Bleaching of Some Vegetable Oils with Acid-Activated Jordanian Bentonite and Kaolinite

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Acid activated Jordanian bentonites and kaolinites (employing hydrochloric, sulfuric, phosphoric and acetic acids) are used for the bleaching of corn, soybean and sunflower oils. The results showed that in general the Jordanian bentonites but not kaolinites, compared favourably with the industrial commercial bleacing earth. The sulfulric acid activated sample showing uniformly best bleachability property. Freundlich's isotherms showed that the acid activated Jordanian bentonites displayed superior adsorption strength, but inferior adsorption capacity, when compared with commercial bleaching earths. This could be due to the low montmorillonite content but high acidity of the activated Jordanian bentonites, which are therefore quite suited for the initial bleaching of pigmented vegetable oils.

Key Words: Acid activation, Kaolinite, Oil bleaching, Freundlich isotherm, Bentonite.

INTRODUCTION

Bentonite and kaolinite are econmic minerals with wide variety of industrial applications[1]. In Jordan bentonites is mainly used in casting metal foundries, in the drilling of wells and as bleaching clay for oils and fats. Kaolinite on the other hand is widely used in the local pottery and ceramic industries. Economical deposits of bentonite in Jordan are mainly found in Al-Azraq area east of the country[2]. This bentonite is characterized by low to moderate montmorillonite content[3]. Good quality kaolinite is found in several locations with the best known is Butn Al-Goul south of the country[4].

Scanty statistics are available about the consumption of bentonite and kaolinite in Jordan. However, the country imports about 1000 MT per year of bentonite to be used mainly in foundries, well drilling and bleaching of oils and fats[5].This figure is expected to rise in the near future if there are no local substitutes.

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Jordanian bentonite as a drilling mud failed to give acceptable results because of its high quartz content[3]. Its use on the other hand, as bleaching clay in the refining of oils and fats has not up till now been investigated.

It is the purpose of the present word to investigate the possible use of Joranian clays as beaching earths for vegetable oils, as industry which has grown rapidly over the last decade maily to meet the local and regional demands[5]. Thus local substitutes to the imported bleaching earths acquire special importance. If proven successful, the ecnomic deposits of native bentonite could become of potential value to Jordan exports contributing the bleaching earth's world market.

Although activated bentonite is known to have the best bleaching power[6-8], kaolinite has historically been used as bleaching clay[1] and because it is abundantly present in the country is considered worth investigating for the same purpose.

The widely accepted technique of the activation of clay[6,7,9] is used in present work to obtain the required performance of the native clays and to compare them with the well known acid activated bleaching earths used in the industry, *viz.*, Tonsil standard 3141 and WAC classic.

Crude sunflower oil is that locally extracted and imported crude soybean and corn oils are selected to test the bleaching performance of the acid activated bentonites and kaolinites.

EXPERIMENTAL

Commercial industrial bleaching earths used in the present work were WAC classic from Taiko Clay Marketing, Malaysia and Tonsil Standard 3141 FF from SUDCHEMI Germany. Crude sunflower oil was extracted and supplied by Chemical Industries Union Co., Jordan, crude corn oil and soybean oil was of USA origin.

Jordanian Natural Resources Authority (NRA) supplied the raw native Jordanian minerals. Bentonite was obtained from Ain Al-Baida at Al-Azraq depression, East of Jordan and kaolinite was from butn Al-Goul region, south of the country. Ground samples that passed the 200 mesh were used.

Acid activation procedure for native minerals: The optimum condition were selected[6,7,9,10] for activation of bentonite and kaolinite with hydrochloric acid, sulfuric acid and phosophoric acid as follows: Acid to clay 0.35 g acid/g clay, acid concentration 5 N, tempareture 95 ± 2 ºC, time 4 h, agitation performed with magnetic stirrer of medium continuous speed sufficient to keep the clay particles suspended without settling. While for activation with acetic acid two acid to-clay ratios were used, 0.42 and 1.47 g acid/g clay.

