Experimental

II-EXPERIMENTAL WORK

II-1-Chemicals

-Base lube oil grade (260/290):

Flash point (open): 204°C *Viscosity index*: 90-95

Pour point: (-5°C). *Origin:* Misr Petroleum Company

-Transformer oil:

Flash point: 135°C *Viscosity at 40°C:* 12.5cSt

Pour point: (-20°C). *Origin:* Misr petroleum Company.

-Microcrystalline wax:

Melting range: 80-81°C *Oil content %*: 0.5

Flash point, open: 288°C

Density at 20°C: 0.9002 g/Cm^3.

Origin: Alexandria Company for Petroleum.

-Polyethylene:

Melt flow rate (2.16) g/10min: 2 *Density*: 920 Kg/m^3

Vicat softening temperature: 93°C.

Origin : Sidi kerir Petrochemicals Co. Alnahda.

-Atactic polypropylene:

Density: 0.92 g/cm^3 *Melting point*: 170°C.

Origin: Egyption plastic and electrical company, Alexandria

-Bitumen:

Viscosity at 135 °C: 390 Cst *Softening point*: 52 °C

Flash point,(open): 250°C.

Origin: Suze Company for petroleum manufacturing.

-Polyvinyl chloride:

K-value: 70 *Viscosity number*: 125 ml/g

Particle size: > 63μm

Origin: Egyption plastic and electrical company, Alexandria.

-Silicon dioxide:

Bulk density: 1.16 Kg/Cm^3 *Particle size*: 140μm.

-Natural rice husk:

Silica: 8.8-13.3 *Ash*: 13.4-20.4% *Fiber*: 65.3-68.9%.

-Behanyl alcohol (1-docosanol) $C_{22}H_{46}O$:

Melting point: 71°C *Boiling point:* 180°C

Molecular weight: 326.6

-Stearyl alcohol (1-octadecanol) $C_{18}H_{38}O$.

Molecular weight ***: 270.50***

Melting Point: ***59.4-59.8*** **°C**

Refractive Index: 1.4346 (45°C)

-Stearic acid $CH_3(CH_2)_{16}COOH$:

Melting point: 69°C *Boiling point (0.013MPa):* 287°C

Refractive index (n_D^{20}): 1.4335

Origin: Sigma- Aldrich Chemie Gm bH, Germany.

-Maleic anhydride $C_4H_2O_3$:

Molecular weight: 98 *Boiling point*: 200°C.

Origin: Sigma- Aldrich Chemie Gm bH, Germany.

-1-octadecene $C_{18}H_{36}$:

Molecular weight: 252 *Boiling point*: 308°C,

Density at 20°C (d_{20}): 0.848.

- Benzoyl peroxide, $C_{14}H_{10}O_4$:

Molecular weight: 242 *Melting point:* 102-150°C.

Origin: Sigma- Aldrich Chemie Gm bH, Germany.

-Ethylene glycol, $HOCH_2CH_2OH$

Molecular weight: 62 *Density at 20°C (d_{20})*: 1.116

Refractive index (n_D^{20}): 1.4319

- P- toluene sulphonic acid $CH_3C_6H_4SO_3H$:

Melting point: 92°C *Molecular weight*: 172.20

Boiling point (0.027-MPa):140°C

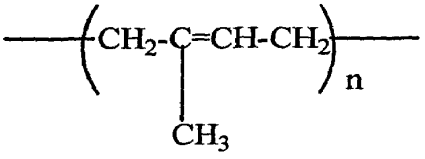
-Triisopropyl phenyl phosphate:

Density at 25°C: 1.131 g/ml *Viscosity at 25 °C*: 95Cst.

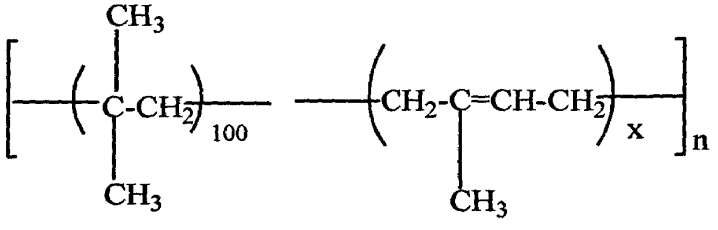
Volume resistivity at 23 °C: 3.3×10^{13} ohm.cm.

