IMPROVEMENT OF CIATIM-201 GREASE PROPERTIES FOR USE UNDER SEVERE CONDITIONS USING SUITABLE ADDITIVES

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ABSTRACT

This work was conducted to improve the undesirable properties of the expired " Ciatim-201". This grease is composed of lithium soap and a mineral oil,its mineral oil that responsible for its deterioration replaced by nutral solvent (SN-500) produced by AL-Dora refinery.

The addition of (10 gm) of neutral solvent (SN-500) to (50 gm) of expired grease was advocated to improve the drop point and penetration.As well as oil separation and corrosiveness in the expired grease were treated by using suitable amount of lithium hydroxide-mono hydrate equal to (0.073 gm),(2wt.%) zinc naphthenate,(2wt%) zinc sulfonate,and (1wt.%) diphenylamine.

INTRODUCTION

Grease as a rule, are viscous plastic products which adhere strongly to the packing material [1,2,3]. Avarity of thickening agents have been proposed for use in such high temperature applications including soap base thickeners, inorganic clay thickeners, and organic thickening agents [4,5].

Lithium soaps in this connection was probably responsible for the advancement by several years of the large scale commercial use of such soaps in the manufacture of lubricating grease[2,6].

Here we must note the appearance of new types of greases based on lithium soaps(Ciatim-201)[7].Cold resistance aviation grease "ciatim-201'' grease is still being used widely today in various types of mechanisms [8].This grease is a general purpose which have a limited operating temperature range[9].

Ciatim –201" grease is one of the aircraft's greases, the originality is Russian [10]. It is composed of a soap thickening agent, dispersed in a base mineral oil, and its containing the necessary chemical additives like anti-oxidant, anti-rust, and corrosion inhibitor [11]. This type of grease may be regarded (by recommendation) as a multi-purpose grease, which is used for ball and roller bearing within the temperature range of (-40 to –100°C) according to the level of military specification (MIL-G-7711A) [11,12].

During along time period of storage, the grease will expose to physical and chemical changes, this is due to the change in its chemical structure, and then, the grease will become out of scale or in other hand, the characteristics of grease are become far from the standard specifications. The true changes occur are the decreasing in Penetration and the increasing in the temperature of drop point, this happened because of oil separation phenomena. Also during storage period, the grease will expose to the occurrence of "syneresis phenomena" which causes to oil separation. Other change occurred in this grease is the formation of acidic compounds as a results of grease oxidation.

The aims of this search are to study the improvement of expired Ciatim-201 properties for use under severs conditions. The main objectives are:

1. Choice of the suitable base oil has an characteristics closely to that base oil of Ciatim-201 grease.
2. Choice of the appropriate chemical additives and their amounts.

EXPERIMENTAL WORK

Expired Grease Ciatim-201

Although this grease is stored under natural conditions such as at an atmospheric pressure and at room temperature, and because of storage this grease for along time period without uses, this result in occurrence some physical and chemical changes in it's structure which causes in loss this grease to it's efficiency during work and so that it would be regarded to be expired and not useful for working.

From the experimental work and from the methods of analysis that are made on the expired grease, we found the following results as shown in table (1).

Table (1) Expired Grease Ciatim-201

Characteristics	Typical Values
Penetration, 1/ 10 mm, Unworked Penetration	251
Penetration, 1/ 10 mm, Worked Penetration (60 strokes)	268
Dropping Point, ^{0} C	192
Copper Corrosion (24 h at 100 ° C)	2C*
Oil Separation (30 h at 100 C)	15.5
Neutralization No. (mg. KOH/g sample)	1.688
Ash Content, % wt.	1.282

** Contain corrosive material*

Base Oil

The better oil that is used in treatment process is called Neutral solvent (SN-500) which is one of the oils that produced in commercial production in Al-Dora Refinery. This oil has characteristics close to the characteristics of oil presene in grease Ciatim-201. Table (2) gives the specifications of the Neutral solvent, as shown below.

Table (2) Specifications of the Neutral Solvent

Specifications	SN-500
Viscosity, at 40° C (cst.)	54 -87
Viscosity, at 100° C (cst.)	5.7-9.6
V.I. (Viscosity Index)	95 minimum
Flash point (° C)	250
Pour point (° C)	-18 maximum
Color	2

Additives

Anti-Oxidation Additives

These additives included phenyldiamine, diphenylamine, or phenylalpha naphthyamine. All of these additives have the same functions, so that, the uses of any one is correct. In treatment process we used diphenylamime, which was added in 1 wt %.

