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Ability of Certain Clays in Decolarization of Lubricant oil

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Abstract

Various types of clays (namely, yellow bentonite, brown bentonite, and Kaolin) of Iraqi Kurdistan have been used for decolorizing dark oils and compared with activated China clay and Iranian clay called (Rose); which are largely used for the industrial processing of mineral oils. Chemical analysis has been made for all clay samples for determination of their chemical compositions then different amounts (percentage) of each clay (3-11%w/v) have been used for the decolorizing process of the lubricant oil, that manufactured at Sulaimani city, at fixed temperature and particle size. Later, studies of different temperatures (150,200,250,300°C) at fixed amount of clay have been carried out to show the temperature effect on adsorption process. The decolorizing efficiency of clays was found to be in the following order; China clay, Iranian clay, Kaolin, yellow clay , brown clay. Also the effect of acid has been studied to activate and improve the adsorption process in the decolorization of dark lubricant oil. The adsorption isotherms data obtained were found to follow the Freundlich adsorption isotherm model.

Keyword: Bentonite, Kaolin, bleaching, mineral oil, adsorption.

Introduction

The importanceof the application of adsorption process in the refining is for the removal of undesirable, highly colored materials of an asphaltic or resinous nature from petroleum fractions. This application is almost as old as the petroleum industry itself. Bone char was used originally, first for the decolorizing of kerosene and finally for heavier oils, but was later displaced by fuller's earth which was found in 1893to be effective for refining steam cylinder stocks[1]. The removal of asphaltic or resinous materials from lubricating oil stocks by acid - activated bentonite, fuller's earth and bauxite is still probably the most important commercial application of adsorption refining [2,3] . However, bentonite is one of the abundant types of soil, its mining and processing is very simple. The mines are open pit and bentonite is crushed, dried, pulverized or

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screened for granular products, classified, and bagged then loaded for many industrial applications [4,5). Bentonite was used as low cost materials in Silica, crude oils and fats as they have drastic power to reduce the levels of colored pigments substances such as carotenoides and chlorophyll, and also residues of phosphatides, soaps, traces of metals and oxidation products. These trace components can have a negative effect on the course of further processing and on the quality of the final product. Dosage of several adsorptions agents should be adapted to ensure the removal of these specific substances [6] However, as a result of widespread use of clay the regeneration of spent bleaching clay by heat treatment in a gas stream is possible and was performed in a thermogravimetric analysis system by SHU - CHEN HOU [7], and Abdulhamid Boukerrou [8].In order to search for method of manufacturing better activated clay in an efficient way, attempt was made to activate local samples of bentonite of Garmian in the Kurdistan region of Iraq. SUN KWANSON[9,10]developed the useful data in deciding activation conditions. The effect of acid activation on some physicochemical properties of bentonite such as chemical composition, cation exchange capacity (CEC), mineralogical distracting , specific area and specific micro pore-meso pore volume by sulphuric acid activation was studied by muserref [11].It was shown by N.Yildiz and A. Calimli[12]that structural changes were observed by treatment of three Turkish bentonite with Na_2C0_3 and H_2S0_4 These changes showed differences depending on exchangeable cations clay type and the ratio of additives. In addition investigation of the surface acidity of a bentonite modified by acid activation and also thermal treatment have been achieved by Muserref and others [13]the obtained result showed that the variations in specific pore volume , specific surface area and surface acidity are functions of the mass percent of the acid used in activation .Huseyin Topallar[14]showed in his study on the bleaching of sunflower- seed oil with bentonite that the forces between the adsorbent and adsorbate appear to be Van der waals forces and the type of adsorption is physical adsorption, it was shown that the Freundlich equation was more applicable than the Langmuir equation to the experimental adsorption isotherms. M.Razae[15]determined the optimum bleaching parameters for decolorizing of edible oils using acid activated bentonite and showed that the acid concentration, activation time and temperature affect the bleaching power. Nevertheless alkaline activation of the montmorillonite [16]clay has been studied and it has been shown that sorption capacity of obtained samples enhance with enhancing retention clay time of activation and alkaline concentration in activation solution. Therefore the main object of this study is that; three different local samples of clays from Kurdistan region of Iraq were used for decolorization and were treated with acids, H_2SO_4 and HCI, to enhance their bleaching power in the process of lubricant oil, which is produced in Sulaimani city of Iraq and also study the isotherms model that fit the experimental findings.

