



## Fabrication and Evaluation of New Multi-Purpose Grease Using Local Raw Materials

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### Abstract

Lithium greases either simple lithium greases or lithium complex greases are most favorable and widely used in manufacturing of multipurpose greases due to their excellent properties such as good temperature resistance, excellent water resistance and excellent mechanical stability. In the last decade the prices of lithium are extremely increased due to the expansion of electric cars and cell phone batteries rather than the lithium rarity in earth. The industrial sectors tend to use alternative grease to substitute lithium greases. In this study calcium sulphonate greases were prepared as multipurpose grease from heavy and light alkyl benzenes via sulphonation, neutralization, carbonation and grease formulation steps. The formulated greases were evaluated compared with lithium greases. The results of the properties of formulated grease showed excellent results of heat resistance, mechanical stability, water resistance, anti wear and extreme pressure properties compared to lithium grease currently used in the market.

*Keywords:* Multipurpose grease; calcium sulphonate grease; thermal grease; lithium grease alternatives; overbased calcium sulphonate.

### 1. Introduction

Multipurpose grease simply defined as grease combines the characteristics of two or more specialized greases, for example the lithium grease can be used as chassis and wheel bearing greases in automotive lubricants [1].

Calcium grease is usually used as chassis grease, while sodium grease is used as wheel bearing grease [2,3]. Calcium grease has good water resistance but has low drop melting point, while sodium grease has higher drop melting point but has low water resistance properties [4]. Lithium grease has both higher drop melting point and water resistance properties and this is the reason why it became the most favorable grease in the market [5,6].

The lithium grease is improved by additives to fulfill the modern applications in industry and works under severe conditions of high temperature, high pressure, high shear and obey environmental legislations [7,8]. Also mechanical equipments suffer from harsh conditions like high/low temperatures,

salt spraying and acidic/alkaline atmosphere which can damage the grease [9]. To face these harsh conditions lithium complex grease was prepared by using dibasic acids like adipic acid, sebacic acid, phthalic acid and dodecanedioic acid [10]. Lithium greases become the most greases used in industry since they have 71% of the grease market, about 51% for lithium greases and 20% for lithium complex greases [11].

In the last decade the prices of lithium hydroxide and lithium carbonate have extremely increased due to the expansion of manufacturing electric cars, batteries of cell phone and power banks [12]. Due to the huge increase in lithium prices the intention turned towards calcium sulphonate grease because its excellent properties like having very high drop melting point, excellent extreme pressure properties, good water resistance and high mechanical stability [11, 13]. The simple calcium sulphonate grease has an important disadvantage because the percent of thickener used is from 40- 60% of the grease

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depending on base oil used so the calcium sulphonate complex grease was developed for lowering the percent of thickener by half and also the complexing acids like 12-hydroxy stearic acid, phosphoric acid, and boric acid improve the properties of the final grease [14].

Calcium sulphonate greases are compatible with lithium greases so simply they can replace each other and also can be blended with each other [10, 15]. Calcium sulphonate greases also has the advantage it can be used as food grade greases [16].

In this study we prepared calcium sulphonate grease and calcium sulphonate complex grease was prepared using locally produced alkyl benzene through multistep reaction. First step was sulphonation of alkyl benzene, second step was preparation of oil soluble calcium sulphonate, third step was preparation of over based calcium sulphonate, and finally in the fourth step was preparation of calcium sulphonate grease and calcium sulphonate complex grease. The formulated greases were evaluated according to ASTM standard methods by testing dropping point to measure heat resistance, worked penetration to measure mechanical stability, washing% to measure water resistivity and four ball test to measure anti-wear/extreme pressure properties compared to lithium grease currently used in the market.