Anti-Rust Additives

In preparation of lithium grease anti-rust additives may be used for this purpose included Zinc, cadmium, or magnesium sulfonates.In treatment of experimental procedure Zinc sulfonate additive is used in (1-3)wt.%.

Oil Bleeding Preventative Additives

Chemical additives include the metals of group IIB of the periodic table of unsaturated fatty acid, such as naphthenic acid. The addition of small proportion of this additive to soap-mineral oil system will stabilize system against "bleeding" and "syneresis".

In our treatment process, we have used Zinc naphthenate to prevent oil bleeding and syneresis. As well as it is used as anti-oxidant, anti-rust and corrosion inhibitors.

Methods of Analysis

Dropping Point (ASTM D2265-78) Test

This test covers the determination of the dropping point of lubricating grease, this point being the temperature at which the first drop of material falls from the cup. So the dropping point is the temperature, at which the grease passes from a semisolid to a liquid state, under the conditions of the test.

Penetration (ASTM D217-82)

The penetration is determined at 25°C by releasing the cone assembly from penetrometer and allowing the cone to drop freely into the grease for 5 second. Penetration of lubricating grease is the depth in tenth of a millimeter, that the standard cone penetrates the sample under prescribed condition of weight, time and temperature

Oil Separation (ASTM D1742-64)

This method covers the determination of the tendency of lubricating grease to separate oil during storage

Corrosive Substances

Copper strip was used for the test method in according to IP test (IP-112/56), this method is intended for the detection of corrosive substances in lubricating grease.

Neutralization Number by Color-Indicator Titration (IP 139/65)

The method of the test is intended for the determination of acidic or basic constituents in petroleum products and lubricants soluble or nearly soluble in mixtures of toluene and iso-propyl alcohol. This method may be used to indicate relative changes that occur in oil during use under oxidizing conditions.

Acid value was determined by dissolving 1g of the sample in 100 ml solvent (blank), which consists of 50% toluene, 49.5% isopropanol and 0.5% distilled water. Then, the mixture was titrated with 0.08-0.1 N alcoholic KOH which prepared by dissolving 3 g of KOH in 500 ml isopropanol. In the titration, phenolphthalein indicator is used by adding 3-5 drops to the mixture (the lubricant and the solvent). In the titration process, the volume of KOH required to neutralize the mixture is measured. Then the acid value is calculated by the following formula:

$$acid\ vlaue\ (mgKOH\ /\ g\ oil) = \frac{(A-B)\times N_{NaOH}\times 561}{w}$$

Where, A = ml of KOH solution required for titration the lubricant. B = ml of KOH solution required for titration the blank. NKOH = the normality of KOH. w = the weight of the lubricant sample.

RESULT AND DISCUSION

Dropping Point

In this set of experiments, the influence of adding the neutral solvent (SN-500) was investigated in order to improve the dropping point of expired grease.

Six experiments were carried out, each 50 gm of grease and different amount of neutral solvent. The results obtained, here, are tabulated in table (3). It is obvious that increasing the solvent added to grease cause decreasing in drop point.

Table (3) Dropping Point Treatment

Weight of Oil Added in (gm)	Dropping Point (^{o} C)
0	192
5	189
6	187
7	186
8	185
9	183
10	181

The results exhibited that (10 gm) of neutral solvent was sufficient for obtain good value of drop point. Figure (1) exhibit the effect of solvent added on drop point.

Penetration

In the suggested oil named neutral solvent (SN-500) was investigated in order to improve the penetration value of expired grease.

Six experiments were carried out, each 50 gm of grease and different amount of neutral solvent.

The results obtained, here, are tabulated in table (4). It is this set of experiments, the influence of adding obvious that amount of neutral solvent is important to achieve the appropriate penetration value.

Table (4) Penetration Results

gm. of Oil Added	Unworked Penetration Value in 1/10 mm
0	251
5	256
6	261
7	264
8	275
9	278
10	280

The results exhibited that (10 gm) of solvent was sufficient for obtain the required penetration value. Figure (2) exhibit the effect of solvent added on penetration value.

Oil Separation

In this set Zinc naphthenate" was investigated in order to improve the oil separation of experiments, the influence of adding "of expired grease.

Six experiments were carried out, each 50 gm of grease and different amount of zinc naphthenate added.