The specified volume of acid solution was mixed with 30 g of clay in a round bottom flask with a reflux condenser and placed in a water bath. The

mixture was continuously agitated and after 4 h the clay was simply filtered and washed with 250 mL of distilled water. The clay cake was then left at room tempreture to dry to equilibrium moisture content under ambient condution. The clay was later grounded with an agate mortar and pestle and sieved, the partciles below 63 µ were collected and used in the bleaching experiments.

Characterization of the clay samples: The mineral constitution of the samples was determined by powder XRD technique, using X'perts instrument operated at 40 kV and 40 mA and fitted with Philips X-ray tube giving CuKα radiation λ = 1.5418 Å.

Chemical compositions of the samples were determinded using Diano-2023 XRF Spectrometer following the manufacturer recommended operating conditions. Accordingly, samples were fluxed with lithium tetraborate with a tempreture program up to 1200 ºC.

Solid infrared spectra were measured over the range 4000-400 cm^{-1} in KBr matrix pressed at 10 tonn m^{-2} using Nicolet-Impact 400 FTIR spectrophotometer.

Moisture content of the air-dried clay samples was determind using Sartorius moisture analyzer model MA 30. Acid values for the clay were determined using the AOCS official method[10] Cd 3d-63 with water as solvent.

Surface area estimation: The surface area of the samples was estimated using the methylene blue (MB) dye adsorption method[10,11]. Two procedures were applied one for kaolinite and the other for bentonite because of the expected significant of their surface areas. Kaolinite samples 15-105 mg were mixed with 100 mL of 25 ppm methylene blue solution in 250 mL stoppered Erlenmeyer flasks and to insure equilibrium adsorption conditions had been reached, samples together with a control one were kept in the dark for 4 weeks. While for bentonite, 15-45 mg samples were mixed with 100 mL of 50 ppm methylene blue solution and the stoppered flasks were shaken in rotary shaker at a moderate speed for 24 h. The equilibrium concentrations of methylene blue were determined spectrophotometrically with UV-Visible cary 100 in 10 mm silica sample cells and using calibration Beer's law polt at λ = 665 nm.

Treatment of crude oils, degumming: For soybean and corn oils the wet acid degumming procedure was applied using phosphoric acid while the sunflower oil because of its low phosphatide content, was not subjected to such treatment[12].

Alkali refining (neutralization): The alkali refining for the oils was conducted following the AOCS official method Ca 9a-52 (points 1-8) and using Ca 5a-40 for the determination of the free fatty acid content[9]. The oil was washed by hot distilled water, 80 °C, untill the soap content did not exceed 0.04 % (w/w), as indicated by the titration of the wash against hydrochloric acid solution using methyl orange indicator.

Drying of the oils: A common practice had been to dry the oil to about 0.1 % H_2O before the addition of the bleaching earth[12]. This level was satisfied by placing the oil in a closed filtration flask under vacuum, at a tempreture 80-85 °C, with continuous stirring for 0.5 h at which the acceptable level of moisture was reduced as determinded with Karl-Fisher moisture analyzer.

Bleaching procedure: In close variance to set standard procedure (AOCS official methods Cc 8a-52, Cc 8b-91)[9], the heating in a glycerol bath was found to be sufficient to give suitable tempeature range (95-110 °C) and a mechanical stirrer with speed regulator to give an adequate agitation rate. The materials were placed in a 150 mL three-necked round bottom flask and continuously purging the oil with a stream of oxygen-free N_2 gas to remove oxygen during the bleaching process.

The bleaching of sunflower, soybean and corn oils was conducted following the recommendations of Patterson[13] and Hodgson[14]. For sunflower oil 0.70 ± 1 g of the prepared oil was placed in the reaction flask and 0.70 ± 0.01 g of the clay was accurately weighed and added to the oil giving 1 % clay dose. The flask was then placed in the glycerol bath at 95 ± 2 °C and a constant slow mechanical strring was started with a stream of N_2 gas passed for 25 min. After filtration while hot, about 50 mL of the oil was collected for colour measurement. The same procedure was followed for soybean and corn oils with 1.5 and 2.0 % clay dose and 95 and 105 ± 2 °C, respectively.