## 2. Experimental

### 2.1 Materials used

Heavy Alkyl Benzene (HAB) produced by Ameria Refining co (Egypt), Light Alkyl Benzene (LAB) produced by E-LAB Co, Sulfuric acid 98%, Calcium oxide, Toluene, and Methanol supplied from El-Gomhoria for medicine and chemicals Co., Boric acid, 12-hydroxy stearic acid supplied from Jetta Trading Co., Acetic acid, Phosphoric acid, and Isopropyl alcohol supplied by Morgan Chemicals Co., Base oils SN150, SN 500 and Fire oil produced by Ameria co for Petroleum Refining, Base oil BS150 produced by Alexandria co. for Petroleum Refining, Carbon dioxide gas produced by Al-Sharkiya company for High Purity Gases.

### 2.2 Synthesis of calcium sulphonate grease

Calcium sulphonate greases were prepared via multisteps procedure: **First step:** Sulphonation of alkyl aryl benzene HAB or LAB. **Second step:** neutralization of the prepared sulphonate with CaO. **Third step:** preparation of over based calcium sulphonate, and **Fourth step:** formulation of calcium sulphonate greases.

#### 2.2.1 First step:

Sulphonation of HAB or LAB with 98% H<sub>2</sub>SO<sub>4</sub>

was carried in an open vessel containing 100g HAB or LAB surrounded by water bath, add (170 g H<sub>2</sub>SO<sub>4</sub> for HAB) or (150 g H<sub>2</sub>SO<sub>4</sub> for LAB) were added drop wisely at temperature between 55-60°C, an increase in temperature was noticed by the addition sulfuric acid because the sulphonation reaction was exothermic and this problem was overcome by addition of ice pieces into the water bath surrounded the vessel of the reaction. The addition time of sulfuric acid was 2 hrs and digestion time for reaction was 4 hrs, then addition of (10-14 ml) of water, and the reaction mixture was transferred into separating funnel and was left overnight. Then the upper layer organic layer contains either HAB sulphonic acid (HABSA) or LAB sulphonic acid (LABSA) was separated from lower layer (aqueous layer contains spent acid) using separating funnel [17-19].

#### 2.2.2 Second step:

Neutralization of the prepared (HABSA) or (LABSA) with CaO [18-21].

**In summary** the reaction was carried out by four trials for both HABSA and LABSA as follows:

**First trial :** In a three neck flask (100g of HABSA or LABSA), 30 ml benzene, 10ml isopropyl alcohol and 25ml methanol, then 20g of CaO suspended in 40 ml methanol was added drop wisely to the reaction mixture at 65°C till complete addition of CaO, the solution was left under reflux with constant stirring for one hr. This procedure yielded compound (A) for (HABSA) and compound (E) for (LABSA).

**Second trial:** The reaction was done using the same procedure of first trial with the same components and solvents but the temperature was raised to 95°C for 15 minutes after complete addition of CaO. This procedure yielded compound (B) for (HABSA) and compound (F) for (LABSA).

**Third trial:** The reaction was done with the same procedure of first trial with the same components but using toluene instead of benzene at reflux temperature 70°C till complete addition of CaO, the solution was refluxed for one hour. This procedure yielded compound (C) for (HABSA) and compound (G) for (LABSA).

**Fourth trial:** the reaction was done with the same procedure of third trial with the same components and solvents but the temperature was raised to 115°C for ten minutes. This procedure yielded compound (D) for (HABSA) and compound (H) for (LABSA).

After neutralization of the reaction mixture of

first, second, third or fourth trials solvent mixtures were removed of by distillation and the reaction mixtures were centrifuged for 30 min at 4000 rpm to get rid of all excess CaO, centrifugation may be repeated if necessary. The prepared Ca sulphonates either from HABSA or LABSA were evaluated by measuring total base number (TBN), Ca% wt and sulphated ash.

