Type and Concentration Effects of some Active Ingredients on the Performance of Bentonite Lubricant Grease

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Abstract

Optimization study was carried out on the materials used in preparation of lubricant grease prepared from bentonite. This study included the selection of the optimum type and percent of quaternary ammonium salts to convert the bentonite from hydrophilic to organophilic; tetraethyl ammonium chloride and tetraethyl ammonium bromide were used for this purpose. It was seen that the quaternary ammonium chloride gave the best results because of the high tendency of calcium ion to react with chloride ion than that with bromide ion; over more it gave a less swelling.

The effect of treated bentonite added in different percentage on the penetration point, NLGI (National Lubricating Greases Institute number), drop point, and water resistance of the prepared grease was studied. It was seen that by increasing the percentage of added organophilic bentonite with the prepared grease caused a decrease in the penetration point which is correspond to increase in the consistency of stiffness of grease. These results were in agreement with the NLGI classification system. It was noticed that by increasing the percentage of added organophilic bentonite in the prepared grease caused an increase in the dropping point and in the degree of water resistance.

Key words: Bentonite, NLGI, Grease Washout.

Introduction:

Bentonite based greases are characterized by high dropping point, good retention to metal and high water resistance. These greases have been used for lubrication of many industrial types of equipments. They have been found suitable for application in steel mills and in glass and rubber plants [1]. The clay greases are showing particular promise as multipurpose materials for service over a wide temperature range; they are, however, sensitive to metal soap greases. In texture, clay base greases are smooth and polished; they are normally transparent [2].

A number of tests are used primarily to ensure uniformity of products while others are intended to predict performance. Grease testing is essentially empirical and the results have to be considered in the light of practical experience. Penetration value is the number indicating the relative consistency (softness or hardness) of grease; grease manufacturers produce a product to certain penetration value for variety of applications [3]. Consistency indicates the softness or hardness of grease. It governed by soap type and content and only secondarily by viscosity of fluid component. Grease varies from semi fluid pourable products to very hard blocked ones [2]. Penetration values measurements are usually taken in (worked) and (unworked) conditions. Worked condition is normally used for quality control and in published standard characteristics. The consistency or penetration test may be carried out on the unworked grease (sample must be manipulated as little as possible when being transferred from the fill package into the test pot) [2]. Greases are classified in the NLGI (National Lubricating Greases Institute) system [4] (oils are classified in the SAE: Society of Automotive Engineers,). The consistency of greases is normally classified by grade system suggested by the NLGI; the normal arbitrary numbering scale devised from most common greases will range from 0 (for soft greases) to 5 (for stiff greases). Beyond the extreme ends of this scale range there are also special additional grade numbers 00 to 000 to donate Simi fluid greases and 6 for very stiff greases, table (1) showing the NLGI grease classification system [5].

Table (1): NLGI Grease Classification System.

Grade number	Structure	Penetration (worked) (mm/10)
000	Semi-fluid	445-475
00	Semi-fluid	400-430
0	Very soft	355-385
1	Soft	310-340
2	Medium soft	265-295
3	Medium	220- 250
4	Stiff	175-205
5	Very stiff	130-160
6	Very stiff	85-115

The ASTM (American Society for Testing and Materials) dropping point is the temperature at which the grease passes from a Simi-solid to a liquid state under the conditions of the test. The drop point is the approximate temperature at which the grease liquefies and is below the melting point of the soap it contains [6]. While drop points are useful for identification and for quality control and specification purposes; they do not indicate maximum service temperature which is generally not less than 40 ˚C below the drop point. For example the drop point of simple calcium grease would be about 100 ˚C but services experience has shown that the maximum temperature for continuous use should be not exceed 60 ˚C [7].Greases in general are subjected to other changes such as bleeding, evaporation, soap phase reorientation ……etc. that markedly affect their performance which occurs well below the dropping point and thus impose more limitations relative to actual performance [8].

A procedure for evaluating the effect of water on the mechanical stability of grease is included as a control test in British Ministry of Defense specification, derived from a MIL (U.S. Military Specifications); it is termed "water stability". Water resistance means the resistance to washing action of water. Small amounts of water can be absorbed by greases with out great harm to the soap structure even when the soap is soluble as in the case of sodium soaps. Larger amounts of water will readily wash away sodium soap greases and although the water insoluble soap greases and non-soap greases are more resistant, the consistency may be reduced very considerably by absorption of water [9]. After a given time, the amount of grease which had been washed out from the bearing is measured by weighing. If large amount of grease is washed out from the bearing the grease will not be suitable for service in very wet conditions [6].