Bleaching isotherms: The bleaching isotherms were conducted using common procedure[14,15] under similar conditions as used in the bleaching experiments except that the temperature was set at 100 ± 2 °C and a dose in the range 0.2-3.0 % (w/w) of the clay, were used.

Colour measurement: The colour was measured[10] acoording to AOCS Official Cc 13b-45 using a Lovibond Tintometer model E.

RESULTS AND DISCUSSION

Characterization of bleaching earths, bentonite samples: The powder XRD data given in Table-1 show the mineral content of the raw and the treated bentonites. It is noted that after the acid-treatment, the montmorillonite contents decrease from minor to trace in all the acid treated samples. Such treatment as reported by others[16], has caused the disruption of montmorlinite structure as revealed by the disappearance of (001) reflection at $2\theta = 5.9°$. More interestingly, the effect of an anorthite is to increase its content in the phosphoric and acetic acid treated samples.

TABLE-1

Sample mineral	BR	BH	BS	BP	BA	BAG	Tonsil	WAC
Quartz	***	***	***	***	***	***	**	***
Montmorillonite	**	*	*	*	*	*	*	***
Kaolinite	*	–	–	*	–	–	–	–
Anorthite	*	*	*	**	**	**	***	–
Muscovite	S	–	*	*	*	*	–	–
Illite	–	–	–	–	–	–	*	–

***Major **Minor *Trace
BR = Raw bentonite; BH = Hydrochloric acid activated bentonite; BS = Sulfuric acid activated bentonite; BP = Phosphoric acid activated bentonite; BA = Acetic acid activated bentonite; BAG = Glacial acid activitaed bentonite; Tonsil = Tonsil Standard 1314 FF bleaching earth; WAC = WAC classic bleaching earth.

The characteristic IR bands for raw Jordanian bentonite are shown in (Table-2). Activation with strong acids causes a clear lowering of the intensity of the montmorillonite characteristic band at *ca.* 1031 cm^{-1}, as indicated by the ascendancy of the quartz band at *ca.* 1091 cm^{-1} mainly with strong acids[17]. This change in the spectra is in complete agreement with the XRD results that indicated the marked depletion of montmorillonite shown in Fig. 1.

TABLE-2
CHARACTERISTIC IR BANDS (cm^{-1}) FOR RAW
JORDANIAN BENTONITE

IR band (cm^{-1})	Mineral	Assignments*
3624 sh	Montmorillonite	OH stretching vibr.
3691 sh	Kaolinite	OH stretching vibr.
1094 sh	Quartz	Ass. Si-O stretching
1032 s	Montmorillonite	Si-O-Si stretching
914 w	Montmorillonite	OH deformation
872 w,sh	Anorthilte	OH deformation
790 m	Quartz	–
51 m	Montmorillonite	Si-O bending
467 m	Montmorillonite	Si-O-Si bending
429 m	Montmorillonite	Si-O-Si bending

*Ref. 14, 16, 18

The XRF data show that the raw bentonite is of intermediate swelling capacity, since it contains both calcium (CaO~0.91 %) and sodium (Na$_2$O~ 0.68 %)[3]. The changes in composition after acid activation expressed as ratios relative to silica are given in Table-3. This shows the chemical composition of the bentonite when treated with different acids and their ratios.
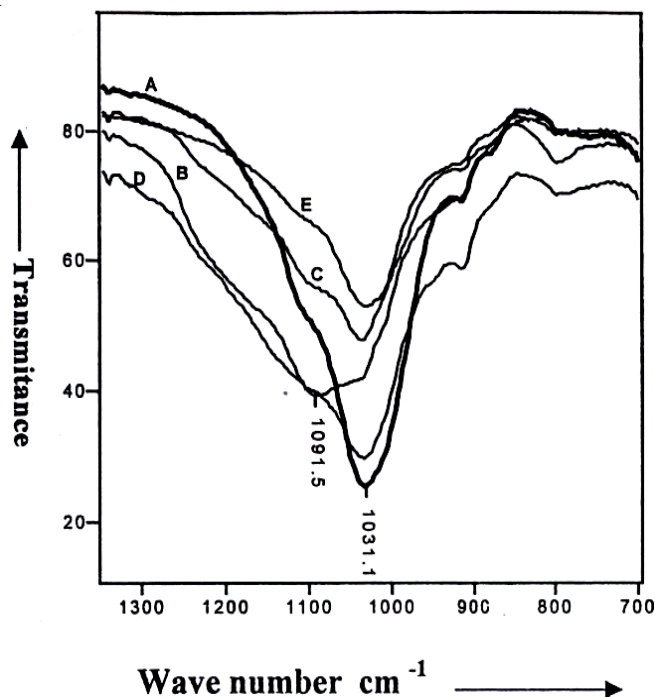
Fig. 1. FTIR spectra of Joranian bentonite samples showing the distinct montmorillonite band at 1031.1 and the quartz band at 1091.5 upon acid treatment, A: BR, B: BH, C: BS, D: BP, E: BAG. (see caption to Table-1 for abbreviations)