### 2.2.3 Third step:

Carbonation of compounds D and H individually by addition of CaO in presence of CO<sub>2</sub> to yield over basic additives 1 and 2 receptively. Preparation of Over-based Calcium Sulphonate (OBCS) was carried out by passing CO<sub>2</sub> gas through mixture of (100 g) Ca sulphonate, 70 g of CaO, 100 ml toluene, 30ml methanol and 40g low viscosity base oil as diluting oil. The addition rate of CO<sub>2</sub> was done very slow 0.02L/min to avoid formation of solid product which was insoluble in oil or water [17,20,21]. The prepared OBCS additives were compared with imported additive.

### 2.2.4 Fourth step:

Formulation of simple calcium sulphonate and calcium sulphonate complex greases.

#### 2.2.4.1 Formulation of simple calcium sulphonate grease

In an open vessel (100 g) of HAB-OBCS was heated for 30 minutes at 60<sup>o</sup>C, then (10 g) LABSA acid and (40 ml) water were added and the reaction temperature was raised to 80<sup>o</sup>C with constant stirring for one hour, then a mixture of acetic acid (5 g) and (15 g) isopropyl alcohol was added with constant stirring at temperature between 85- 95 <sup>o</sup>C until densification of solution [16, 21]. After densification the mixture was heated continuously at 85 -95 <sup>o</sup>C for 1hr after, then 80 g of base oil added to the mixture at 95 <sup>o</sup>C with continuous stirring for 50 min then the temperature was raised gradually up to 140<sup>o</sup>C for one hr to remove water. The mixture was cooled at 100 <sup>o</sup>C then the rest amount of base oil (20-30 g) was added to adjust the required grease consistency according to NLGI grade. The formed grease was cooled then test for drop melting point and cone penetration were measured. The above procedure was done using different base oils to get NLGI grade2 grease. Different base oils and percentages of OBCS were used to prepare NLGI grade 1, 2 and 3.

#### 2.2.4.2 Preparation of calcium sulphonate complex grease

In an open vessel (100 g) of HAB-OBCS was

heated for 30 minutes at 60<sup>o</sup>C, then (10 g) LABSA acid and 40 ml water were added and the reaction temperature was raised to 80<sup>o</sup>C with constant stirring for 1 hr, then a mixture of acetic acid (5 g) and (15 g) isopropyl alcohol was added with constant stirring at temperature between 85- 95 <sup>o</sup>C until densification of solution [16,21]. After densification the mixture was heated continuously at 85 -95 <sup>o</sup>C for 45 min, then 150 g of base oil was added with continuous heating at 95 <sup>o</sup>C for 50 min then (3g) boric acid, (11g) CaO and 15 ml water were added with continuous stirring, then temperature was raised gradually to 140<sup>o</sup>C for one hour to evaporate water. After removing of water, (9g) of 12-hydroxy stearic acid and (5g) phosphoric acid were added with continuous stirring and heating at 140<sup>o</sup>C, then (100 g) of base oil was added with continuous stirring and heating at 140<sup>o</sup>C until complexation. After complexation the mixture was left to cool till 100<sup>o</sup>C and the rest amount of base oil usually (40-50 g) was added according to the required NLGI grade. This procedure was done to produce NLGI grade2 grease. Different base oils and percentages of OBCS were used to prepare NLGI grade 1, 2 and 3.

## 3. Results and discussion

### 3.1 Investigation of (HAB) and (LAB)

The investigation of HAB and LAB as raw materials was done using GC/MS. The chromatogram of (HAB) in Figure 1 reveals all components of substitution with branched side chains (C<sub>12</sub>- C<sub>16</sub>) either  $\alpha$ ,  $\beta$  positions or both to the benzene ring, these groups included -CH<sub>3</sub>, C<sub>2</sub> H<sub>5</sub> -C<sub>3</sub> H<sub>7</sub>, -C<sub>4</sub> H<sub>9</sub>, and -C<sub>5</sub> H<sub>11</sub>, all components gave rise to base peak with m/e 91 except those with  $\alpha$ -methyl substituted group which gives a base peak m/e 105. Component (1-methyl -2-propyl-nonyl benzene) with molecular weight 260 represents the highest percentage 27.6%, this component has 13 carbon atoms in its side chain. All components which have side chain contain 13 carbon atoms percent about 80.65%wt of HAB.