Origin: CIBA- Geigy. Industrial chemical division.

-Polyisoprene rubber:



-Butyl rubber (isobutylene-isoprene copolymer).



Where x =0.6 n=350-1000

- Nano-magnesium silicate, (talc) $Mg_2Si_4O_{10}(OH)_2$

Particle size: 100 nm *Bulk density*: 350 g/l

Specific gravity: 2 g/cm^3.

-Nano-kaolin, Al_2O_3. $2SiO_2.2H_2O$:

Specific gravity: 2.6 *Aluminum silicate, SiO_2*: 43.5%,

Particle size: 70 nm.

-Ultramarine, $Na_{8-10}\ Al_6\ Si_6\ O_{24}\ S_{2-4}$:

Melting point: >1000°C (decompose) *PH*: 6-9

Specific gravity: 2.25 g/cm^3 *Particle size*: 125μm.

– Polyoxy ethylene sorbiton-nano-palmitate, Tween (20):

Specific gravity: 1.1 *Boiling Point:* >100°C

Specification number: 43-49. *Refractive index:* 1.4685

pH of 1% Aqouse solution : 5-7

-2, 2` methylen-bis(4-methyl-6-tertiary butyl phenol).

- Sodium silicate, Na_2SiO_3:

Molecular weight: 122 *Density*: 2.4 Kg/L.

-Xylene, $C_6H_4\ (CH_3)_2$:

Density: 0.864-0.861 *Boling point*: 139-144°C

Refractive index (n_D^{20}): 1.5004.

- Benzene C_6H_6:

Density (d_{20}): 0.869 *Boiling point*: 80°C

Refractive index (n_D^{20}): 1.5017.

II-2-Preparations and Measurements

II-2-1-Preparations

II-2-1-1-Preparation of wax gel (wax-oil mixture)

Two types of oils, the first was a base lube oil grade (260/290) and the second was transformer oil.

Lube base oil and transformer oil in the ratio 2:1 by weight were mixed under stirring at 110°C for 30 minutes to produce the lube oil blend. The physico-chemical characteristics of these oils were carried out using ASTM standard methods, tables (12, 13) and their IR spectra are shown in figs (9, 10).

The lube oil blend, table (14), fig (11) was mixed with microcrystalline wax (Alexandria for petroleum company), table (15), fig (12) under stirring at 100-120°C for 30 minutes with different ratios 1:1, 1.85:1, 2.3:1 and 4:1 respectively [85]. After cooling the mixture was thickened to a gel. The obtained samples are denoted by WG_1, WG_2, WG_3 and WG_4 respectively. The formulation and specification were given in tables (17, 16).

II-2-1-2-Preparation of copolymer esters

II-2-1-2-1-Preparation of 1-octadecene-maleic anhydride copolymer

1-octadecene- maleic anhydride copolymer (A) was synthesized by reacting (189g, 0.75 mole) of 1- octadecene with (88.5g, 0.9 mole) of maleic anhydride in the presence of 300 ml. xylene as a solvent and 0.375g. benzoyl peroxide as an initiator. The reaction mixture was then refluxed for 6 hour with vigorous stirring in the temperature range 120-135°C [63]. The specification of the prepared copolymer (A) was shown in the table (11) and fig (5).

II-2-1-2-2-Preparation of poly (1-octadecene- co- maleic anhydride) bis behanate ester

In a three neck flask with a Dean-stark trap, thermometer, and over head mechanical stirrer, the produced 1-octadecene- malice anhydride copolymer (A) (75g, 0.04 mole) was then estrified with (343.2g, 0.96 mole) of behanyl alcohol in the presence of 1 g p- toluene sulfonic acid at 140°C until no further water of reaction was produced . At the end of full estrification processes, the reaction mixture was purified and dried to produce poly (1-octadecene-co-maleic anhydride) bis behanate ester (AC_{22})[63]. The specification of copolymer was shown in table (11) and fig. (6).