The activation causes an extensive dealumination from raw benonite particularly in the case of hydrochloric acid and sulfuric acid treated samples. The general effect of the acid on the alumina and the iron oxide, which may contribute largely to the structure of the octahedral layer in montmorillonite, follows the depletion order.

$$BH > BS > BP \sim BA \sim BAg^1$$

Exchangeable (Ca^{2+}, Na^+) cations, that affect the swelling properties of montmorillonite, have both decreased with the Ca^{2+} showing the largest reduction (Table-3).

Moisture content (MC) increased after acid treatment (Table-3) expect for acetic acid treated samples. This increase may due to the enhanced water adsorption power of the clay after activation. Similarly, loss-on-ignition (LOI) of all treated samples showed an increase. The loss-on-ignition may be considered as an indication of total water, *i.e.* bound and unbounded, while moisture content is a good measure of unbound water.

TABLE-3

Sample ratio	BR	BH	BS	BP	BA	BAG	WAC	Tonsil
SiO_2	58.090	68.31	63.700	52.820	59.210	59.330	57.640	55.0100
Al_2O_3/SiO_2	0.256	0.132	0.182	0.227	0.238	0.236	0.167	0.1970
Fe_2O_3/SiO_2	0.155	0.051	0.097	0.144	0.144	0.131	0.130	0.0820
MgO/SiO_2	0.057	0.015	0.029	0.035	0.046	0.051	0.015	0.0370
CaO/SiO_2	0.016	0.007	0.012	0.009	0.012	0.011	0.002	0.0231
Na_2O/SiO_2	0.012	0.010	0.010	0.121	0.108	0.108	0.000	0.0000
K_2O/SiO_2	0.043	0.028	0.035	0.043	0.042	0.043	0.000	0.0480
$(Al_2O_3+Fe_2O_3)/SiO_2$	0.411	0.183	0.279	0.371	0.382	0.368	0.279	0.2800
$(Na_2O+K_2O)/SiO_2$	0.043	0.028	0.035	0.043	0.042	0.043	0.000	0.0480
$(CaO+MgO)/SiO_2$	0.072	0.022	0.041	0.044	0.057	0.061	0.017	0.0600
$(CaO+Na_2O)/SiO_2$	0.027	0.017	0.021	0.021	0.021	0.022	0.002	0.0230
$E.I*/SiO_2$	0.019	0.470	0.731	0.460	0.681	0.652	0.141	1.2700
MC**	6.620	10.520	8.350	9.510	3.920	6.680	13.330	7.3400
LOI	8.870	13.040	11.070	9.050	9.810	10.290	22.560	23.1800
MC/LOI	0.075	0.810	0.750	1.000	0.400	0.650	0.580	0.3300

*E.L = $(MgO+CaO+Na_2O+K_2O)$
**Obtained from Sartorious moisture analyzer at 110 ºC.

The ratio MC/LOI gives an idea about the relative amount of unbounded water to total water in structure. It is interesting to observe that this ratio shows an increase for hydrochloric acid and phosphoric acid treated samples and decreases with the others. It appears that there is general decrease in the amount of bounded water, which is quiet clear in the case of the phosphoric acid treated sample, in which activation seems to have removed almost all bound water from the crystal structure.