Experimental preparation

a. Oil sample: the oil sample was taken directly from the oil refinery (Asia company) after a stage of acid treatment, the general properties of which were as follows

Specific gravity at 15.6 °C = 0.8878
A.P.I gravity= 27.88
Viscosity at 100 °C/Cst= 11.65
Flash point (c.o.c) °C= 230
Pour point°C= 0.0

b. Kaoline yellow bentonite and brown bentonite were taken from Garmian beds in Kurdistan crushed and screened. The mesh sizes were chosen to be 200um.

Instruments

a. spectrophotometer model Jenaway 6405 uv /vis. Single beam.
b. Flame photometer, Jenaway PFP7, model 9003.
c. Atomic absorption spectrophotometer model, braic WFX - 120.
d. Stuart rotary evaporator RE 300. UK. e. Stuart water bath RE 300B /UK.

Chemicals and reagents

All chemicals and reagents used were from Merck and Fluka and all are of analytical grade: hydroflouric acid, ammonium molybedate, quinoline, EDTA, E.B.T, ethanol, ammonium thiocyanate, ammonium ferric sulfate, hydrochloric acid, sodium hydroxide, sulphuric acid.

Calibration curves

Calibration curves have been obtained for the determination of chemical composition using standard materials (GSB G 62020 - 9) and standard international conditions for atomic absorption [17] calibration curve for Na and K by flame photometer are with R^2=0.999 and by Atomic absorption spectroscopy for Fe with R^2 = 0.9928.

Procedures:

a. Fixing λ_{max} : The dark oil was diluted ten times with light naphtha and scanned in thespectrophotometer through wavelength light (350 - 800)nm. The spectrum plotted, and the wavelength at which maximum absorbance occurs detected, as shown Fig (1).

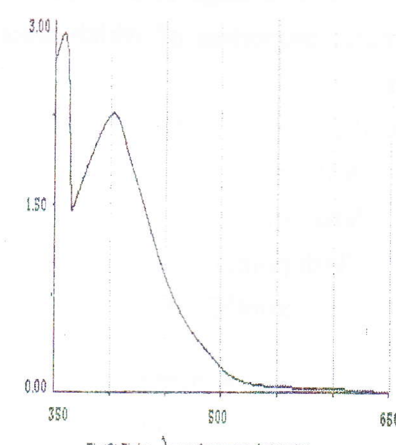
b. Clay treatment : 100 ml. dark oil was mixed with the adsorbent (3 - 11%W/V) in a round bottom flask of the rotary evaporator, the temperature of the oil bath fixed at the study temperature (150, 200, 250, and 300 °C).The vacum pump was connected and mixed at this temperature continued for one hour and the slurry was cooled to 90 - 100 °C, filtered through a special cloth filter, the filtrate was diluted with light naphtha ten times, the absorbance was measured using 410 nm wavelength light.

c. Acid activation :100grams of the ground clay (dry basis, 110°C) were placed in a one liter flask, heated by an electrical mantle ,400ml of 1:3.2(v/v) hydrochloric acid (d=1.16 g/ml, %w/v(HCl)=32) solution were added so that the HCl concentration is 10% (w/v) and the dry acid /clay ratio (w/w) was 0.40 kept for 24 hours then refluxed at 80 C° for 3 hours with constant stirring while hot, the clay suspension was transferred to a 5-liter beaker and washed by decantation with distilled water for at least 5 times until it is Cl^- and Fe^{+3} free (pH =3.5-4.0). The acid activated was filtered in a

Buchner funnel, dried at 110 °C and reground to pass No. 200 mesh sieve (74mm)for the decolorization test.(the same method repeated for 20%HCl ,10% and 20% H_2SO_4)*.

d) Oil and active clay treatment :100ml dark oil was mixed with the active clay (3-11%w/v) in a round bottom flask of the rotary evaporator the temperature of the oil bath fixed at 150 °C.The vacuum pump connected, mixing continued for one hour and then the slurry was cooled to 90-100 °C, filtered through a special cloth filter.