II-2-1-2-3- Preparation of poly (1-octadecene- co- malice anhydride) bis stearate ester

1-octadecene- malice anhydride copolymer (A) (75g, 0.04 mole) was estrified with (259.68 g, 0.95 mole) of stearyl alcohol in the presence of 1 g p- toluene sulfonic acid at 140°C using a (Dean-stark apparatus) under stirring, until no further water of reaction was produced. At the end of full estrification processes, the reaction mixture was purified and dried to produce poly (1-octadecene- co-malice anhydride) bis stearate ester (AC_{18}) [63]. The specification of copolymer was shown in table (11) and fig.(7).

II-2-1-3-Preparation of ethylene glycol bis stearate ester

Ethylene glycol bis stearate ester (EGS) was synthesized by reacting stearic acid (2 moles) with ethylene glycol (1 mole) in the presence of 1 g p-toluene sulfonic acid as catalyst. The reaction mixture was refluxed for 7 hour with vigorous stirring at 210°C using a Dean-stark apparatus until no further water of reaction was produced. At the end of estrification processes, the reaction mixture

was purified by benzene and dried to produce the ethylene glycol bis stearate ester (EGS) [121]. The specification of (EGS) was shown in table (11) and fig (8).

II-2-1-4-Preparation of silica from rice husk

Natural rice husk, a by-product of the rice milling industry, is used for preparation a series of modified rice husk using various concentration of KOH according to the following steps: natural rice husk was stirred with KOH (0.5-7.0%)at weight ratio 1:12 and heated to boiling for 30 minutes then the mixture was left over night. The filtrate is washed twice with doubly distilled water and 10% HCl was added (~ 100 ml).

The formed precipitate of silica was washed, dried at 105°C and thus the silica extracted by alkaline treatment has pH 3.3 with small surface area 13 m^2/g and large pore volume 35.5 ml/g, the product was burned at (700-800°C) to obtain a powdered of silica (SiO_2) [122] .Physico-chemical properties of silicon dioxide was observed in table (1) and fig (13).

Table (1): Physico- chemical properties of prepared silicon dioxide

Properties	Value
Bulk Density,Kg/m^3	1.16
Particle size, µm.	140
Colour	Light grey
Component	
SiO_2 %	95.20
Al_2O_3 %	0.29
CaO %	0.24
Na_2O %	0.96
K_2O %	0.60
Fe_2O_3 %	0.57

II-2-1-5-Preparation of several types of insulator greases

II-2-1-5-1–Preparation of greases containing wax gel (S_0)

Oil blend 250.9g (base lube oil 167.27g and transformer oil 83.63g) was heated to 110-120°C and the microcrystalline wax 109 g was added portion wise under stirring for 30 minutes, following by adding 2, 2` methylen bis(4-methyle-6-tertiary butyle phenol) 1.4 g as antioxidant and Polyoxyethelen sorbiton-nano-palmitate 1.4 g as anticorrosion additives and stirring was continued to disperse the additives .After cooling the mixture was thickened to grease S_0. The formulation of sample S_0 grease was shown in table (6). The specification of the resulted S_0 grease is given in table (18).

II-2-1-5-2-Preparation of greases containing polymers S_1, S_2, S_3 and S_4

II-2-1-5-2-1-Preparation of grease S_1

16.4g polyethylene were mixed with 250.9g lube oil blend and heated to 92°C until complete dissolution of polyethylene has taken place. The mixture hold for 30 minutes at 100-110°C, 92.6g of microcrystalline wax was then added with continued agitation followed by adding 1.4g of antioxidant 2, 2` methylen bis(4-methyle-6-tertiary butyle phenol) and 1.4g of anticorrosion Polyoxyethylene sorbiton-nano-palmitate and there allowed to cool to ambient temperature to produce a coherent, homogenous S_1 greases, its formulation and pacification are shown in tables (6, 18).The physical properties of polyethylene included in S_1 grease was observed in table (2).

