

Preparation of Some Dielectric Greases from Different Types of Polymers

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ABSTRACT: At this research the grease (S_0) containing [base lube oil grade 260/290, transformer oil, microcrystalline wax, Polyoxyethylene sorbiton-mono-palmitate antioxidant 2,2'-methylenebis(4-methyl-6-tertiary-butyl phenol) anticorrosion] was prepared. To improve its physicochemical & dielectric properties (dielectric constant, dielectric loss, and volume resistivity) and giving it a good electrical insulator property, we add to the wax gel a nonpolar polymer as polyethylene, atactic polypropylene, unplasticized polyvinyl chloride, and plasticized polyvinyl chloride include triisopropyl vinyl phosphate in certain concentration. We conclude that the best insulation properties was achieved by adding atactic polypropylene. © 2010 Wiley Periodicals, Inc. *J Appl Polym Sci* 119: 1026–1033, 2011

Key words: polyethylene grease; polyvinyl chloride grease; atactic polypropylene grease; polymer greases

INTRODUCTION

Lubricating grease consists of base oil, performance additives, and a thickener which forms a matrix that retains the oil in a semisolid state, however, a smaller number of greases, restricted to very special applications are manufactured with nonsoap thickener such as polymer or compounds.¹ A cable lubricant was composed of 20% atactic polypropylene, 10% transformer oil, 10% cylinder oil, 80% petrolatum wax.²

Lubricating grease usually consist lubricating oil and a high molecular weight (co-or homo-) polymer of propylene. Also, the invention relates to the use of such a polymer thickener in the preparation of lubricating grease with improved oil bleeding characteristics at low temperature, noise characteristics, and/or mechanical stability.³ The greases prepared of atactic polypropylene (3–18%), petroleum wax (10–40%), petroleum tar (5–30%), antirust (1–7.5%) and mineral oil have been widely utilized in many fields of industry. Because heavy polymer is a type of atactic polypropylene, which can be dissolved in the oil used, the dripping spot rises above 80°. ⁴

Dielectric greases, insulating fluids and transformer oils have high dielectric strength and have been used in transformers, capacitors, and other electrical devices applications.⁵

In this study, we attempt to obtain dielectric greases through improvement the properties of wax gel by adding different types of polymers thickeners.

EXPERIMENTAL WORK

Materials

Base lube oil grade (260/290): Flash point (open): 204°C, Viscosity index: 90–95, Pour point: (–5°C).

Transformer oil: Flash point: 135°C, Viscosity at 40°C: 12.5 cSt, Pour point: (–20°C).

Microcrystalline wax: Melting range: 80–81°C, Oil content: 0.5%, Flash point, open: 288°C Density at 20°C: 0.9002 g/cm³.

Atactic polypropylene: Density: 0.92 g/cm³, Melting point: 170°C, Volume resistivity: 10¹⁶ ohm cm.

Polyethylene: Melt flow rate (2.16) g/10 min: 2, Density: 920 Kg/m³, Vicat softening temperature: 93°C.

Polyvinyl chloride: *K*-value: 70, Viscosity number: 125 mL/g, Particle size: >63 μm.

Tri-isopropyl phenyl phosphate: Density at 25°C: 1.131 g/mL, Viscosity at 25°C: 95 Cst. Volume resistivity at 23 °C: 3.3 × 10⁻¹³ ohm cm.

Polyoxyethylene sorbitan-monopalmitate, Tween (20): Specific gravity: 1.1 Boiling Point: >100°C

n_D^{20} : 1.4685 Specification number: 43–49. pH of 1% Aqueous solution : 5–7

2,2'-methylenebis(4-methyl-6-tertiary-butylphenol): Melting point: 125°C, Specific gravity: 1.08, Molecular weight: 340.4989.

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METHODS

Dielectric and resistivity measurement

The computerized LRC (Hioki model 3531 Z Hi Tester) was used to measure 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 (ϵ''), $\tan(\delta)$ from 10^{-5} up to 10^1 .

