



**Results  
&  
Discussion**

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### III-RESULTS AND DISCUSSION

Until comparatively recently, individual greases were required for specific purposes. However, with the advent of new types of formulations, a wide range of applications is now covered. This has resulted in the emergence of multipurpose industrial greases which are replacing the very numerous specialized materials formerly required. Greases are products composed of a liquid phase (mineral and synthetic oils) and a thickening phase.

The known greases used in insulators <sup>(85)</sup> service are classified on the basis of the formulations into hydrocarbon type (thickened by paraffin, petrolatum, and ceresin), polymer type (thickened by high molecular weight compounds as polymers, rubbers, resins and copolymers) and inorganic type.

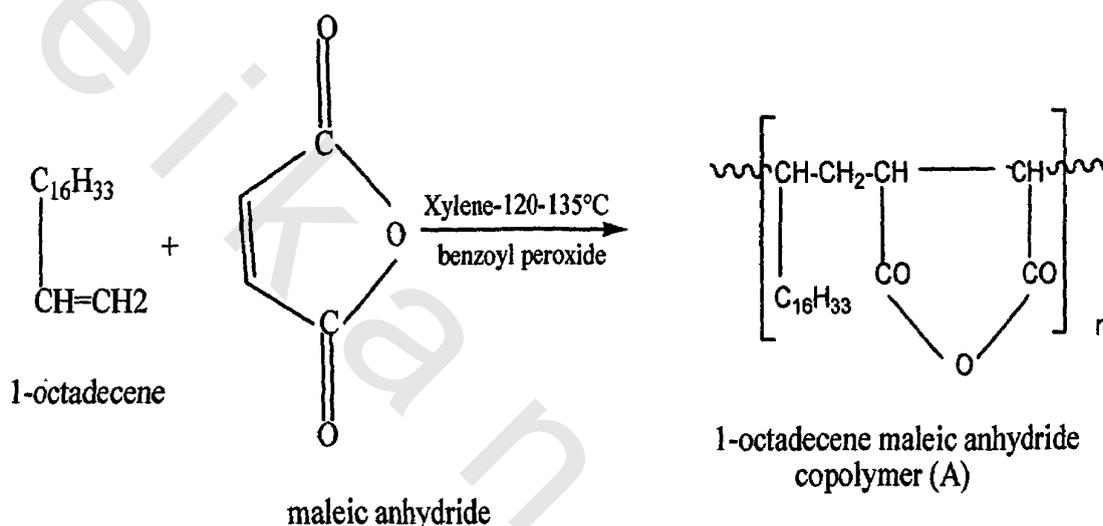
Such types of greases should have particular characteristics such as: high flash point, high dielectric properties, high resistance to water, and high dropping point to overcome the problem of grease sliding in insulation in hot weather. They should adhere to insulation and in the same time have mobility at either ambient or discharge temperature, proper formulation which provides resistance to oxidation and corrosion and which allows also stability on storage.

A grease thus produced, is properly thickened in order that it remains in contact with the surface and does not leak out, or be squeezed out. A grease is a two-phase dispersed system of oil gelled with wax.

### III-1- Synthesis of copolymers and ester

#### III-1-1- The Synthesized (1-octadecene-maleic anhydride copolymer) (A)

The copolymer (A) was prepared through the reaction of 1-octadecene with maleic anhydride in the presence of xylene as a solvent and benzoyl peroxide as an initiator through the following equation Fig (1):

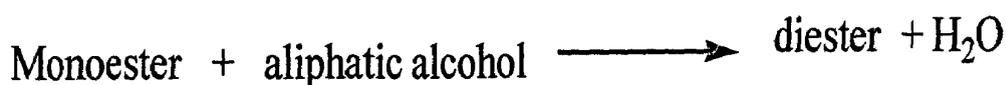
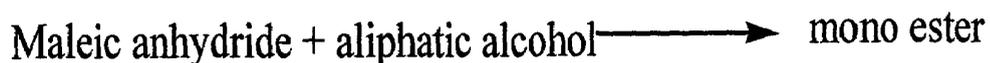


**Fig (1): 1-octadecene- maleic anhydride copolymer (A)**

The specification of the prepared copolymer (A) was shown in the table (11) and fig (5).

#### III-1-2- The Synthesized poly(1-octadecene- co- maleic anhydride) bis behanate ester ( $\text{AC}_{22}$ )

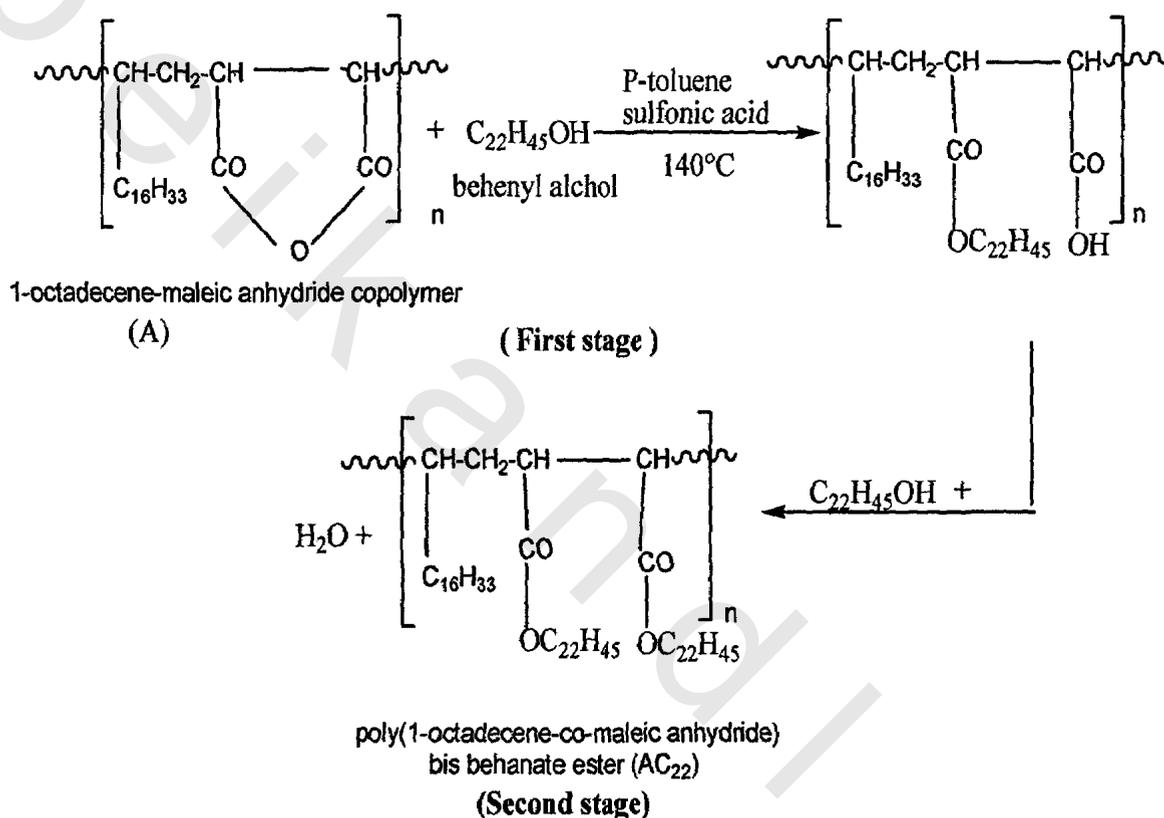
Estrification reactions of maleic anhydride using aliphatic alcohol run as two stages reactions.



It was established that the second stage of the esterification of maleic anhydride with n- alcohol in the

presence of catalyst is considered of a first order reaction in a relationship to the monoester <sup>(123-127)</sup>.

The copolymer (AC<sub>22</sub>) was prepared through the reaction of 1-octadecene- maleic anhydride copolymer (A) with behanyl alcohol in the presence of p- toluene sulfonic acid through the following equation Fig (2):

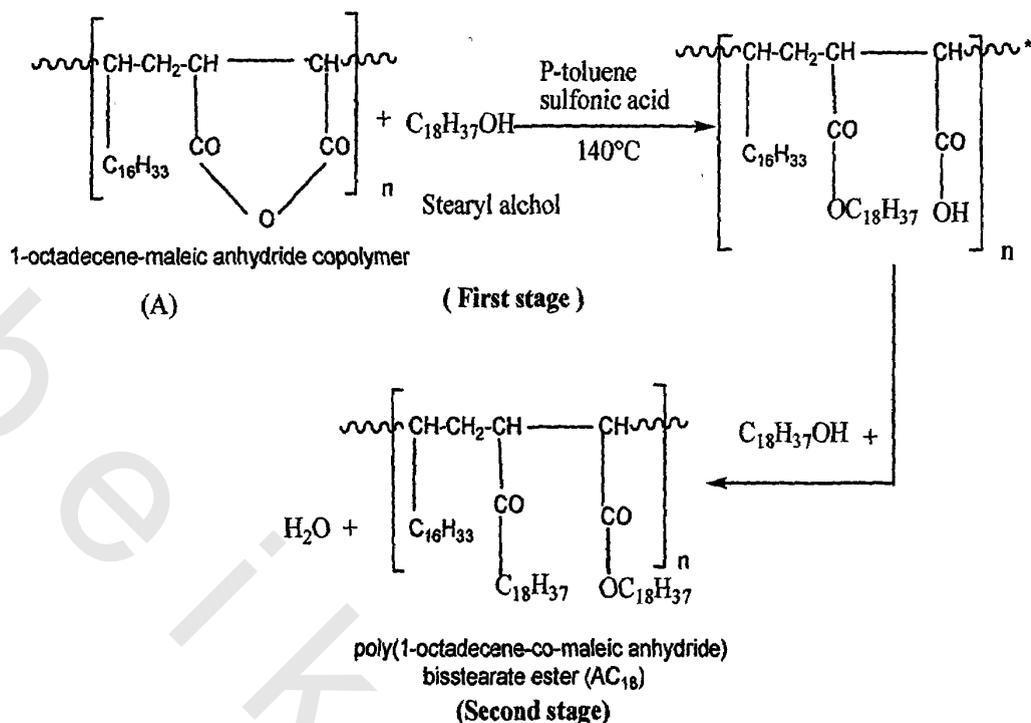


**Fig (2): poly (1-octadecene- co- maleic anhydride) bis behanate ester (AC<sub>22</sub>)**

The specification of copolymer was shown in table (11) and fig. (6).

### III-1-3- The Synthesized poly(1-octadecene- co- maleic anhydride) bis stearate ester (AC<sub>18</sub>)

The copolymer (AC<sub>18</sub>) was prepared through the reaction of 1-octadecene- maleic anhydride copolymer (A) with stearyl alcohol in the presence of p- toluene sulfonic acid through the following equation Fig (3):

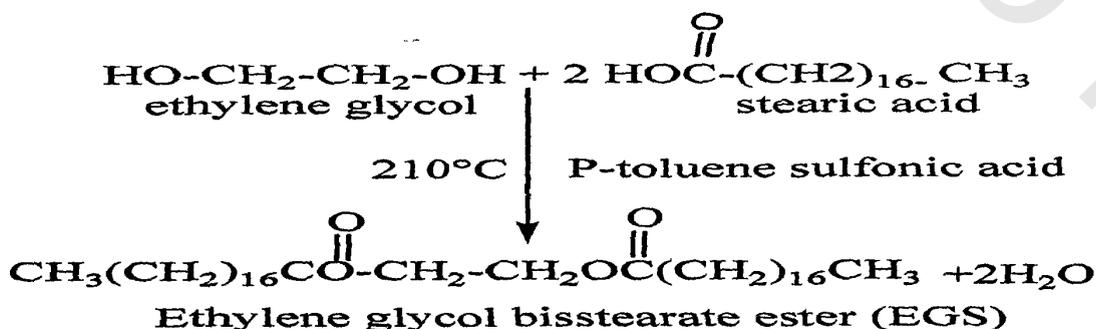


**Fig (3): poly (1-octadecene-co-maleic anhydride) bis stearate ester ( $\text{AC}_{18}$ )**

The specification of copolymer  $\text{AC}_{18}$  was shown in table (11) and fig (7).

### III-1-4- The Synthesized ethylene glycol bis stearate ester (EGS)

Ethylene glycol bis stearate ester (EGS) was synthesized by reacting stearic acid with ethylene glycol in the presence of p-toluene sulfonic acid as catalyst through the following equation Fig (4):



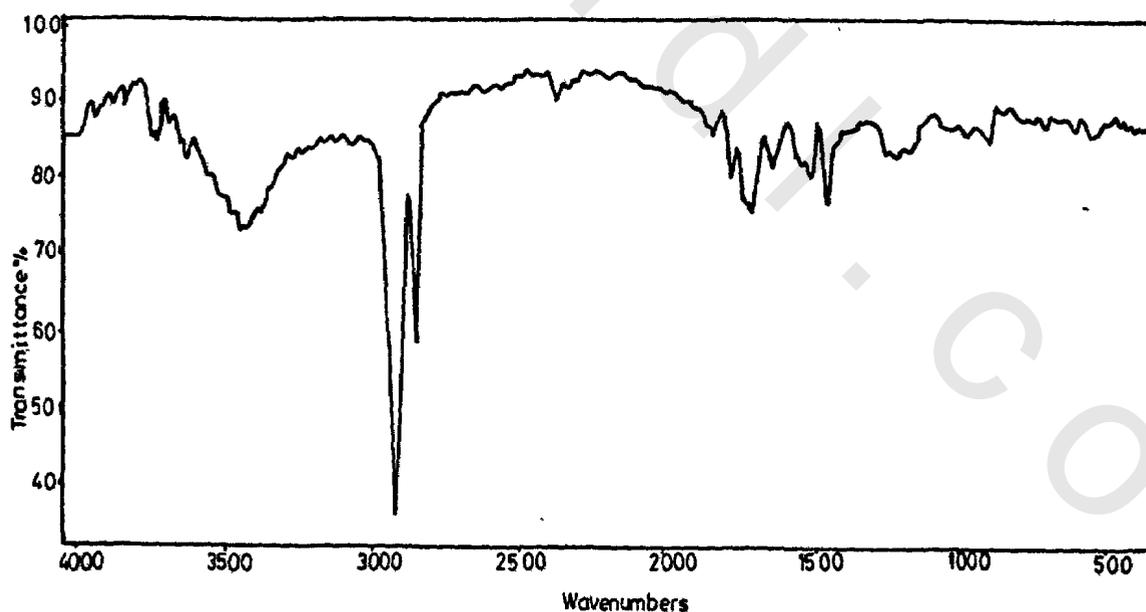
**Fig (4): Ethylene glycol bis stearate ester (EGS)**

The specification of (EGS) was shown in table(11) and fig (8).

### III-2- Characterization of the prepared copolymers and ester

#### III-2-1- FTIR of 1-octadecene- maleic anhydride copolymer (A)

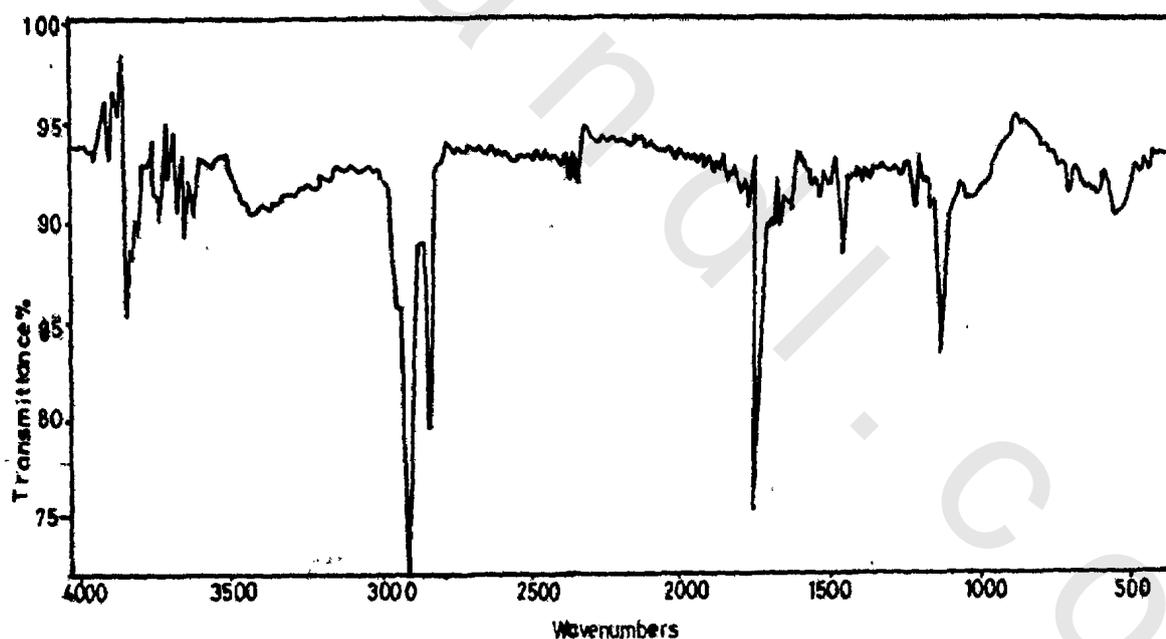
The FTIR spectra of (1-octadecene maleic anhydride copolymer), fig (5), show band in the region of (1300-1000  $\text{cm}^{-1}$ ) for C-O of anhydride group, band at (1709  $\text{cm}^{-1}$ ) for C=O of anhydride group, band at (3000-2850  $\text{cm}^{-1}$ ), for stretching vibration of C-H of aliphatic group, band at (1459  $\text{cm}^{-1}$ ) for methylene ( $\text{CH}_2$ ) group.



**Fig (5): IR spectrum of 1-octadecene- malice anhydride copolymer (A)**

### III-2-2-FTIR of poly (1-octadecene-co-maleic anhydride) bis behanate ester (AC<sub>22</sub>)

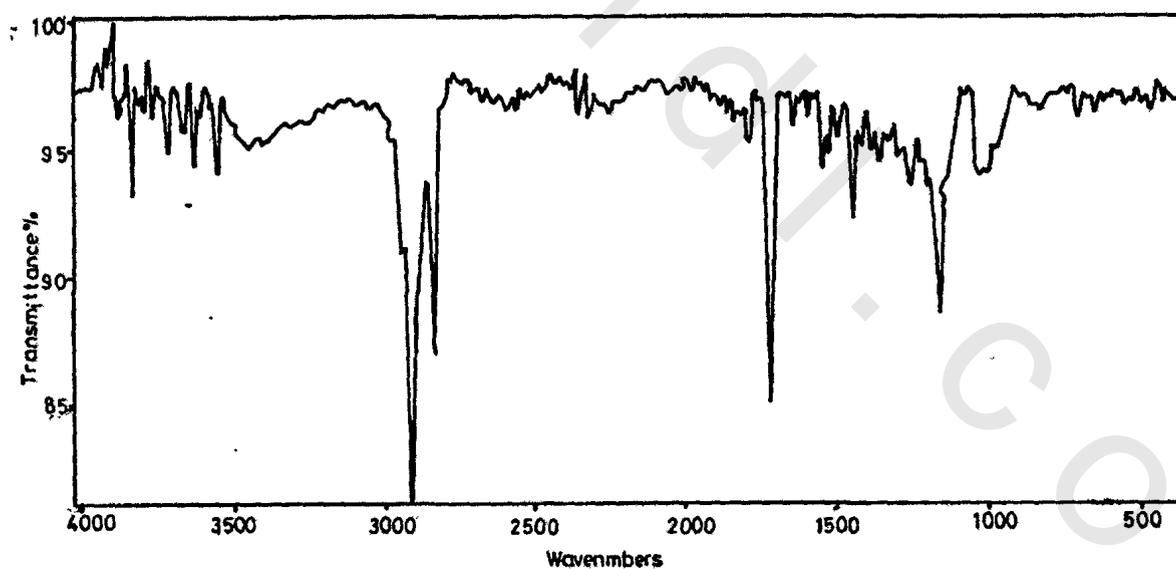
The FTIR spectra of poly (1-octadecene-co-maleic anhydride) bis behanate ester, fig (6), show band in the region of (3000-2850  $\text{cm}^{-1}$ ) for stretching vibration of C-H of aliphatic group, band in (1737  $\text{cm}^{-1}$ ) for carbonyl group (C=O) of ester, band in (1174  $\text{cm}^{-1}$ ) for C-O of ester group and band at (1460  $\text{cm}^{-1}$ ) for bending vibration of methylene group (CH<sub>2</sub>).