Table (2): Physical properties of polyethylene

Properties	polyethylene
Type	LL022 OKJ
Melt flow rate (2.16), g/10min.	2
Density, Kg/m3.	920
Vicat softening T°, °C	93
Tensile strength at yield MD/TD, MPa.	10/11
Elongation strength MD/TD, g. /25mic.	100/270
Volume resistivity, ohm.cm.	10^{16}

II-2-1-5-2-2-Preparation of grease S_2

This type was prepared from microcrystalline wax, lube oil blend, atactic polypropylene in the ratio 5.65:15.3:1 by weight respectively in the following manner:

Atactic polypropylene was added portion wise to the lube oil blend (base lube oil and transformer oil) at 170°C with stirring for 60 minutes until complete dissolution of polypropylene has taken place. The temperature was lowered to 110-120 °C and the microcrystalline wax was then added portion wise and the stirring was continued for 30 minutes, 1.4g of 2, 2` methylen bis(4-methyle-6-tertiary butyl phenol) as antioxidant and 1.4g of Polyoxyethylene sorbiton-nano-palmitate as anticorrosion were then dispersed in the mixture. The mixture formed homogenous smooth grease, its formulation and pacification are shown in tables (6, 18). The physical properties of atactic polypropylene included in S_2 grease was observed in table (3).

Table (3): Physical properties of atactic polypropylene

Properties	Atactic polypropylene
Type	atactic
Appearance	Translucent
Density,g./cm^3	0.92
Melting point	170
Water absorption	Nil
Hardness (shore)	60-75
Flammable	Ignites on flame contact, will burnes
Volume resistivity, ohm.cm.	10^{16}

II-2-1-5-2-3-Preparation of grease S_3

A grease composition was made by blending 250.9 g lube oil blend with microcrystalline wax in an amount of 92.7 g, polyvinyl chloride 16.4 g; an antioxidant 1.4g and an anticorrosion inhibitor 1.4g added. then heating the mixture to about 80-85°C with agitation followed by cooling and milling at about 30°C for 15 minutes. The resulted greases S_3 have the formulation and characteristics shown in tables (6, 18). The physical properties of polyvinyl chloride included in S_3 grease was observed in table (4).

Table (4): Physical properties of polyvinyl chloride

Properties	Value
K value	70
Viscosity number ml./g.	125
Inherent viscosity ml./g.	100
Apparent bulk density , g./ml	0.30
Particle size > 63 μm. , % by wt.	< 1

II-2-1-5-2-4-Preparation of grease S_4

Lube oil blend (250.9g), 8.2g of Ttriisopropyl phenyl phosphate as plasticizer and 8.2g of polyvinyl chloride were mixed at 30°C then the mixture was stirring for 20 minutes at temperature of 80-85°C. Then 92.6g of microcrystalline wax were added gradually under strong and constant stirring for obtaining a gelatinous mass, an additive mixture of (antioxidant 1.4g and anticorrosion 1.4g) were added for obtaining a grease S_4 having great extensibility and adherence, its formulation and pacification are shown in tables (6, 18). The general properties of Ttriisopropyl phenyl phosphate included in S_4 grease was observed in table (5)

Table (5): General properties of triisopropyl phenyl phosphate

properties	Value
Specific gravity at 25/25°C	1.36
Density at 25°C, g/ml	1.131
Viscosity at 25°C, Cst	95
Volume resistivity at 23°C ohm.cm.	3.3×10^{13}

Table (6): Formulation of the prepared greases S_0, S_1, S_2, S_3 and S_4

Constituent, parts by weight, g.	Sample Notation				
	S_0	S_1	S_2,	S_3	S_4
Base lube oil	167.27	167.27	167.27	167.27	167.27
Transformer oil	83.63	83.63	83.63	83.63	83.63
Microcrystalline wax	109	92.6	92.6	92.6	92.6
Polyethylene	—	16.4	—	—	—
Atactic polypropylene	—	—	16.4	—	—
Poly vinylchloride	—	—	—	16.4	8.2
Ttriisopropyl phenylphosphate	—	—	—	—	8.2
Polyoxyethylene sorbiton-nano-palmitate	1.4	1.4	1.4	1.4	1.4
2,2` methylen-bis(4-methyle-6-tertiary butyle phenol)	1.4	1.4	1.4	1.4	1.4

II-2-1-5-3- Preparation of greases containing ester and copolymer esters S_5, S_6 and S_7