However the chromatogram of (LAB) in Figure 2 reveals all components of substitutions with branched side chain (C<sub>11</sub>-C<sub>13</sub>) at either  $\alpha$ ,  $\beta$  or both positions to the benzene ring. These group were included, -CH<sub>3</sub>, -C<sub>2</sub>H<sub>5</sub>, -C<sub>3</sub> H<sub>7</sub>, C<sub>4</sub> H<sub>9</sub>, and -C<sub>5</sub> H<sub>11</sub>, all components gave rise to base peak with m/e 91 except those with  $\alpha$ -methyl substituted group which gave a peak with m/e 105. Component 2-pentyl heptyl benzene with molecular weight 246 represents the highest

percentages 22.2 % component which has 12 carbon atoms in the side chain. All components which have side chain 12 carbon atoms percent about 56.11%wt of LAB.

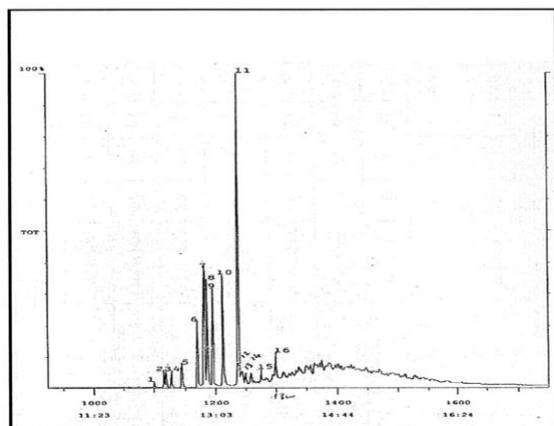


Figure 1: chromatogram of Heavy Alkyl Benzene (HAB)

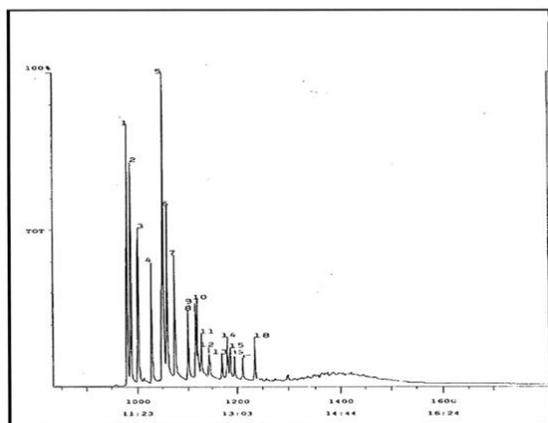


Figure 2: Chromatogram of Light Alkyl benzene (LAB)

### 3.2 First step (sulphonation process)

Since the alkyl benzene sulphonic acids present in our market are suitable for household and laundry but unsuitable for preparation of oil additives or grease formulation, so we had to start from sulphonation step. The sulphonation conditions of HAB and LAB were listed in Table 1. These conditions were used to get the maximum yield and best quality of manufacturing over-based calcium sulphonates (OBCS).

Evaluation results of the prepared alkyl benzene sulphonic acids (HABSA and LABSA) using Kinematic viscosity, color, water%, free sulfuric acid, unsulphonated matter and anionic active matter in Table 2

Table 1: The optimum sulphonation conditions of HAB and LAB.

Condition	HAB	LAB
Acid/ sample ratio	1.7 : 1	1.5 : 1
Reaction temperature	55°-60°C	55°-60°C
Stirring speed, rpm	150-200	150-200
Digestion time, hr	4	4
Yield %	96.7	95.1

Table 2: Analysis results of the two synthesized sulphonic Acid.