The aim of this research is to select the optimum percentages of ingredients affecting the performance of bentonite grease.

The influencing factors are taken constant in the preparation of grease except the concentrations of reacting ingredients which were optimized.

Methodology

Materials used:

1. Mineral oils stock 60 and 150 furnished from Al- Dora Refinery were selected on the basis that they were widely used in commercial production with specifications listed in table (2):

Table (2) : Specifications of the used Base Oils.

Specifications	Stock 60	Stock 150
Viscosity (cSt) At 40 ˚C At 100 ˚C	60- 90 8- 10	460 minimum 30- 35
Viscosity Index (VI)	95 minimum	93 minimum
Pour Point ˚C	-6 maximum	-3 maximum
Color*	1.5	3
Flash Point ˚C	220 minimum	260 minimum

* The color of oil is measured by a Lovibond apparatus, thus it is a comparison with standard colors; it gives knowledge of products pollution and the difference of physical and chemical properties from international specifications.

2. The used bentonite soil was an Iraqi soil purchased from the State Company for Geological Survey Exploration with the specifications listed in table (3).

Table (3): Chemical Composition and Mineralogy of Iraqi Soil [10]

Metal%	**Range%**	**Average%**
Chemical Composition		
SiO_2	54.61 – 59.83	56.77
$Al2O_3$	13.70 – 16.69	15.67
Fe_2O_3	4.88 – 5.66	5.12
CaO	2.80 – 5.80	4.48
MgO	3.06 – 3.76	3.42
Na_2O	0.65 – 1.80	1.11
K_2O	0.40 – 0.90	0.60
P_2O_5	0.47 – 0.91	0.65

SO_3	0.10 – 1.95	0.59
Cl	0.10 – 1.22	0.57
L.O.I	8.37 – 13.27	9.49
C (Total Carbon)	0.24 – 1.20	0.56
Impurities	0.95 – 1.0	0.97
		Total = 100%
Mineralogy		
Montimorlonite	70 – 85	80
Palygorskite	5 – 10	7
Quartz and Chalcedony	3 – 7	5
Calcite	1.6 – 5.59	4
Apatite	1.4 – 2.2.7	2
Gypsum	0.2 – 3.9	1
Halite	0.2 – 1.8	1
		Total= 100%

3. Quaternary ammonium salts, quaternary ammonium chloride and quaternary ammonium bromide were supplied from Fluka with purity of 98%, anhydrous, colorless, odorless, hygroscopic, free soluble in water and alcohol, and slightly soluble in benzene and ether.

4. The polar liquid used as dispersion agent was methyl alcohol with specific gravity 0.7924, flash point of 65 ˚C.

5. The medium of ionic exchange was 200 ml. of distilled water for every 100 gm.

6. The used additives were 7% wax to raise the solidity and adherence of the grease and 5% asphalt in some greases to increase the viscosity and to be used as a sealant rather than grease.

7. Certain ratios of oil stock 60 (viscosity 67.25 cSt) and oil stock 150 (viscosity 414.73 cSt) were blended at 40 ˚C to get the desired viscosity. The desired viscosity obtained by blending 50% of stock 60 and 50% 0f stock 150. The resulting optimum viscosity was 175 cSt.

Measurements techniques:

1. The selection procedure of the quaternary ammonium salt was carried out according to [12].

2. The penetration point measurements were done according to ASTM-(D 217) [13]. The penetrotmeter used was surBerline, Ablesung 1/10 mm. type.
3. The drop point measurements were done according to ASTM D- 566 [14].
4. Water resistance measurements were carried out according to ASTM D- 1264 [14].

Experimental procedure:

The prepared sample of grease was carried out by three steps, the first step was that a pre-estimated proportion of quaternary ammonium salt [6] and bentonite were mixed in the presence of a suitable volume of water (200 ml.) as a medium for ion exchange and in a temperature fixed that no boiling occurs, it was chosen to be 75 ˚C, accompanied with mixing for 30 min. Then the flocculated clay is dried in oven at 110 ˚C, pulverized, and milled to 200 meshes.

Secondly, and in order to mix the organophilic clay with the base oil, a suitable dispersion agent should be used; methanol was used and the weight ratio was equal to 1%. Then treated bentonite was added to the mixture with continuous mixing (5000 rpm) for 30 min. in order to have a homogenous mixture; some times heat is needed to ensure efficient process and the temperature could be raised up to 40 ˚C. Additional mixing for 30 min. was necessary to sweep away bubbles of air formed during the treatment.