The relative dielectric permittivity was calculating using the relations:

$$\begin{aligned}\epsilon' &= C_m/C_0 \\ \epsilon'' &= \epsilon' \tan \delta\end{aligned}$$

where C_m : the measured capacitance of the used material, C_0 : the capacity of the empty condenser, ϵ'' : the dielectric loss, $\tan \delta$: the loss tangent.

The resistivity is calculating using the equation

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

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.⁶

Dynamic viscosity

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

Dropping point test

The test covers the determination of the dropping point of lubricating grease, this point being the temperature at which the first drop of material falls from the cup. In general, the dropping point is the temperature at which the grease passes from a semi-solid to a liquid state under condition of test. This change in state is typical of grease containing as thickener soap of a conventional types. Greases containing as a thickener material other than conventional soap may be without change in state and separate oil. The method is usually to assist in identifying the grease as to type and/or establishing and maintaining bench marks for quality control ASTM D-566.

Penetration

Penetration was determined using ASTM D-217.

Infrared spectroscopy

FTIR spectra were obtained using (Fourier transform infrared, ATI Maston Genesis series (USA) infrared

spectrophotometer adopting KBr technique. For all samples, the KBr technique was carried out approximately in a quantitative manner as the weight of sample and that of KBr, were always kept constant. At wave number from 500 to 4000 cm^{-1} , transmittance from 0–100% and resolution was 4 mm. Number of scans used during the collection of FTIR is one scan.

Flash point

Flash point measurements were made by open system of ASTM D-92.

FORMULATIONS

Formulation of oil blend

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 min to produce the lube oil blend.⁶

Formulation of greases containing wax gel (S_0)

Oil blend 250.9 g (base lube oil 167.27 g and transformer oil 83.63 g) was heated to 110–120°C and the microcrystalline wax 109 g was added portionwise under stirring for 30 min, followed by adding 1.4 g of antioxidant [2,2'-methylenebis(4-methyl-6-tertiary-butylphenol)] and 1.4 g as anticorrosion (Polyoxyethylene sorbiton-nano-palmitate). Stirring was continued to disperse the additives. After cooling the mixture was thickened to grease S_0 .⁷ The formulation of sample S_0 grease was shown in Table I. The specification of the resulted S_0 grease is given in Table II.

Formulation of grease S_1

Polyethylene (16.4 g) was mixed with 250.9 g lube oil blend and heated to 92°C until complete dissolution of polyethylene has taken place. The mixture hold for 30 min at 100–110°C, then 92.6 g of microcrystalline wax was then added with continued agitation followed by adding 1.4 g of antioxidant; 2,2'-methylenebis(4-methyl-6-tertiary-butylphenol) and 1.4 g of anticorrosion Polyoxyethylene sorbiton-mono-palmitate. They were allowed to cool to ambient temperature to produce a coherent, homogenous S_1 greases,⁷ its formulation and specification are shown in Tables I and II.

Formulation of grease S_2

This type was prepared from microcrystalline wax, lube oil blend, atactic polypropylene in the ratio 5.65

TABLE I
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
Tri-isopropyl phenylphosphate	—	—	—	—	8.2
Polyoxyethylene sorbiton-mono-palmitate	1.4	1.4	1.4	1.4	1.4
2,2' methylen-bis(4-methyle-6-tertiary-butyl phenol)	1.4	1.4	1.4	1.4	1.4

: 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 min 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 min. the antioxidant 1.4 g of 2,2' methylenebis(4-methyl-6-tertiary-butyl phenol) was added as well as 1.4 g of Polyoxyethylene sorbiton-mono palmitate as anticorrosion, then dispersed in the mixture. The mixture formed homogenous smooth grease,⁷ its formulation and specification are shown in Tables I and II.

Formulation 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 1.4 g, and an anticorrosion inhibitor 1.4 g added. then heating the mixture to about 80–85°C with agitation followed by cooling and milling at about 30°C for 15 min.⁷ The resulted greases S_3 have

the formulation and characteristics shown in Tables I and II.