II-2-1-5-3-1-.Preparation of grease S_5

The lube oil blend 250.9g with the added atactic polypropylene 14.4g is heated to 170°C in a 1 L Pyrex beaker equipped with a stainless steel paddle type stirrer 30 minutes. The mixture is held for 30 minutes at 110°C, wax then added (92.6g) with continued 15 minutes agitation following by adding 2g of poly (1-octadecene-co-malice anhydride) bis behanate ester (AC_{22}). While the agitated mixture is maintained at temperature of 110°C for a minimum of 30 minutes, then the addition of 1.4g of both antioxidant and anticorrosion additives. Then it allowed cooling to ambient temperature to produce coherent, homogenous grease S_5, its formulation and pacification are shown in tables (7, 19).

II-2-1-5-3-2-.Preparation of grease S_6

This grease was prepared from lube oil blend, microcrystalline wax and atactic polypropylene in the ratio 17.4: 6.4: 1 by weight respectively in the following method: The wax was blended with the lube oil blend and the mixture was heated to 170 °C under stirring for 30 minutes until it become homogenous.

After decreasing the temperature to 100-110°C, the poly (1-octadecene-co-malice anhydride) bis stearate ester (AC_{18}) 2g was added portion wise with stirring for further 20 minutes. 1.4g of both antioxidant and anticorrosion were added and stirring was continued to disperse the copolymer and the additives. On cooling to room temperature, the mixture was evaluated; its formulation and pacification are shown in tables (7, 19).

I-2-1-5-3-3-Preparation of grease S_7

lubricating greases S_7 was formulated from wax (92.7g), lube oil blend (250.9g), and atactic polypropylene (14.4g) in the ratio 17.4:6.4:1 by weight respectively.

The polymer (atactic polypropylene) was added portion wise to the gel under stirring at 170 °C, followed by adding 2g of Ethylene glycol bis stearate ester (EGS), 1.4g of both antioxidant and anticorrosion where added to obtain a stable grease . On cooling, the mixture formed smooth grease; its formulation and pacification are shown in tables (7, 19).

Table (7): Formulation of the prepared greases S_0, S_5, S_6 and S_7

Constituent, parts by weight, g.	Sample Notation			
	S_0	S_5	S_6	S_7
Base lube oil	167.27	167.27	167.27	167.27
Transformer oil	83.63	83.63	83.63	83.63
Microcrystalline wax	109	92.6	92.6	92.6
Ac_{22}	—	2	—	—
Ac_{18}	—	—	2	—
EGS	—	—	—	2
Atactic polypropylene	—	14.4	14.4	14.4
Poly oxyethylene sorbiton-nano-palmitate.	1.4	1.4	1.4	1.4
2,2` methylen-bis (4-methyle-6-tertiary butyle phenol).	1.4	1.4	1.4	1.4

II-2-1-5-4- Preparation of greases containing bitumen and rubber S_8, S_9 and S_{10}

II-2-1-5-4-1-Preparation of grease S_8

A glass container was charged with 250.9g of lube oil blend and 1.4g of both antioxidation and anticorrosion where added then the content was heating to the temperature of 50°C with stirring for 15 minutes. Heating was raised to 70°C followed by adding 16.4g bitumen under stirring, when the content was melted and become homogenous, the temperature was raised to 100-120°C for 30minutes, 92.6g of microcrystalline wax were added and stirring was continued to obtain a viscous smooth rubbery grease, followed by cooling to the normal temperature, its formulation and pacification are shown in tables (9, 20). The phsico-chemical properties of bitumen included in S8 grease was observed in table (8)

Table (8): Physico- chemical properties of Bitumen

Test	Value
Penetration at 25°C , 0.1 mm	64
Viscosity at 135°C , cst	390
Softening point, °C	52
Ductility at 25°C, cm.min.	+100
Flash point, °C ,(open) °C	250
Asphaltene	13.8
Resins, %	59.1
Oils, %	29.9
Wax, %	3.5
Specific gravity 25/25°C	1.01-1.06

II-2-1-5-4-2-Preparation of grease S_9

A lubricating grease S_9 Consisting essentially of about 99g microcrystalline wax, 250.9 lube oil blend, 10g butyl rubber (isobutylene-isoprene copolymer) together with 1.4g of both antioxidant and anticorrosion were mixed under stirring for 1/2 hour at 85-90°C until it become homogenous. On cooling coherent and homogenous grease S_9 was obtained, its formulation and pacification are shown in tables (9, 20).