The filtrate was diluted with light naphtha ten times; the absorbance was measured using 410nm wave length light.



Fig(2) Fixing λmax by spectrophotometer

* (i) 40o ml of 1:1.6 HCI acid added to 100gm clay so that the HCl concentration is 20%(w/v) and the dry acid/clay ratio (w/w) was 0.80.

(ii) H_2SO_4 =98% w/w H_2SO_4,d=1.84 400ml of 1:10 acid added to 100gm clay so that the H_2SO_q concentration is 10%(wlv) and the dry acid /clay ratio (w/w) was 0.40

(iii)400 ml of 1:5 acid added to 100 gm clay so that the H_2SO_4 concentration is 20%(w/v) and the dry acid/ clay ratio (w/w) was 0.80

Procedures for chemical compositions analysis,the procedures were adapted as mentioned on ref [18].

a. determination of SiO_2 : by gravimetric method as (quinoline silico molybdate) .

b. determination of Al_2O_3 : by gravemtric method as (aluminum oxinate).

c. Determination of Fe_2O_3 : (i) by sepctrophotometric method as (thiocyanate complexation) (ii) by atomic absorption spectrophotometer .

d. Determination of CaO and MgO : by titration method (complexometric titration with EDTA). Determination of otal carbonate :as free carbonate .

Results and discussion

The chemical compositions of all clay samples used in this work are shown in table (1). This table shows that the various components of the chemical analysis are between 99.12 and 99.8% which indicates the correctness of the analysis and the lack of considerable content of other components as occasionally observed in clays. The analysis shows great difference in $Si0_2$, $A1_20_3$, and $CaC0_3$ content of the local clays (brown clay, yellow clay) in comparison with Kaolin, Iranian clay and China clay, that may be due to the contamination of local clays with organic substances in addition to their different genesis composition, that brown and yellow clays are dominantly CaC03 content. The presence of the above could effect on their decolorization abilities of dark oils. The results shown in Fig (2) indicate clearly the efficiency of clays in the decolorization in the following order: China clay>Iranian clay>Kaolin clay> yellow clay>brown clay. As $Si0_2$ and $A1_2O$content increase and $CaCO_3$ decreases the efficiency of decolorizing increases. While the results shown in Figures. (3 - 7) indicate that, increasing temperature causes to increase the decolorizing abilities of the clays. Chemically speaking the speed of chemical reactions will double for every increase of ten degrees Celsius. However, the rate of oxidation of the oil is increased approximately three times for each increase of the same ten degrees Celsius [19]. Hence, the oil temperature in the process and operation should be kept at optimum. Vacuum helps to protect the oil from oxidation by contact with air at high temperature.

Table (1) The chemical composition of clay samples.

Costituents	Brown clay	Yellow clay	Kaolin clay	Iranian clay	China clay
SiO_2	16.8	7.46	47.07	55.7	53.42
Al_2O_3	3.33	3.86	35.05	13.13	10.06
Total CaO & MgO	8.34	8.81	1.37	3.77	10.18
K_2O	0.28	0.57	0.1	0.3	0.12
Na_2O	1.82	2	1.6	1.4	0.7
$CaCO_3$	30.86	42.23			
Fe_2O_3	10.57	8.81	1.34	1.34	3.58
TiO_2	0.27	0.21	1.19	0.33	0.52
Uncombined water	7.35	4.37	0.08	15.77	11.79
Loss on ignition	19.5	21.3	12	7.48	9.4
Total	99.12	99.62	99.8	99.22	99.77