The methylene blue method gave an approximate indication of the general effect of acid activation on surface areas, especially for the strong acid activated samples[19,20]. Thus the surface areas of raw bentonite *ca.* 325 m^2/g was founded to be comarable to that obtained from the ethylene glycol method[3]. It appears that there is a considerable decrease in the surface area after treatment with stong acids, falling to *ca.* 93 m^2/g with the hydrochloric acid sample.

The surface areas in m^2/g follow the sequence :

WAC (674.1) > Tonsil (328.7) > BR (324.9) > BAg (312.8) >
BA (281.6) > BP (180.1) > BS (176.4) > BH (93.2)[1].

As to be expected, treatment with the weak acetic acid in small changes and the surface area remainted close to the untreated bentonite.

Kaolinite samples: In general, the acids have affected kaolinties much less than bentonites. This is clearly revealed by the XRD results, which show quartz as major, kaolinite and alunite as minors and muscovite as trace for the treated and untreated kaolinites.

Also, their characteristic bands for kaolinite have not been altered. Furthermore, the compositions as given by XRF results reveal that only minor changes have occurred upon acid treatment.

The moisture content is low in comparison with bentonites, which reflects the lower adsorption capacity of kaolinite relative to bentonite. The MC/LOI ratio shows an increase in the unbounded water and demonstrating that acid treatment has somewhat decreased the relative amounts of bound water with the greatest effect observed in the case of sulfuric acid sample. The unbound water in the samples decrease along the sequence:

KS (0.213) > KH (0.087) > KAg (0.061) ~ KP (0.057) > KR (0.03)[2].

Kaolinites that have lower surface areas than bentonites have also been affected by the strong acid treatment. The most noticeable effect is that of hydrochloric acid as shown by the following order of decreasing surface areas:

KR (62.8) > KAg (58.2) > KS (47.3) > KP (42.3) > KH (37.0)[2]

Oil bleaching: The measured red colour is used for calclating the relative bleachabilities (RB) of samples as defined by:

$$RB = \frac{(Cc - Cx)}{(Cc - Cf)} \times 100\%$$

where Cc and Cx are the lovibond red colour index values for the crude and the clay-bleaching oil respectively, while Cf is that for the clay that has the maximum reduction in the oil colour as indicated by (Cc-Cf) value.

For the industrial clays, WAC is found to be the best clay for the bleaching of sunflower, soybean and corn oils with red colour reduction of 52.4, 71.0 and 71.8 %, respectively.

In general, the acid activated Jordanian bentonites and especially the sulfuric acid sample performed comparably with industrial cays as shown in the histogram displayed in Fig. 2.

In the case of soybean and corn oils the sulfuric acid activated clay had RB 90 % with reference WAC, while for sunflower oils it is similar to WAC and better than Tonsil. In the case of soybean and sunflower it is similar to WAC and better than Tonsil. In the case of soybean and sunflower oils the hydrochloric acid activated clay showed close RB value to that of the sulfuric acid, but was quite inferior in the bleaching of corn oil. The phosphoric acid activated clay has performed similar to the sulfuric acid traeted clay in the bleaching of sunflower oil slightly less for corn oil but was quiet inferior for soybean oil. Interestingly, the acetic acid activated clays performed quiet well, comparing favourably in the bleaching of corn and sunflower oils and even for soybean it has RB more than 80 %.

