through recycling of the spent clay, thus, reducing the amount of the imported clays. Furthermore, the recovered oil can be either recycled or sold for other uses such as soap industry.

Solvent extraction processes for recovery of oil from bleaching residues have been suggested because they give higher yields and better quality of oil compared to other methods, provided that the process is carried out on fresh residues[1-5]. The extracted clays may be used in bleaching, either directly or after activation with acids. Solvent extraction followed by burning at 400ºC is reported as a method which produces more active clay than the original [2-3]. A low molecular weight ketone was found to be the most effective solvent[4]. Funayama[5] reported the removal of sorbed oil from the spent catalysts and other inorganic materials. Kheoh[6] studied the reactivation of spent bleaching clay. The reactivation process involves washing of the spent earth from edible-oil bleaching with organic solvents to remove fats, colored compounds and organic impurities with subsequent solvent removal. The preferred solvents are acetone and methylethylketone (MEK). Sakakura[7], described a solvent extraction method to recover oil and fats which can be used for food. DeFilippi and Chung[8] studied the extraction of silicone oil and soybean oil from bleaching clays using halocarbon and hydrocarbon solvents. In their study[8], they also reported an economical evaluation of a plant based on critical fluid extraction for recovery of silicone oil from spent bleaching clay. They concluded that such a plant will pay out favorably due to the credits for recovered oil, however, in their economical evaluation they did not consider the possibility of a process in which both oil recovery and regeneration of the spent clay are carried out simultaneously. The authors have performed exhaustive laboratory studies[9-10] to determine the optimum conditions for oil recovery from the spent bleaching clay and regeneration of the deoiled clay.

In this paper, a process for the recovery of oil from the spent bleaching clay by solvent extraction and regeneration of the deoiled clay by acid treatment is described. A preliminary economical analysis of the process to estimate the return on capital investment is also presented.

2. Plant Location

The major edible-oil refiner in Saudi Arabia is SAVOLA company with a production capacity of about 200,00 ton/year. SAVOLA plant is located in the industrial district of Jeddah City. Other producers are Best Foods in Yanbu City and Nabati in the Eastern region. Each plant produces about 30,000 ton/year of refined edible-oil. Nabati company is planning to double the production capacity to 60,000 ton/year. The total amount of spent clay used by these manufacturers, including declared future expansions, was estimated to be 6000 ton per year. The proper location for the suggested plant is in the Western region, preferably in Jeddah, since over 75% of edible oil is refined in this part of the Kingdom. It is also essential for the plant to be close to the source of the raw materials since delays in extracting the oil from the spent clay will result in the degradation of the oil and consequently the recovered oil will be of poor quality. Being close to the source of the raw material will also reduce the transportation costs.

3. Process Description

The suggested process consists of two major parts namely oil recovery by solvent extraction using methylethylketone (MEK) and regeneration of the deoiled clay by sulfuric acid treatment. The authors' experience in this area[9,10] suggests that a process based on solvent extraction is very appropriate. Due to the large difference in the boiling points of the oil and the solvent, the oil can be easily separated and the solvent can be regenerated for reuse with minimum losses. MEK was found to give the highest oil recovery amongst the other solvents (acetone, hexane, petroleum ether) tested[10]. Furthermore, spent clay deoiled by MEK at optimum conditions (solvent to clay ratio = 4 ml/g and, extraction time = 18 minutes) has the highest bleaching power compared to those deoiled by other solvents. The operating conditions are chosen such that maximum oil recovery and maximum bleaching power are obtained according to the laboratory studies reported previously[10].