The results obtained, here, are tabulated in table (5). It is obvious that increasing the amount of zinc naphthenate up to 2 wt % causes the weight percent of oil separation decrease, beyond that the weight percent of oil separation increase.

Appropriate amount of zinc naphthenate was 2 wt % to obtain good weight percent of oil separation.

Figure (3) exhibit the relation between weight percent of oil separation and weight percent of zinc naphthenate added.

Table (5) Oil Separation Treatment

wt. % of Zinc Naphthenate Additives	wt. % of Oil Separation
0	15.5
1	5
2	4
3	6
4	6
5	6

The Corrosion

In this set of experiments, the influence of adding zinc naphthenate, anti-rust additive called zinc sulfonate, and anti-oxidant additive called diphenylamine was investigated in order to change the behavior expired grease from corrosive (2c) to non-corrosive (1b).

Six experiments were carried out, an amount of grease, 2 wt.% of zinc sulfonate, 1 wt. % of diphenylamine, and different amount of zinc naphthenate.

The results obtained, here, are tabulated in table (6) and illustrated in Fig. (4).

The results clearly demonstrate that increasing the amount of zinc naphthenate weight percent up to 2 wt %, the change occur from corrosive to non-corrosive according to copper corrosion test.

Table (6) Corrosion Treatment

wt. % of Zinc Naphthenate Additives	Strip Copper Corrosion Test
0	2c
1	2c
2	1b
3	1b
4	1b
5	1b

The Acidity

In this set of experiments, the influence of adding an alkali named lithium hydroxide-monohydrate was investigated in order decrease the acidity of the expired grease.

Six experiments were carried out, each 1 gm of grease and different amount of lithium hydroxide-monohydrate added.

The results obtained, here, are tabulated in table (7). It is clearly demonstrate that increasing the amount of alkali causes the acidity of grease decrease.

The results exhibited that (0.008 gm) of lithium hydroxide-monohydrate was sufficient for obtaining the required neutralization number.

Figure (5) show the effect of alkali on acidity of grease.

Table (7) Acidity Treatment

Weight of Lithium Hydroxide Added (gm)	ml. of KOH Used for Titration	Neutralization No. (mg KOH/gm Sample)
0.000	0.45	1.6880
0.002	0.260	0.7225
0.004	0.255	0.6967
0.006	0.250	0.6709
0.008	0.200	0.4129
0.010	0.180	0.3096

The Effect of Additives on Grease Acidity

In this set of experiments, the influence of previous additives (zinc naphthenate, zinc sulfonate, and diphenylamine) on grease acidity was investigated.

Six experiments were carried out, each 1 gm of grease and different amount of zinc naphthenate added.

The results obtained, here, are tabulated in table (8). A gradual increase in neutralization number were observed with increasing the amount of zinc naphthenate added.

Figure (6) exhibit the effect of these additives on grease acidity.

(73 gm) was sufficient to achieve the required neutralization number.

The results obtained, here, are tabulated in table (9)

Table (8) The Effect of Additives on Grease Acidity

Wt. % of Zinc Naphthenate Additives	wt. % of Diphenylamine Additives	wt. % of Zinc Sulfonate Additives	ml. of KOH Used For Titration =A	Neutralization No. (mg KOH/gm. Sample).
0	0	0	0.45	1.688
1	1	2	0.500	1.961
2	1	2	0.700	2.993
3	1	2	0.800	3.510
4	1	2	0.920	4.120
5	1	2	1.060	4.827

The Effect of Both Additives and Lithium Hydroxide on Grease Acidity

Finally, the influence of both additives (i.e. 2 wt. % zinc naphthenate, 2 wt. % zinc sulfonate, and 1 wt. % diphenylamine) and lithium hydroxide on grease acidity was investigated.
Seven experiments were carried out. The results exhibited that increasing the amount of lithium hydroxide up to (0.illustrated in Fig. (7).