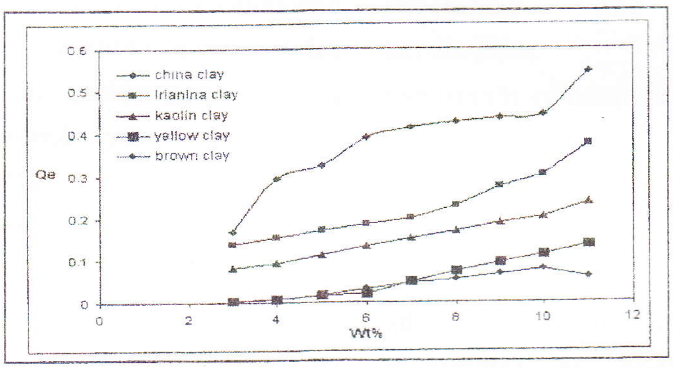


Figure (2) comparison of adsorption on different clays at 200 °C (Where Qe = X/m)

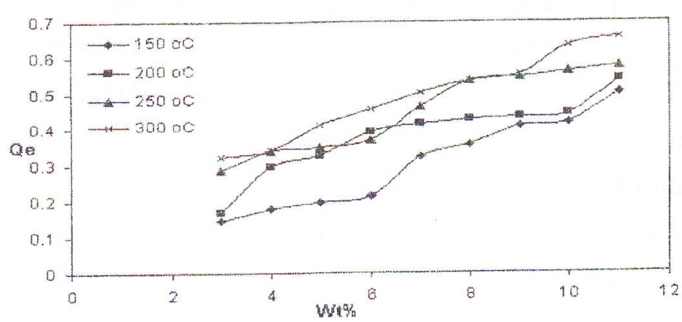
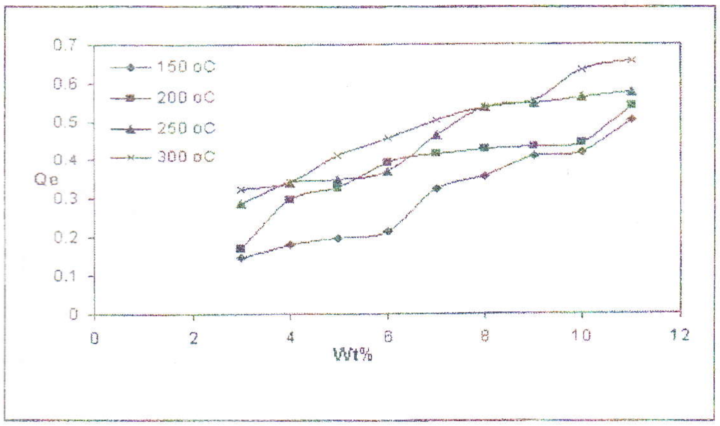
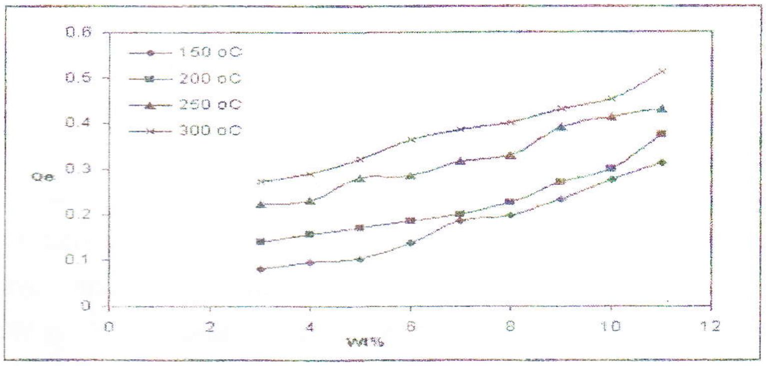


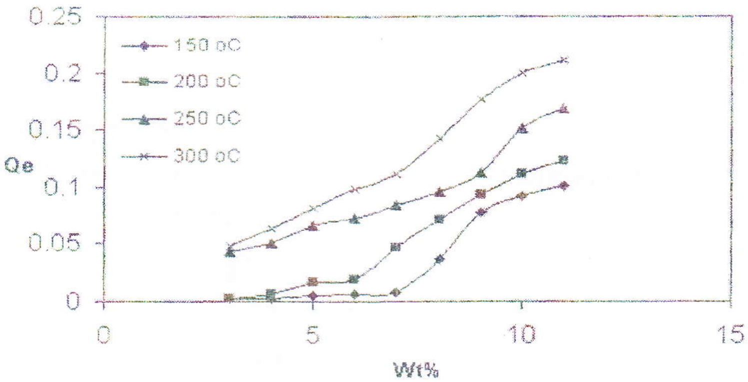
Figure (3) Comparison of adsorption by China clay at different temperature(where Qe=x/m)