A flowsheet for the suggested process is presented in figure 1. The process consists of two major sections namely the oil extraction and the clay activation sections. A batch process is employed except for the distillation column which is operated in a continuous mode. In the extraction section, the spent clay and the solvent are introduced into the extraction vessel where they are contacted at room temperature under agitation for 18 minutes. The solvent to clay ratio (SCR) is kept at 4 milliliters of solvent to 1 gram of spent clay. After 18 minutes of extraction, the slurry is pumped through one of the filters (plate and frame type) while the cake from the previous batch is being collected from the other filter. The filtrate is collected in the holding tank where it is pumped to the distillation column for separation of the solvent from the oil. The recovered oil is sent to the oil holding tank and the solvent is recycled to the solvent tank. The cake removed from the filters is transferred to a tray type dryer. The hot air leaving the dryer passes through a condenser where the solvent is recovered and recycled to the solvent tank. The dried clay is transferred to a storage tank.

In the activation section 10 wt. % sulfuric acid is used. The activation is carried out at the boiling point of the mixture (about 103ºC) in a jacketed reactor equipped with an agitator. The reactor is heated using steam. The water to clay ratio (WCR) is kept at 5 milliliters water to 1 gram of spent clay. After 20 minutes of reaction, the slurry is pumped through one of the plate and frame type filters while the cake from the previous batch is being collected from the other filter. The filtrate is then recycled to the dilute-acid tank. The filters used in the activation section are provided with through washing arrangement and water is used to remove the residual acid from the cake. The cake removed from the filters is transferred to a tray type dryer where it is dried using hot air. Finally, the activated clay is sent for packaging and storage.

4. Plant Capacity

The current production of the refined edible oils in Saudi Arabia is about 260,000 ton per year. The estimated amount of the spent clays produced annually (assuming 2 wt.% is used in bleaching) is 5200 ton/year. Since it is not possible to collect all the spent, the plant capacity was taken as 4800 ton/year. Assuming 300 days operation per year, 7 days per week and 3 shifts, the plant capacity will be 666 kg/hour. The process is assumed to be batch except for the distillation column which operates in continous mode. The time per cycle (batch) is one hour.