**Fig (6): IR spectrum of poly (1-octadecene-co-maleic anhydride) bis behanate ester (AC<sub>22</sub>)**

### III-2-3- FTIR of poly (1-octadecene-co-maleic anhydride) bis stearate ester

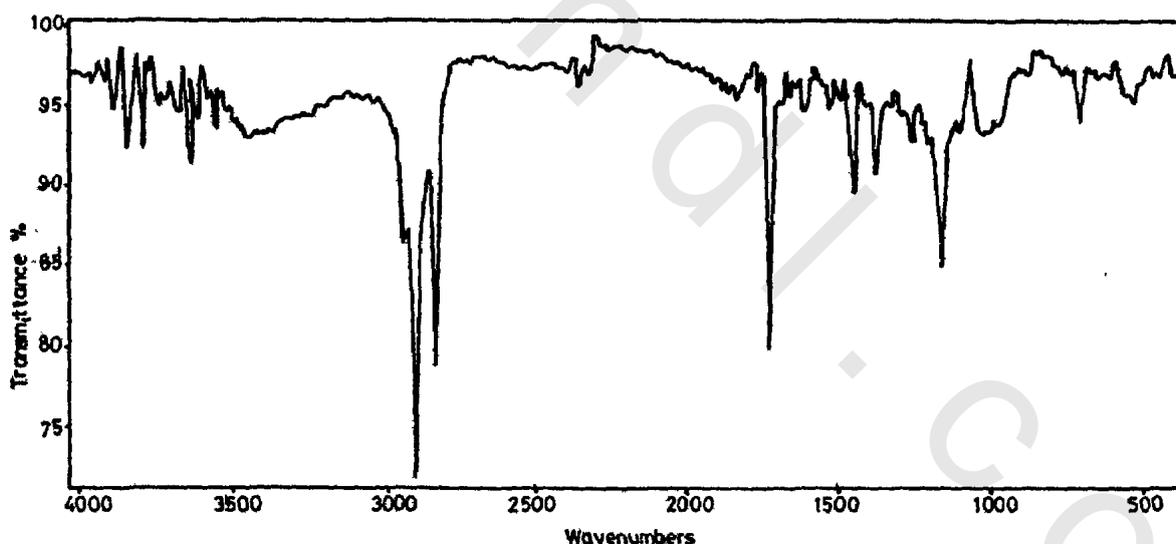
The FTIR spectra of poly (1-octadecene-co-maleic anhydride) bis stearate ester, fig (7), show band in the region of (3000-2850  $\text{cm}^{-1}$ ) for stretching vibration of C-H of aliphatic group, band in ( $1737 \text{ cm}^{-1}$ ) for carbonyl group (C=O) of ester, band in ( $1174 \text{ cm}^{-1}$ ) for C-O of ester group, band at ( $1459 \text{ cm}^{-1}$ ) for bending vibration of methylene group ( $\text{CH}_2$ ) and band at ( $1370 \text{ cm}^{-1}$ ) for bending vibration of methyl ( $\text{CH}_3$ ) group.



**Fig (7): IR spectrum of poly (1-octadecene-co- maleic anhydride) bis stearate ester (AC<sub>18</sub>).**

### III-2-4- FTIR spectra of ethylene glycol bis stearate ester

The FTIR spectra of ethylene glycol bis stearate ester, fig (8), show band in the region of (3000-2850  $\text{cm}^{-1}$ ) for stretching vibration of C-H of aliphatic group, band in (1737  $\text{cm}^{-1}$ ) for carbonyl group (C=O) of ester, band in (1174  $\text{cm}^{-1}$ ) for C-O of ester group and band at (1462  $\text{cm}^{-1}$ ) for bending vibration of methylene group ( $\text{CH}_2$ ).



**Fig (8): IR spectrum of ethylene glycol bis stearate ester (EGS)**

**Table (11): Specification of the prepared ester EGS and Copolymers esters A, AC<sub>22</sub> and AC<sub>18</sub>**

Product	Sample Notation			
	A	AC <sub>22</sub>	AC <sub>18</sub>	EGS
Appearance	Solid-transparent	Solid-Opaque	Solid-Opaque	Solid-Opaque
Colour	Yellow	Brown	Brown	Brown
Average molecular weight	975	2755	2616	594
Average Repeating unit (n)	2.8	2.8	9.7	-
Melting range, °C	56-62	48-54	39-43	47-53
Density, g/cm <sup>3</sup> , at 20 °C <sup>(144)</sup>	0.9167	0.8600	0.8499	0.8766
Solubility at 25°C (g/100ml)	Cyclohexan, toluene, xylene, methanol and chloroform			

### **III-3-Characterization of oils (Dispersion medium)**

#### **III-3-1-Physico-Chemical Characterization**

Data in tables (12, 13, 14) presenting the physico – chemical characteristics of the above oils show that the viscosity of base lube oil grade 260/290 – transformer oil blend is suitable to be used as fluid part in the preparation of grease 27.6 cSt, where 50-55 cSt. for base lube oil grade 260/290 at 40° C which was very large compared with the required value for the insulating oils [transformer oils which are mainly used today ~ 20 cSt]. The chief point of difference between the types of greases is the viscosity of the oil used as an ingredient of the grease. A base lube oil grade 260/290 and transformer oil, tables (12, 13) were mixed and used for this purpose, table (14).

The suitable flash point of the oils blend is (198°C) according to ASTM D-93, where the flash point for good insulating oil is not less than 135°C. The pour point for the base lube oil grade 260/290 (-5°C) is not suitable according to ASTM D-97 where it is high, but after treating with transformer oil (-20°C) it became (12°C) for base lube oil – transformer oil blend, i.e. it is better than base lube oil only.

In addition the distribution of %C<sub>A</sub>, %C<sub>P</sub>, %C<sub>N</sub> was shown in tables (12, 14) and it was deduced that as the %C<sub>P</sub> is greater than 50%, both the base lube oil grade (260/290) and base lube oil – transformer oil blend is considered as paraffinic oils, in the same time, this blend is better than the base lube oil (260/290).

The naphthenic percentage of C<sub>N</sub> for the oil blend is high and so, the dielectric properties for this oil is better than the first oil grade (260/290)<sup>(128)</sup>. This means that the base lube oil grade (260/290) is not suitable as insulating oil, but after blending it with transformer oil (12.5cst) it improved i.e. became suitable as insulating medium.

From the above discussion, it may be pointed out that the first oil under study is not suitable for using as fluid part as insulating before carrying out blending (with transformer oil) to overcome the high aromatic contents and to decrease the pour point.

**Table (12): Physico- chemical properties of base lube oil grade (260 / 290).**

Test	Base lube oil grade (260/290)
Kinematic viscosity <sup>(129)</sup> At 40°C, cSt.	50-55
At 100°C, cSt.	6.5-7
Viscosity index <sup>(130)</sup>	90-95
Flash point, °C (open) <sup>(131)</sup>	204
Pour point, °C <sup>(132)</sup>	-5
Colour <sup>(133)</sup>	4.5
Total hydroxyl number ,	Max. 0.05
Carbon residue , wt. % <sup>(134)</sup>	Max. 0.05
Sulphur content , wt. % <sup>(135)</sup>	Max. 0.01
Average molecular weight <sup>(136)</sup>	392
Refractive index at 20°C $n_D^{20}$ <sup>(137)</sup>	1.4880
Density at 20°C, g/cm <sup>3</sup> <sup>(138)</sup>	0.8799
n-d-m	
C <sub>A</sub> %	10.92
C <sub>N</sub> %	26.48
C <sub>P</sub> %	62.60

**Table (13): Specification of transformer oil**

Specifications	transformer oil
Kinematic viscosity <sup>(139)</sup> At 40°C cSt	12.5
Flash point, °C <sup>(140)</sup>	135
Pour point, °C <sup>(141)</sup>	-20
Density, g/cm <sup>3</sup> , at 20 °C <sup>(142)</sup>	0.8768Max.
Saponification number ,mg. KOH/g	0.60 Max.
Dielectric strength , KV	30

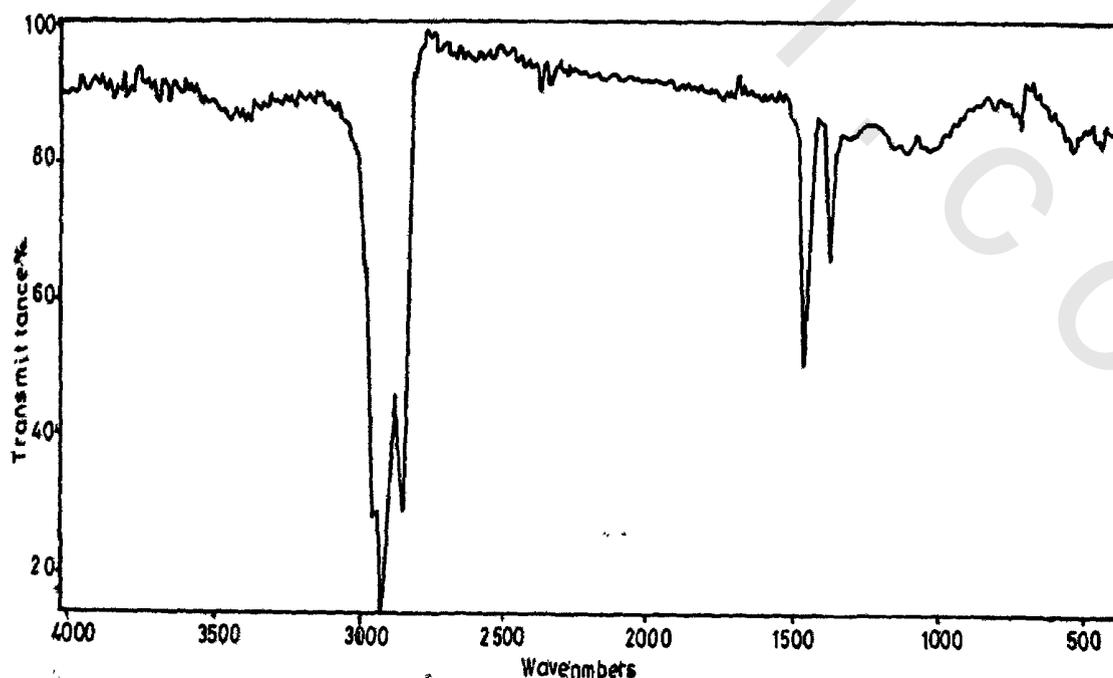
**Table (14): Specification of base lube oil (260/290) and transformer oil after blending**

Specifications	Oil blending
Kinematic viscosity <sup>(129)</sup> At 40°C, cSt.	27.6
Pour point, °C <sup>(132)</sup>	-12
Flash point, °C (open) <sup>(133)</sup>	198
Density, g/cm <sup>3</sup> , at 20 °C <sup>(138)</sup>	0.8773
Average molecular weight <sup>(136)</sup>	400
Refractive index ,n <sub>D</sub> <sup>20</sup> <sup>(137)</sup>	1.4859
n-d-m	
C <sub>A</sub> %	8.99
C <sub>N</sub> %	27.39
C <sub>P</sub> %	63.92
Transformer oil :Base lube oil	2:1

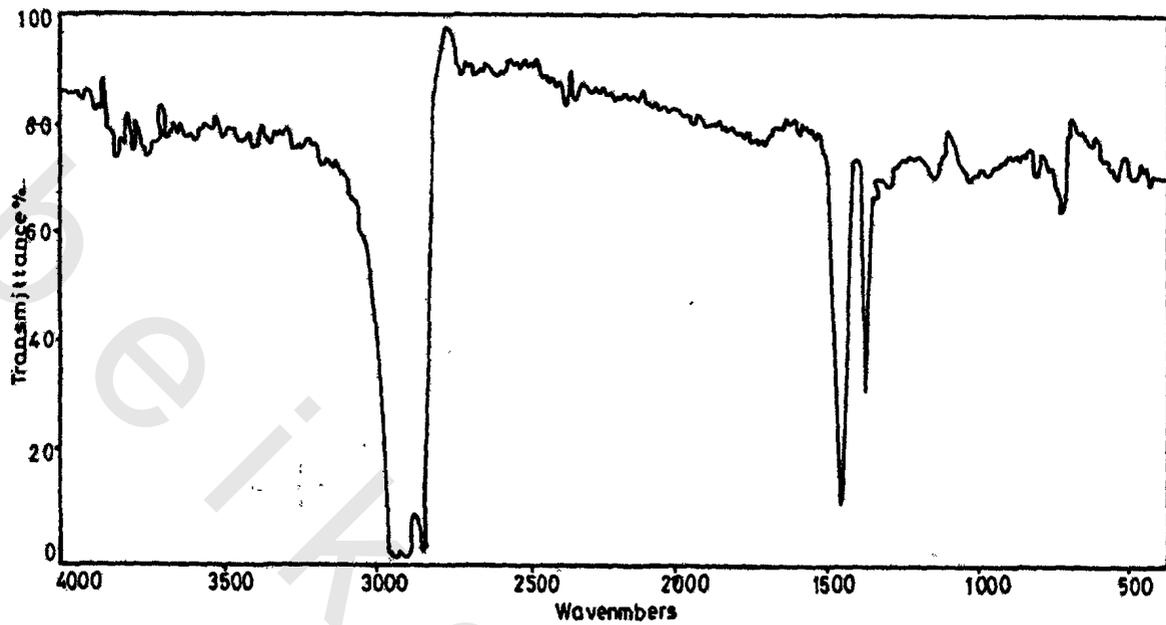
### III-3-2- FTIR Characterization

Infrared absorption spectrometry has been applied to determine the functional groups of base lube oil (first oil), transformer oil (second oil) and base lube oil- transformer oil blend. The measurement of IR spectra in the range from ( $4000 - 500 \text{ cm}^{-1}$ ) figs (9, 10, 11) shows that the above oils have low intensity bands in the region ( $3431 - 3436 \text{ cm}^{-1}$ ), indicating low concentrations of  $-\text{OH}$  and  $-\text{NH}$  groups which have important role in the polarity of oils.

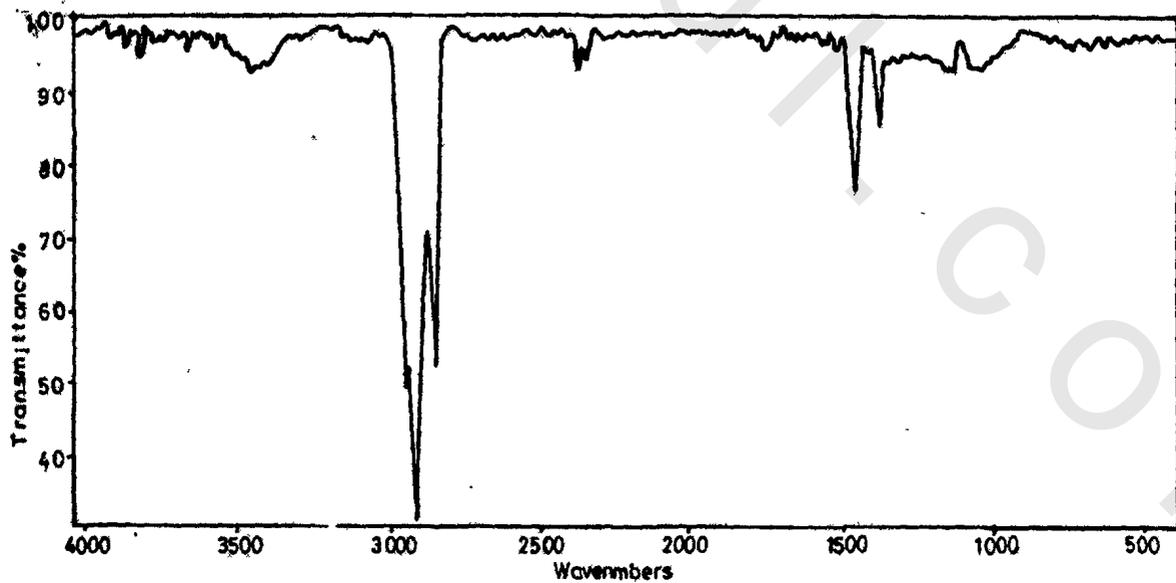
The spectra also show strong two bands at ( $2850-3000 \text{ cm}^{-1}$ ) resulting from C-H bending vibrations asymmetric of methyl and methylene groups, the strong band at ( $1459 \text{ cm}^{-1}$ ), which due to  $\text{CH}_2$  asymmetric bending vibration. In addition, the weak band obtained at ( $1374 \text{ cm}^{-1}$ ) may be attributed to C-N stretching vibration for aromatic amines or  $\text{CH}_3$  bending vibration symmetric for methyl groups and a weak band at ( $1605 \text{ cm}^{-1}$ ) is due to the stretching vibration of C=C aromatic rings.



**Fig (9): IR Spectrum for base lube oil grade (260/290)**



**Fig (10): IR Spectrum for transformer oil**



**Fig (11): IR Spectrum for base lube oil grade (260/290) and transformer oil after blending**

### III-4-Characteristics of Microcrystalline wax

#### III-4-1-Physico-Chemical Characterization

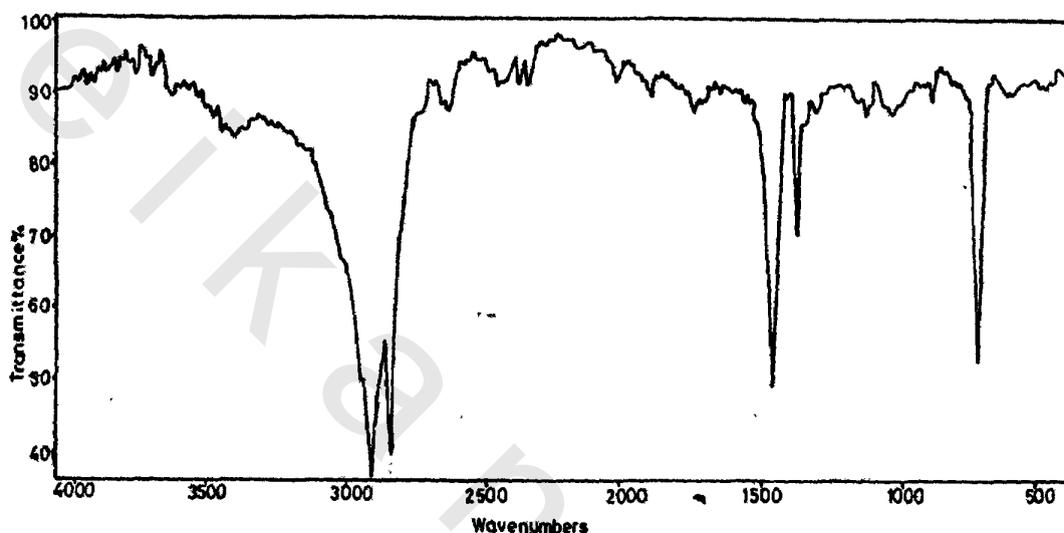
The data in table (15) presenting the physico-chemical properties of microcrystalline wax shows that having high molecular weight (large hydrocarbon chain ~40) 560 and have a melting point of range of 80-81 which give rise for its use in coating applications, also the fine crystal structure enables microcrystalline wax to bind solvent or oil and thus prevent the sweating-out of compositions.

**Table (15): Physical properties of microcrystalline Wax**

Properties	wax
Melting range, °C <sup>(139)</sup>	80-81
Oil content, % <sup>(140)</sup>	0.5
Needle penetration, 0.1/mm at 25°C <sup>(141)</sup>	10-11
Flash point, °C (open) <sup>(142)</sup>	228
Colour	0 (white)
Mean molecular weight <sup>(143)</sup>	560
Average number of carbon atoms.	~40
Density, g/cm <sup>3</sup> , at 20 °C <sup>(144)</sup>	0.9002
Appearance	Odorless, Malleable, Opaque
Solubility	Benzene, ether, chloroform and mineral oil

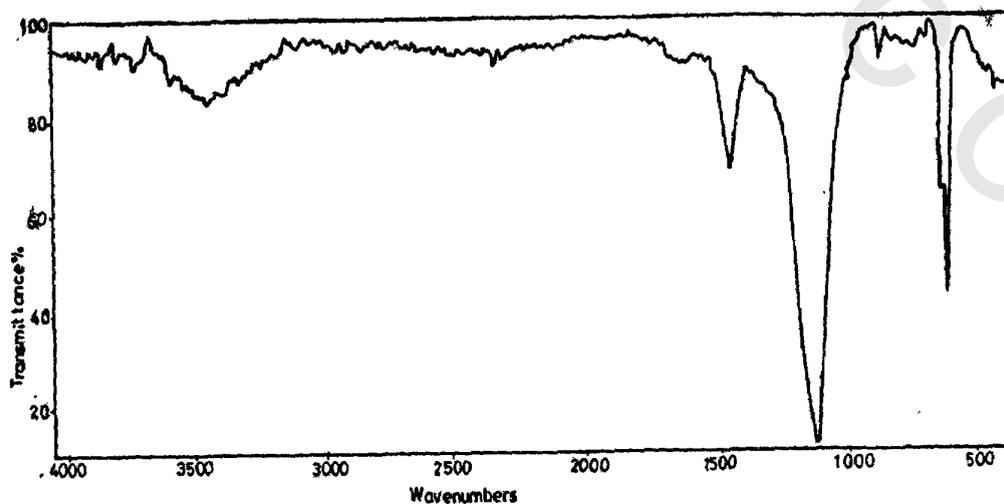
### III-4-2-FTIR Charactrization

The FTIR spectra of the microcrystalline wax fig (12), indicates sharp band in the region of ( $3000-2850\text{ cm}^{-1}$ ) for stretching vibration of C-H of aliphatic and band in the region ( $1450-1375\text{ cm}^{-1}$ ) for stretching vibration of methyl and methylene groups.