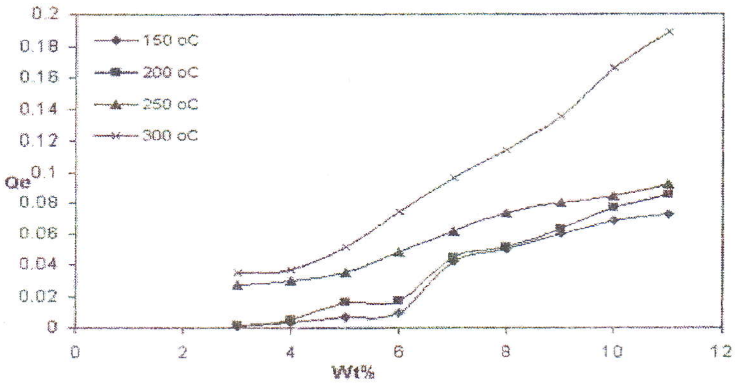
Figure(4)Comparision of adsorption by Iranian clay at different temperature (where Qe = X/m)



Figure(5)Comparision of adsorption by Kaolin clay at different temperature (were Qe = X/m)



Figure(6)Comparision of adsorption by Yellow clay at different temperature (where Qe = X/m)



Figure(7)Comparision of adsorption by Brown clay at different temperature

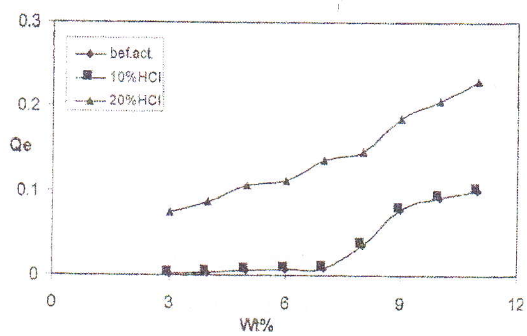
(were Qe = X/m)

The clays were applied as powders of 200 mesh size and mixing was performed either with the dry clay or as mud or pulp with excess water. The last one is more convenient as the subsequent conversion of the water to steam helps to protect the oil from oxidation by air at high temperature. Then cooled slurry was mixed with light naphtha which makes easier the removal of spent clay by filter or may the diluted mixture is subjected to dewaxing process after removal of clay. On lighter lubricating distillate SAE 30, SAE 20 and lower the slurry was filtered without naphtha at a temperature of 90 - 100 $^{\circ}$C. The time required for this treatment is generally about half to one hour and no great advantage was recognized for longer period treatment.

The clay treatment was often applied after acid treatment, which serves to neutralize and to decolorize at the same time. This is useful because neutralization of highly viscous stocks containing the products of the reaction with sulphuric acid almost impossible with aqueous alkali that emulsify the stock contents.

Therefore, different clays with different level of acid activation were applied to adsorb colored compounds in lubricant oil. Decolorizing capacity is quoted here as the quality criterion of the activated products, as it is an important index to evaluate the efficiency of the activated clays in the decolorization. Figure (8) to [10] show the improvement of the efficiency of the activated clays using 10% and 20% HCl and also results shown through figure (11) to (13) indicate the improvement of the efficiency of the activated clays using 10%,and 20 %H_2SO_4. However, the adsorption capacity of the acid (HCl or H_2SO_4.) activation of the clays was significant; As the intensity of the acid treatment is increased decolorizing ability for a given oil increases up to an optimum value, but with further treatment there is a decline in that ability. So in this an acid dependence pH may enhances and latter inhibits the sorption processes of adsorbate and in this physicochemical process may accompany by H^{+} and other cations release in