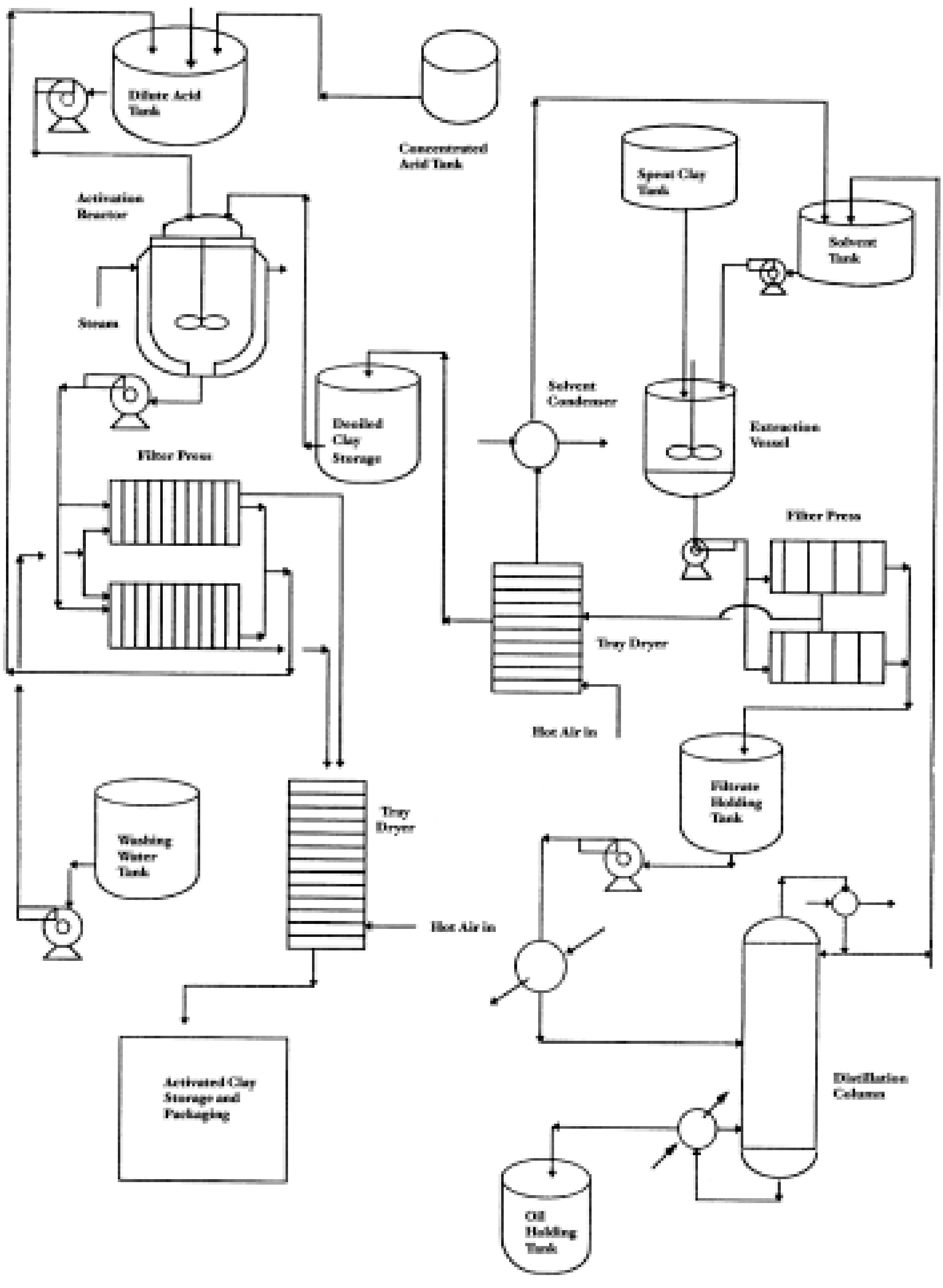

FIG. 1. Process flowsheet.

5. Equipment Sizing

Equipment sizing was essential to estimate the cost of purchased equipment. The calculations for sizing of the major equipment are outlined below.

5.1 Extraction Vessel

666 kg of spent clay is extracted per batch. The volume of MEK solvent needed for extraction is 2.7 m^3 using a solvent to clay ratio of 0.004 m^3 solvent/kg spent clay. The volume of the clay and the solvent is 3.5 m^3 using a clay density of 816 kg/m^3. Assuming 14% free space in the extractor, the volume of the extraction vessel is estimated to be 4 m^3 (1057 gal).

5.2 Acid Treatment Reactor

The total oil content of each spent clay (oil content = 23 wt.%) batch is 153.2 kg. The degree of oil extraction using MEK at the optimum conditions (i.e. 0.004 m^3 solvent / 1 kg spent clay and extraction time of 18 minutes) was determined experimentally to be 73%. Therefore, the weight of the clay leaving the filter press is estimated to be 554 kg/batch. The acid treatment of the deoiled clay is carried out using a water to clay ratio of 5 kg water / 1 kg deoiled clay (1 m^3 of 10% H_2SO_4 / 192 kg deoiled clay), an acid concentration of 10% H_2SO_4 and a reaction temperature of 103ºC.

The bulk density of the deoiled clay as determined experimentally is 727 kg/m^3. Therefore, the volume of the slurry is 3.66 m^3 (the volume of acid is 2.88 m^3 and the volume of the deoiled clay is 0.762 m^3). Assuming 36% free volume, the reactor volume is estimated to be 5 m3 (1321 gal).

5.3 Solvent Filter Press

For an incompressible cake, the filtration time (θ) is given by the equation below[11].

$$\theta = (\mu\, \alpha\, w / g_c A^2 (-\Delta P_t))\ (V^2/2 + V_e w) \tag{1}$$

μ = viscosity of the solvent = 0.39×10^{-3} N.s/m^2 ,

w = weight of the solids in feed slurry per volume of liquid in this slurry = 250 kg spent clay / m^3 solvent,

V = volume of filtrate that has passed through the filter cake,

ΔP_t = total pressure drop across the filter (assumed to be 25 psia),

V_e = The volume of filtrate necessary to build up a fictitious filter cake, the resistance of which is equal to the resistance of the filtration medium and the piping between the pressure taps used to measure ΔP_t,

α = specific cake resistance = $5(1 - \in)S_o^2/\rho_s \in$,

$\in$ = porosity of the cake (assumed to be 0.3),

ρ_s = density of the cake (deoiled clay) = 1800 kg/m^3 as measured by porosometer,

S_o^2 = $6/d_p$; where d_p is the diameter of the solid particles – 48×10^{-6} m.