**Fig (12): IR Spectrum for microcrystalline wax**

### III-5-FTIR Characterization of Silica from Rice Husk



**Fig (13): IR spectrum of silicon dioxide**

The FTIR spectra of the prepared silica SiO<sub>2</sub> from rice husk fig (13), in the frame work region (4000–500 cm<sup>-1</sup>), indicate absorption observed at (1090 – 1115 cm<sup>-1</sup>). This band is associated with the Si-O stretching modes in silica Si-O unit.

### **III-6-Comparison of wax – oil mixtures (wax gel)**

The chief points of difference between the types of wax – oil mixtures tables (16, 17) WG<sub>1</sub>, WG<sub>2</sub>, WG<sub>3</sub> and WG<sub>4</sub> are the viscosity, penetration and dropping point.

From the data in the table (17) reveals that the sample WG<sub>3</sub> (comprising about 250.9 g of lube oil blend and 109g of microcrystalline wax i.e. 2.3:1) proved advantageous to the other samples WG<sub>1</sub>, WG<sub>2</sub> and WG<sub>4</sub> because of :It has apparent viscosity 47.88 cP at 66°C, penetration 190 and dropping point 50°C.

This means that the ratios of the lube oil blend and wax used in the formulations of the prepared wax gel proved that it is the best since other attempt to mix these ingredient in other ratios failed to produce good quality wax gel (wax-oil blend), where as higher amount of oil defected them (caused oil bleeding, lowering dropping point and viscosity). i.e. excessive oil separation. More over, higher ratios of wax lead to a decrease in the cohesive force of the components (resistance to motion, more difficult to handle). For this, wax – oil mixture WG<sub>3</sub> is suitable for formulation greases S<sub>0</sub> (wax gel).

In this investigation, a base lube oil blend (base lube oil 260/290 and transformer oil 2:1), microcrystalline wax (2.3:1) and 2,2` methylen-bis (4-methyle-6-tertiary butyl phenol) as antioxidant, and Polyoxyethelene sorbiton-nano-palmitate as anticorrosion additives were formulated together to form wax gel (S<sub>0</sub> greases) that is found to have inconveniencing dropping point, viscosity, penetration, water resistance, flash point and dielectric properties.

In an attempt to improve these properties of sample  $S_0$  and directing it to have a good dropping point, viscosity, penetration, water resistance, flash point and electrical insulation properties, any of the following .Polyethylene, atactic polypropylene, polyvinyl chloride, plasticized PVC, poly(1-octadecene-co-malice anhydride) bis behanate ester, poly(1-octadecene-co-malice anhydride) bis stearate ester, ethylene glycol bis stearate ester, butyl rubber, polyisoprene rubber, bitumen ultramarine, silicon dioxide, nano kaolin, nano-talc or sodium silicate are formulated together in certain concentrations.

In general, each of these greases was made from the prepared hydrocarbon wax gel with one of the above mentioned polymers or rubbers as well as bitumen, inorganic compounds and antioxidant as well as anticorrosion additives.

**Table (16): Formulation of the prepared samples WG<sub>1</sub>, WG<sub>2</sub>, WG<sub>3</sub> and WG<sub>4</sub> wax gel**

Constituent parts by weight ,g	Sample Notation			
	WG <sub>1</sub>	WG <sub>2</sub>	WG <sub>3</sub>	WG <sub>4</sub>
Base lube oil grade (260 / 290) } Transformer oil } Ratio	167.26	167.26	167.26	167.26
Lube oil blend } Microcrystalline wax } Ratio	250.9	250.9	250.9	250.9
	250.9	135.62	109	62.73
	1:1	1.85:1	2.3:1	4:1

Table (17): Specification of the prepared wax gels WG<sub>1</sub>, WG<sub>2</sub>, WG<sub>3</sub> and WG<sub>4</sub>

Specification	Sample Notation			
	WG <sub>1</sub>	WG <sub>2</sub>	WG <sub>3</sub>	WG <sub>4</sub>
Colour	Pale yellow	Pale yellow	Pale yellow	Pale yellow
Oil bleeding	Non	Non	Non	Non
Extensibility	Slight	Large	Large	Large
Penetration, 25°C, 10mm /cone ( unworked) <sup>(145)</sup>	125	250	190	180
Dropping point ,°C <sup>(146)</sup>	72	59	50	48
Dynamic viscosity, at 66°C , cp <sup>(147)</sup>	83.35	29.08	47.88	50.81
Behavior at high temperature	Melted	Melted	Melted	Melted

### III-7-Physico-chemical characteristics of the prepared greases

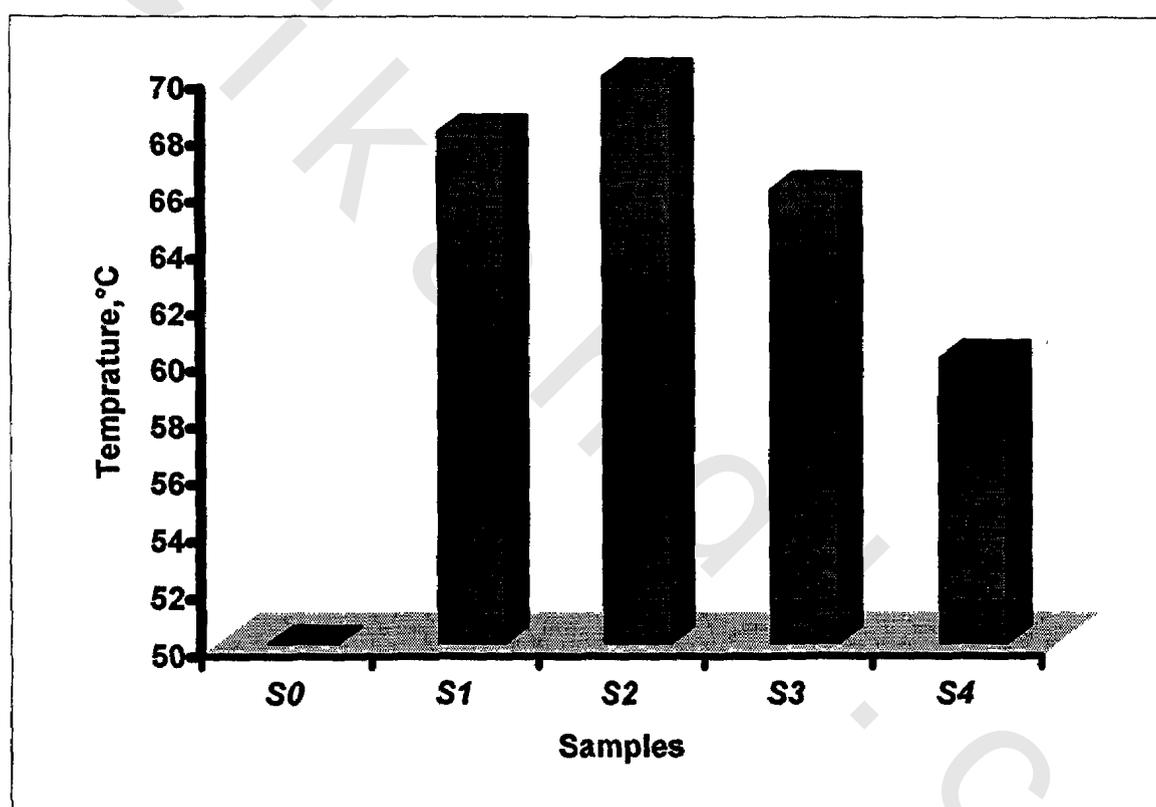
The data in tables (18, 19, 20, 21) and figures (14, 15, 16, 17) indicate that, the samples of greases can be arranged according to the best of dropping point (highest dropping point) are as follows:  $S_2 > S_1 > S_3 > S_5$ ,  $S_{10}$ ,  $S_{11} > S_{12}$ ,  $S_{13}$ ,  $S_{15} > S_4$ ,  $S_9$ ,  $S_7 > S_0$ . While samples  $S_6$ ,  $S_8$ ,  $S_{14}$  appear a lowest dropping point.

It is clear that from table (18) and fig (14) the samples  $S_3$  proved to be advantageous of dropping point than the sample  $S_4$  (containing polyvinyl chloride and triisopropyl phenyl phosphate) as a plasticizer. This may be due to PVC are known to have kinetically rigid chains. On the addition of plasticizer as TIPPP, they became effective enough to penetrate inside the molecular bundles of PVC and to separate the polymeric chains. Accordingly, the mutual interaction between plasticizer and PVC becomes appreciable, leading to aggregates or segments having size smaller than that of PVC, <sup>(148)</sup> i.e. greases  $S_4$  should exhibit higher mobility than greases  $S_3$ .

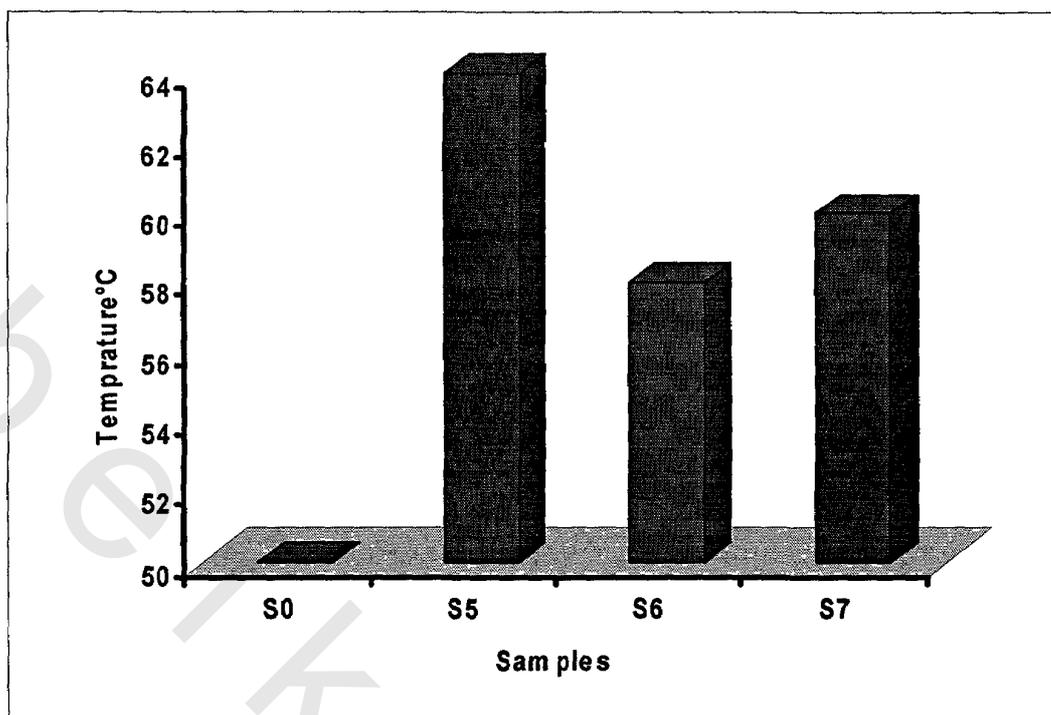
In case of grease  $S_2$  (including atactic polypropylene) has dropping point better than  $S_1$  (including polyethylene), due to a type of atactic polypropylene which can be dissolved in the oil is used while polyethylene, upon swelling in oils, forms plastic structurized systems, the thickening effect of polyethylene is due to its crystalline structure. i.e. This is attributed to the ability of oil to fortify the binding force with polypropylene structure which is higher than P.E., also leading to heavier consistency which provides higher resistance to flow of  $S_2$ .

Also, sample  $S_1$  (including PE) is better than sample  $S_9$  (including butyl rubber), this indicates that, polyethylene upon swelling in oils, forms plastic structurized systems, while butyle rubber forms viscous liquids. The thickening effect of polyethylene due to its crystalline structure is

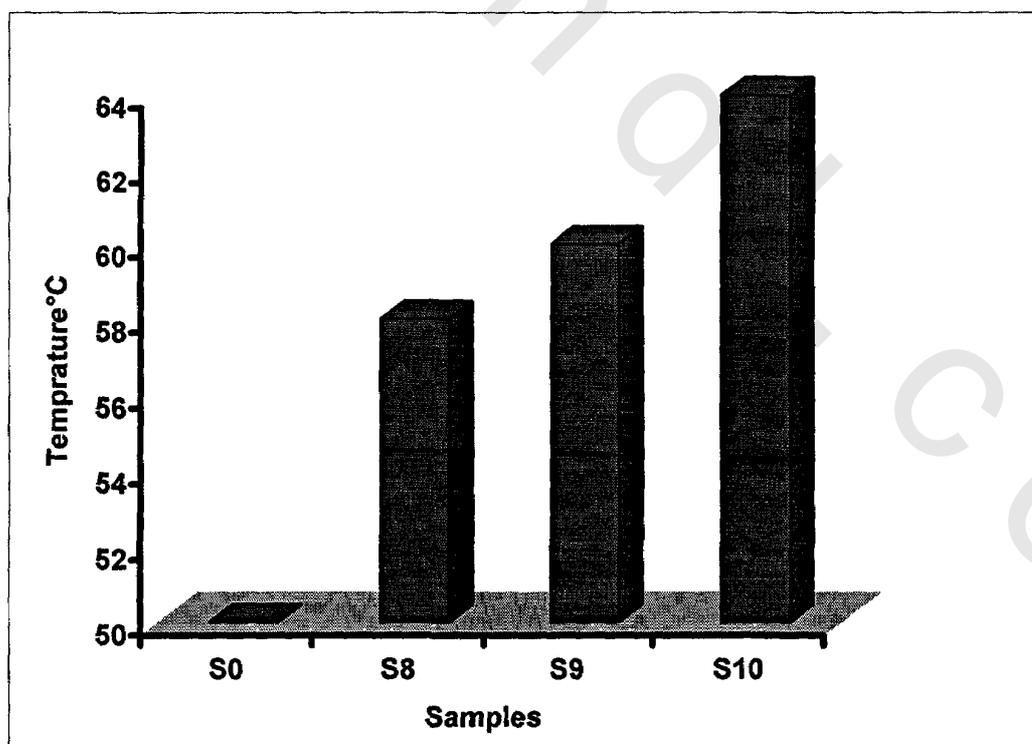
substantially greater than that of butyle rubber <sup>(149)</sup> i.e. S<sub>2</sub>, S<sub>1</sub> and S<sub>3</sub>, shall remain stable on the insulation surface in the highest ambient temperature (about 66-70°C).



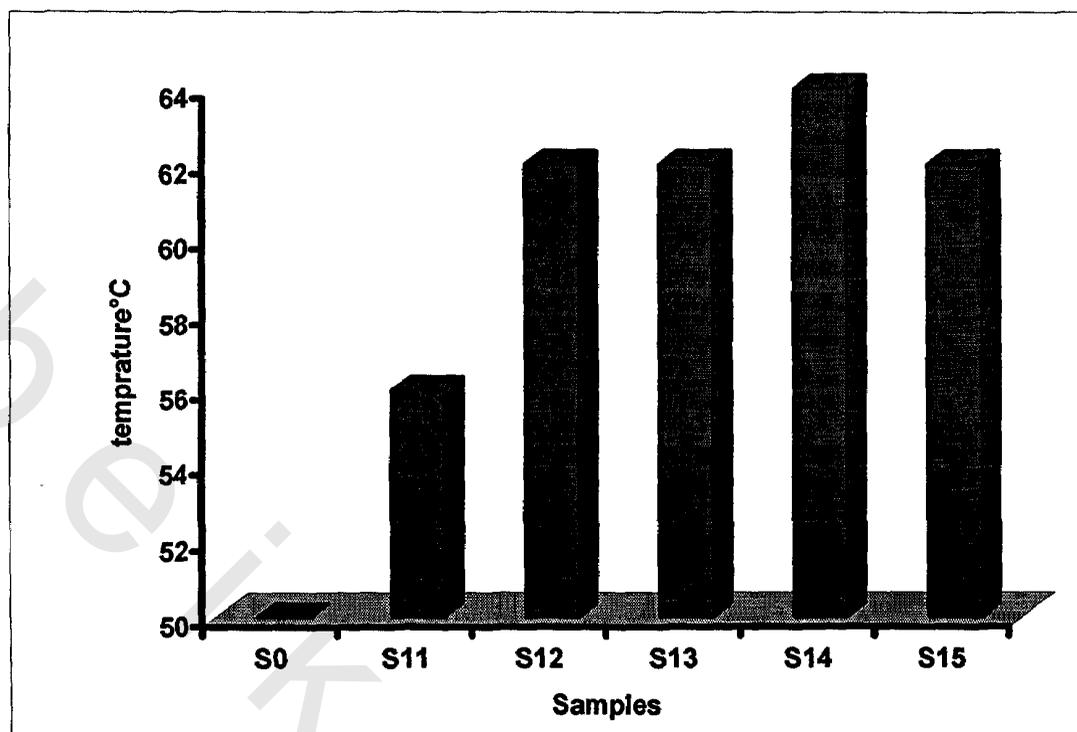
**Fig (14): Comparison between greases S<sub>0</sub>, S<sub>1</sub>, S<sub>2</sub>, S<sub>3</sub> and S<sub>4</sub> containing different thickener of wax, poly ethylene (PE), atactic polypropylene (App), polyvinyl chloride (PVC) and plasticized polyvinyl chloride (PPVC) according to their dropping point.**



**Fig (15): Comparison between greases S<sub>0</sub>, S<sub>5</sub>, S<sub>6</sub> and S<sub>7</sub> containing different thickener of wax, AC<sub>22</sub>, AC<sub>18</sub> and EGS according to their dropping point.**



**Fig (16): Comparison between greases S<sub>0</sub>, S<sub>8</sub>, S<sub>9</sub> and S<sub>10</sub> containing different thickener of wax, bitumen, butyl rubber and isoprene rubber according to their dropping point.**



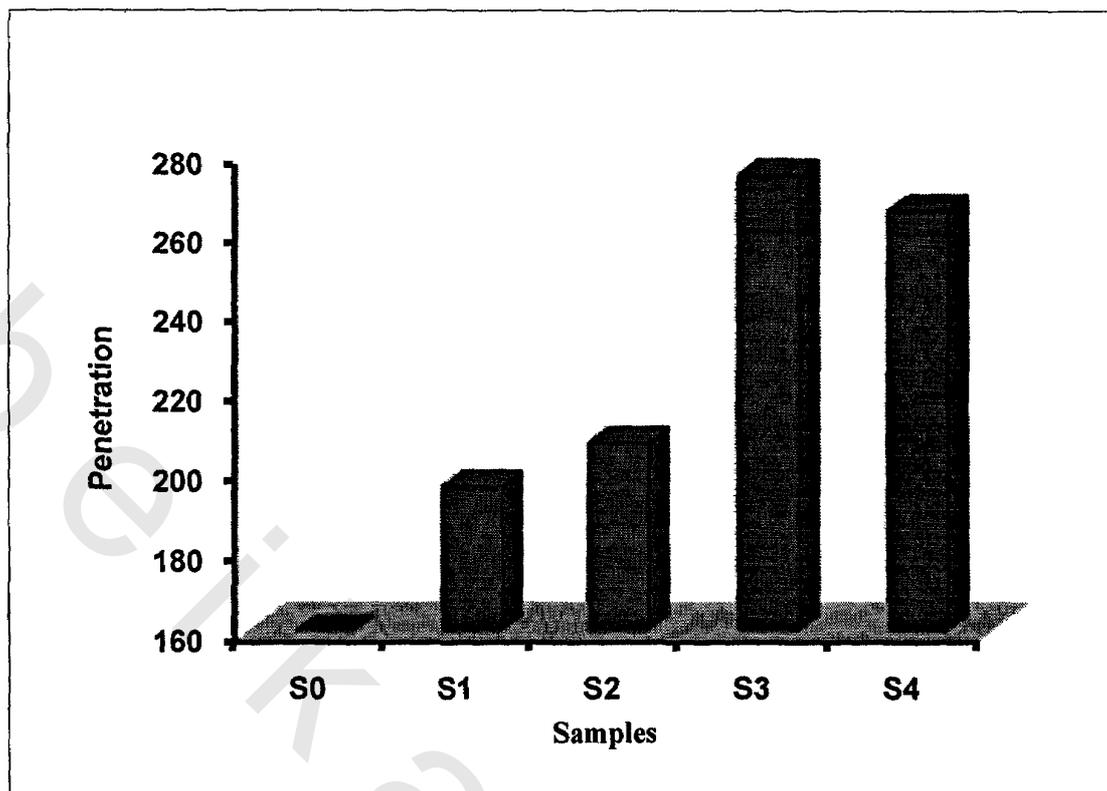
**Fig (17): Comparison between greases S<sub>0</sub>, S<sub>11</sub>, S<sub>12</sub>, S<sub>13</sub>, S<sub>14</sub> and S<sub>15</sub> containing different thickener of wax, ultramarine, sodium silicate, silica from rice husk, talc and kaolin according to their dropping point.**

The data in the tables (18, 19, 20 and 21) and Fig (18, 19, 20 and 21) shows the results of the penetration test for (S<sub>0</sub>- S<sub>15</sub>) greases. These tests showed that the difference of penetration values between these greases are in the order S<sub>3</sub> and S<sub>9</sub> > S<sub>8</sub> > S<sub>4</sub> > S<sub>5</sub> > S<sub>6</sub> > S<sub>13</sub> > S<sub>11</sub> > S<sub>14</sub> > S<sub>7</sub> and S<sub>10</sub> > S<sub>15</sub> > S<sub>2</sub> > S<sub>1</sub> > S<sub>0</sub> respectively.