Test	ASTM Method	HABSA	LABSA
Kinematic viscosity @ 40°C	D 445	864	186
Kinematic viscosity @ 100°C	D 445	220	71
Color	D 150	8.5	8
Water, ppm	*KFT	26	11
Free sulfuric acid, %wt	D 4711	1.4	1.2
Unsulphonated matters, %wt	D 483	2.8	2.4
Anionic active matter, %wt	D 4251	89	93

\*KFT Karl Fisher Test.

From Table 2 HABSA had greater viscosity than LABSA. HABSA was more favorable in manufacturing OBCS as a thickener of grease.

### 3.3 Second step (neutralization of sulphonic acid with CaO)

The process of neutralization was proceeded under dilution using different solvents to reduce viscosity during reaction and to control the reaction temperature since the reflux temperature of benzene is 65°C while in toluene is 110°C. Four trials were done to determine the suitable solvent and reaction temperature of neutralization reaction as reported in experimental part [17].

During neutralization reaction the viscosity was increased due to formation of calcium sulphonate paste. It is insoluble either in oil or water, so diluting oils with low viscosity like fire oil from El-Ameria Company have to be used. Diluting oil was added at the end of neutralization reaction before cooling and centrifugation steps to prevent consuming of it by the action of continuous heating during neutralization step. The action of the fire oil with the low kinematic viscosity (4 Cst at 40°C), to reduce viscosity of calcium sulphonate paste and facilitate the miscibility of sulphonate in lube oil blending, i.e. coupling of additive as detergent dispersant additive. The neutralization should be carried out in presence of promoter such as methanol which is an ionizing solvent (water like solvent), which increase the

dielectric constant of neutralization reaction media. Methanol should be in vapor state to promote reaction. Some authors used ethanolamine instead of methanol [20]. In this study methanol was used due to its availability and lower cost [17-19].

Neutralization reaction of HABSAs by four trials yielded compounds A, B, C and D respectively. These compounds were evaluated toward TBN (Total Base number) test, Ca %wt, sulphated ash and appearance. Analysis results of compounds A, B, C and D are shown in Table 3.

**Table 3: Analysis results for products A-D**

Test	Method	A	B	C	D
TBN, mg KOH/g sample	D 2896	6	46	6.2	48
Ca, %wt	D 5185	0.214	1.64	0.22	1.7
Sulphated ash	D 874	0.72	5.68	0.75	5.8
Appearance	visible	clear	Clear	clear	clear

Neutralization of LABSAs by four trials yielded products E, F, G and H respectively. These compounds were evaluated toward TBN (Total Base number) test, Ca %wt, sulphated ash and appearance. Analysis results of compounds E, F, G and H are shown in Table 4.

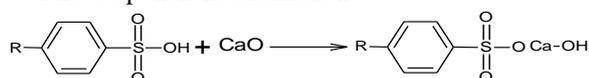
**Table 4: Analysis results for products (E-H).**

Test	Method	E	F	G	H
TBN, mg KOH/g sample	D 2896	5	37	5.4	39.6
Ca, %wt	D 5185	0.178	1.3	0.19	1.38
Sulphated ash	D 874	0.6	4.42	0.65	4.7
Appearance	Visible	clear	clear	clear	clear

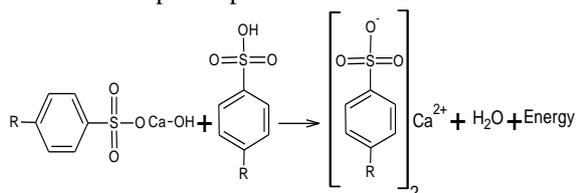
From results in Tables 3 and 4 compounds A, C, E, and G have relatively small base numbers compared to compound B, D, F and H. Therefore compounds A, C, E and G are neutral sulphonate while compounds B, D, F and H are basic sulphonates.

