

# NASA Technical Paper 1439

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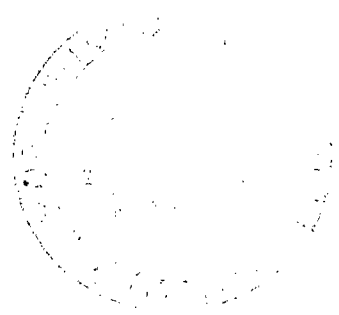


## Control of Volume Resistivity in Inorganic-Organic Separators

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# Control of Volume Resistivity in Inorganic-Organic Separators

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## SUMMARY

Control of resistivity in NASA inorganic-organic separators is achieved by incorporating small percentages of high-surface-area, fine-particle silica with other ingredients in the separator coating. The volume resistivity appears to be predictable from coating composition, that is, from the surface area of filler particles in the coating.

The approach has been applied to two polymer-"plasticizer"-filler coating systems, where the filler content of each is below the generally acknowledged critical pigment volume concentration of the coating. Application of these coating systems to 0.0254-centimeter-thick (10-mil) fuel-cell-grade asbestos sheet produces inexpensive, flexible, microporous separators that perform at least as well as the original inorganic-organic concept, the Astropower separator.

## INTRODUCTION

The usefulness of the inorganic-organic separators developed at the Lewis Research Center for alkaline batteries (i.e., Ag-Zn, Ni-Zn, and Ni-Cd batteries) depends on an optimum balance of resistivity and microporous-structure uniformity. Over the past few years separators have been developed with longer cycle life, more uniform current density, greater flexibility, and lower cost than the original Astropower inorganic-organic separator.

The Astropower separator (ref. 1) and several other microporous separators contain relatively high filler volumes such that the amount of binder is insufficient to completely coat the filler particles and fill the interstices. That is, their filler volume is above the critical pigment volume concentration (CPVC) of 40 to 45 volume percent. These separators derive their ionic conductivity through the pathway created by the particle-to-particle contact of the fillers. Bozek (ref. 2) described the structure and function of the Astropower separator. Philipp and May (ref. 3) studied the reaction of the plasticizer with the electrolyte, the ionic resistivity of simulated inorganic-organic separators, and the zincate diffusion through various separators. Sheibley (ref. 4) discussed factors that influence the flexibility, resistivity, and zinc dendrite penetration rate of the Astropower separator and NASA improved inorganic-organic separators. A significant conclusion of that work was that the volume resistivity of the improved separators could be maintained in an acceptable range (10 to 25  $\Omega$ -cm) while keeping the volume percentage of fillers in the polymer below the CPVC (ref. 5). This also ensured good coating flexibility. Another significant conclusion of reference 4 was that the zinc

dendrite penetration rate is controlled principally by the tortuosity produced by the presence of inert fillers in the coating. The acceptable range of resistivity was achieved by judicious selection of fillers that combined inertness and reactivity with the potassium hydroxide (KOH) electrolyte while providing a multiplicity of tortuous conduction paths. These paths were created by filling the spaces between larger particles with smaller particles (a filler packing effect).

Ion permeability of coatings rises very rapidly by several orders of magnitude at the CPVC (ref. 5). The key to film permeability below the CPVC in these improved inorganic-organic separators was the use of a proper polyester plasticizer. Philipp and May (ref. 3) pointed out that the plasticizer must be a polar compound that preferentially adsorbs on the filler particles, which are also polar compounds. Sheibley (ref. 4) showed that one of the products of the plasticizer reaction with the electrolyte must be a short-chain (two or three carbons) glycol. It was also shown that resistivity could be further improved by using an electrolyte-reactive filler in the coating.

This paper discusses the development of resistivity control in NASA microporous separators that contain filler volumes significantly less than the CPVC. First, the effect of filler volume on volume resistivity is shown for a typical inert filler. Then the effect of an electrolyte-reactive filler on volume resistivity is illustrated. Next, the effect on volume resistivity of combinations of fillers plus a selected organic additive (plasticizer) in the polymer is demonstrated. Finally, the incorporation of high-surface-area, fine-particle fillers is shown to be a predictable means of controlling resistivity.

## EXPERIMENTAL PROCEDURE

### Preparation of Coating Mixture

The coatings used for the separator materials were prepared by weighing an amount of polymer (either polyphenylene oxide<sup>1</sup> (PPO) or Kraton G<sup>2</sup> resin) into a ball mill, adding chloroform or another solvent, and then adding the weight of filler or filler plus organic additive (plasticizer) needed to provide the desired total volume percentage of filler in the resin. Before ball milling, the Kraton G must be cut into solution with the solvent in a high-shear mixer. After the materials were milled for 20 hours, chloroform was added to reduce the viscosity of the mixture to 15 to 16 seconds as measured with a number 3 Zahn cup at ~22° C.

The fillers used in these studies were magnesium silicate (Microtalc), aluminum silicate (kaolinite), synthetic magnesium aluminosilicate (NAS-100), magnesium titanate

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<sup>1</sup>General Electric Co., Schenectady, N. Y.