If filtration time is taken to be 15 minutes and V_e was assumed to be 0.2V, the filtration area can be estimated using equations (1) and (2) to be 1.8 m^2.

The filtration area for the acid filter press is estimated in a similar fashion. However, in the case of acid, a filter press equipped with a through washing arrangement is recommended. The filter press materials recommended is PVC coated iron to avoid corrosion problems due to contact with acid.

5.4 Distillation Column

The relative volatility of MEK with respect to oil is very high, therefore, a small distillation column is adequate. The maximum allowable superficial vapor velocity (V_m) needed for the estimated of the column diameter was calculated using the following equation[12].

$$V_m = K_v\,[(\rho_L - \rho_G) / \rho_G]^{1/2} \tag{3}$$

ρ_L , ρ_G = densities of liquid and gas (0.725 and .00249 g/ cm^3), respectively,
K_v = an empirical constant = 0.18 ft/s (assuming tray spacing of 12 inches)[12].

The column is operated at 1 atmosphere with a feed rate of 2.8 m^3/hour and a temperature of 80ºC (boiling point of MEK). In applying equation (3), the properties of MEK were used since its oil content is only 5%. V_m was found to be 3.07 ft/s and the corresponding column diameter is 2 ft. The height of a 4 tray column is 5 ft.

5.5 Dryers

Tray dryer is recommended because of its low cost, flexibility and applicability for small quantities. The volume of the clay to be dried per batch is 0.736 m^3. Assuming a layer thickness of 5 cm/tray, the required drying area is calculated at 158 ft^2. However, the drying area of 200 ft^2 was considered.

6. Cost Estimation

The preliminary estimation of capital investment and total product cost was made using the design criteria listed in Table 1. The cost estimation was made for a plant processing 4800 tons of spent clay per year to produce 3600 tons of regenerated clay and 800 tons of oil per year. The selling price of the regenerated clay is assumed to be 50% of the virgin clay price ($US 500) and the oil is assumed to be sold for $US 150 per ton (i.e. about 10% of the selling price of the refined corn oil).

6.1 Purchased Equipment Cost

The specifications, and purchase and installation costs of major equipments outlined in the flowsheet shown in figure 1 are summarized in Table 2. Equipments cost ($US) estimation was based on data available for the year 1990[12]. As can be seen from Table 2, the purchased equipments cost is $US 171000 and the installation cost is $US 73100.

6.2 Capital Investment

Direct cost components were estimated as a percentage of the purchased equipments cost and the indirect costs were calculated as a percentages of the direct cost[12]. Table 3 summarizes the different components of capital investment and the percentages used in their estimation. The total capital investment was estimated to be \$US 940448 as shown in Table 3. Different components of the annual total product cost were estimated as percentages of different cost components as outlined in Table 4[12].

TABLE 1. Design criteria.