Although the acid activated kaolinite showed in general enchanced bleachability yet they fall well behind when compared with the bentonite clays as shown in Figs. 3 and 4. This rules out their use in the bleaching of vegtable oils.
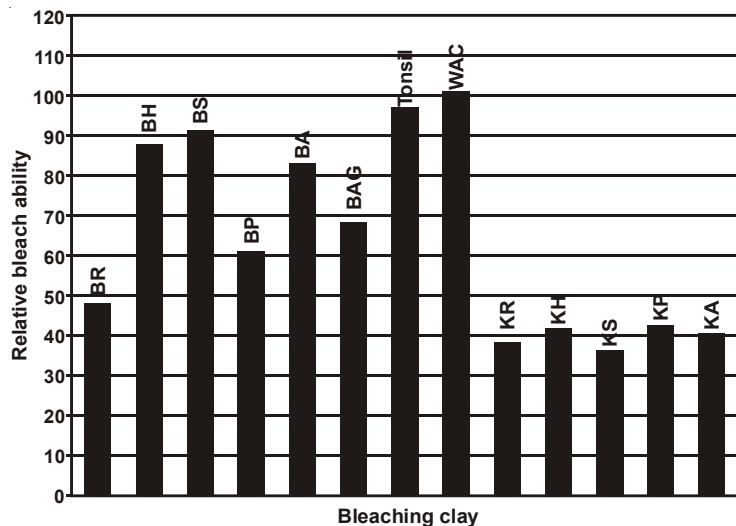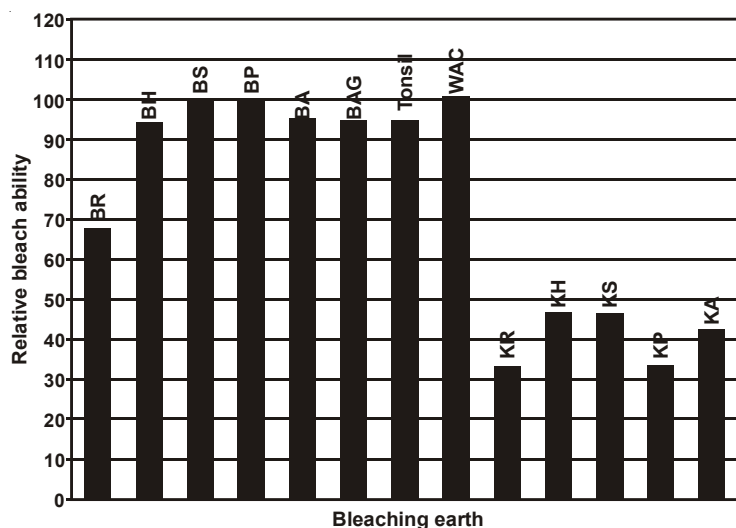
Fig. 2. A histogram showing the relative bleaching of the clays with sunflower oil rela-
tive to WAC-classis as 100 %. The initial colour values for the oil (4.2 R, 40.0Y)
and for WAC bleaching oil (2.0 R,17.0), where: Br Rwa bentonite. BH:
Hyrochloric acid activated bentonite. BA: Acetic acid activated bentonite. BAG:
Glacial acid activated bentonite. Tonsil: Tonsil standrad 1314 FF bleaching earth
WAC: WAC classis bleaching earth. KP: Rwa, Kaolinite. KH: hydrochloric acid
activated kaolinite. KS: Sulfuric acid activated kaolinite KP: Phosphosric acid
activated kaolinite. KA: Acetic acid activated kaolinite.

Fig. 3. A histogram showing the relative bleachability of the clay with soyabea oil
relative to WAC-Classic as 100%. The initial colour values for the oil (7.6 R,
40.0Y) and for WAC bleached oil (2.2R, 20.0Y). (See caption to Fig. 2 for
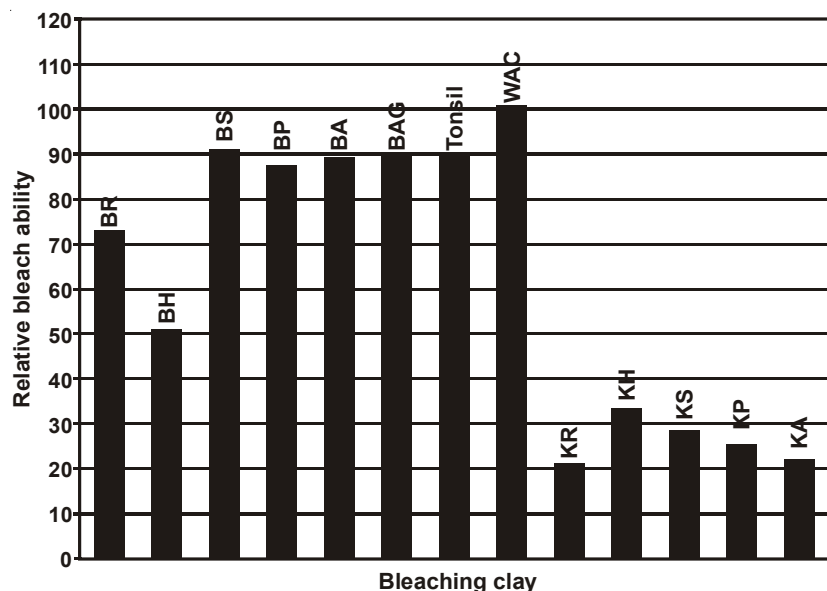abbreviations)