Thirdly, 7% wax was added to raise the solidity and adherence of the prepared grease. Wax addition is respected to be very important to prevent separation between oil and the thickener. Asphalt in a percent of 5% was added in some grease samples to increase the viscosity of the grease and to be used as a sealant rather than grease [11].

Results and Discussions

1. Selection of Quaternary salt:

20-40 % of chloride and bromide ammonium salts were used to treat the bentonite. The swelling measurements indicated that chloride ammonium salt [$(C2H5)_4NCl)$] is more preferable than bromide ammonium salt, for it gave less swelling indicating that the degree of changing the bentonite into organophilic is higher. Fig.(1) shows a comparison between the increases in volume of treated bentonite due to water for both salts used, The upper curve shows the volume increase with ammonium bromide salt while the lower one is with the ammonium chloride salt.

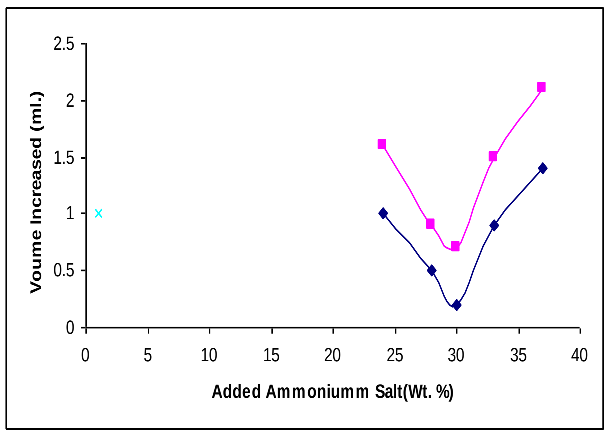

Fig. (1): Swelling measurement of treated bentonite Using Bromide and Chloride Ammonium salts.

It is noticed that 30% of the added ammonium chloride salt proved the best minimum swelling measurement. This fact was ensured by the highest possible separation of calcium ion from bentonite to convert it from hydrophilic to organophilic using atomic absorption spectrophotometer. The behavior of calcium ion exchange process is shown in figure (2) explaining the weight of calcium ion exchanged on 100 gm.

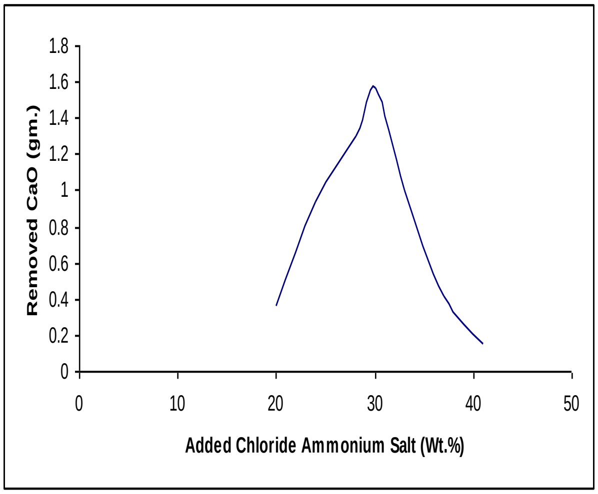

Fig. (2): Calcium ion exchange on treating bentonite with tetraethyl ammonium chloride.

It is seen that the greatest proportion of CaO achieved is 1.57 gm. or 62.8% from absorption spectrophotometer, which indicates that the separation is 50%, therefore, the result of atomic absorption advocate the result noticed in swelling measurements concerning the choice of 30% as optimum ratio that gave the highest possible separation of calcium ion from bentonite.

2. Effect of different percent weight bentonite on NLGI and penetration point:

Table (4) and figure (3) showing the effect of added treated bentonite on the NLGI grade number and the behavior of penetration point of the prepared grease at different percentage of the organophilic bentonite respectively; the grease was prepared using blended oil consisting from 50% stock 60 and 50% stock 150 with a viscosity of 175 cSt at 40˚C.

Table(4): NLGI grade of prepared grease as a function of different weight percentage of treated bentonite with base oil 50% stock 60 and 50% stock 150.