Processing capacity	4800 ton per year of spent clay.
Operating factor	300 day per year, 7 days per week, 3 shifts
Oil content of spent clay	23% of the spent clay weight
Solvent	Methylelthylketone (MEK)
Acid	10 wt.% sulfuric acid
Solvent to clay ratio	4 milliliters of solvent per gram spent clay
Extraction temperature/time	Room temperature / 20 minute
Degree of extraction	73% of the total oil content of the spent clay
Water to clay ratio	5 milliliters water per gram of deoiled-dried clay
Degree of reactivation of the clay	100%
Weight of the spent clay per batch	666 kg of spent clay
Reaction temperature / time	boiling point of the acid / clay mixture (about 103ºC)/20 minute
Particle size of the clay	325 mesh
Weight of activated clay produced	3600 ton/year (assuming 77% of the spent clay)
Selling value of the activated clay	\$US 500/ton
Weight of oil recovered	800 ton per year (73% total oil content)
Selling value of oil recovered	\$US 150 (10% of the selling value of refined corn oil)
Solvent requirements	33 tons per year (assuming 0.2 wt.% loss of solvent)
Make up acid	418 tons per year of 100% sulfuric acid (assuming 20% loss of the acid strength per batch)
Solvent cost	\$US 1300 per ton
Acid cost (100% sulfuric acid	\$US 75/ton

TABLE 2. Equipment sizing, specifications, purchase cost and installation cost.

Ser. no.	Equipment	Size	No. of units	Installation cost / unit (\$US)	Purchase cost / unit (\$US)	Specifications
1	Extraction vessel	4 m3 (1057 gal)		4500	1500	Cast iron, jacketed, with agitator
2	Filter press for solvent removal	1.8 m^2 (19 ft2)	2	650	1000	Cast iron
3	Tray dryers	(200 ft^2)	2	3500	10500	Tray type, atmospheric, carbon steel dryer

TABLE 2. Contd.

Ser. no.	Equipment	Size	No. of units	Installation cost / unit ($US)	Purchase cost / unit ($US)	Specifications
4	Reactor for acid treatment	5 m^3 (1321 gal)	1	20000	40000	Glass lined iron, installation cost includes auxiliaries, agitator, drive and jacket
5	Filter press for acid removal	1.5 m^2 (17 ft2)	2	650	2000	PVC coated iron. Provided with through washing arrangement for the cake
6	Distillation column	Diameter = 2 ft Height = 5 ft	24000	36000		Steel shell, stainless steel sieve tray. Total cost includes installation and auxiliaries
7	Storage holding tanks	5 m^3 (1321 gal)	3	2000	7000	Carbon steel
8	Deoiled activated clay storage	3 m^3 (800 gal)	2	1500	5000	Carbon steel
9	Concentrated acid tank	2 m^3 (528 gal)	1	1500	5000	Stainless steel
10	Dilute (10%) acid mixing tank	5 m^3 (1321 gal)	1	20000	70000	Stainless steel with agitator
11	Pumps	2042 gal / minute at 40 psia	5	500	2000	Stainless steel, acid resistant and chemical resistant centrifugal pump
Total				**73100**	**171000**	

TABLE 3. Estimation of Total Capital Investment.

Estimation of Total Capital Investment		
I. Direct Costs (DC) A. Equipment + installation + instrumentation + piping + electrical + insulation + painting	Assumed (%)	$US
1. Purchased Equipment Cost (PEC)	100	171000
2. Installation (25-55% of PEC)	43	73000
3. Instrumentation and control (6-30% of PEC)	0	17100
4. Piping, installed (10-80% of PEC)	31	53010
5. Electrical, installed (10-40% PEC)	30	51300

TABLE 3. Contd.

Estimation of Total Capital Investment		
B. Buildings, process and auxiliaries (10-70% of PEC) C. Service facilities and yard improvement (40-100% of PEC) D. Land (4-8% of PEC)	55 50 4	94050 85500 6840
Total Direct Costs (TDC)		
II. Indirect Costs (IDC) A. Engineering and supervision (5-30% of DC) B. Construction expenses & contractor fees (6-30% of DC) C. Contingency (5-15% FCI)	 18 18 5	 99324 99324 50000
Total Indirect Costs (TIC)		248648
III. Fixed-Capital Investment (FCI) = (TIC + TIDC)		800448
IV. Working Capital (WC): 10-20% of TCI	10	140000
V. Total Capital Investment (TCI) = (FCI + WC)		940448

TABLE 4. Estimation of Total Product Cost per year.