Fig. 4. A histogram showing the relative bleachability of the clays with corn oil relateive to WAC-Classic as 100%. The intial colour values for the oil (8.5R, 40.0Y) and for WAC bleacjed oil (2.4R, 25.0Y) (see caption to Fig. 2 for abbreviations)

Bleaching isotherms: In order to understand the variation in the performance of the bleaching clays. Their adsorption isotherms are studied. The beaching of oils by activated clays is known to be best-investigated using freundlich adsorption isotherm[18].

$$(C_c–C_x)/m = K (C_x)^n \qquad (2)$$

or in its logarithmic form:

$$\log (C_c–C_x)/m = \log K + n \log C_x \qquad (3)$$

where C_c and C_x are defined as in eqn. 1 and m is the wt % of clay-to-oil.

The parameters K and n are good indicators of the adsorption capacity and sterngh of adsorption, respectively[12]. Therefore in order to explore the behaviour of the acid Jordanian bentonites *vis-á-vis* the industry commercial earths, *viz.*, Tonsil and WAC, recourse is made to the adsorption isotherms for the bleaching of locally extracted sunflower oil. The bleaching of the highly pigmented soybean oil was also studied by comparing the best performing clay WAC with the sulfuric acid activated clay that has the highest relative bleachability.

The freundlich adsorption plots for sunflower oil, shown in Fig. 5, reveal that the Jordanian clay samples activated by sulfuric acid, phosphoric acid and glacial acetic acid have high adsorption strength with n value 2.4, 2.1 and 2.7, respectively, as compared with WAC 1.2 and Tonsil 1.5.
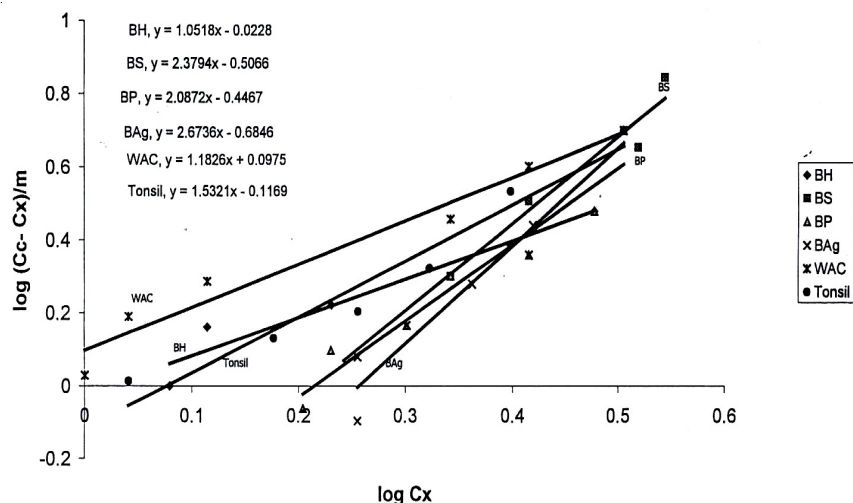
Fig. 5.   Sunflower oil bleaching Isotherm-plots. (See caption to Table-1 for
        abbreviations)

Interestingly, the hydrochloric acid activated clay with n = 1.1 behaves similarly to WAC. Also the adsorption capacity K = 0.95 falls between the Tonsil value of 0.76 and 1.25 for WAC. All the other activated Jordanian clays, on the other hand, had quite low k values of 0.31, 0.36 and 0.21 for the sulfuric acid, phosphoric acid and glacial acetic acid activated samples, respectively Jordanian clay, which is close to those of WAC, may be due to the extensive dealumination occurring with this sample.