to media. As a consequence of increasing acid % concentration, the exchangeable cation between the 2:1 layer leave first and are replaced by the H^{+} ions; Since only a small quantity of cations Mg^{+2},Fe^{+3} and Al^{+3} leaves from the 2:1 layers, the change in the composition percentage of oxide R_XO_y% {$R_XO = Al_2O_3+Fe_2O_3+MgO$}has been shown small[11]before activation, but as the acid concentration (HCl% or H_2SO_4.%) increases the cation dissolve easily from the clay layers, so the decrease in R_XO_y% is great and has asignificant effect. Again it was observed the variation in specific pore volume , specific area, and surface acidity are modified by function of the acid that is used in activation [13]The variation of the metal cation exchange capacity (CEC) by the R_XO_y% was studied also by Mu~erref [11]and he showed that, as the R_XO_y% decrease by progressing activation, the CEC decreases linearly. Also the disappearance of some of the octahedron, whose in clay structure Mg^{+2} cations in their centers are dissolved, leads to the disappearance of the excess negative charge originating from them and as a result the CEC decrease as the R_XO_y% decreases.In addition to chemical composition and CEC, the specific surface area and micro pore-Meso pore volume of bentonite as important properties that will vary by acid activation and also vary by alkaline treatment The (BET) surface areas of bentonite sample show to decrees [12]with alkali Na_2CO_3 activation because Ca^{+2} ions were exchanged by Na^{+} ion and this caused a decrease in the micro pore area and low surface areas for Na_2CO_3 treated samples, while the surface areas for acid H_2SO_4treated sample has been shown to increase and reach a maximum and then decrease. This is in according to the our experimental results which indicates that the observed chemical structural and composition change after treatment with higher acid concentration caused an alteration of the morphology and consequently the surface properties of the local clay samples.



Fig(8) comparision of decolorization of lube oil by kaoline before and after activaiton by HCl at 150C$^{\circ}$ (were Qe = X/m)

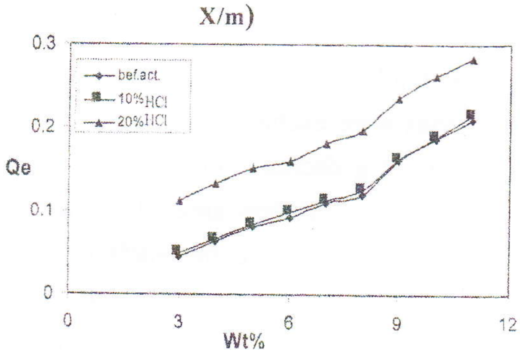
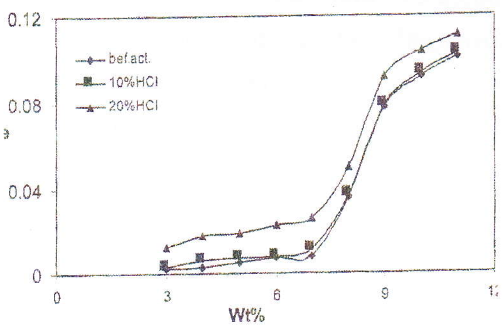
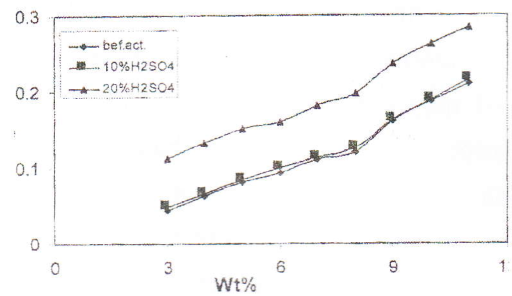


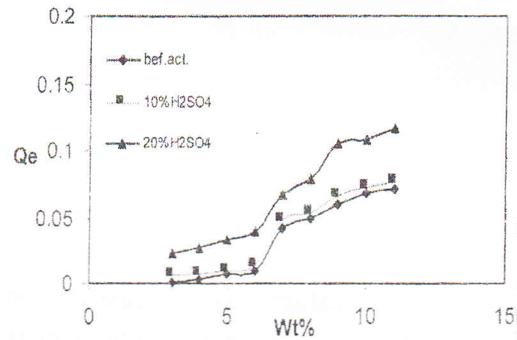
Fig. (9) Comparison of decolorize lube oil by brown clay before and after activation by HCl at 150° C (were Qe = X/m