Estimation of Total Product Cost		
I. Manufacturing Cost (MC)	Assume (%)	$US
A. Direct Production Costs (DPC)		
1. Raw materials		
Methylethylketone : 33 ton/year at $US 1300/ton (Assuming 0.2 wt.% losses per day)		42900
Sulfuric acid: 418 tons (100% H_2SO_4) at $US 75/ton		31350
2. Operating labor: 15 workers/shift, 3 shifts at $US 5/h (24 hours, 300 days/year)		540000
3. Direct supervisory and clerical order (10-25% of operating labor)	12	64800
4. Utilities (10-20% of total product cost)	10	160000
5. Maintenance and repairs (2-10% of FCI)	5	40022
6. Operating supplies (0.5-1% of FCI)	1	8004
7. Laboratory charges (10-20% of operating labor)	10	54000
8. Patents and royalties (0-6% of total product cost)	0	0
B. Fixed charges		
1. Depreciation 10% of installed equipment, 3% of buildings)		26110
(3% of buildings)	3	8721
2. Local taxes (0-4% of FCI)	0	0
3. Insurance (0.4-1% of FCI)		8004
4. Rent (8-12% of land or building rented)		0
C. Plant-overhead costs (50-70% of, operating labor, supervision and maintenance costs)	50	322411
Total Manufacturing Costs		1306323
II. General expenses		

TABLE 4. Contd.

A. Administrative costs (15% of, operating labor, supervision and maintenance cost)	15	96723
B. Distribution and selling costs (2-20% of total product cost)	2	40000
C. Research and development costs (5% of total product cost)	0	0
D. Financing (0-10% of total capital investment	10	94045
Total General Expenses		230768
III. Total product cost = (manufacturing cost + general expenses)		1537091
IV. Gross earnings = Total income – total product cost		382909
Return on investment (%)		25
Payoff time (Years)		4

The price of the solvent used in this work is the current market price including transportation cost. The solvent losses were estimated using data available for soybean plant in which the oil seeds are extracted by using hexane as a solvent. According to Myers[13], for an average soybean plant, the solvent loss is about 0.7 US gal/ton. This is equivalent to about 0.2 wt.% of the total solvent used. A good system for solvent recovery is essential for the economics of the process. Further optimization of the process and energy saving through heat recovery from various streams of the process will further improve the economics of the process.

As shown in Table 4, the total product cost is $US 1,537,091 per year. The annual return on the capital investment is 25% and the payoff time is 4 years. Based on the cost analysis presented in this paper, the installation of a plant to recover oil from the spent bleaching clay followed by reactivating the clay is economically sound and profitable.

Conclusions

A process for the recovery of oil from spent bleaching clay by solvent extraction and the regeneration of the deoiled clay by acid treatment was found to be profitable. A preliminary cost estimation for a plant which can process 4800 ton/year of spent bleaching to produce 3600 ton/year of reactivated spent bleaching and 800 ton/year of recovered oil indicate that the process is economically viable with a 25% return on investment.

References

[1] **Ong, John, T.L.,** Oil Recovery from Spent Bleaching Earth and Disposal of the Extracted Material, *JACOS,* **60**(2): (1983).

[2] **Hamza, A.,** *An Investigation on the Utilization of Egyptian Clays in Bleaching of Cotton Seed Oil,* M.Sc. Thesis, Alexandria University, Egypt, pp. 16-48 (1966).

[3] **Kheok, S.C.,** Mechanism of Palm Oil Bleaching by Montmorillonite Clay Activated at Various Acid Concentration, *JAOCS,* **59:** 129 (1982).

[4] **Fenge, R.** and **Janssen, H.,** Bleaching of Cottonseed Oil in Hexane, *JAOCS,* **28:** 429 (1951).

[5] **Funayama, S.,** Japan Pat. 7871, 109 (1978).

[6] **Kheoh, S.L.,** *Reviving of Spent Earth from Edible Oil Refining by Washing by Organic Solvents,* UK Pat. G.B. 2, 189233 (CA 108: 74055s), 21 Oct. (1987).