Formulation of grease S_4

Lube oil blend (250.9 g) of tri-isopropyl phenyl phosphate 8.2 g as plasticizer and 8.2 g of polyvinyl chloride were mixed at 30°C then the mixture was stirring for 20 min at temperature of 80–85°C. Then 92.6 g of microcrystalline wax were added gradually under strong and constant stirring for obtaining a gelatinous mass, an additive mixture of (antioxidant 1.4 g and anticorrosion 1.4 g) were added for obtaining a grease S_4 having great extensibility and adherence,⁷ its formulation and specification are shown in Tables I and II.

RESULTS AND DISCUSSION

Any oil mixed with either organic or inorganic fillers is called grease. Modern greases contain one or more fillers, base oil, additives, and an optional tacky fire, the later makes the grease sticky and increases adherence. The fillers hold the oil in its interstitial spaces.^{8–10}

TABLE II
Specification of the Prepared Greases S_0 , S_1 , S_2 , S_3 , and S_4

Specifications	Sample notation				
	S_0	S_1	S_2	S_3	S_4
Appearance color	Pale brown	Pale brown	Pale yellow– brown	Pale yellow	Pale yellow
Oil bleeding	None	None	None	Slight	Slight
Penetration, 25°C, 10 mm/cone (unworked)	190	196	207	275	266
Dropping point (°C)	50	68	70	66	60
viscosity, at 66°C, cP Apparent	47.88	5213	5548.82	2927.38	2005.29
Behavior at high temperature	Melted	Melted	Melted	Melted	Melted
Water repel at 25°C, 1 h, (%)	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				

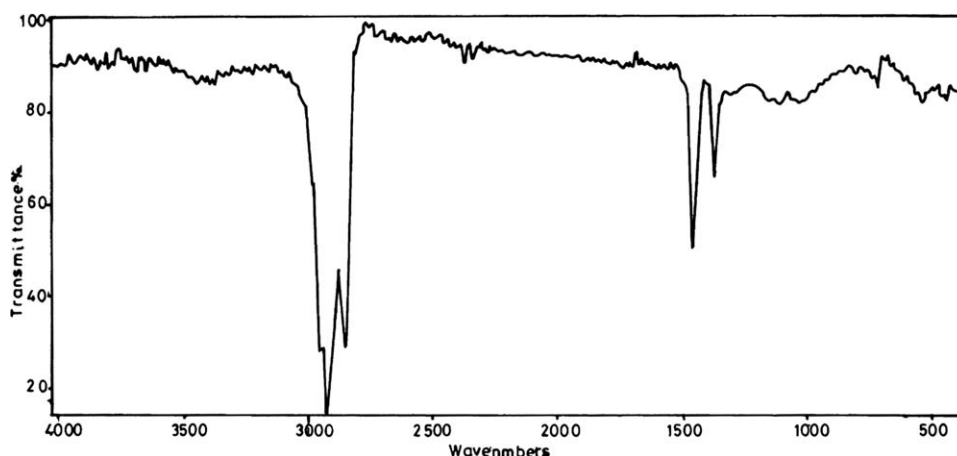


Figure 1 IR Spectrum for base lube oil grade (260/290).

Oil blend

The physicochemical characteristics of the used 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, were mixed and used for this purpose.

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 alone.

In addition the distribution of %C_A, %C_P, %C_N was shown and it was deduced that as the %C_P 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_N for the oil blend is high and so, the dielectric properties for this oil is better than the first oil grade (260/290).¹¹ This means that the base lube oil grade (260/290) is not suitable as insulating oil, but after blending it with transformer oil (12.5 cst) it has 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 blend-

ing (with transformer oil) to overcome the high aromatic contents and to decrease the pour point.

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 Infrared (IR) spectra in the range from 4000 to 500 cm⁻¹ Figures 1–3 shows that the above oils have low intensity bands in the region (3431–3436 cm⁻¹), indicating low concentrations of –OH and –NH groups, which have important role in the polarity of oils.