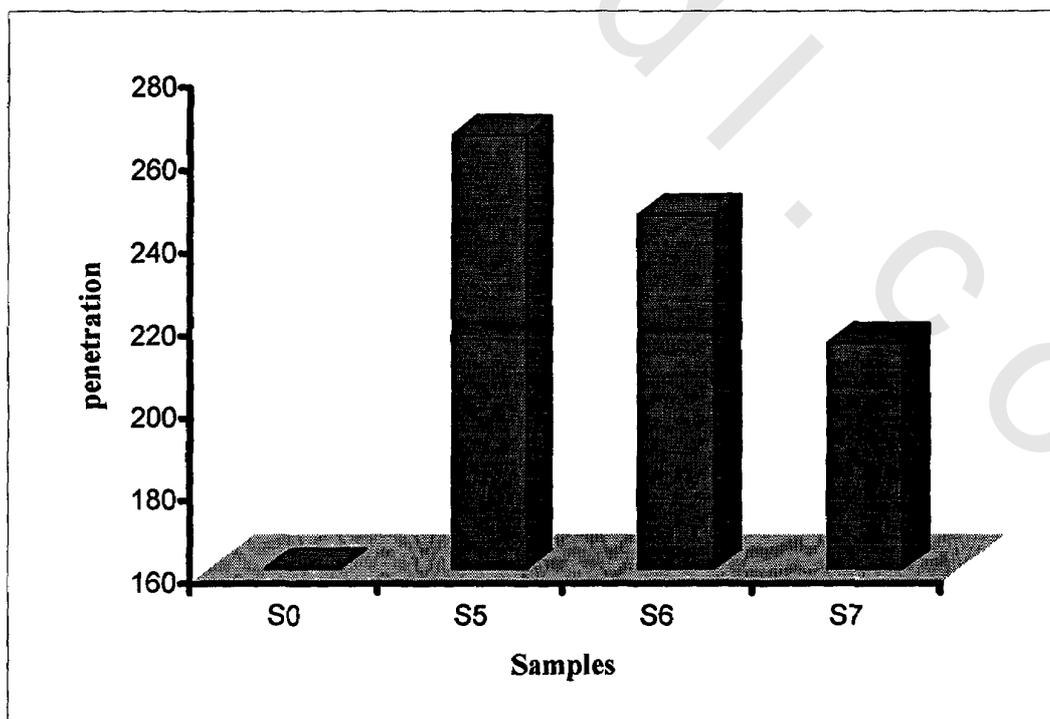
It has been established that the addition of butyl rubber to grease S<sub>0</sub> improved the penetration of S<sub>9</sub> (including butyl rubber) because butyl rubber forms viscous liquids.

The increase of penetration of S<sub>8</sub> (including bitumen) reveals that the compatibility of S<sub>0</sub> greases (including wax gel) with the thickening agent bitumen.

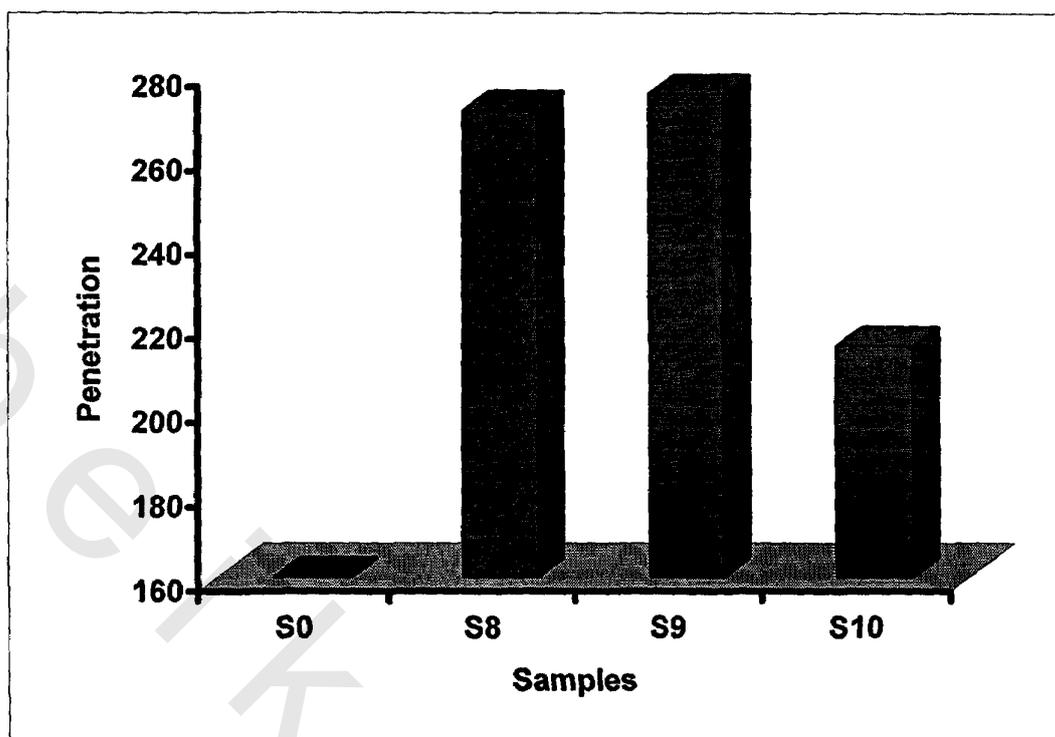
Also, greases S<sub>4</sub> (including PVC and a plasticizer) should exhibit higher penetration because of the higher mobilities.



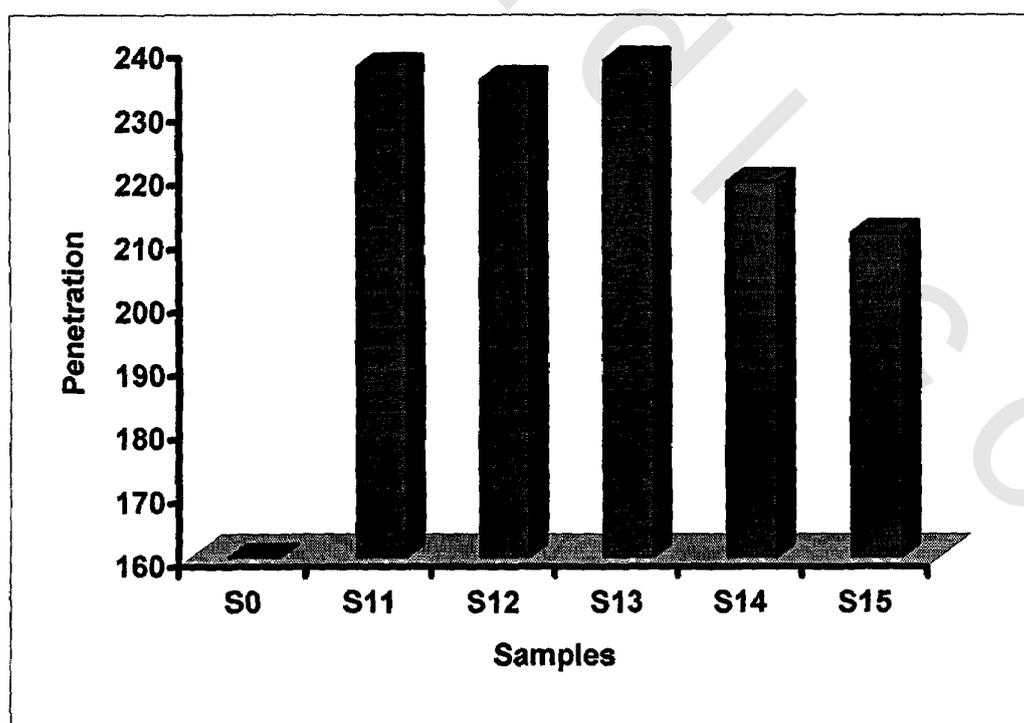
**Fig (18):** Comparison between greases  $S_0$ ,  $S_1$ ,  $S_2$ ,  $S_3$  and  $S_4$  containing different thickener of wax, PE, APP, PVC and PPVC according to their penetration at 25°C.



**Fig (19):** Comparison between greases  $S_0$ ,  $S_5$ ,  $S_6$  and  $S_7$  containing different thickener of wax,  $AC_{22}$ ,  $AC_{18}$  and EGS according to their penetration at 25°C.



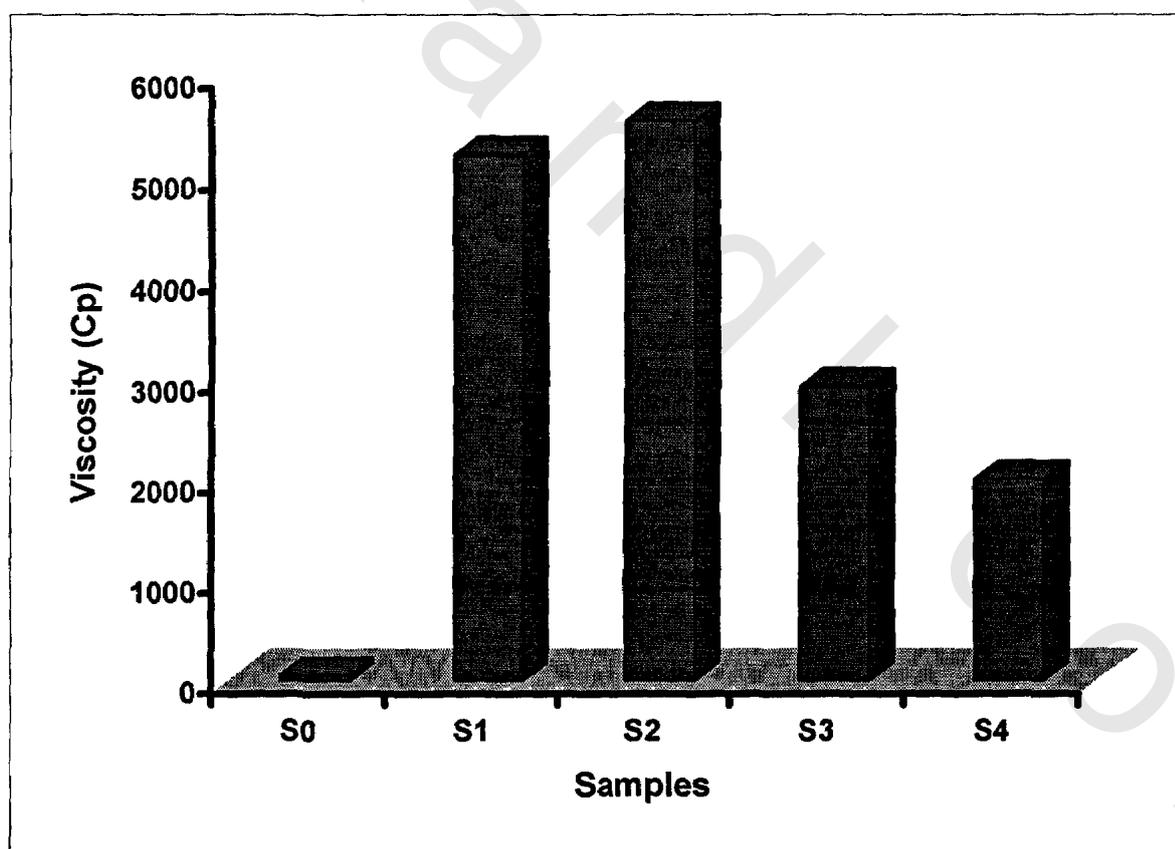
**Fig (20): Comparison between greases  $S_0$ ,  $S_8$ ,  $S_9$  and  $S_{10}$  containing different thickener of wax, bitumen, butyl rubber and isoprene rubber according to their penetration at  $25^\circ\text{C}$ .**



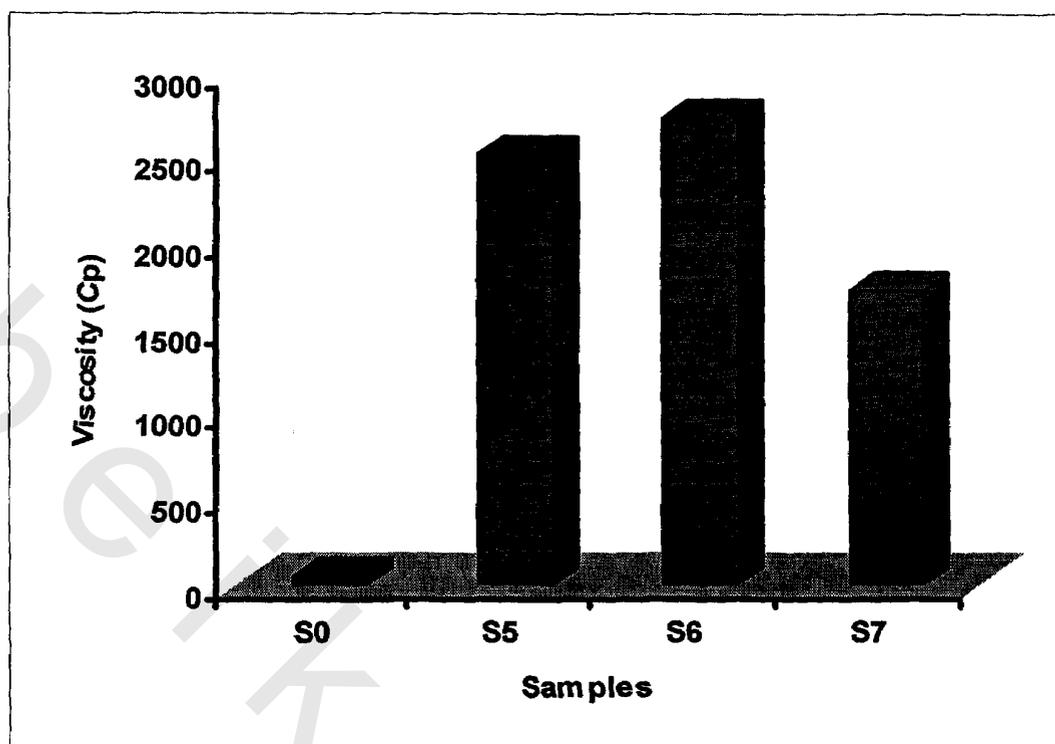
**Fig (21): Comparison between greases  $S_0$ ,  $S_{11}$ ,  $S_{12}$ ,  $S_{13}$ ,  $S_{14}$  and  $S_{15}$  containing different thickener of wax, ultramarine, sodium silicate, silica from rice husk, talc and kaolin according to their penetration at  $25^\circ\text{C}$ .**

Data in tables (18, 19, 20 and 21) and fig (22, 23, 24 and 25) shows that the greases  $S_2$ ,  $S_1$  and  $S_3$  exhibit a marked improvement in apparent viscosity when compared with the other prepared greases. The difference of viscosity values between samples are in the order  $S_2 > S_1 > S_3 > S_6 > S_5 > S_4$ .

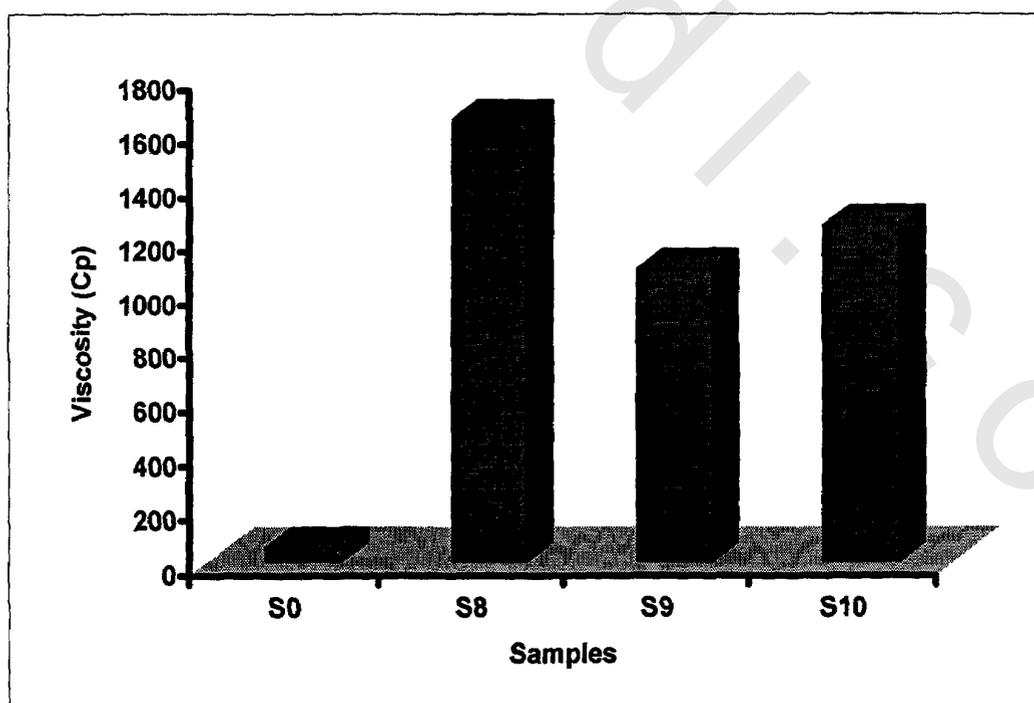
The increase in viscosity for  $S_2$  due to the compatibility of oil with the thickening agent atactic polypropylene i.e. its viscosity should be stable in the highest ambient temperature ( $\approx 66^\circ\text{C}$ ).