II-2-1-5-4-3-Preparation of greases S_{10}

10g of polyisoprene rubber, 250.9g lube oil blend, were mixed with 99g of microcrystalline wax, 1.4g of antioxidant and 1.4g of anticorrosion, the mixture being kept at 100-120°C for 1/2 hour during stirring, and then allowed to cool to ambient temperature to produce coherent and homogenous rubbery greases S_{10}. Formulation and pacification of the finished grease were shown in tables (9, 20).

Table (9): Formulation of the prepared greases S_0, S_8, S_9, and S_{10}

Constituent, parts by weight, g.	Sample Notation			
	S_0	S_8	S_9	S_{10}
Base lube oil	167.27	167.27	167.27	167.27
Transformer oil	83.63	83.63	83.63	83.63
Microcrystalline wax	109	92.6	99	99
Bitumen	—	16.4	—	—
Butyl rubber	—	—	10	—
Poly isoprene rubber	—	—	—	10
Polyoxyethylene sorbiton-nano-palmitate.	1.4	1.4	1.4	1.4
2, 2` methylen-bis (4-methyle-6-tertiary butyle phenol).	1.4	1.4	1.4	1.4

II-2-1-5-5-Preparation of greases containing inorganic compounds S_{11}, S_{12}, S_{13}, S_{14} and S_{15}

II-2-1-5-5-1-Preparation of grease S_{11}

This type of greases that employs a lube oil blend (250.9g), microcrystalline wax 103.5g. The composition were formulated by heating the mixture to 100°C followed by adding 1.4g of both antioxidant and of anticorrosion will mixing in a beaker under stirring for 20 minutes until the additives dissolved and completely dispersed.

Next, the ultramarine powder 5.5g was added and mixed by hand until all the powder was wetted out and then mixed for 15 minutes. The product was then cooled to room temperature and set aside for testing; its formulation and pacification are shown in tables (10, 21).

II-2-1-5-5-2-Preparation of grease S_{12}

Grease S_{12} was prepared by heating blend of base lube oil and transformer oil (250.9g) to 90-100°C and mixed with 103.5g of microcrystalline wax under stirring for 40 minutes until wax was dissolved and completely dispersed. Then the sodium silicate 5.5g was added and mixed until all the powdered was wetted out and then mixed for 1/4 hour under strong stirring. The antioxidant 1.4g and anticorrosion 1.4g were added and mixed for 10 minutes. After cooling to room temperature, the formulation and specification of the resulted grease S_{12} was given in tables (10, 21).

II-2-1-5-5-3-Preparation of grease S_{13}

The prepared silica 5.5g was incorporated into 250.9g of lube oil blend and mixed with spoon until all the powder was wetted out and then mixed for 1/4 hour at room temperature. The temperature was raised to 90-100°C for 1/2 hour and the 103.50g of wax was added under stirring until the wax was dissolved and completely dispersed. Next the antioxidant and anticorrosion additives

1.4g for each were added and mixed for 10 minutes followed by cooling the resulting blend to obtain a stable greases, its formulation and pacification are shown in tables (10, 21). The characteristics if the prepares silica from rice husk was shown in table (1) and fig. (13)

II-2-1-5-5-4-Preparation of grease S_{14}

Talc powder, 5.50g based on the weight of microcrystalline wax and 250.9g lubricating oil base blend were introduced into a glass beaker in the indicated proportions and mixed thoroughly with a spoon at room temperature 25°C. Mixing continued for sufficient time to produce a homogenous mass. Microcrystalline wax 103.5g was added portion wise to the above mixture at 100°C with stirring for 1/2 hour until the mixture turned thick.