**The neutralization reaction proceeds according to the following two steps:**

First step: half neutralization



Second step: complete neutralization

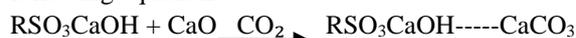


R is a substituted alkyl group in HAB or LAB as described by GC/MS in section 3.1

Neutralization reaction was exothermic thus in order to produce half neutralization (basic sulphonates) higher temperature was applied using toluene as a solvent to retard the second step of reaction. To produce over basic detergent additive, the second and fourth trials in neutralization reaction were more favorable for preparation [24,25]. The fourth trial was the most favorable since toluene was used instead of benzene due to its higher boiling point and to avoid highly toxic effect of benzene [26].

**3.4 Third step (Carbonation to prepare overbasic additive)**

Compounds D and H that were prepared in the second step have higher Total base number (TBN). Therefore they were selected to undergo carbonation process individually by addition of CaO in presence of CO<sub>2</sub> which yielded over basic additive 1 and 2 respectively. The reaction proceeds according to the following equation:



Calcium carbonate is attached with calcium sulphonate by electrostatic bond. The rate of CO<sub>2</sub> gas should be very slow to avoid precipitation of solid product which insoluble in oil or water. To get better results very low rate of CO<sub>2</sub> 0.02 L/min was used [27]. Evaluation results of the prepared OBCS additives 1 (HAB-OBCS) and 2 (LAB-OBCS) were analyzed and compared to the imported additive using TBN test, sulphated ash, detergency test [27], dispersive power [27], oxidation stability test, copper strip corrosion test and solubility in oil test in Table 5.

**Table 5: Analysis figure of the prepared additives and imported one.**

Test	Method	Prepared additives		Imported Additive
		Add. (1)	Add. (2)	
TBN, mg KOH/g sample	D 2896	477	422	498
Ca, %wt	D 5185	16.8	14.9	16.92
*Sulfated ash, %wt	D 874	56.8	54.17	57.5
*Detergency%	Spot method	93	90.2	93.5
*Dispersive power	Sedimentation test	12	10.4	13
*Oxidation stability, (min.)	D 2272	200	263	246
*Copper strip corrosion	D 130	1B	1A	1A
Solubility in oil	visual	Completely soluble		
Appearance	visible	Clear mass		
Colloidal stability		1.1	1.3	1.17

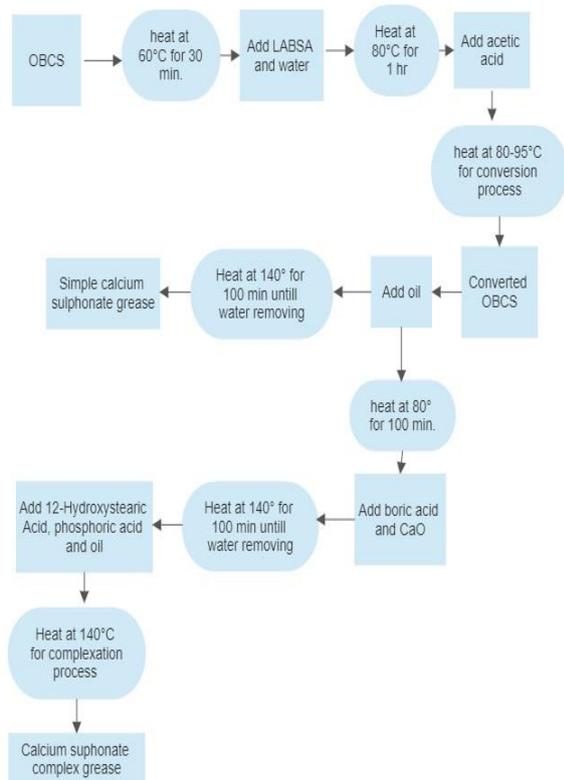
\* These tests were done on a blend of 4% additive and 96% base oil SN150.