The spectra also show strong two bands at (2850–3000 cm⁻¹), which is due to CH₂ asymmetric of methyl and methylene groups, the strong band at (1459 cm⁻¹), which due to CH₂ asymmetric bending vibration. In addition, the weak band obtained at (1374 cm⁻¹) may be attributed to C–N stretching vibration for aromatic amines or CH₃ bending vibration symmetric for methyl groups and a weak band at (1605cm⁻¹) is due to the stretching vibration of C=C aromatic rings.

Physicochemical properties

It is clear from the Table I that sample S₃ proved to be advantageous of dropping point than sample S₄ (containing polyvinyl chloride and tri-isopropyl 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, which was 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,¹² i.e. greases S₄ should exhibit higher mobility than greases S₃.

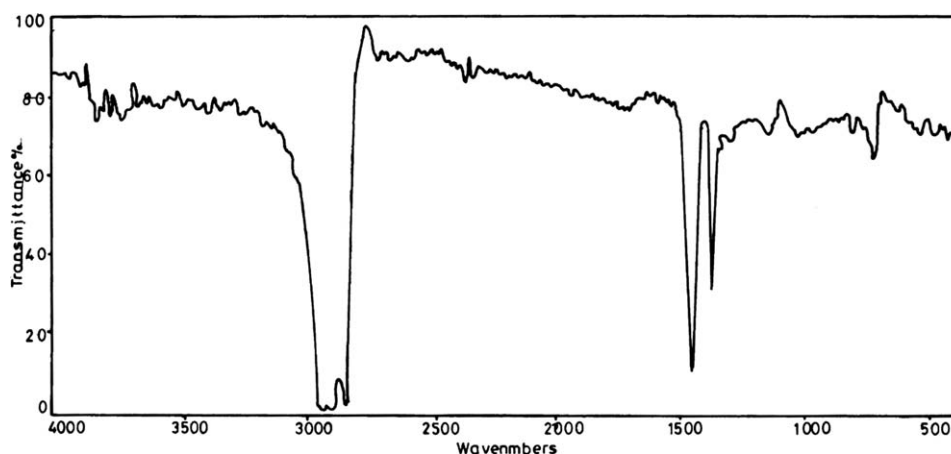


Figure 2 IR Spectrum for transformer oil.

In case of grease, S_2 (including atactic polypropylene) has better dropping point than S_1 (including polyethylene), due to type of atactic polypropylene which can be dissolved in the oil is used whereas polyethylene, upon swelling in oils, forms plastic structured 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, grease S_4 (including PVC and a plasticizer) should exhibit higher penetration because of the higher mobilities.

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

Sample S_4 (containing plasticized PVC) has flash point better than sample S_3 unplasticized due to the presence of plasticizer TIPPP (has high fire resistance property).¹³

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 char layer may make the surface of some sample containing phosphates.

Dielectric properties of the prepared greases

The dielectric constant ϵ' , dielectric loss ϵ'' and volume resistivity for the prepared samples (S_0 – S_4) over the frequency range from 1 to 1000 KHz at 35°C were studied.

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 hindrance Good insulating greases have low dielectric constant, low dielectric loss, and high volume resistivity.¹⁴

It is evident from Table III and Figures 4 and 5 that dielectric constant and dielectric loss decrease with increasing frequency for samples (S_0 , S_1 , S_2 , S_3 ,