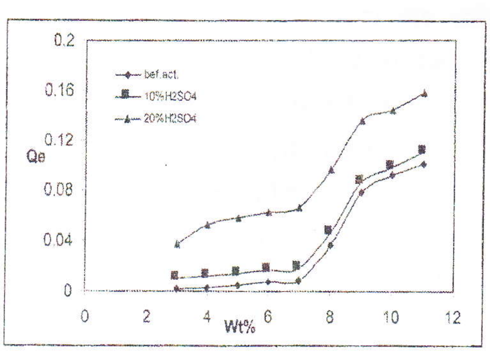
Fig(10) comparison of decolorization of lube oil by yellow clay before and after activation by HCl at 150C$^{\circ}$ (were Qe = X/m)



Fig(11) comparision of decolorization of lube oil by kaoline before and after activation by H_2S04 at 150C$^{\circ}$ (were Qe = Xlm



Fig(12) Comparision of decolorization of lube oil by brown clay before and after activation byH_2SO_4 at150°C (were Qe = Xlm)



Fig(13) comparision of decolorization of lube oil by yellow clay before and after activation by H_2SO_4 at 150°C (were Qe = Xlm)

Adsorption Isotherm:

The experimental data have been tested, for bleaching of lubricant oil on to the Bentonite clays surface before activation and after activation with %10 HCl , on the Langnuir and Freunedich isotherm models [7].

Langmuir:

$$\frac{C}{\frac{X}{m}} = \frac{1}{b X_e} + \frac{C}{X_e} \qquad \text{------} (1)$$

Where X/m is the out of substance adsorbed pre gram of adsorbent, C is equilibration concentration for given amount of substance adsorbed (residual substance) and X_e represent the maximum amount of adsorbed per amount of clay (retention capacity of clay).

Freundich:

$$\frac{X}{m} = k C^n \qquad \text{---------} (2)$$

where K and n are Constants.

Since the absorbance measurements were taken from experiments for the bleaching process of colored solution the relative amount substance adsorbed (X) and that at equilibrium are obtained from the following equations (3 and 4):

$$X = \frac{Ao - At}{Ao} \qquad \text{--------} (3)$$

$$X_e = \frac{A_t}{A_0} = 1 - X \qquad \text{--------} (4)$$

Where A_O is the absorbance of unbleached lube. oil and A_t is the absorbance of bleached oil at time t. Thus by, means of equations (3 and 4) and by writing X_e instead of equilibrium residual substance C, the equations (1) and (2) are rearranged as following (la) :

$$\frac{X_e}{X/m} = \frac{1}{a} + \left[\frac{b}{a}\right] X_e \qquad \text{------} (5)$$

$$X/m = K X^n_e \qquad \text{------} (6)$$

By taking the logarithm of eq. (6) we get eq.(7)

$$\log X/m = \log K + n \log X_e \qquad \text{------} (7)$$

A plot of versus X_e and log X/m versus log X_e should give straight lines and the last squares analysis can be used to calculated the parameters (a, b, K) and (n) from intercept and slope of straight lines of the isotherms. Therefore the experimental data were plotted according to equations (5) and (7) give the relations shown at figs (14-15) which indicate very clearly, according to the above interpretation that the adsorption process before and after activation are in accord with the Freundlich model more than langmuur model (straight lines for freundich, but all points of the Langmuir isotherm plot were present randomly on the line). This means that the Freundlich equation is more applicable than the Langmuir equation to the adsorption isotherms in the process of bleaching of lube oil with all three types of clay used under our experimental condition. This shows also that there are a physical adsorption of one Layer with Van der waals forces between the adsorbent and adsorbate.

Conclusion

Studies show that the local clays (yellow clay and brown clay) have weak decolorizing power comparable with China clay and Iranian clay so they need acid activation to improve their decolorizing ability. While Kaolin can be used as good bleaching clay as its decolorizing power was shown as to be efficient for bleaching lubricating oils, that is probably result from well packed structure that not easily break down during sorption process on the clay surface.