From Table 5 additives numbers 1 and 2 have Total Base Numbers higher than 400 as the imported one. Additive number 1 has higher detergency/dispersive power and higher viscosity than additive number 2 while additive 2 has higher oxidation stability and corrosion inhibition than additive 1.

### 3.5 Fourth step (formulation of calcium sulphonate grease)

From Tables (2) and (5) HAB-OBCS could be used as a grease thickener in calcium sulphonate grease due to its high thickening power in grease formulation process because it has higher viscosity and base number compared with LAB-OBCS. However LABSA could be used as auxiliary thickener (free from  $\text{CaCO}_3$ ) because it has better oxidation stability compared with HABSA.

The preparation of both simple calcium sulphonate, and calcium sulphonate complex greases was done as shown in Figure 3 as reported in experimental part.



**Figure 3: Steps of preparations of calcium sulphonate greases**

Figure 3 illustrates the two main processes. The first one was conversion process which applied in

simple and complex greases in which the amorphous  $\text{CaCO}_3$  converted into calcite structure (wafer-like platelets) with a much larger particle size (15–500 nm) [28].

Conversion process should be done at 80-95°C to avoid increasing in temperature since amorphous  $\text{CaCO}_3$  will converted into vaterite instead of calcite which is undesirable crystalline form since it gives poor quality to the grease [29].

The second main process was complexation process (reaction with other acids added to grease mixture in order to making complex structure with original thickener OBCS and HABSA) which applied only in complex grease in order to give better properties and to lower the percentage of thickener in the prepared grease [21-23].

### 3.6 Evaluation of the prepared calcium sulphonate grease compared to lithium grease

The prepared calcium sulphonate greases were evaluated compared to three samples of lithium grease with NLGI grades 1, 2 and 3 which are the most commonly used. The tests used and results of the comparative evaluation are shown in Table 6. These are the most common test used in evaluation of multipurpose greases.

Evaluation results of lithium grease samples are shown in Table 6.

**Table 6: Analysis results of the three lithium grease samples**

Property	Test method	NLGI Grade		
		(1)	(2)	(3)
*Total thickener%		8.5	10	12.2
**E.P additive%		2	2	2
Filler %		1	1	1
Base oil content%		88.5	87	84.8
Base oil type	Blend	SN 150	SN 500	SN 500
		70%	80%	40%
		SN 500	BS 150	BS 150
		30%	20%	60%
Dropping point °C	D 2265	175	190	195
Un-worked penetration, mm	D 217	329	286	233
Worked penetration, mm	D 217	341	292	241
Rust	D 665	nil	nil	nil
Four ball welding load, kg	D 2596	230	250	262
Four ball scar diameter, mm	D 2266	0.58	0.51	0.52
Washing, %	D 1264	2.5	2.2	1.8

\* Total thickener refers to soap content (lithium hydroxide and 12-hydroxy stearic acid).

\*\* E.P (Extreme Pressure) additive.

### 3.6.1 Evaluation of the prepared simple Calcium sulphonate grease

The prepared simple calcium sulphonate greases with NLGI 1, 2 and 3 grades.

Evaluation results of the formulated simple calcium sulphonate grease were shown in Table 7.

**Table 7: Analysis results of the prepared simple Ca sulphonate greases**

Property	Test method	NLGI Grade		
		(1)	(2)	(3)
Over Based Calcium Sulphonate %		38	50	55
*Total thickener%		47	62	68
Base oil content		52	38	32
Base oil type	Blend	SN 150 70% SN 500 30%	SN 500 80% BS 150 20%	SN 500 40% BS 150 60%
Dropping point °C	D 2265	272	289	301
Un-worked penetration, mm	D 217	335	282	248
Worked penetration, mm	D 217	341	283	248
Rust	D 665	nil	nil	nil
Four ball welding load, kg	D 2596	365	374	379
Four ball scar diameter, mm	D 2266	0.58	0.51	0.52
Washing, %	D 1264	1.5	1.5	1.5

\*Total thickener refers to OBCS plus auxiliary LABSA (free from CaCO<sub>3</sub>).