<sup>2</sup>Shell Chemical Co., Houston, Tex.

( $\text{Mg}_2\text{TiO}_4$ ), natural calcium silicate (wollastonite), lead titanate, reprecipitated calcium silicate (Silene EF), and fumed silicon dioxide (Cab-O-Sil). The organic additives (plasticizers) were a polymeric polyester (P-9750), used with the PPO, and an epoxidized soya bean oil (Paraplex G-62), used with the Kraton G resin. The trade names and manufacturers of these fillers and additives are given in table I. Coating compositions are given in table II and discussed later.

### Coating the Asbestos Substrate

Fuel-cell-grade asbestos sheet 0.0254 centimeter thick (10 mils) and impregnated with 2-weight-percent PPO was the substrate for all experimental materials. The coating mixtures were applied to the asbestos with an adjustable knife-blade applicator with the blade height set typically 0.0254 centimeter above the substrate. Two layers were applied at a wet thickness of 0.0254 centimeter for each coating. The second layer was applied after the first had dried. In the laboratory coating method the substrate was held flat by means of a vacuum plate while the knife blade was pulled over the fixed substrate. On factory production equipment the knife blade was fixed and the substrate moved beneath it. The final dry coating thickness was 0.0762 to 0.1016 centimeter (3 to 4 mils) which gave a total separator thickness, including substrate, of 0.0330 to 0.0355 centimeter (13 to 14 mils).

### Determination of Volume Resistivity

Volume resistivities were measured with a test apparatus and procedure based on those described by Salkind and Kelley in reference 6. In most cases, resistivities were measured on at least two samples to establish precision, which ranged from  $\pm 3$  to  $\pm 6$  percent. Measurements were made in  $\sim 45$ -weight-percent KOH at room temperature. The two samples (3.8 cm by 3.8 cm) of each material were pretreated by soaking them in a 45-weight-percent KOH overnight at  $90^\circ$  to  $100^\circ$  C. The test area in the conductivity cell was 0.95 square centimeter. The dry thickness of the separator was used in calculating volume resistivity because it was difficult to measure the wet thickness of the separator once it was positioned in the resistivity cell and the cell halves were tightened together. The wet thickness of some separators is 25 percent greater than their dry thickness in an uncompressed state.

## Determination of Plasticizer Associated with Fillers

The amount of plasticizer associated with the fillers was estimated from the ratio of plasticizer to PPO in the liquid phase. It is known (ref. 3) that the plasticizer (polar compound) is preferentially adsorbed on the fillers (also polar compounds), but the PPO is not. Any selective adsorption on the balls of the ball mill used to prepare the coatings should not affect the result because the surface of a ball is about  $5 \times 10^{-6}$  square meter and the surface contribution from fillers was always at least 1 square meter.

Samples of various coating mixtures were allowed to stand until all the filler particles had settled out and a relatively clear supernatant was left. The ratio of plasticizer to PPO in the supernatant was determined by infrared spectroscopy. A calibration curve was obtained from a series of solutions of known ratios; and a straight line was fit to the data by regression analysis.

The spectrometer used was a Beckman model IR 18A. The samples were diluted 6:1 with chloroform and run against a chloroform blank in sodium chloride cells. The peak at  $1595 \text{ cm}^{-1}$  (distinctive for aromatic substituted compounds and easily measured) was used to determine the PPO content. The peak at  $1725 \text{ cm}^{-1}$  (carbonyl stretching) was used to determine the plasticizer content. A typical infrared spectrum is shown in figure 1. Table II contains information on the composition of formula X39NF. The precision of this method was estimated to be from 3 to 5 percent.

## Measurement of Filler Surface Area

The specific surface area of the dry fillers was determined by the standard multi-point Brunauer-Emmett-Teller (B. E. T.) technique (ref. 7) with krypton and nitrogen gas adsorption. This work was performed by a contractor<sup>3</sup>, who used an Orr model 2100 surface-area pore-volume analyzer.

## RESULTS AND DISCUSSION

The effect of filler volume content on volume resistivity was studied initially with various fillers in PPO. Some fillers were reactive with the electrolyte; some were unreactive (inert). The fillers comprised a large range of particle sizes as well as different chemical compounds, both synthetic and natural. The effect was studied with and without a saponifiable organic additive (plasticizer) in the filler-PPO mixture. Table II presents information on PPO coatings discussed in this section. Table III presents data on particle sizes and surface areas of all fillers used in the PPO coatings (table II) and

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<sup>3</sup>Micromeritics Instrument Corp., Norcross, Ga.

in the Kraton G resin-base coatings (table IV). Similar, but limited, studies on the volume content of fillers in Kraton G were also performed to establish that the resistivity results were not unique to PPO but were a more general result and usable with other resin-filler-plasticizer systems.