Treated Bentonite (wt.%)	NGLI Number	Penetration point (1/10)mm
10	1	313
15	2	294
20	2	286
25	2	277
30	2	269
40	3	248

It was noticed that by increasing the percentage of added organophilic bentonite in the prepared grease causes a decrease in the penetration point which is correspond to increase in the consistency of grease stiffness. These results are in agreement with the NLGI classification system given in table (1) and (4).

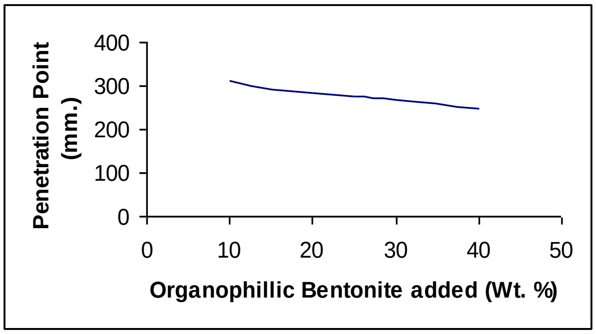

Figure 3: The behavior of Penetration point of prepared grease as a function of different weight percentage of treated bentonite with base oil 50% stock 60 and 50% stock 150

3. Effect of different percent weight of treated Bentonite on the drop point of grease:

Greases samples were prepared using stock 60 alone to optimize the drop point. Results indicate that increasing the amount of organophilic Bentonite produced grease specimens having smooth touch, no odor, moderate consistency in between liquidity and solidity with pale brown color.

The drop point of the prepared greases was also studied as shown in figure (4).

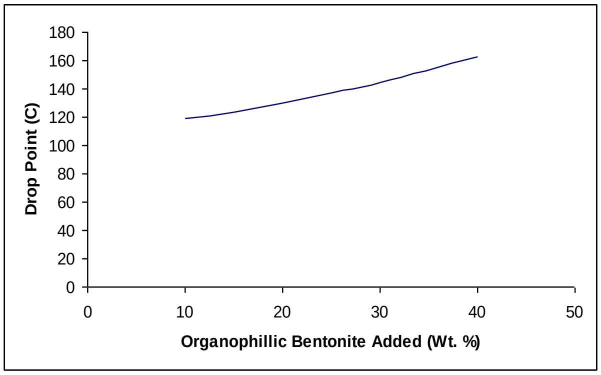

Figure (4): Drop point behavior of prepared grease as a function of different weight percentage of treated bentonite with base oil 100% stock 60

4. Effect of different percent weight of treated Bentonite on water resistance:

Stock 150 was used alone in the preparation of grease to optimize the water resistance. Figure (5) is showing the grease resistance to wash out by tap and raw river water. The upper curve is showing the washed grease with raw river water, while the lower one is showing the washed grease with the tap water.

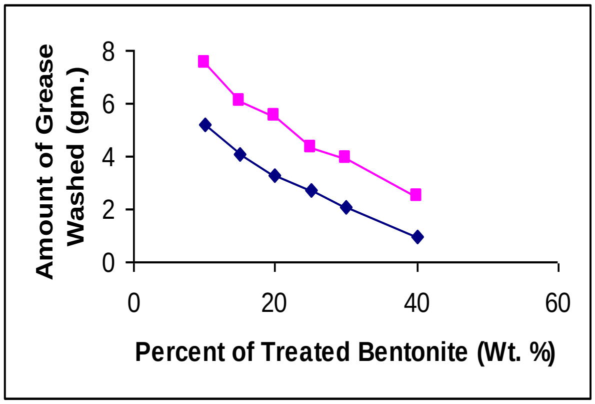

Figure (5): Behavior of Water resistance for prepared grease as a function of different weight percentage of treated bentonite with base oil 100% stock 150.

It is noticed that an increase in the percent of treated organophilic Bentonite causes increase in the resistance to grease washout by water due to the increase in the adherence of the prepared grease. Another reason is that the quantity of oil will decreases and then emulsified in water and as a result grease will be more massive.

The results obtained from washed grease with raw river water (the upper curve) were expected because of salt content which have a destructive effect on grease composition and consequently on the efficiency of grease.

Conclusions:

The following conclusions were drawn from the present work:

1. Iraqi Bentonite was successfully used in the preparation of grease; the used Bentonite was proved to be technically feasible.
2. It was found that by increasing the percentage of the thickener or the viscosity of the base oil resulted in increasing the drop point and decreasing the penetration point (higher consistency) which fitted well with the NLGI specification.
3. It is noticed that an increase in the percent of treated organophilic Bentonite causes increase in the resistance to grease washout by water due to the increase in the adherence.

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