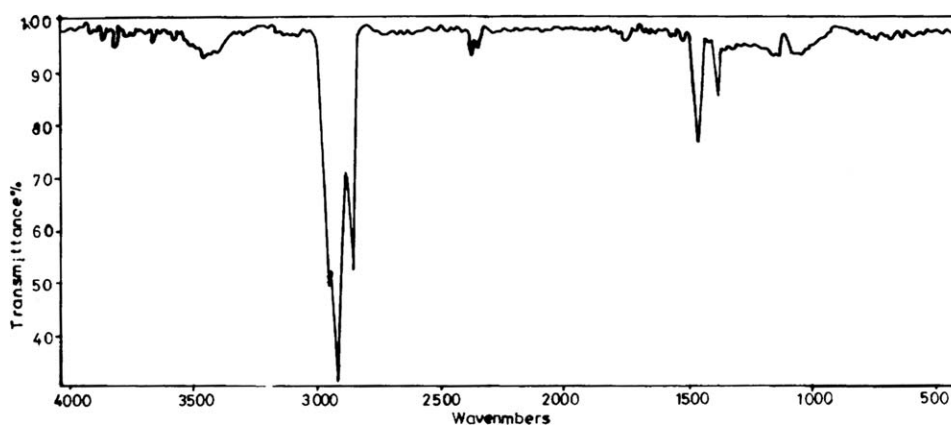


Figure 3 IR Spectrum for base lube oil grade (260/290) and transformer oil after blending.

TABLE III
Dielectric Measurement of the Prepared Greases S_0 , S_1 , S_2 , S_3 , and S_4

Specification (KHz)	Sample notation				
	S_0	S_1	S_2	S_3	S_4
Permativity (ϵ') at frequency,					
1	2.0798	1.8451	1.7921	2.0056	2.0140
10	2.0761	1.8286	1.7796	1.9393	2.0055
100	2.0716	1.7619	1.5715	1.95	1.9976
1000	2.0589	1.6975	1.551	1.898	1.9387
Dielectric loss (ϵ'') at frequency,					
1	0.6429	0.0449	0.0365	0.4242	0.5835
10	0.2344	0.0376	0.0202	0.1878	0.2219
100	0.0813	0.0435	0.0380	0.0588	0.0669
1000	0.0216	0.0062	0.0023	0.0075	0.0120

and S_4). Similar behavior was noticed before in the literature.^{15,16} The decrease of dielectric constant and ϵ'' with frequency shows an anomalous dispersion.

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

On the other hand, the volume resistivity values of these samples represented in table (IV) and Figure 6 shows that the value of volume resistivity decreases with increasing frequency from 1 to 1000 KHz. It is in order $S_2 > S_1 > S_3 > S_4 > S_0$ respectively. As S_2 has the highest value of volume resistivity (0.9×10^{12} ohm cm) at 1 KHz at 35°C.

This proves that, the dielectrical properties (lowest value of ϵ' and ϵ'' and highest value of volume resistivity) make a good insulator of sample S_2 grease (formulated from wax gel S_0 and atactic polypropylene) with chemical structure. This could be attrib-

uted to the presence of branched methyl group $-\text{CH}_3$ in atactic polypropylene, the presence of sigma bonds (σ bond) in its structure, which are usually stronger than π bond; beside atactic polypropylene has volume resistivity 10^{16} ohm cm

The nonpolar 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 nonpolar 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 nonpolar and this determines many of the dielectric properties of the plastic.

For nonpolar plastics the dielectric constant is independent of the alternating current frequency because the electron polarization is effectively instantaneous. Nonpolar plastic always have dielectric

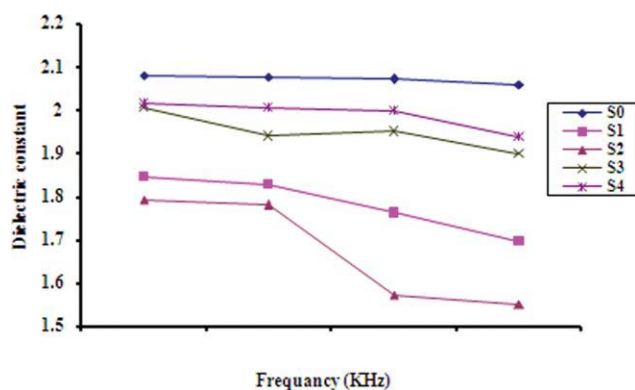


Figure 4 Dielectric constant vs. frequency at 35°C for S_0 , S_1 , S_2 , S_3 , and S_4 greases. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