Table (9) The Effect of Additives & Lithium Hydroxide on Grease Acidity

Weight of Lithium Hydroxide Added (gm)	ml. of KOH Used For Titration =A	Neutralization No.(mg KOH/gm. Sample)
0.000	0.80	3.5096
0.010	0.65	2.7354
0.024	0.55	2.2193
0.026	0.50	1.9612
0.033	0.45	1.7031
0.040	0.40	1.4441
0.073	0.20	0.4128

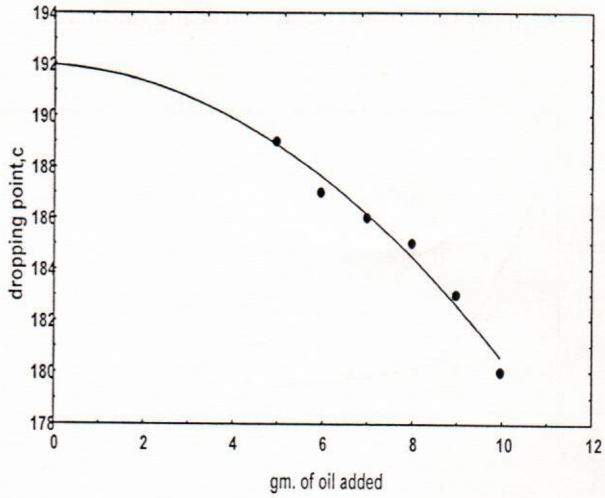

Fig. (2) Relation between Penetration Value and Amount of Neutral Solvent Added

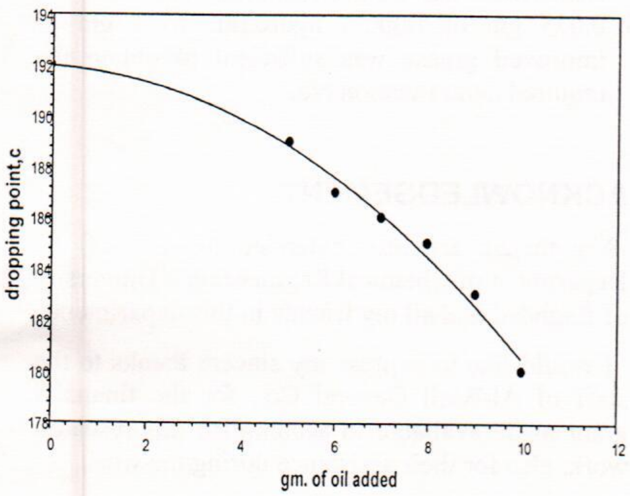

Fig. (1) Relation between Dropping Point and Amount of Neutral Solvent Added

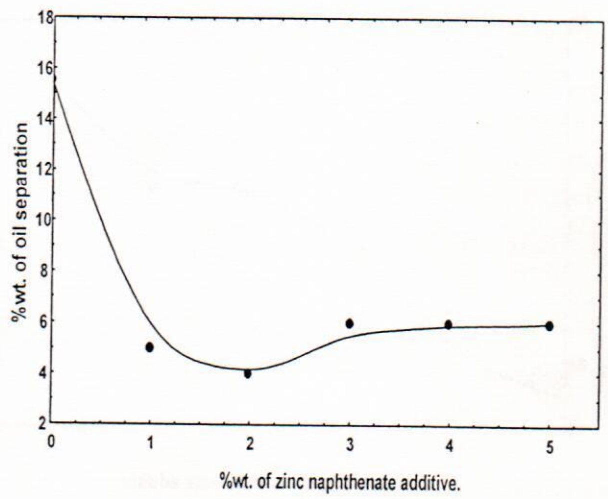

Fig. (3) Relation between Weight Percent of Oil Separation and Weight Percent of Zinc Naphthenate Added