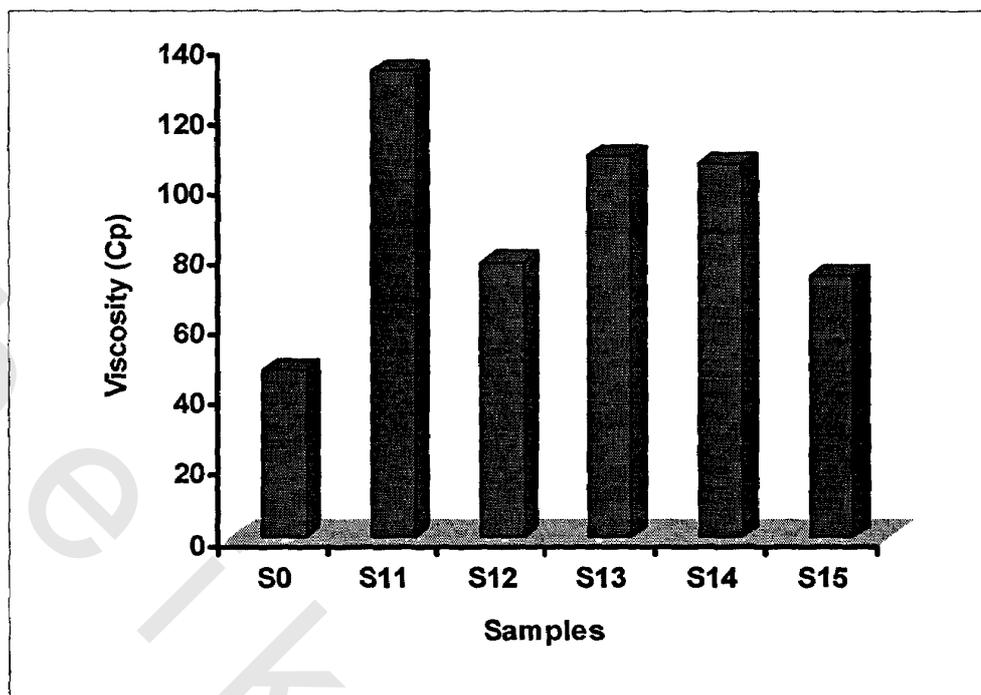
**Fig (22): Comparison between greases  $S_0$ ,  $S_1$ ,  $S_2$ ,  $S_3$  and  $S_4$  containing different thickener of wax, PE, APP, PVC and PPVC according to their viscosity in (Cp) at  $66^\circ\text{C}$ .**



**Fig (23): Comparison between greases S<sub>0</sub>, S<sub>5</sub>, S<sub>6</sub> and S<sub>7</sub> containing different thickener of wax, AC<sub>22</sub>, AC<sub>18</sub> and EGS according to their viscosity in (Cp) at 66 °C.**



**Fig (24): Comparison between greases S<sub>0</sub>, S<sub>8</sub>, S<sub>9</sub> and S<sub>10</sub> containing different thickener of wax, bitumen, butyl rubber and isoprene rubber according to their viscosity in (Cp) at 66 °C.**



**Fig (25): Comparison between greases S<sub>0</sub>, S<sub>11</sub>, S<sub>12</sub>, S<sub>13</sub>, S<sub>14</sub> and S<sub>15</sub> containing different thickener of wax, ultramarine, sodium silicate, silica from rice husk, talc and kaolin according to their viscosity in (Cp) at 66 °C.**

Tables (18, 19, 20 and 21), shows that greases S<sub>6</sub>, S<sub>7</sub>, S<sub>14</sub>, S<sub>15</sub>, S<sub>13</sub>, and S<sub>2</sub> have the highest value of water repel test. i.e. It should not be washed off by heavy rain and wind.

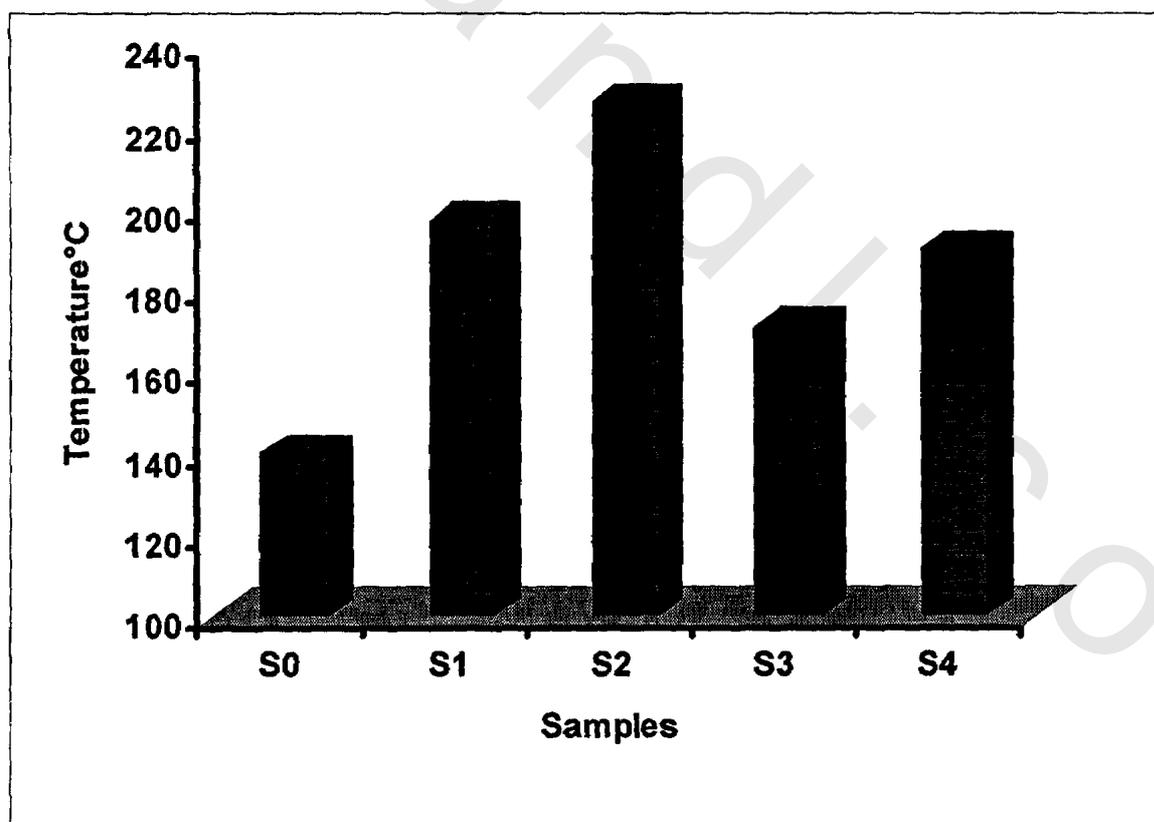
It is indicated from tables (18, 19, 20 and 21) and fig (26, 27, 28 and 29) that the samples can be arranged according to the best of flash point (highest flash point) as follows: S<sub>2</sub>>S<sub>8</sub>>S<sub>12</sub>>S<sub>1</sub>>S<sub>14</sub>>S<sub>4</sub>>S<sub>11</sub>>S<sub>9</sub>>S<sub>13</sub> δ S<sub>10</sub>>S<sub>15</sub>>S<sub>5</sub>, S<sub>0</sub> while samples S<sub>3</sub> appear a lowest flash point. So it is clear that from these tables also that samples (S<sub>1</sub>- S<sub>15</sub>) proved to be advantageous flash point than the prepared sample S<sub>0</sub> containing wax gel only.

The sample S<sub>4</sub> (containing plasticized PVC) has flash point better than sample S<sub>3</sub> unplasticized due to the presence of plasticizer TIPPP (has high fire resistance property)<sup>(63)</sup>.

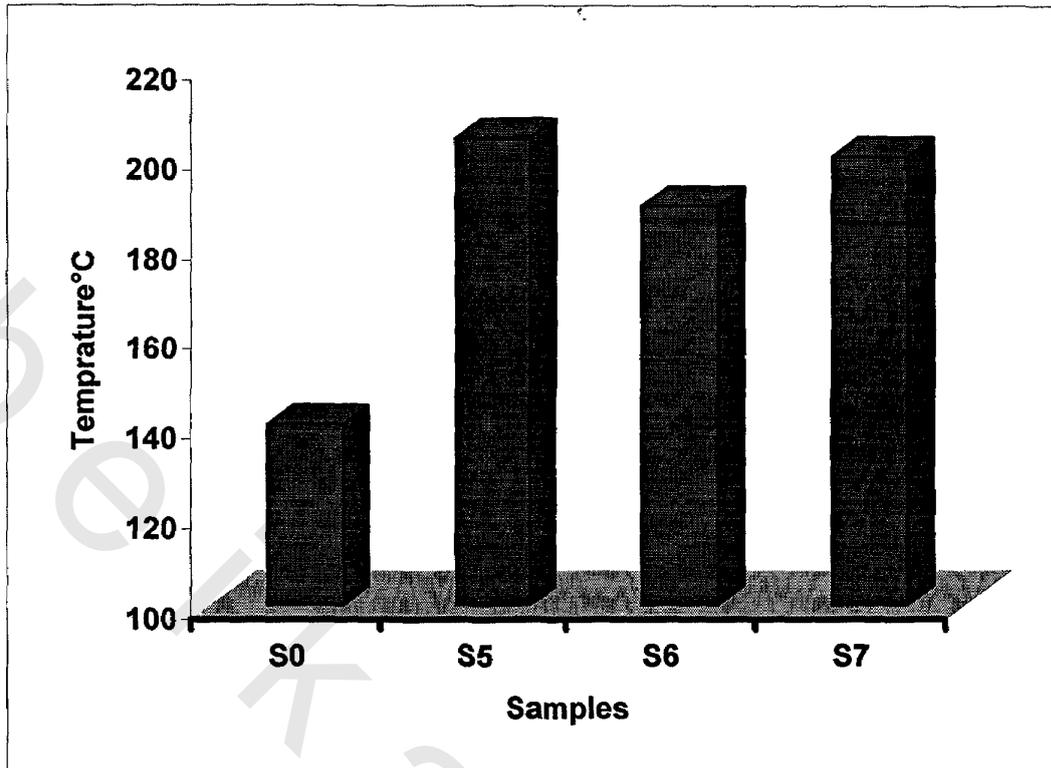
Due to the presence of plasticizer TIPPP containing phosphate compounds, it plays the following role:

- 1) Promotion of char formation, by reducing the production of combustible greases during greases decompositions.
- 2) The formulation of glassy coating and a chair layer may make the surface of some sample containing phosphates.

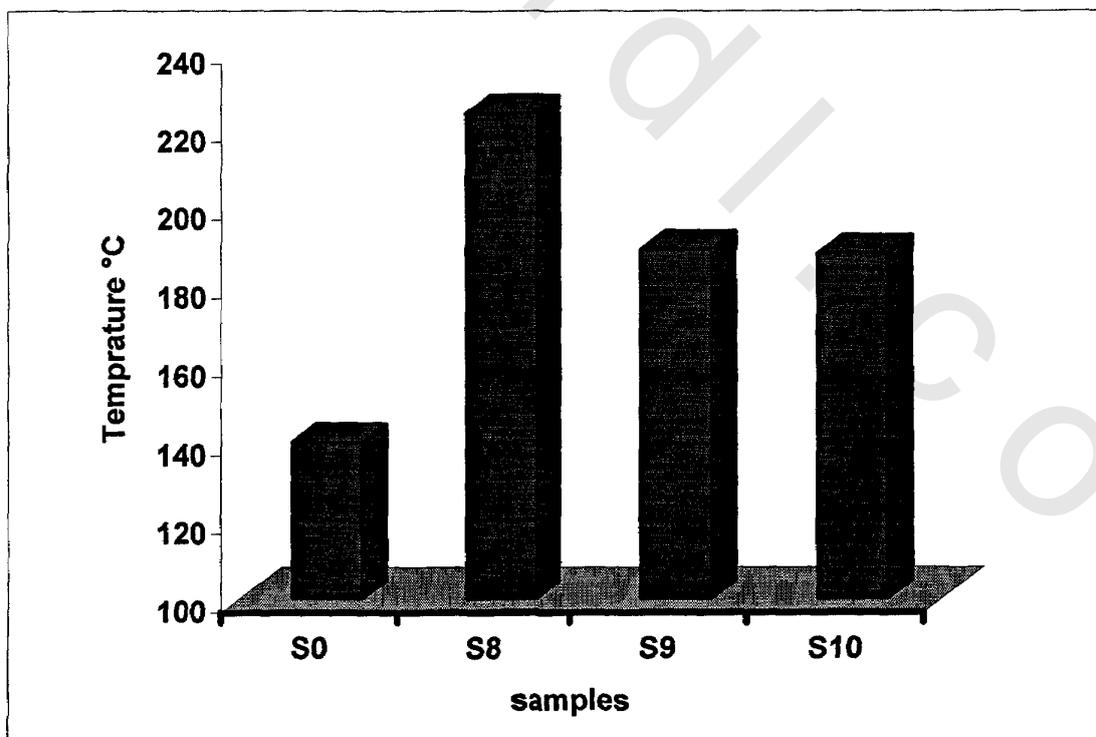
These samples indicate that the flash point was found to be greatly enhanced than that of their analogs of samples S<sub>5</sub>, S<sub>15</sub>, S<sub>10</sub>, due to separation of some inorganic materials (as SiO<sub>2</sub>, ultramarine, sodium silicate) from the greases (wax and oil) at high temperature. i.e. greases shall not ignite but carbonization may be allowed to take place at high temperature. i.e. the flash point must high as much as possible about (184-250°C).



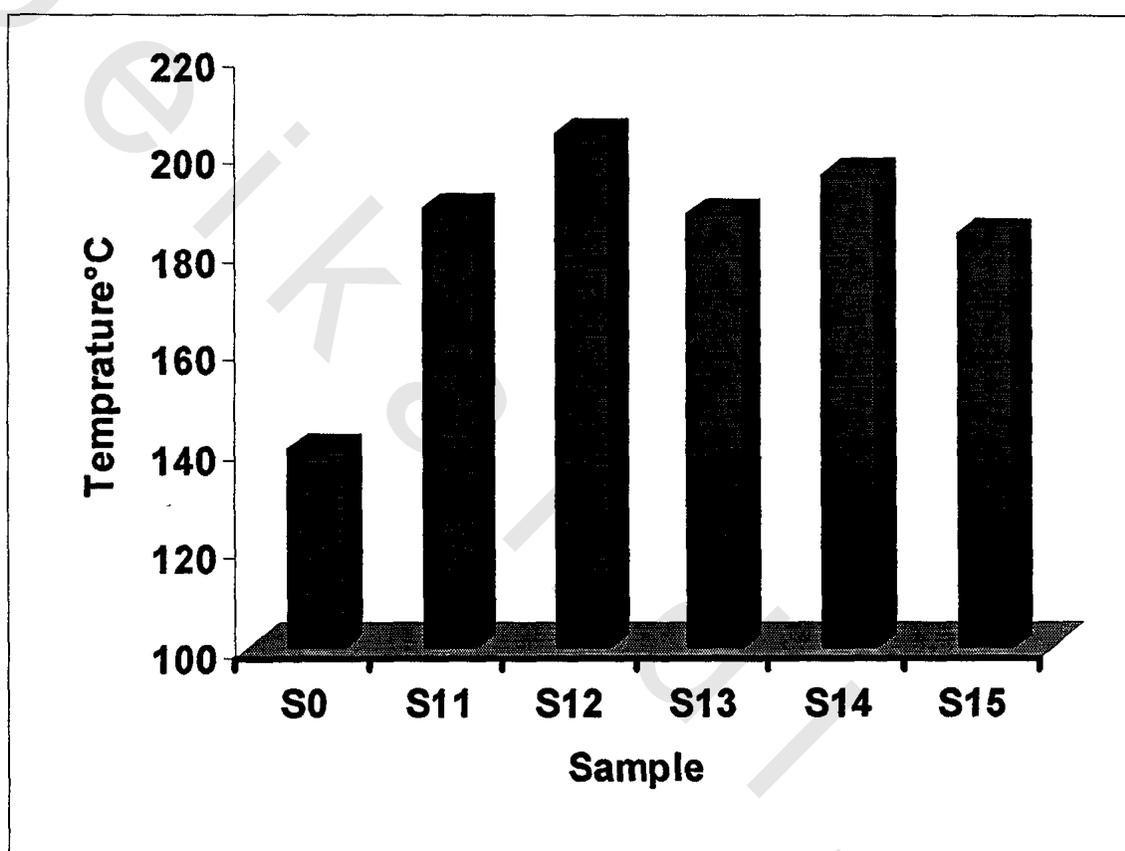
**Fig (26): Comparison between greases S<sub>0</sub>, S<sub>1</sub>, S<sub>2</sub>, S<sub>3</sub> and S<sub>4</sub> containing different thickener of wax, PE, APP, PVC and PPVC according to their flash point.**



**Fig (27):** Comparison between greases  $S_0$ ,  $S_5$ ,  $S_6$  and  $S_7$  containing different thickener of wax,  $AC_{22}$ ,  $AC_{18}$  and EGS according to their flash point greases.



**Fig (28):** Comparison between greases  $S_0$ ,  $S_8$ ,  $S_9$  and  $S_{10}$  containing different thickener of wax, bitumen, butyl rubber and isoprene rubber according to their flash point.



**Fig (29): Comparison between greases S<sub>0</sub>, S<sub>11</sub>, S<sub>12</sub>, S<sub>13</sub>, S<sub>14</sub> and S<sub>15</sub> containing different thickener of wax, ultramarine, sodium silicate, silica from rice husk, talc and kaolin according to their flash point.**

**Table (18): Specification of the prepared greases S<sub>0</sub>, S<sub>1</sub>, S<sub>2</sub>, S<sub>3</sub> and S<sub>4</sub>**

Specifications	Sample Notation				
	S <sub>0</sub>	S <sub>1</sub>	S <sub>2</sub>	S <sub>3</sub>	S <sub>4</sub>
Appearance colour	Pale brown	Pale brown	Pale yellow- brown	Pale yellow	Pale yellow
Oil bleeding	Non	Non	Non	Slight	Slight
Penetration, 25°C, 10 mm/cone (unworked)	190	196	207	275	266
Dropping point, ° C	50	68	70	66	60
Apparent viscosity, at 66°C, cp	47.88	5213	5548.82	2927.38	2005.29
Behavior at high temperature	Melted	Melted	Melted	Melted	Melted
Water repel at 25 °C , 1 hour, %	98.5	98.98	99.6	96.9	96.7
Flash point, °C,(open)	140	197	226	171	190
Code grease according to, NLGI	4	4	3	2	2
Encapsulation rate	Slow	Fast	Fast	Fast	Fast
Removing with at, 35°C	Benzene, butyl acetate, tetrachloro ethylene, toluene and xylene				

**Table (19): Specification of the prepared greases S<sub>0</sub>, S<sub>5</sub>, S<sub>6</sub>, and S<sub>7</sub>**

Specifications	Sample Notation			
	S <sub>0</sub>	S <sub>5</sub>	S <sub>6</sub>	S <sub>7</sub>
Appearance colour	Pale brown	White- yellow	White- yellow	White- yellow
Oil bleeding	Non	Non	Non	Non
Penetration, 25°C, 10 mm/cone ( unworked)	190	265	246	215
Dropping point ,° C	50	64	58	60
Apparent viscosity, at 66°C , cp	47.88	2528	2744	1713
Behavior at high temperature	Melted	Melted	Melted	Melted
Water repel at 25 ° C 1 hour, %	98.5	99.5	99.96	99.9
Flash point, °C ,(open)	140	182	204	200
Code grease according to, NLGI	4	2	3	3
Encapsulation rate	Slow	Fast	Fast	Fast
Removing with at, 35°C	Benzene, butyl acetate, tetrachloro ethylene, toluene and xylene			

**Table (20): Specification of the prepared greases S<sub>0</sub>, S<sub>8</sub>, S<sub>9</sub>, and S<sub>10</sub>**

Specifications	Sample Notation			
	S <sub>0</sub>	S <sub>8</sub>	S <sub>9</sub>	S <sub>10</sub>
Appearance colour	Pale brown	Black	Pale yellow- brown	Yellow
Oil bleeding	Non	Non	Non	Non
Penetration,25°C,10 mm/cone ( unworked)	190	271	275	215
Dropping point ,° C	50	58	60	64
Apparent viscosity, at 66°C , cp	47.88	1643.65	1080	1244.73
Behavior at high temperature	Melted	Melted	Melted	Melted
Water repel at 25 °C, 1 hour, %	98.5	98.98	99.6	99.4
Flash point, °C ,(open)	140	224	189	188
Code grease according to, NLGI	4	2	2	3
Encapsulation rate	Slow	Fast	Fast	Fast
Removing with at, 35°C	Benzene, butyl acetate, tetrachloro ethylene, toluene and xylene			

Table (21): Specification of the prepared greases S<sub>0</sub>, S<sub>11</sub>, S<sub>12</sub>, S<sub>13</sub>, S<sub>14</sub> and S<sub>15</sub>

Specifications	Sample Notation					
	S <sub>0</sub>	S <sub>11</sub>	S <sub>12</sub>	S <sub>13</sub>	S <sub>14</sub>	S <sub>15</sub>
Appearance colour	Pale brown	Blue	Grey-green	Pale yellow-brown	Pale yellow-brown	Pale yellow-brown
Oil bleeding	Non	Slight	Non	Non	Slight	Slight
Penetration, 25°C, 10 mm/cone (unworked)	190	237	235	238	219	211
Dropping point, °C	50	56	62	62	64	62
Apparent viscosity, at 66°C, cp	47.88	133.4	78.8	109.3	107.3	74.9
Behavior at high temperature	Melted	Melted	Melted	Melted	Melted	Melted
Flash point, °C (open)	140	189	204	188	196	184
Water repel at 25 °C, 1 hour, %	98.5	99.5	99.2	99.8	99.9	99.8
Code grease according to, NLGI	4	3	3	3	3	3
Encapsulation rate	Slow	Fast	Slow	Fast	Fast	Fast
Removing with at, 35°C	Benzene, butyl acetate, tetrachloro ethylene, toluene and xylene					

### III-8-Dielectrical properties of the prepared greases

In this investigation, the greases ( $S_0$ ) containing (lube base oil grade (260/290), transformer oil, microcrystalline wax, antioxidant and anticorrosion additives) was found to have inconvenient dielectrical properties. In an attempt to improve the electrical properties of sample  $S_0$  and directing it to be a good electrical insulators, polyethylene, atactic polypropylene, polyvinyl chloride, plasticized PVC, atactic polypropylene including [poly (1-octadecene-co-maleic anhydride) bis behanate ester, poly(1-octadecene-co-maleic anhydride) bis stearate ester and ethylene glycol bis stearate ester], butyle rubber, polyisoprene rubber, bitumen, ultramarine, silicon dioxide, nano kaolin, nano talc or sodium silicate are formulated together in certain concentration.