The analysis results in Table 7 reveal the formulated simple calcium sulphonate greases as multipurpose greases have excellent heat resistant, mechanical stability, extreme pressure properties and water resistant, but with high concentration of thickener. It can also be used as thermal grease at higher temperature without the need to add additives.

Calcium sulphonate complex grease was needed to lower the thickener percent which retards pumpability of grease and lubrication at low temperatures.

### 3.6.2 Evaluation of the prepared Calcium sulphonate complex grease

The prepared calcium sulphonate complex greases with NLGI 1, 2 and 3 grades.

The results of evaluation of the formulated calcium sulphonate complex grease were shown in Table 8.

**Table 8: Analysis results of the prepared Ca sulphonate complex greases**

Property	Test method	NLGI Grade		
		(1)	(2)	(3)
Over Based Calcium Sulphonate %		20	26	29
*Total thickener%		35	41	44
Base oil content		65	59	56
Base oil type	Blend	SN 150 55% SN 500 45%	SN 500 65% BS 150 35%	SN 500 32% BS 150 68%
Dropping point °C	D 2265	≥ 320	≥ 320	≥ 320
Un-worked penetration, mm	D 217	329	278	238
Worked penetration, mm	D 217	331	283	239
Rust	D 665	nil	nil	nil
Four ball welding load, kg	D 2596	422	435	464
Four ball scar diameter, mm	D 2266	0.38	0.35	0.35
Washing, %	D 1264	2	2	2

\* Total thickener refers to OBCS, LABSA (free from CaCO<sub>3</sub>), 12-hydroxy stearic acid and any complexing acids.

The analysis results in Table 8 reveal that the formulated calcium sulphonate complex greases as multipurpose greases have excellent heat resistant, mechanical stability, extreme pressure properties and water resistant compared to both simple calcium sulphonate greases and lithium greases. Calcium sulphonate complex greases have lower concentration of thickener compared to simple calcium sulphonate greases. The addition of complexing acid like boric acid works as heat resistance improver [30]. Addition of phosphoric acid improves extreme pressure properties through formation of calcium phosphate, while 12-hydroxy stearic acid is used as co-thickener to reduce OBCS percentage in grease formulation [14,31].

### 4. Conclusion

Simple and complex calcium sulphonate grease can be prepared from local materials via multisteps procedure. The first step was sulphonation of HAB and LAB with conc. H<sub>2</sub>SO<sub>4</sub> 98% at acid/HAB ratio 1.7/1 or acid/LAB ratio 1.5/1, digestion time 4 hrs and temperature 55-60°C. These sulphonation conditions were carried out in order to produce heavy and light alkyl benzene sulphonic acids (HABSA & LABSA) suitable and reliable for preparation of overbased calcium sulphonate OBCS. The second step was neutralization process of sulphonic acids should be carried out in presence of promoter (methanol) with solvent that has boiling point over

100°C (toluene) at reflux temperature then raising temperature over 100°C, in order to obtain half neutralized sulphonate of linear alkyl benzene with CaO to qualify prepared sulphonates for carbonation process (production of OBCS). The third step was carbonation process in which CO<sub>2</sub> gas should be passes on a mixture of basic sulphonate and CaO in presence of methanol as promoter with very low rate 0.02L/min. in order to produce OBCS. HAB-OBCS was better than LAB-OBCS as detergent additive and grease thickener, while LABSA was more favorable than HABSAs to use as auxiliary thickener (free from CaCO<sub>3</sub>) in grease formulation step because it has better oxidation stability. The formulated calcium sulphonate greases either simple or complex were multipurpose greases with excellent heat resistant, mechanical stability, extreme pressure properties and water resistant compared to lithium greases. The complex grease has better analysis results than the simple grease in addition to lower thickener content i.e. better pump ability at low temperatures.

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