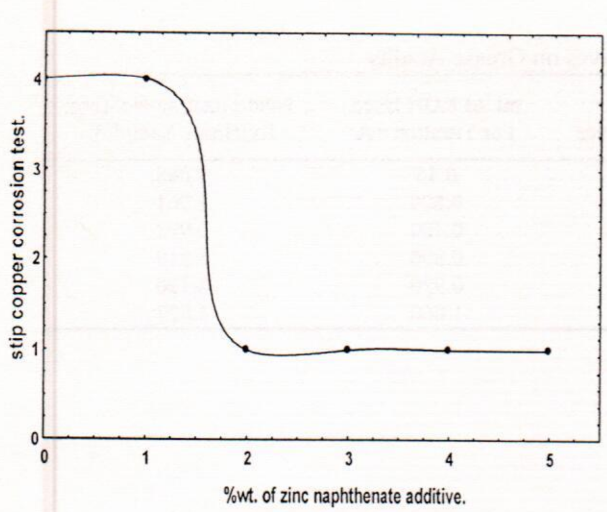

Fig. (4) Corrosion treatment using additives

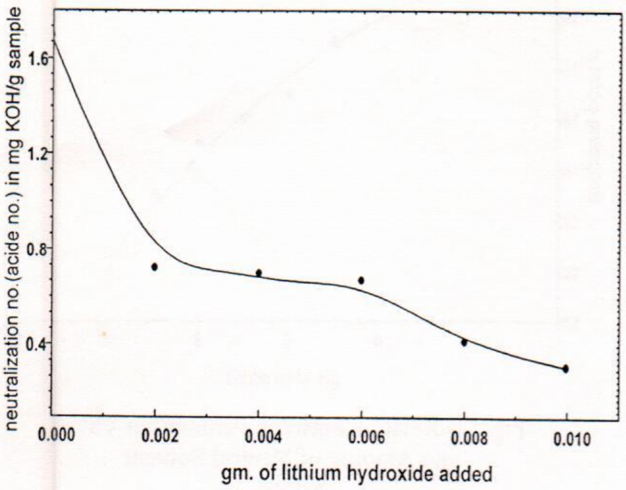

Fig. (5) Acidity Treatment by Using Lithium Hydroxide

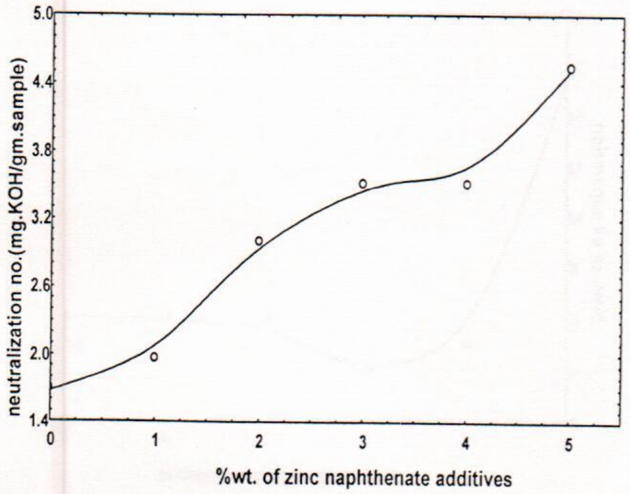

Fig. (6) The Effect of Zinc Naphthenate on Grease Acidity

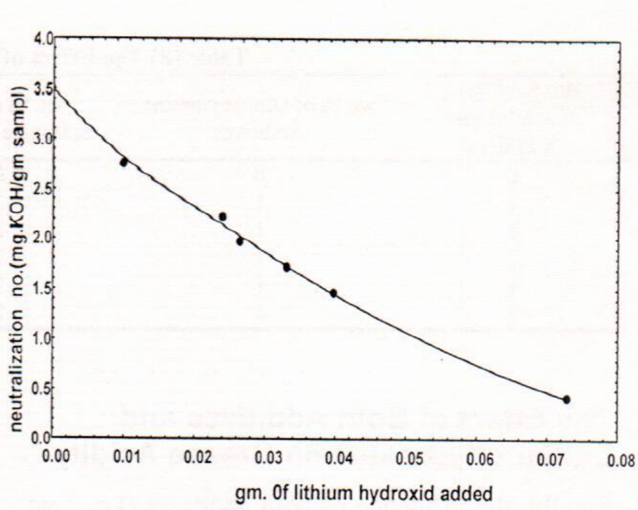

Fig. (7) The Effect of Additives And Lithium Hydroxide on Grease Acidity

CONCLUSIONS

1. The penetration will increase with increasing the mount of oil added and 10 gm of neutral solvent (SN-500) to each 50 gm of this expired grease was sufficient to obtain the required property.
2. The dropping point will decrease with increasing the mount of oil added, also 10 gm of neutral solvent (SN-500) to each 50 gm of this expired grease was sufficient to achieve the required property.
3. 2 wt. % of zinc naphthenate was sufficient to improve the oil separation of this expired grease.
4. 2 wt. % of zinc naphthenate, 2 wt. % of zinc sulfonate, and 1 wt. % of diphenylamine was sufficient to change the expired grease from corrosive to non-corrosive matter.
5. 0.073 gm of lithium hydroxide to 1 gm of improved grease was sufficient to obtain the required neutralization No.

ACKNOWLEDGEMENT

My thanks are also extended to staff of the Department of Chemical Engineering – University of Baghdad and all my friends in this department.

I would like to express my sincere thanks to the staff of Al-Basil General Co., for the financial grant made available to accomplish this research work, also for their assistance during the work.

I'm thankful also to the staff of Al-Dura refinery for their help and cooperation during the present work..

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