The dielectric constant  $\epsilon'$ , dielectric loss  $\epsilon''$  and volume resistivity for the prepared samples ( $S_0$ - $S_{15}$ ) over the frequency range from 1 KHz to 1000 KHz at  $35^\circ\text{C}$  were studied.

The results obtained for  $\epsilon'$ ,  $\epsilon''$  and volume resistivity vs. the frequency for these samples at  $35^\circ\text{C}$  are show in figs (30-41) and tables (22- 29).

Dielectric loss of polymers depends on the chemical constitution of the repeating unit in the chain. The nature and number of polar groups, substitute size, size radical isomerism and steric.

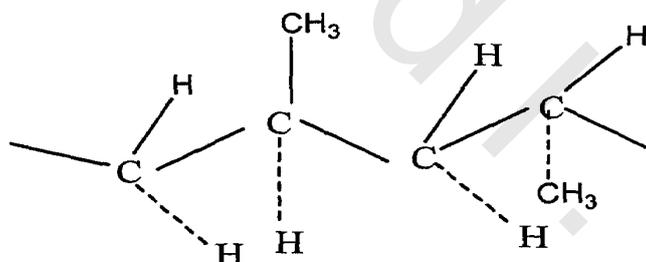
Good insulating greases have low dielectric constant, low dielectric loss and high volume resistivity.

It is evident from table (22) and figs (30, 31) that  $\epsilon'$  and  $\epsilon''$  decrease with increasing frequency for samples ( $S_0$ ,  $S_1$ ,  $S_2$ ,  $S_3$  and  $S_4$ ). Similar behavior was noticed before in the literature<sup>(150, 151)</sup>. The decrease of  $\epsilon'$  and  $\epsilon''$  with frequency shows an anomalous dispersion.

The data, show that the  $\epsilon'$  of the samples ( $S_0$ - $S_4$ ) at frequency 1 KHz are in the order  $S_2$  is more dielectric properties than  $S_1$ ,  $S_3$ ,  $S_4$  and  $S_0$ . Since  $S_2$  has the lowest value of dielectric constant (1.7921) at 1 KHz at 35°C. At the same time, the dielectric loss ( $\epsilon''$ ) decreases with increasing frequency. The  $\epsilon''$  of the sample is in the order  $S_2$  more dielectric properties than  $S_1$ ,  $S_3$ ,  $S_4$  and  $S_0$ . Since  $S_2$  has the lowest value of dielectric loss (0.0365) at 1 KHz at 35°C.

On the other hand, the volume resistivity of these samples represented in table (23) and fig (32) shows that the value of volume resistivity decreases with increasing frequency from 1 KHz to 1000 KHz. It is in order  $S_2 > S_1 > S_3 > S_4 > S_0$  respectively. Since  $S_2$  has the highest value of volume resistivity ( $0.9 \times 10^{12}$  ohm.cm) at 1 KHz at 35 °C.

This proves that, the dielectrical properties (lowest value of  $\epsilon''$ ,  $\epsilon'$  and highest value of volume resistivity) make good insulator of sample  $S_2$  grease (formulated from wax gel  $S_0$  and atactic polypropylene) with chemical structure

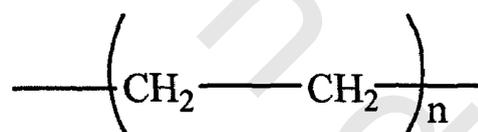


This could be attributed to the presence of branched methyl group  $-CH_3$  in atactic polypropylene, and due to the presence of sigma bonds ( $\sigma$  bond) in its structure which are usually stronger than  $\pi$  bond; beside atactic polypropylene has volume resistivity  $10^{16}$  ohm.cm, table (3).

The non polar plastics are truly covalent and generally have symmetrical molecules. In these materials there are no polar dipoles present and the application of an electric field does not try to align any dipoles. The electric field does, however, move the electrons slightly in the direction of the electric field to create (electron polarization)

in this case the only movement is that of electrons and this is effectively instantaneous. Examples of non-polar plastics are Polytetrafluoro ethylene, polyethylene, polypropylene and polystyrene. These materials tend to have high resistivities and low dielectric constants. The structure of the polymer determines if it is polar or non polar and this determines many of the dielectric properties of the plastic.

For non polar plastics the dielectric constant is independent of the alternating current frequency because the electron polarization is effectively instantaneous. Non polar plastic always have dielectric constant of less than 3<sup>(152)</sup>. In the same time, the value of greases S<sub>1</sub> has good insulating properties (formulated from S<sub>0</sub> and polyethylene) due to the presence of saturated bonds in its structure. Polyethylene has volume resistivity 10<sup>16</sup> ohm.cm as shown in table (2).



On the other hand, the sample S<sub>3</sub> (formulated from S<sub>0</sub> and PVC unplasticized) has good dielectric properties better than sample S<sub>4</sub> (formulated from S<sub>0</sub>, PVC and triisopropyl phosphate), this may be due to that PVC is known to have kinetically rigid chains. On the addition of plasticizer they are effective enough to penetrate inside the molecular bundles of PVC and to separate the polymeric chain. Accordingly, the mutual interaction between plasticizer and PVC becomes appreciable, leading to high molecular mobility, therefore, the PVC plasticized should exhibit higher mobility<sup>(114)</sup>. i.e. if a polymer containing plasticizer (polar group) is placed in an electric field, orientation of its segments and smaller kinetic units will be observed to field frequency ratios, and this give rise to definite values of dielectric constant and dielectric loss<sup>(114)</sup>.

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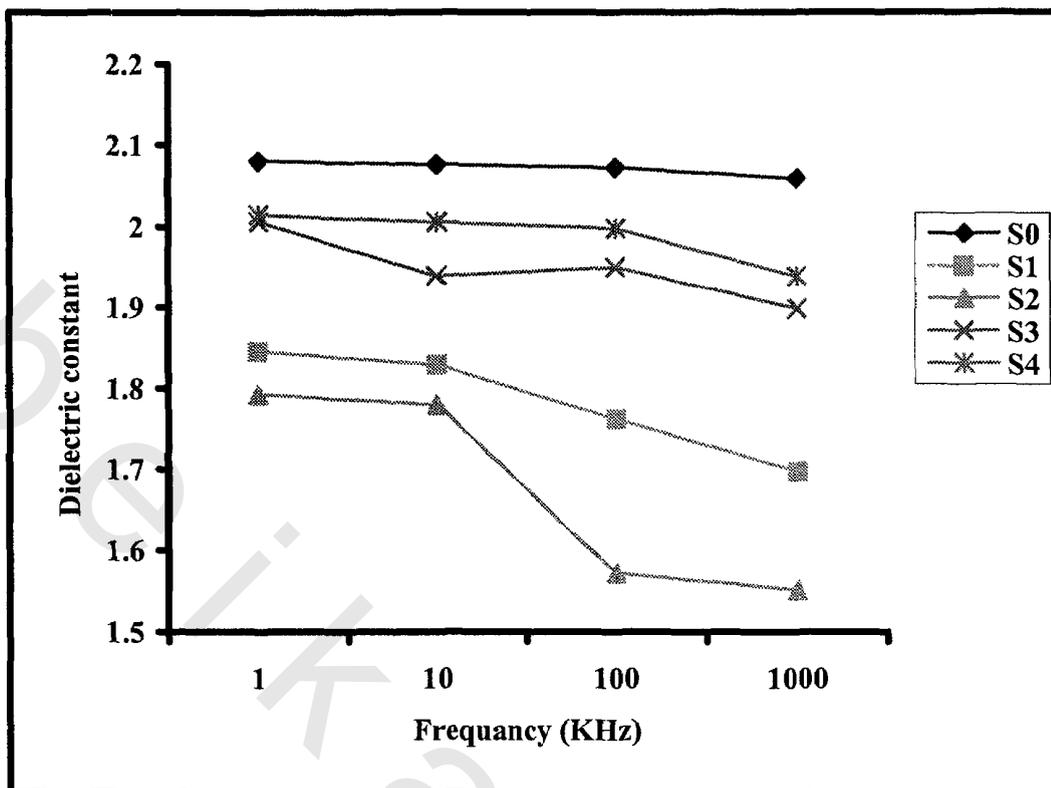
This proves that, the dielectric properties improve gradually with adding atactic polypropylene or polyethylene or unplasticized PVC to  $S_0$ , i.e. all samples have dielectric properties better than sample  $S_0$ .

**Table (22): Dielectric measurement of the prepared greases S<sub>0</sub>, S<sub>1</sub>, S<sub>2</sub>, S<sub>3</sub> and S<sub>4</sub>**

Specification	Sample Notation				
	S <sub>0</sub>	S <sub>1</sub>	S <sub>2</sub>	S <sub>3</sub>	S <sub>4</sub>
Permativity ( $\epsilon'$ ) at frequency,					
1 KHz	2.0798	1.8451	1.7921	2.0056	2.0140
10 KHz	2.0761	1.8286	1.7796	1.9393	2.0055
100 KHz	2.0716	1.7619	1.5715	1.95	1.9976
1000 KHz	2.0589	1.6975	1.551	1.898	1.9387
Dielectric loss ( $\epsilon''$ ) at frequency,					
1 KHz	0.6429	0.0449	0.0365	0.4242	0.5835
10 KHz	0.2344	0.0376	0.0202	0.1878	0.2219
100 KHz	0.0813	0.0435	0.0380	0.0588	0.0669
1000 KHz	0.0216	0.0062	0.0023	0.0075	0.0120

**Table (23): Volume resistivity measurements of the prepared greases S<sub>0</sub>, S<sub>1</sub>, S<sub>2</sub>, S<sub>3</sub> and S<sub>4</sub>**

Specification	Sample Notation				
	S <sub>0</sub>	S <sub>1</sub>	S <sub>2</sub>	S <sub>3</sub>	S <sub>4</sub>
Volume resistivity. Ohm.cm, at 35°C at frequency,					
1 KHz	$0.23 \times 10^{10}$	$0.5 \times 10^{11}$	$0.9 \times 10^{12}$	$0.2 \times 10^{12}$	$0.9 \times 10^{11}$
10 KHz	$0.913 \times 10^9$	$0.5 \times 10^{11}$	$0.4 \times 10^{12}$	$0.3 \times 10^{11}$	$0.36 \times 10^{11}$
100 KHz	$0.305 \times 10^9$	$0.26 \times 10^{11}$	$0.49 \times 10^{11}$	$0.13 \times 10^{11}$	$0.11 \times 10^{11}$
1000 KHz	$0.116 \times 10^9$	$0.96 \times 10^{10}$	$0.04 \times 10^{11}$	$0.38 \times 10^{10}$	$0.01 \times 10^{10}$



Fig(30): The dielectric constant ( $\epsilon'$ ) vs. frequency at 35° C for S<sub>0</sub>, S<sub>1</sub>, S<sub>2</sub>, S<sub>3</sub> and S<sub>4</sub> greases

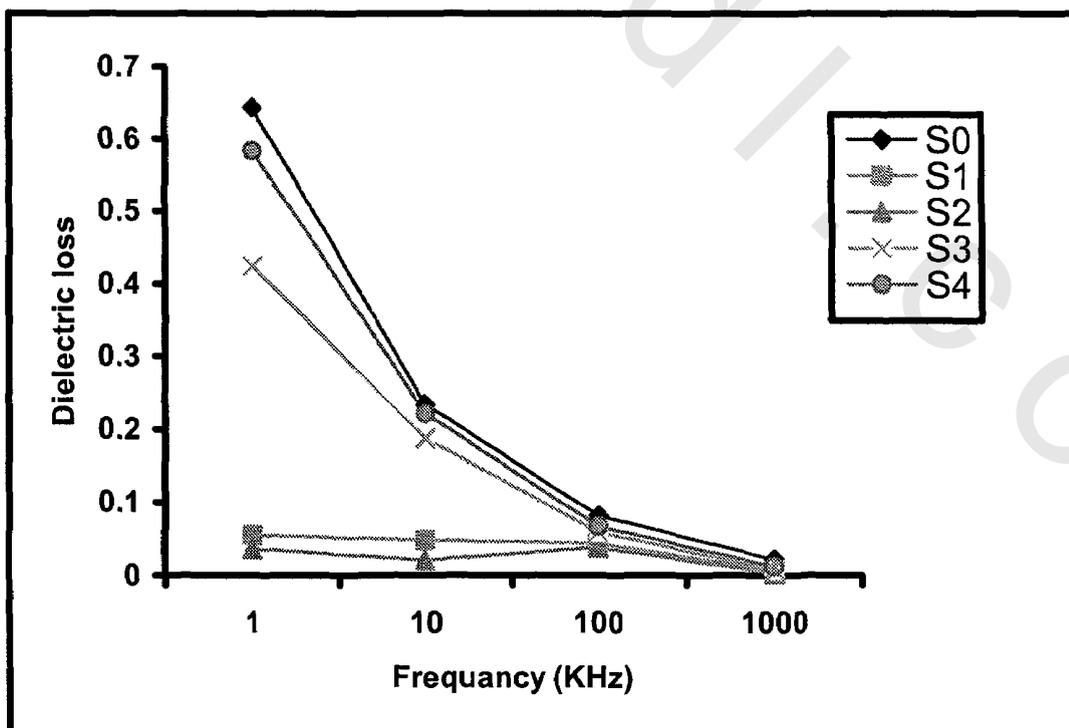


Fig (31): The dielectric loss ( $\epsilon''$ ) vs. frequency at temperature 35° C for S<sub>0</sub>, S<sub>1</sub>, S<sub>2</sub>, S<sub>3</sub> and S<sub>4</sub> greases

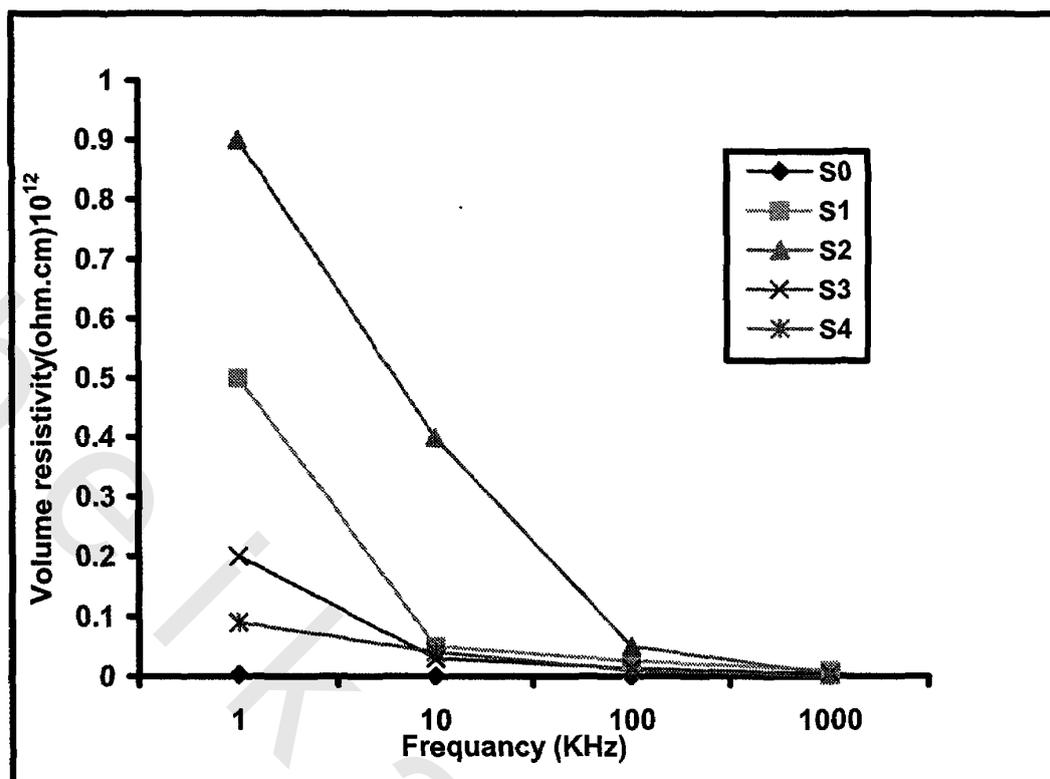


Fig (32): The relation between volume resistivity and frequency at 35° C for S<sub>0</sub>, S<sub>1</sub>, S<sub>2</sub>, S<sub>3</sub> and S<sub>4</sub> greases

It is evident from table (24) that, the value of  $\epsilon''$  decreases with increasing frequency from 1 KHz to 1000 KHz at 35°C. Table (24) shows that the values of  $\epsilon''$  and  $\epsilon'$  decrease with adding poly(1-octadecene-co-maleic anhydride)bis behanate ester AC<sub>22</sub> or poly (1-octadecene-co-maleic anhydride)bis stearate ester AC<sub>18</sub> or ethylene glycol-bis stearate ester EGS in mixture with atactic polypropylene to sample S<sub>0</sub>, especially at low frequency region 10 KHz.

Table (24) and figs (33, 34), shows that the values of  $\epsilon''$  and  $\epsilon'$  for the samples S<sub>0</sub>, S<sub>5</sub>, S<sub>6</sub> and S<sub>7</sub> are in the order of S<sub>5</sub> < S<sub>6</sub> < S<sub>7</sub> < S<sub>0</sub>. Since S<sub>5</sub> and S<sub>6</sub> have the lowest value of  $\epsilon''$  and  $\epsilon'$  i.e. good insulator.

Also, the table (25) and fig (35) Show the volume resistivity of samples S<sub>5</sub>, S<sub>6</sub> and S<sub>7</sub> decreases with increasing frequency from 1 KHz to 1000 KHz at 35°C. Since the value of volume resistivity of S<sub>5</sub>, S<sub>6</sub> and S<sub>7</sub> are higher and better than sample S<sub>0</sub>.

$S_7$  respectively. i.e. these samples have the lowest value of  $\tilde{\epsilon}$  and  $\epsilon''$  beside the highest value of volume resistivity at 1 KHz. this could be attributed to steric hindrance due to the presence of largely alkyl radicals in copolymer  $AC_{22}$  or  $AC_{18}$  or chemical EGS ( $S_7$ ). Despite the presence of a number of ester groups that are capable of orientation in an electric field. This restricts the rotation the large aggregates of the polymeric chain.

It is significant to point out that the effect of the presence of one  $C_{16}H_{33}$  group in the copolymer A or two groups of  $C_{22}H_{45}$  in copolymer  $AC_{22}$  or two groups of  $C_{18}H_{37}$  in copolymer  $AC_{18}$  and ester EGS increase steric hindrance and accordingly, the decrease of  $\tilde{\epsilon}$  and  $\epsilon''$  is more pronounced when copolymer  $AC_{22}$  or  $AC_{18}$  is introduced (samples  $S_5$  or  $S_6$ ) at 1 KHz. Also the volume resistivity of samples ( $S_5$ -  $S_7$ ) is higher and better than sample  $S_0$  table (25) and fig (35).

Hence, the addition of EGS or copolymer ( $AC_{22}$  or  $AC_{18}$ ) in mixture with atactic polypropylene to the sample  $S_0$  improves the  $\tilde{\epsilon}$ ,  $\epsilon''$  and volume resistivity. i.e. sample  $S_5$ ,  $S_6$  and  $S_7$  have low  $\tilde{\epsilon}$  and  $\epsilon''$  and high volume resistivity due to the ability of the copolymer groups (bulky groups) to switch orientation in phase with an alternating current may be large.