Such clay parameters as dealunmination, calcium ion content, moisture content, acidity and surface area are quiet valuable in evaluating their performance in the bleaching of vegetable oils[6,13,14]. Interestingly as revealed by the data in Table-4 which shows the Freundlich parameter obtained from sunflower oil bleaching for the acid activated samples with the clay charactersitic propereties for comparison purpose show that for the bleaching of sunflower oil, the general trend of increased bleaching capacity with these prameters is opposite to that for adsorption strength. The exception in the clay acidity, where both parameters increase, but not for sulfuric acid activated clay that has the lowest capacity with quite high adsorption strength.

With the crude soybean oil the best Jordanian clay, *viz.*, the sulfuric acid activated, gives Freundlich plot closely similar to the best performing commercial clay WAC as shown in Fig. 6. The adsorption strength 0.40 and adsorption capacity 1.31 for the former compare well with 0.52 and 1.33 for the later, resectively.

TABLE-4

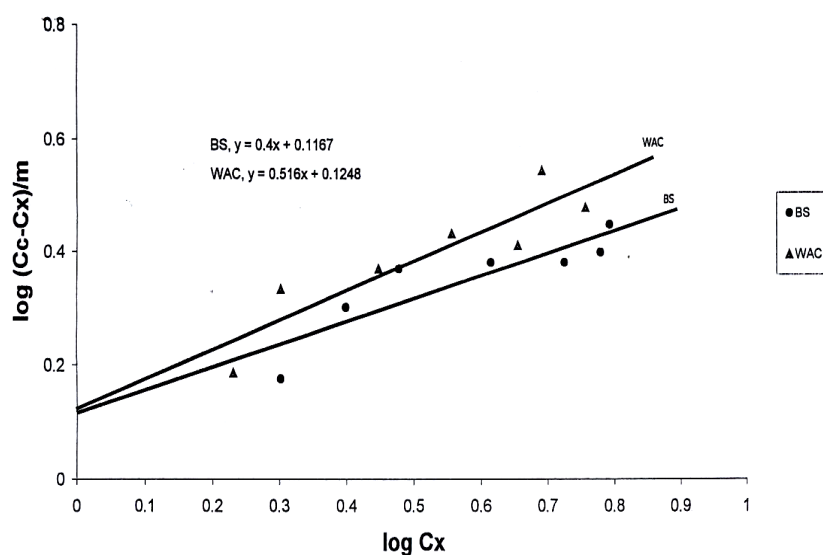| Sample ratio	K	n	Surface area (m$_2$ g^{-1})	Acid value (mg KOH/g clay)	MC/LOI	CaO/ SiO$_2$	Al$_2$O$_3$/ SiO$_2$
BH	0.95	1.1	93.2	36.5	0.81	0.0069	0.132
BS	0.31	2.4	176.4	40.6	0.75	0.0115	0.182
BP	0.36	2.1	180.0	29.5	1.00	0.0087	0.227
BAG	0.21	2.7	312.8	11.3	0.65	0.0110	0.236
WAC	1.82	1.2	674.0	32.0	0.58	0.0024	0.167
Tonsil	0.76	1.5	328.7	16.5	0.33	0.0231	0.197

Fig. 6.   Soybean oil bleaching isotherms plots (See caption to Table-1 for abbreviations)

In general, the activated Jordanian bentonite show quite high adsorption strength with relatively low adsorption capacity except for the hydrochloric acid activated sample that behave in a similar way to the high capacity industry commercial bleaching earths.

As revealed by the relative bleachability and bleaching isotherms, the activated Jordanian bentonite perform generally quite well in the bleaching of sunflower oil and the sulfuric acid treated sample is the best in the bleaching of sunflower oil, soybean and corn oils. For the soybean oil the sulfuric acid treated sample has adsorption capacity and adsorption strength quite close to the industry standard, WAC but not for sunflower for which the hydrochloric acid treated sample show the best values. Because of their rather high adsorption strength, the activated Jordanian clays seem to be best suited in the bleaching of the highly pigmented vegetable oils, especially in the initial stages, to be followed by high capacity clays for finishing.

ACKNOWLEDGEMENTS

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