[7] **Sakakura, N.,** *Recovery of Oils and Fats from Spent White Clay Used for Decolorization,* Japan Pat. 80 12, 160, (CA 93: 93859V), 31st Mar. (1980).
[8] **DeFelippi, Richard O.** and **Chung, Marie E.,** *Laboratory Evaluation of Critical Fluid Extractions for Environmental Applications,* EPA/600/2-85/045, EPA, Office and Research and Development, Air and Energy Engineering Research Laboratory, Research Triangle Park NC 27711, April (1985).
[9] **Al-Zahrani, A.A.** and **Alhamed, Y.A.,** Regeneration of Spent Bleaching Clay and Oil Recovery by Boil-Off Method and Acid Treatment, *Middle East FORUM,* Issue **1,** 17-28 (1996).
[10] **Al-Zahrani, A.A.** and **Alhamed, Y.A.,** Regeneration of Spent Bleaching Clay and Oil Recovery by Solvent Extraction and Acid Treatment, *Eleventh International Conference in Solid. Waste and Technology and Management,* Philadelphia, USA, Nov. 12-15 (1995).
[11] **Foust, A.S., Wensel, L.A., Clump, C.W., Maus, L.** and **Anderson, L.B.,** *Principles of Unit Operations,* 2nd edition, John Wiley & Sons Inc., New York, p. 669 (1980).
[12] **Peters, M.S.** and **Timmerhaus, K.D.,** *Plant Design and Economics for Chemical Engineers,* 4th Ed., McGraw-Hill Inc., New York (1991).
[13] **Myers, N.W.,** Solvent Recovery, *JACOS,* **60**(2): (1983).

Notation

d_p the diameter of the solid particles
K_v an empirical constant
V volume of filtrate that has passed through the filter cake
Vm maximum allowable superficial vapor velocity
w weight of the solids in feed slurry per volume of liquid in this slurry
ΔP total pressure drop across the filter
α specific cake resistance
μ viscosity of the liquid
$\in$ porosity of the cake
ρ_s density of the deoiled clay
ρ_G density of gas
ρ_L density of liquid

تقـويم اقتصـادي وتقـني لإسترجـاع الزيت من طينـة التبيـيض المستهلكـة وإعـادة تنشيطهـا

يحيى أبو بكر الحامد و عبدالرحيم أحمد الزهراني

قسم الهندسة الكيميائية وهندسة المواد ، كلية الهندسة

جامعة الملك عبدالعزيز ، جـــدة - المملكة العربية السعودية

المستـخلص . لقـد شهـدت صناعـة تكرير زيوت الطعـام في المملكة العربية السعودية توسعًا كبيرًا خلال العقدين الماضيين ، وتقدر كمية طينة التبييض المستهلكة التي تنتجها هذه المصانع بحوالي ٦٠٠٠طن سنويًا ويتم التخلص منها برميها في مرادم النفايات العامة ، مما قد يسبب تلوثًا بيئيًا بالإضافة إلي كونها مصادر للروائح المزعجة ومخاطر الحريق . كما أن الطينة المستهلكة لايمكن استخدامها مرة أخرى ويُفقد محتواها من الزيت .

هذه الدراسة تعرض عملية لاسترجاع الزيت من طينة التبييض المستهلكة وإعادة تنشيطها بالإضافة إلى دراسة اقتصادية مبدئية لمصنع يعالج ٤٨٠٠طن سنويًا من طينة التبييض المستهلكة لينتج ٣٦٠٠ طن من طينة التبييض المنشطة التي يمكن استخدامها في تبييض زيوت الطعام ، كما ينتج ٨٠٠طن من الزيت المسترجع سنويًا .

لقد بينت هذه الدراسة أن العملية المقترحة مجدية من الناحية الاقتصادية ، ويُقدر العائد على الاستثمار بحوالي ٢٥٪ .