Table (24): Dielectric measurement of the prepared greases  $S_0$ ,  $S_5$ ,  $S_6$  and  $S_7$ 

Specification	Sample Notation			
	$S_0$	$S_5$	$S_6$	$S_7$
Permativity ( $\epsilon'$ ) at frequency,				
1 KHz	2.0798	1.7565	1.789	1.792
10 KHz	2.0761	1.5686	1.775	1.779
100 KHz	2.0716	1.4452	1.560	1.571
1000 KHz	2.0589	1.0136	1.018	1.811
Dielectric loss ( $\epsilon''$ ) at frequency,				
1 KHz	0.6429	0.2865	0.353	0.465
10 KHz	0.2344	0.0316	0.088	0.090
100 KHz	0.0813	0.0323	0.035	0.038
1000 KHz	0.0216	0.0046	0.0018	0.002

•At temperature 35°C

**Table (25): Volume resistivity measurements of the prepared greases S<sub>0</sub>, S<sub>5</sub>, S<sub>6</sub> and S<sub>7</sub>**

Specification	Sample Notation			
	S <sub>0</sub>	S <sub>5</sub>	S <sub>6</sub>	S <sub>7</sub>
Volume resistivity. Ohm.cm, at 35°C at frequency,				
1 KHz	$0.23 \times 10^{10}$	$0.638 \times 10^{11}$	$0.60 \times 10^{11}$	$0.40 \times 10^{11}$
10 KHz	$0.913 \times 10^9$	$0.630 \times 10^{11}$	$0.48 \times 10^{11}$	$0.42 \times 10^{11}$
100 KHz	$0.305 \times 10^9$	$0.766 \times 10^{10}$	$0.44 \times 10^{10}$	$0.25 \times 10^{10}$
1000 KHz	$0.116 \times 10^9$	$0.549 \times 10^{10}$	$0.23 \times 10^{10}$	$0.11 \times 10^{10}$

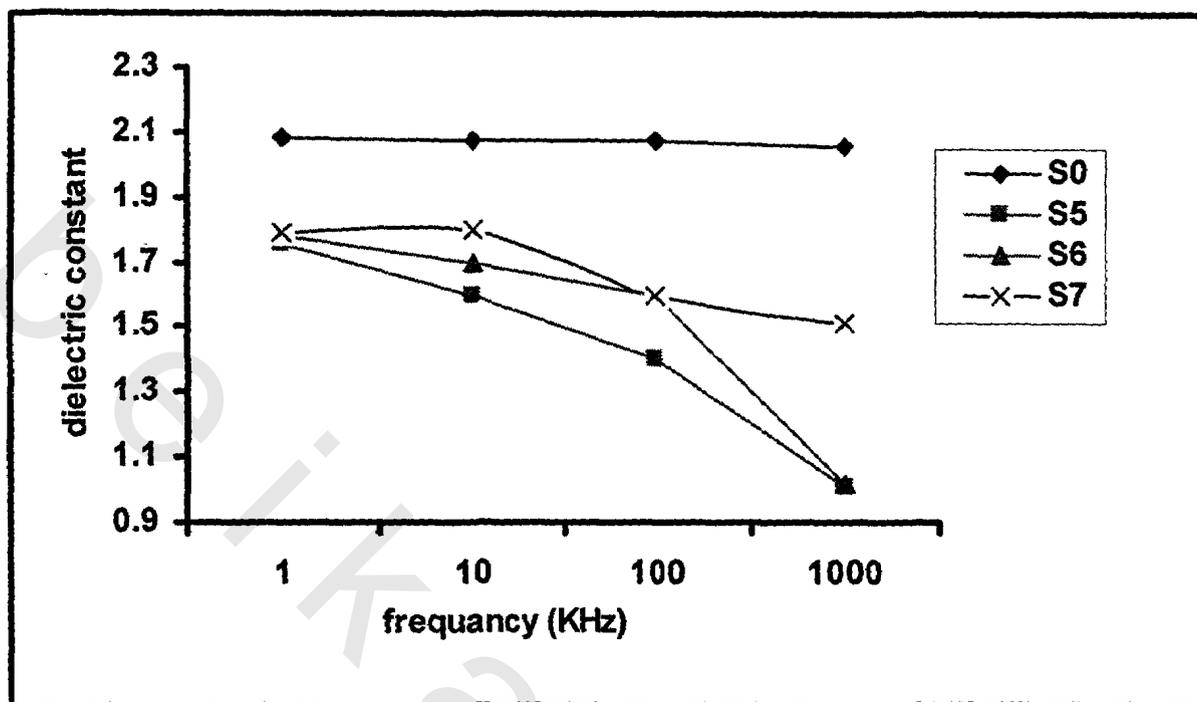


Fig (33): The dielectric constant ( $\epsilon'$ ) vs. frequency at temperature 35° C for S<sub>0</sub>, S<sub>5</sub>, S<sub>6</sub> and S<sub>7</sub> greases

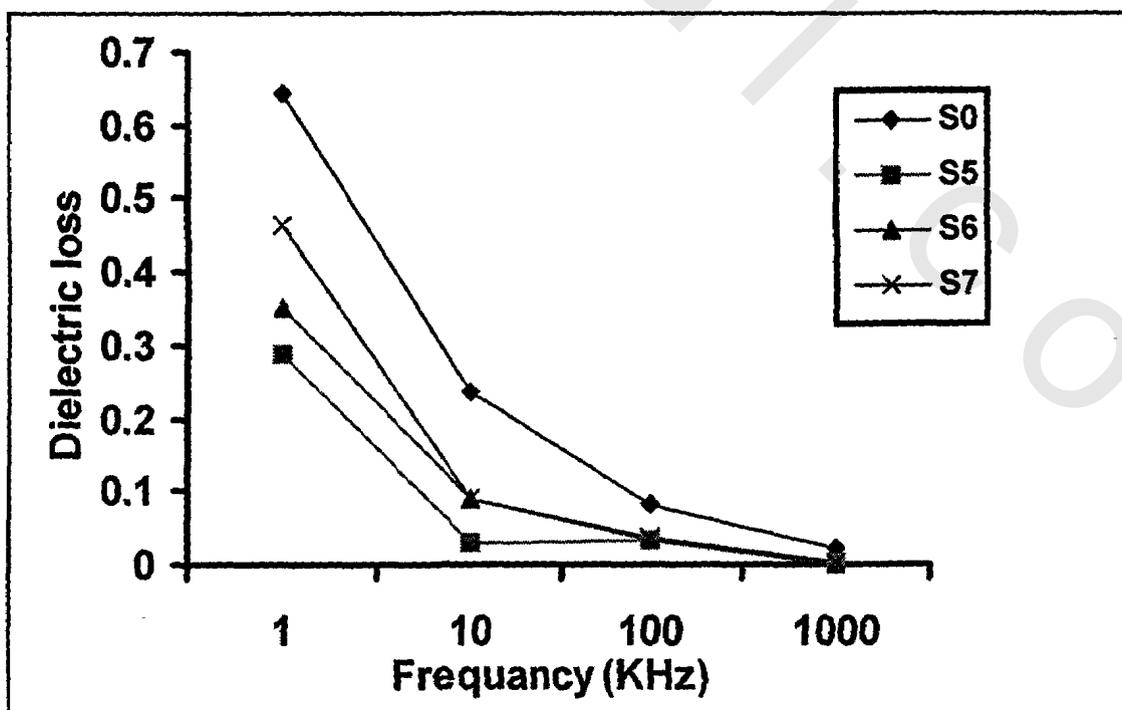


Fig (34): The dielectric loss ( $\epsilon''$ ) vs. frequency at temperature 35° C for S<sub>0</sub>, S<sub>5</sub>, S<sub>6</sub> and S<sub>7</sub> greases

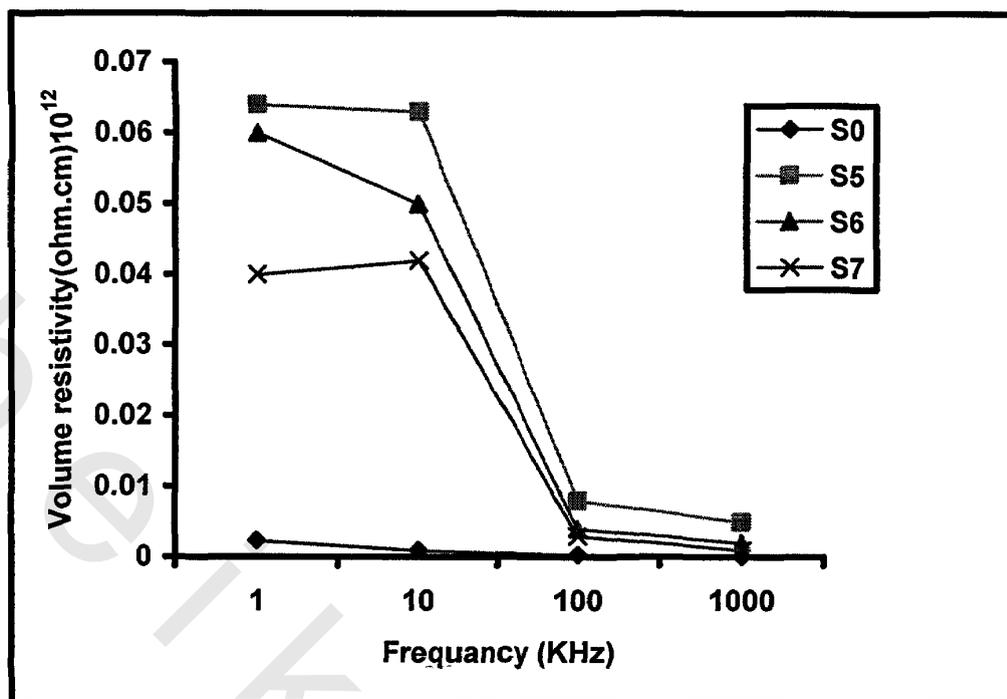


Fig (35): The relation between volume resistivity and frequency at temperature 35° C for S<sub>0</sub>, S<sub>5</sub>, S<sub>6</sub> and S<sub>7</sub> greases

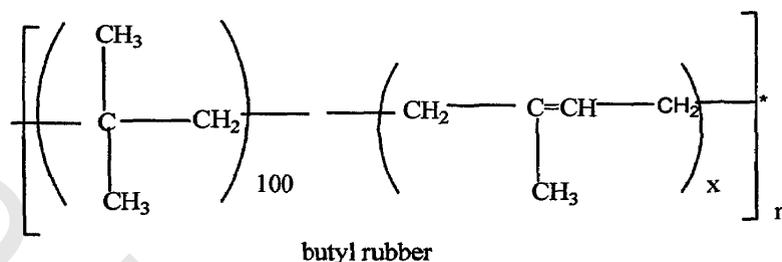
The presenting data given in table (26) and figs (36, 37) show that the  $\epsilon'$  and  $\epsilon''$  of the samples S<sub>0</sub>, S<sub>8</sub>, S<sub>9</sub> and S<sub>10</sub>. Decrease with increasing frequency from 1 KHz to 1000 KHz at 35 °C.

It is shown that the  $\epsilon'$  and  $\epsilon''$  of the samples as following: S<sub>9</sub> has more dielectric properties than S<sub>8</sub>, S<sub>10</sub> and S<sub>0</sub>. Since the sample S<sub>9</sub> (containing isobutylene-isoprene copolymer) butyl rubber has good electrical insulation i.e. least value of  $\epsilon'$  and  $\epsilon''$ .

Also, the volume resistivity of these samples which are represented in table (27) and fig (38) show that the values of volume resistivity decrease with increasing frequency (1 KHz to 1000 KHz). It is in the order S<sub>9</sub> > S<sub>8</sub> > S<sub>10</sub> > S<sub>0</sub>. Since S<sub>9</sub> has the highest value of volume resistivity.

This means that the dielectric properties (electrical insulation) of these samples are in the order S<sub>9</sub> > S<sub>8</sub> > S<sub>10</sub> > S<sub>0</sub>. This indicates that the presence of methyl groups as branched groups and presence of saturated bonds ( $\sigma$  bond) in

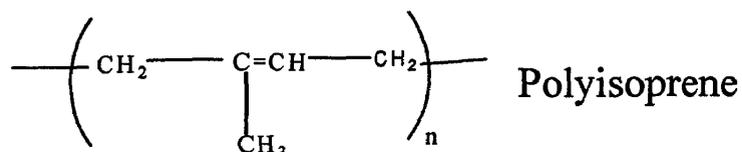
sample S<sub>9</sub>, decreases the moving of units of butyl rubber and leading to the decreasing of  $\epsilon'$  and  $\epsilon''$  and increasing volume resistivity (despite of the presence of isoprene with small proportions) <sup>(153)</sup>. i.e. improving dielectric properties of sample S<sub>9</sub>.



Where  $x=0.6-3$   $n=350-1000$

It is also clear that from tables (26, 27) and figs (36, 37, 38) that sample S<sub>8</sub> (containing bitumen) has the highest dielectric properties than sample S<sub>10</sub> (containing polyisoprene) this may be due to that sample S<sub>8</sub> has (83% carbon, 10% hydrogen, 7% oxygen, nitrogen, sulfure) i.e. bitumen contains a much higher proportions of relatively high molecular weight paraffin, naphthenic hydrocarbon and their derivatives (having carbon number greater than C<sub>25</sub> with a high carbon to hydrogen ratio). <sup>(153)</sup>.

On the other hand sample S<sub>10</sub> (containing polyisoprene), has unsaturated bond in its structure ( $\pi$  bond). Due to  $\pi$  bonds are usually weaker than sigma bonds because of their (negatively charged) electron density farther far from the positive charge of the atomic nucleus and thus it requires less energy to be free and cause rupture of the double bond.



The rupture of the double bonds ( $\pi$  bonds) may be due to the excitation by heat with the formation of free

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electrons that are mobile under the influence of an electric field and, thus greatly increases the degree of electrical conductivity in this sample. So the dielectrical properties of sample S<sub>9</sub> is superior than that of the other samples, which may be due to the presence of alkyl group and less the unsaturation bond in the structure of this sample.

This means that, the dielectrical properties (lowest value of  $\epsilon'$ ,  $\epsilon''$  and highest value of volume resistivity, ohm.cm. at 1 KHz at 35 °C) are improved with adding butyl rubber, bitumen or polyisoprene with certain amounts to sample S<sub>0</sub>, i.e. all samples S<sub>9</sub>, S<sub>8</sub> and S<sub>10</sub> have dielectric properties better than sample S<sub>0</sub> respectively.

Table (26): Dielectric measurement of the prepared greases  $S_0$ ,  $S_8$ ,  $S_9$ , and  $S_{10}$ 

Specifications	Sample Notation			
	$S_0$	$S_8$	$S_9$	$S_{10}$
Permativity ( $\epsilon'$ ) at frequency, 1 KHz 10 KHz 100 KHz 1000 KHz Dielectric loss( $\epsilon''$ ) at frequency, 1 KHz 10 KHz 100 KHz 1000 KHz	2.0798	1.8988	1.806	2.050
	2.0761	1.8898	1.768	2.041
	2.0716	1.8690	1.690	2.0119
	2.0589	1.6958	1.525	1.946
	0.6429	0.3163	0.025	0.545
	0.2344	0.1431	0.034	0.1909
	0.0813	0.0428	0.011	0.053
	0.0216	0.0198	0.0069	0.029

• At temperature 35°C

**Table (27): Volume resistivity measurements of the prepared greases S<sub>0</sub>, S<sub>8</sub>, S<sub>9</sub> and S<sub>10</sub>**

Specifications	Sample Notation			
	S <sub>0</sub>	S <sub>8</sub>	S <sub>9</sub>	S <sub>10</sub>
Volume resistivity.Ohm.cm, at35°C at frequency,				
1 KHz	$0.23 \times 10^{10}$	$0.48 \times 10^{11}$	$0.9 \times 10^{12}$	$0.28 \times 10^{11}$
10 KHz	$0.913 \times 10^9$	$0.15 \times 10^{11}$	$0.05 \times 10^{12}$	$0.46 \times 10^{10}$
100 KHz	$0.305 \times 10^9$	$0.57 \times 10^{10}$	$0.62 \times 10^{11}$	$0.11 \times 10^{10}$
1000 KHz	$0.116 \times 10^9$	$0.02 \times 10^{10}$	$0.21 \times 10^{10}$	$0.86 \times 10^9$

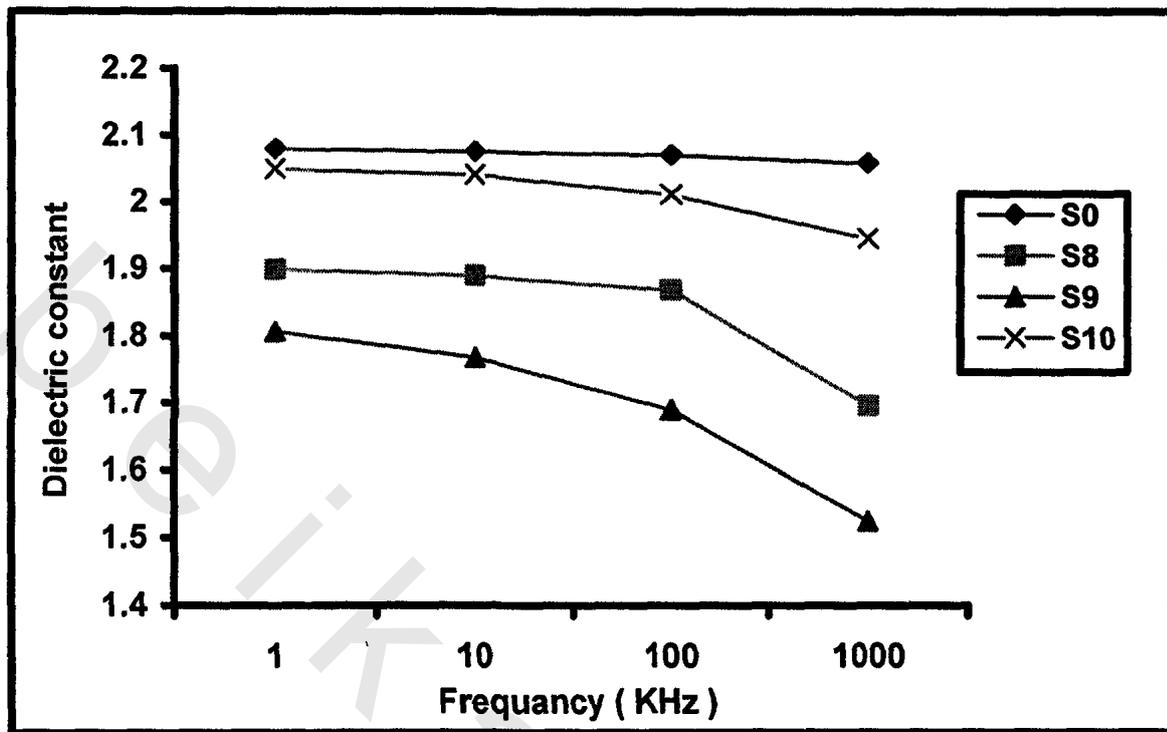


Fig (36): The dielectric constant ( $\epsilon'$ ) vs. frequency at temperature 35 °C for S<sub>0</sub>, S<sub>8</sub>, S<sub>9</sub> and S<sub>10</sub> greases