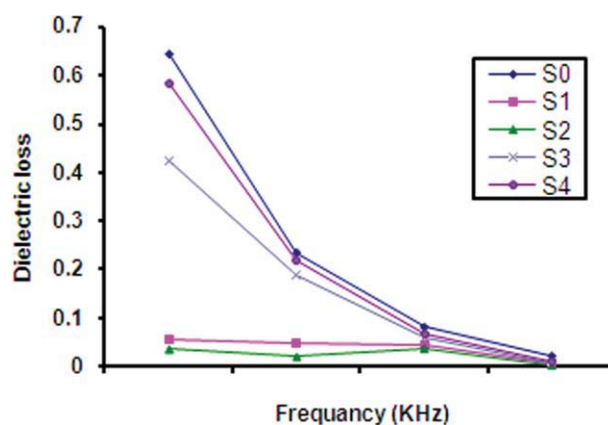


Figure 5 Dielectric loss vs. frequency at 35°C for S_0 , S_1 , S_2 , S_3 , and S_4 greases. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

TABLE IV
Volume Resistivity Measurements of the Prepared greases S_0 , S_1 , S_2 , S_3 , and S_4

Specification (KHz)	Sample notation				
	S_0	S_1	S_2	S_3	S_4
Volume resistivity.Ohm cm, at 35°C at frequency					
1	0.0023×10^{10}	0.05×10^{11}	0.9×10^{12}	0.2×10^{12}	0.09×10^{11}
10	0.000913×10^9	0.05×10^{11}	0.4×10^{12}	0.03×10^{11}	0.036×10^{11}
100	0.000305×10^9	0.026×10^{11}	0.049×10^{11}	0.013×10^{11}	0.011×10^{11}
1000	0.000116×10^9	0.0096×10^{10}	0.004×10^{11}	0.0038×10^{10}	0.00001×10^{10}

constant of less than 3.¹⁷ In the same time, the value of grease S_1 has good insulating properties (formulated from S_0 and polyethylene) due to the presence of saturated bonds in its structure. Polyethylene has volume resistivity 10^{16} ohm cm.

On the other hand, the sample S_3 (formulated from S_0 and PVC unplasticized) has good dielectric properties better than sample S_4 (formulated from S_0 , PVC, and tri-isopropyl 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, 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.^{17,18}

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 the original sample S_0 .

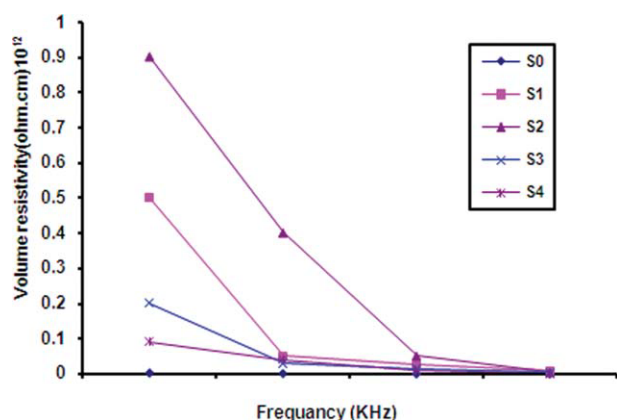


Figure 6 The relation between volume resistivity and frequency at 35°C for S_0 , S_1 , S_2 , S_3 , and S_4 greases. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

CONCLUSION

- The results of the physicochemical properties indicate that all prepared greases has better physicochemical properties (dropping point, flash point, viscosity, penetration, and water resistance) and dielectrical properties (dielectric constant, dielectric loss, and volume resistivity) than the prepared wax gel (base oil blend and microcrystalline wax) which can overcome the leakage from surface.
- The grease (S_4) containing (tri-isopropyl phenyl phosphate compounds) has shown to promote char formation by reducing the production of combustible grease during greases decomposition.
- Dielectric measurements indicate that, greases containing atactic polypropylene, and polyethylene are the best and this may be due to their non polarity and high volume resistivity 10^{16} .
- Grease of unplasticized PVC has better dielectric properties than that of the plasticized PVC & due to that plasticizer can penetrate the molecular bundle of PVC and reduce it to several segments, which can be rotated in an electric field.

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