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توانای چەند جۆرە گڵێك لە لابردنی ڕەنگی ڕۆنی چەور کردن

عبدالسلام رحیم کریم و محمد علی عبدالله و کریم جمعه جبرائیل

پوختە

چەند جۆرێکی جیاوازلە لە گڵی کوردستانی عیراق، بە ناوی بنتونایتی زەرد ،بنتونایتی قاوەیی و گلە سەر بەکارهێنران لە کرداری لابردنی ڕەنگی ڕۆنی ڕەش چەور کردن وە بەراورد کران لە گەڵ گڵی چینی چالاککراو گڵی ئێرانی بە ناوی (ڕۆز) کە هەردووکیان زۆر بە فراوانی بەکار دەهێنرێ لە گرداره پیشەسازی یەکانی ڕۆنی کانزاییدا.
لێکۆڵینەوەی کیمیایی تەواو بۆ هەموو جۆرە گڵە بەکار هاتووەکان ئەنجام درا وە بە مەبەستی زانینی پێکهاتی کیمیاوییان و بڕی جیاواز لوم گڵانە بە ڕێژەی (٣-١١٪) بەکار هێنران بۆ لابردنی ڕەنگی ڕۆنێکی چەور کردن کە لە شاری سلێمانی بەرهەم دەهێنرێت ئەویش جێگرکردنی پلەی گەرمی (١٥٠، ٢٠٠ ، ٢٥٠ ،٣٠٠ س) لەسەر کرداری ڕوومژینی گڵەکان بە بەکارهێنانی بڕێکی جێگیر لەو گڵانە . بینرا کە توانای ڕەنگ لابردنی گڵەکان بەم زنجیرەیە :گڵ چینی ،گڵی ئێرانی، گلە سەر بنتونایتی زەرد ،بنتونایتی قاوەیی .هەروەها لێکۆڵینەوەیەکی تر ئەنجام درا لەسەر کاریگەری ترشی بۆ چالاك کردن و چاك کردنی کرداری ڕوومژینەکە پەیوەست بە البردنی ڕەنگی تاریکی ڕۆنی چەورکردنەکە وە لە ئەنجامەکەی کۆڵرایەوە. پاشان ئەنجامی هێڵکاری ڕوومژینەکە لە پلەی گەرمی نەگۆڕاوودا دەریخست کە پەیڕەوی مۆدێلی ڕوومژینی فرندلچ دەکات.

قابلية الاطيان المختلفة في ازالة لون زيت التزييت

عبدالسلام رحيم كريم و محمد على عبدالله و كريم جمعه جبرائيل

الخلاصة

تم استخدام انواع عديدة من اطيان كردستان العراق ، تحديدا(البنتونايت الاصفر ،البنتونايت البني ،و الكاؤلين)في عملية ازالة اللون للزيوت الداكنة وتم مقارنتها مع الطين الصيني المنشط و الطين الايراني المسمى (روز)،اللذان غالبا ما يستخدمان في العمليات الصناعية للزيوت المعدنية.
و قد انجز التحليل الكيميائي الكامل لكل انواع الا طيان المستخدمة لمعرفة التركيب الكيميائي و بعدها استخدمت نسب مختلفة (٣-١١٪و/ح) من هذه الاطيان لازالة لون زيت التزيت المصنع في مدينة السليمانية بثبوت درجة الحرارة و حجم الحبيبات ،كما تم دراسة تاثير درجة الحرارة (١٥٠، ٢٠٠ ، ٢٥٠ ،٣٠٠م)لكميات ثابتة من هذه الطيان على عملية الامتزاز ، وقد كانت تسلسل كفاءة ازالة اللون اللا طيان كالاتى (الطين الصيني ،الطين الايراني ؛الكاؤلين ،البنتونايت الاصفر ،البنتونايت البني).
و كذللك تم دراسة تاثير الحوامض على تنشيط الاطيان و تحسين كفاءتها لازالة لون زيوت التزيت الداكنة ،و اخيرا اضهرت النتائج بان عملية الامتزاز تكون بموجب معادلة (فرندلش).

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