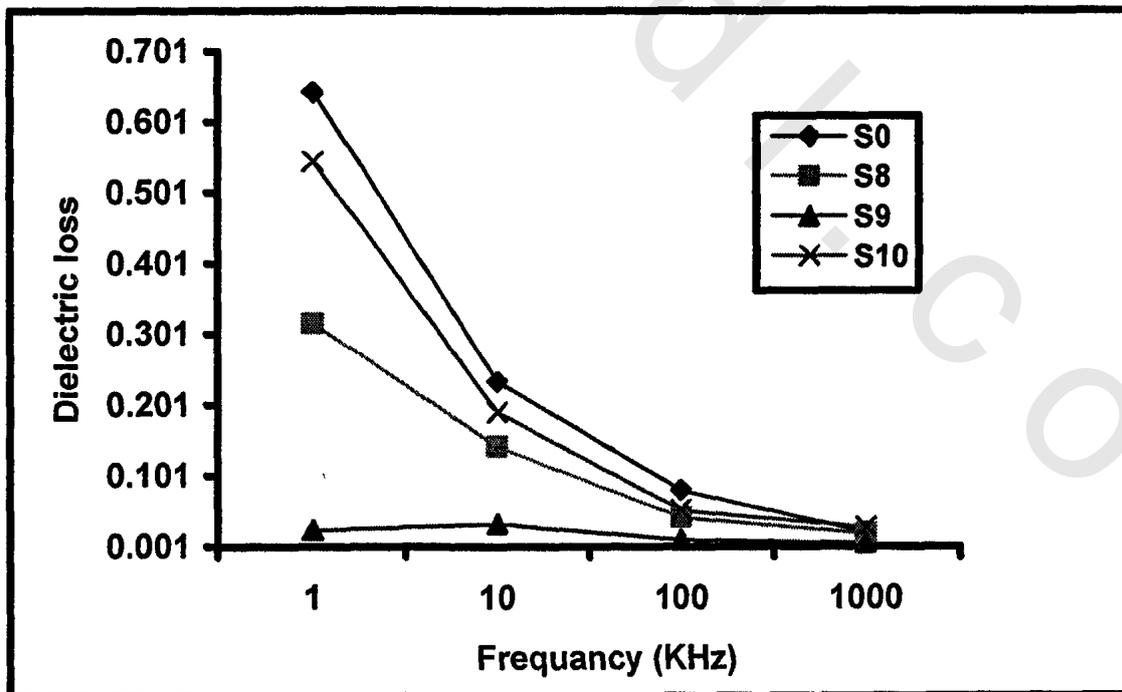
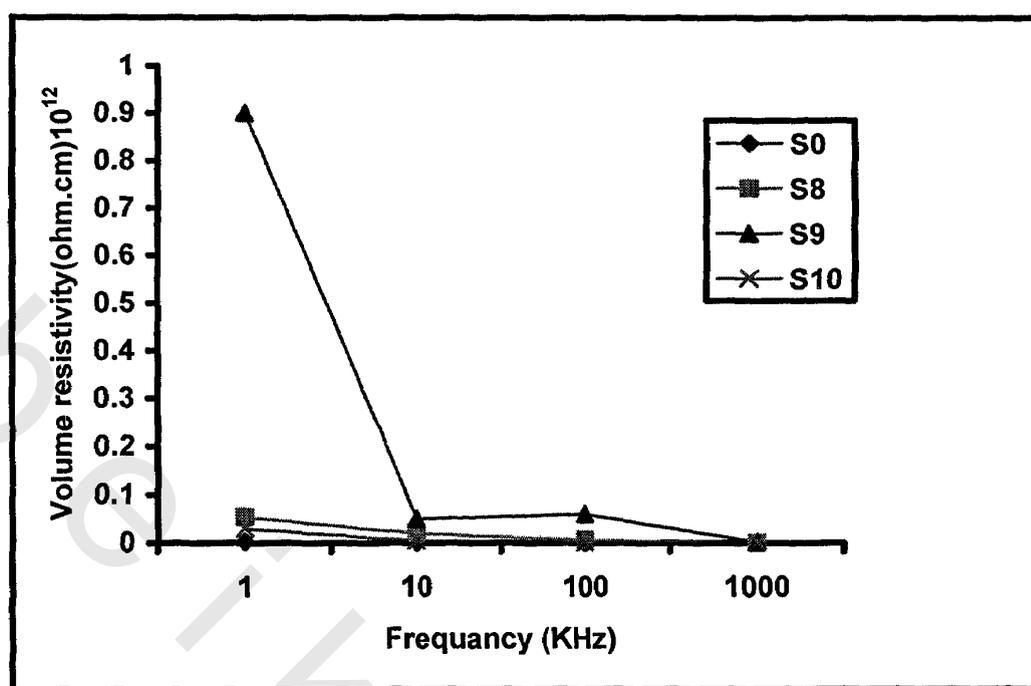


Fig (37): The dielectric loss ( $\epsilon''$ ) vs. frequency at temperature 35 °C for S<sub>0</sub>, S<sub>8</sub>, S<sub>9</sub>, and S<sub>10</sub> greases



**Fig (38):** The relation between volume resistivity and frequency at temperature 35° C for S<sub>0</sub>, S<sub>8</sub>, S<sub>9</sub> and S<sub>10</sub> greases.

The dielectric constant  $\epsilon'$ , dielectric loss  $\epsilon''$  and volume resistivity for the prepared greases S<sub>0</sub>, S<sub>11</sub>, S<sub>12</sub>, S<sub>13</sub>, S<sub>14</sub>, and S<sub>15</sub> are over the frequency range from 1 KHz to 1000 KHz at temperature 35°C were studied. The results obtained for of  $\epsilon'$ ,  $\epsilon''$  and volume resistivity vs. frequency for these samples are shown in tables (28, 29) and figs (39, 40, 41). It is evident from figures and tables that  $\epsilon'$  decreases with increasing frequency.

The fundamental properties of insulating greases are the electrical properties (dielectric constant  $\epsilon'$ , dielectric loss  $\epsilon''$  and volume resistivity).

The data obtained in tables (28, 29) and figs (39, 40) show that the dielectric constant of the samples are in the order S<sub>14</sub> more electrical insulating than S<sub>13</sub> > S<sub>15</sub> > S<sub>12</sub> > S<sub>11</sub> > S<sub>0</sub>. Since S<sub>14</sub> has the lowest value of dielectric constant (1.9011) at frequency 1 KHz at 35°C. The dielectric loss decreases with increasing frequency. The  $\epsilon''$  of the samples is in order S<sub>14</sub> more electrical insulating than S<sub>13</sub> > S<sub>15</sub> > S<sub>12</sub> >

$S_{11} > S_0$ . Since  $S_{14}$  has the lowest value of  $\epsilon''$  (0.773) at 1 KHz.

On the other hand the change of the volume resistivity of these samples  $S_0$ ,  $S_{11}$ ,  $S_{12}$ ,  $S_{13}$ ,  $S_{14}$ , and  $S_{15}$  represented in table (29) and fig (41), show that the volume resistivity decreases with increasing frequency. Since  $S_{14}$  has the highest value of volume resistivity ( $0.7 \times 10^{12}$  ohm.cm.)

The volume resistivity of the samples is in the order  $S_{14}$  more electrical insulating than  $S_{13} > S_{15} > S_{12} > S_{11} > S_0$ .

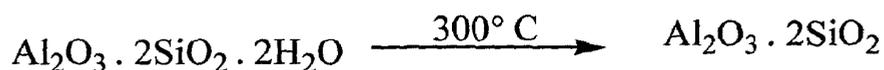
This proves that, the dielectrical properties of sample  $S_{14}$  greases formulated from nano-talc powder (magnesium silicate) with the structure  $Mg_2Si_4O_{10}(OH)_2$  or  $H_2Mg_2(SiO_3)_4$  which composed from (MgO 31.88%,  $SiO_2$  63.77%,  $H_2O$  4.75 %) i.e.  $Mg^{+2}Si^{+4}O^{-2}OH^{-1}$  due to ion  $Mg^{+2}$  blocks the flow of current from one part of the device to another i.e. blocks the migration path of the electrons.

The improvement occur in electrical insulation due to the presence of silicate group, where silicate consists of silicon with oxygen as the legand silicate anions, with a negative net electrical charge, it must have to change balanced by other cations to make an electrically neutral compound. Moreover, talc consists of a two silicate layers are bonded together by weak Vander Waals forces.

The data obtained in tables (28, 29) and figs (39, 40, 41) show that sample  $S_{13}$  has silica dioxide good dielectrical properties. This may be attributed to the  $SiO_2$  produced from rice husk is a giant covalent structure. The strong bonds in three dimensions make it a hard, does not conduct electricity, there are not any delocalized electrons. All the electrons are held tightly between the atoms, and are not free to move.

It is evidence from tables (28, 29) and figs (39, 40 and 41) that the sample  $S_{15}$  (formulated from  $S_0$  and nano

kaolin) is dielectric this may be due to the structure of nano kaolin



(39%  $\text{Al}_2\text{O}_3$ , 46.3% Silica, 13.9  $\text{H}_2\text{O}$ ). The dielectric is attributed to the presence of  $\text{Al}_2\text{O}_3$ , because of possessing strong interatomic bonding; it gives to its desirable material characteristics. Also, this may be due the presence of silica group in the structure of nano kaolin.

On the other hand, the dielectric properties of  $S_{12}$  (formulated from  $S_0$  and sodium silicate  $\text{Na}_2\text{SiO}_3$ ) are better than the sample  $S_{11}$  (formulated from  $S_0$  and ultramarine,  $\text{Na}_{8-10}\text{Al}_{16}\text{Si}_6\text{O}_{24}\text{S}_{2-4}$ ) i.e. structure of ultramarine contains  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ .

The sample  $S_{11}$  and  $S_{12}$  have dielectric properties, despite the presence of sodium ions  $8\text{Na}^+$  in case of ultramarine,  $2\text{Na}^+$  ion in case of sodium silicate (sodium are classified as conductors because their outer electrons are not tightly bound valance electrons).

This could be attributed to Mott insulation theory<sup>(154-156)</sup> which say that there is class of materials that are expected to conduct electricity under conventional band theories, but which in fact turn out to be insulators when measured. This effect is due to electron-electron interactions which are not considered in the formulation band theory.

The presence of silicate group and silica group beside alumina in its structure improve the electrical insulating character.

This proves that, the dielectric properties of grease  $S_{14}$  improves gradually with adding nano - talc or  $S_{13}$  with silica,  $S_{15}$  with nano-kaolin,  $S_{12}$  with sodium silicate or  $S_{11}$  with ultramarine. i.e. all samples have dielectric properties better than  $S_0$ .

**Table (28): Dielectric measurement of the prepared greases  $S_0$ ,  $S_{11}$ ,  $S_{12}$ ,  $S_{13}$ ,  $S_{14}$  and  $S_{15}$**

Specifications	Sample Notation					
	$S_0$	$S_{11}$	$S_{12}$	$S_{13}$	$S_{14}$	$S_{15}$
Permativity ( $\epsilon'$ ) at frequency,						
1 KHz	2.0798	2.074	2.065	1.92	1.9011	1.9314
10 KHz	2.0761	2.070	2.05	1.916	1.9018	1.92.09
100 KHz	2.0716	2.026	2.010	1.919	1.9023	1.923
1000 KHz	2.0589	2.010	1.957	1.860	1.7044	1.9113
Dielectric loss( $\epsilon''$ ) at frequency,						
1 KHz	0.6429	0.4946	0.4455	0.3759	0.0773	0.3946
10 KHz	0.2344	0.2911	0.2600	0.153	0.0396	0.229
100 KHz	0.0813	0.0638	0.0496	0.046	0.0048	0.0519
1000 KHz	0.0216	0.0200	0.0014	0.0065	0.0066	0.0021

At temperature 35°C

**Table (29): Volume resistivity measurements of the prepared greases S<sub>0</sub>, S<sub>11</sub>, S<sub>12</sub>, S<sub>13</sub>, S<sub>14</sub> and S<sub>15</sub>**

Specifications	Sample Notation					
	S <sub>0</sub>	S <sub>11</sub>	S <sub>12</sub>	S <sub>13</sub>	S <sub>14</sub>	S <sub>15</sub>
Volume resistivity .Ohm.cm, at 35°C at frequency,						
1 KHz	$0.23 \times 10^{10}$	$0.1 \times 10^{11}$	$0.5 \times 10^{11}$	$0.7 \times 10^{11}$	$0.7 \times 10^{12}$	$0.2 \times 10^{11}$
10 KHz	$0.913 \times 10^9$	$0.039 \times 10^{11}$	$0.44 \times 10^{11}$	$0.53 \times 10^{11}$	$0.99 \times 10^{11}$	$0.06 \times 10^{11}$
100 KHz	$0.305 \times 10^9$	$0.13 \times 10^{10}$	$0.5 \times 10^{10}$	$0.5 \times 10^{10}$	$0.05 \times 10^{11}$	$0.28 \times 10^{10}$
1000 KHz	$0.116 \times 10^9$	$0.88 \times 10^9$	$0.07 \times 10^{10}$	$0.01 \times 10^{10}$	$0.008 \times 10^{10}$	$0.02 \times 10^{10}$

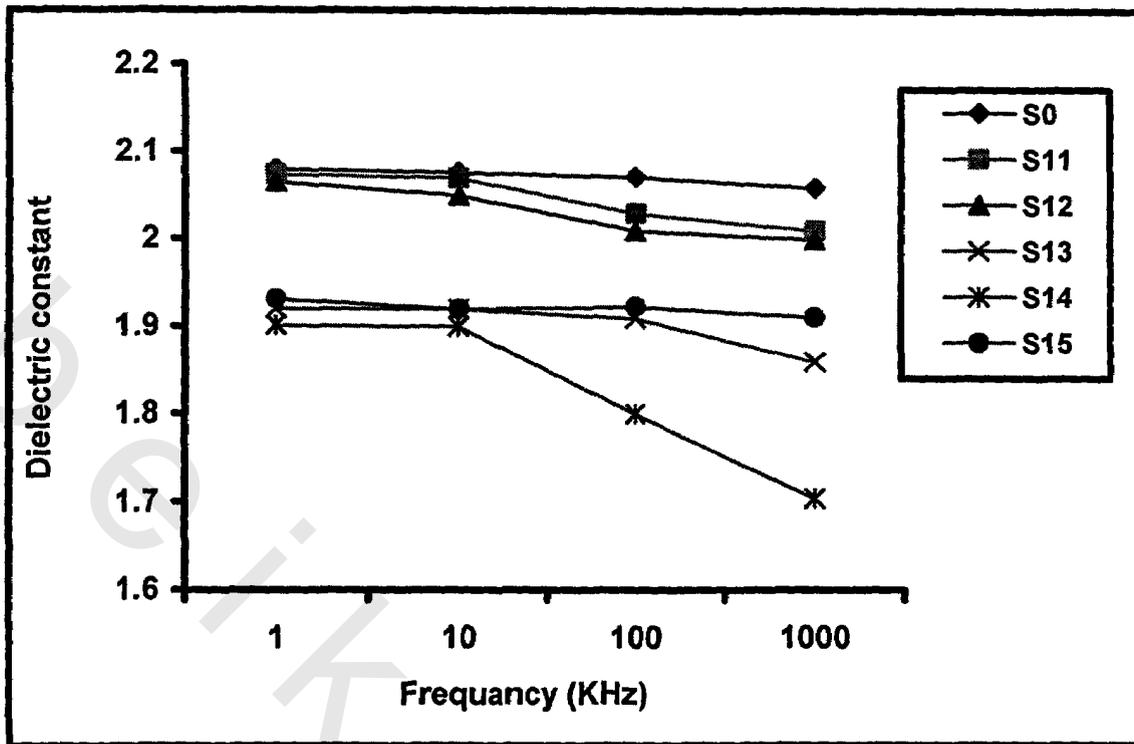


Fig (39): The dielectric constant ( $\epsilon'$ ) vs. frequency at temperature 35° C for S<sub>0</sub>, S<sub>11</sub>, S<sub>12</sub>, S<sub>13</sub>, S<sub>14</sub> and S<sub>15</sub> greases.

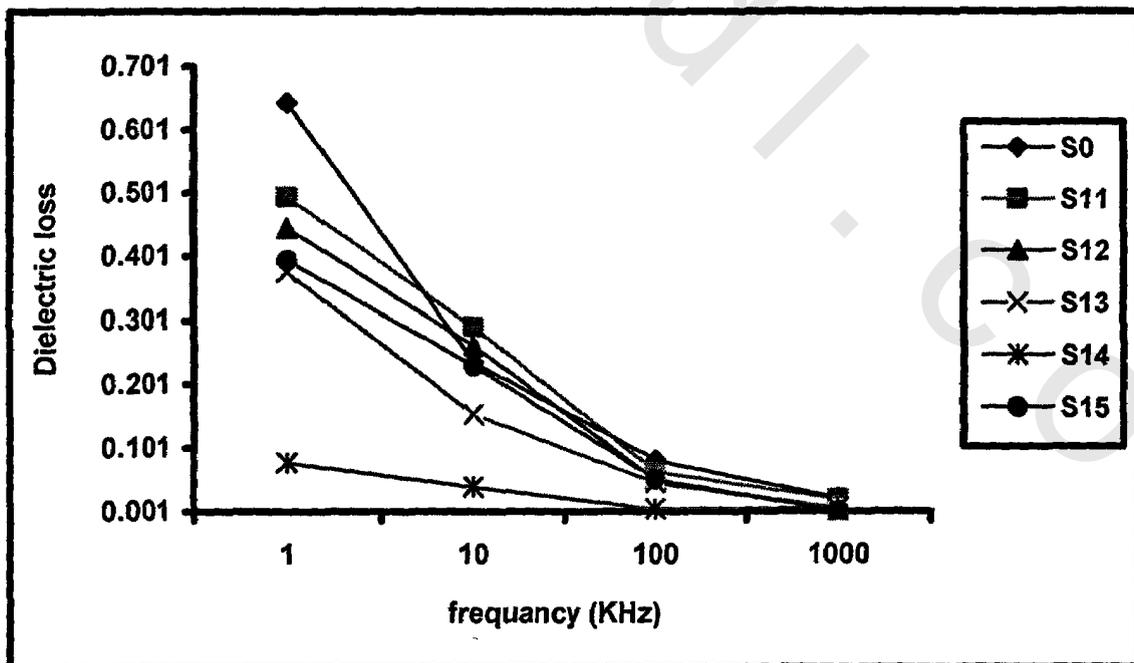


Fig (40): The dielectric loss ( $\epsilon''$ ) vs. frequency at temperature 35°C for S<sub>0</sub>, S<sub>11</sub>, S<sub>12</sub>, S<sub>13</sub>, S<sub>14</sub> and S<sub>15</sub> greases

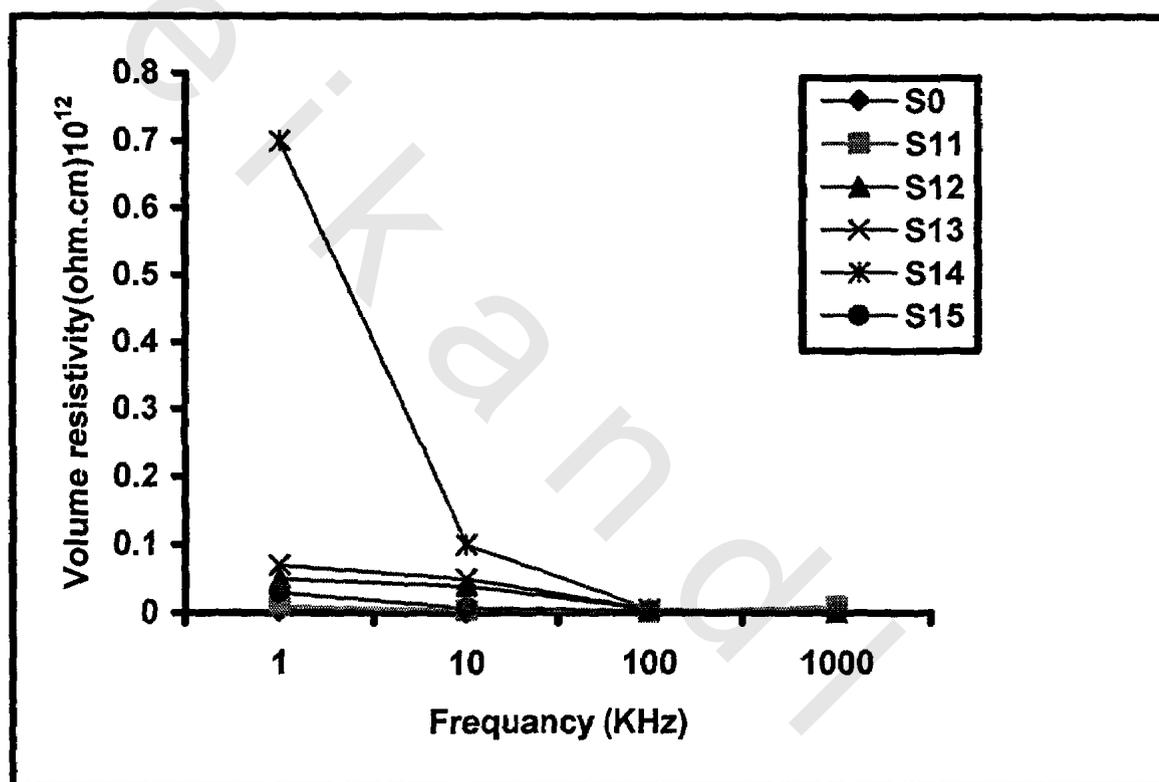


Fig (41): The relation between volume resistivity and frequency at temperature  $35^{\circ}\text{C}$  for  $S_0$ ,  $S_{11}$ ,  $S_{12}$ ,  $S_{13}$ ,  $S_{14}$  and  $S_{15}$  greases