Heating was stopped to 70°C, when the content was melted and become homogenous, 1.4g of both antioxidant and anticorrosion were added followed by cooling to the normal temperature to obtain the lubricant product greases S_{14}, its formulation and pacification are shown in tables (10, 21).

I-2-1-5-5-5-Preparation of grease S_{15}

Powdered nano-kaolin was incorporated into 250.9g of lube oil blend in amount of 5.5g by weight at room temperature with stirring for 15 minutes to disperse the powdered.

The grease S_{15} was formulated from the above mixture at 110°C which had been thickened with about 103.5g of microcrystalline wax with agitation followed by adding 1.4g of antioxidant, 1.4g of anticorrosion additives under stirring and heating the mixture to about 110°C followed by cooling, its formulation and pacification are shown in tables (10, 21).

Table (10): Formulation of the prepared greases S_0, S_{11}, S_{12}, S_{13}, S_{14} and S_{15}

Constituent, parts by weight, g.	Sample Notation					
	S_0	S_{11}	S_{12}	S_{13}	S_{14}	S_{15}
Base lube oil	167.27	167.27	167.27	167.27	167.27	167.27
Transformer oil	83.63	83.63	83.63	83.63	83.63	83.63
Microcrystalline wax	109	103.5	103.5	103.5	103.5	103.5
Ultramarine	—	5.50	—	—	—	—
Sodium silicate	—	—	5.50	—	—	—
Silicon dioxide	—	—		5.50	—	—
Nano-Magnesium silicate (talc powder).	—	—	—	—	5.50	—
Nano-Kaolin	—	—	—	—	—	5.50
Polyoxyethylene sorbiton-nano-palmitate.	1.4	1.4	1.4	1.4	1.4	1.4
2, 2` methylen-bis (4-methyle-6-tertiary butyle phenol).	1.4	1.4	1.4	1.4	1.4	1.4

II-2-2-Measurements

II-2-2-1-Dielectric measurements

Permativity (ε`) and dielectric loss (ε``) for the denoted samples were measured at different frequencies ranging from 1 KHz to 1000 KHz.

The computerized LRC (Hioki model 3531 Z Hi Tester) was used to conduct the electrical properties of the investigated samples. The bridge measures the capacitance from 19 pF up to 370 mf, the resistance from 100 mΩ up to 200 MΩ and the dielectric loss, tan (δ) from 10^{-5} up to $10^{1.}$

The bridge is good earthed and all connected cables are good shielded and connected to earth, the accuracy of measurement is better than 1%. The dielectric constant (ε`) for the investigated samples was carried out at room temperature at different frequencies ranging from (42) Hz to (1) MHz. The sample used in the measurement in the form of grease, using a cell have 0.65cm in diameter and 0.2 cm thickness, pressed at room temperature. The cell is consisting of stanlesteel disk coated inside with teflon.

The relative dielectric permativity was calculating using the relations:

$$\varepsilon` = C_m/C_0$$
$$\varepsilon`` = \varepsilon` \tan \delta$$

Where C_m :is the measured capacitance of the used material

C_0 : is the capacity of the empty condenser.

ε``:is the dielectric loss,

tan δ :is the loss tangent.

II-2-2-2-Resistivity measurement

Resistivity ρ of investigated samples were measured at room temperature using the computerized LRC (Hioki

model 3531 Z Hi tester). The resistivity is calculating using the equation

$$R = \rho L / A \quad , \quad \rho = 1/\sigma$$

Where ρ is the resistivity ohm.cm. L is the length of the sample in mm, A is the cross sectional area, and σ is the electrical conductivity.

II-2-2-3-Dynamic viscosity

The apparent viscosity of the prepared greases were carried out by digital Rheometer LVDV-III- Ultra ASTM.

II-2-2-4-FTIR spectroscopy

FTIR spectrometer (Fourier transform infrared, ATI Maston Gensis FTIR™).at wave number from 4000 to 500 cm^{-1} and transmittance from 12 to 26 %.

II-2-2-5-Dropping point

The dropping point tests of the prepared greases measured according to ASTM D - 566 methods.

II-2-2-6-Penetration

Penetration was determined by using ASTM D – 217.

II-2-2-7-Flash point

Flash point was determined by using open system of ASTM D – 92