#### Effect of Volume Content of Fillers in PPO on Volume Resistivity

The volume resistivities of separators made with the fillers used in this evaluation are shown in figure 2. These fillers were milled into PPO-chloroform solutions without an organic additive. The curve for Microtalc in PPO shows a decreasing volume resistivity with increasing volume percent. This material has a surface area of  $\sim 3 \text{ m}^2/\text{g}$  (table III). The curve for synthetic magnesium aluminosilicate (NAS-100) in PPO is similar to that for Microtalc except that the larger surface area of NAS-100 ( $280 \text{ m}^2/\text{g}$ ,  $\sim 100$  times greater than that of Microtalc) produces a correspondingly greater reduction in volume resistivity as filler volume content increases. The curve for P-9750 shows the effect of just a polyester plasticizer in PPO without any fillers. Since P-9750 is only slightly soluble in a dry PPO film ( $<10$  percent by weight, ref. 3), it was assumed that when it was dispersed in the dry PPO film, it would segregate into discrete globules similar to filler particles. The plasticizer volume content without fillers had little effect on the volume resistivity until its volume percent was greater than 50 percent (greater than the CPVC). Reprecipitated (synthetic) calcium silicate filler (Silene EF), with a surface area of  $\sim 57 \text{ m}^2/\text{g}$  (table III), reacts with the electrolyte and leaves voids filled with gelatinous, hydrated calcium oxide. This curve is described in reference 4 and is shown here for comparison as typical of reactive filler formulations.

#### Effect of Volume Content of Filler with Organic Additive

##### (Plasticizer) in PPO on Volume Resistivity

The data in figure 3 provide an insight into the interaction that results from combining the proper (ref. 4) organic additive (P-9750) with an inert filler. Magnesium titanate ( $\text{Mg}_2\text{TiO}_4$ ) has a surface area of approximately  $3.5 \text{ m}^2/\text{g}$  and a mean particle diameter of 1.7 micrometers, about the same as Microtalc (fig. 2). However, the  $\text{Mg}_2\text{TiO}_4$  with plasticizer results in a volume resistivity an order of magnitude lower than that for Microtalc without plasticizer (data from table II for formulas X47NF, X37NF, and X39NF). As the filler surface area increases the resistivity decreases. When the filler volume percent exceeds the CPVC ( $\sim 40$  to 45 percent), the resistivity is lower than would be expected from a linear extrapolation of the other two points.

Figure 3 also shows the effect of a fixed amount of fine-particle fumed silicon dioxide (Cab-O-Sil) combined with a larger amount of magnesium titanate with plasticizer. The curve represents the total for both fillers. The addition of electrolyte-reactive Cab-O-Sil, which has a mean particle size of 0.015 micrometer and a surface area of  $\sim 210 \text{ m}^2/\text{g}$  (table III), decreased the volume resistivity of  $\text{Mg}_2\text{TiO}_4$  plus P-9750 by an order of magnitude. The data for formulas X31, X37, and X39 from table II show that adding Cab-O-Sil increased the surface area significantly so that doubling the  $\text{Mg}_2\text{TiO}_4$  content had little effect (20 percent) on surface area. Volume resistivity did not decrease significantly over the range investigated, even when the CPVC was exceeded (X39).

Figure 3 also shows the effect of an inert filler combination of magnesium titanate, natural calcium silicate (wollastonite P-15), and an increasing content of electrolyte-reactive Cab-O-Sil (0 to 3 vol%) with the plasticizer present. Data from table II (formulas X47W, X47W1, and X47W2) show a pronounced drop in volume resistivity ( $445 \text{ } \Omega\text{-cm}$  to  $21 \text{ } \Omega\text{-cm}$ ) and at the same time a sharp increase in surface area ( $183 \text{ m}^2/\text{g}$  to  $1025 \text{ m}^2/\text{g}$ ) as the Cab-O-Sil content increased. The actual relationship of volume resistivity to filler surface area is described in detail later.

#### Effect of Organic-Additive (Plasticizer) Content on Volume Resistivity

To more fully understand the role of the plasticizer and to estimate the thickness of the adsorbed layer on the filler particles, we attempted to measure the amount of P-9750 associated with the filler by using the ratio of P-9750 to PPO determined as described in the section EXPERIMENTAL PROCEDURE. The calibration data, the regression line, and the results for the various formulas are shown in figure 4. The formulas are shown in table II. Except for two points (X37 and X47W in fig. 4), the data indicate that from 28 to 32 cubic centimeters of P-9750 remained in solution. The remaining 12 to 16 cubic centimeters are assumed to be associated with the settled filler particles.

From the surface areas given for the fillers in table III, the total filler surface areas of the coating materials shown in figure 3 range from  $1.8 \times 10^6$  to  $1.5 \times 10^7$  square centimeters. Since an average 14 cubic centimeters of P-9750 is assumed to be associated with the fillers, the organic-additive film deposited on the particles is 8 to 23 nanometers (80 to 230 angstroms) thick. This is in the same range as the average pore size (5 to 15 nm)<sup>4</sup> for PPO-based separators as determined by water permeability (ref. 6) on KOH-soaked separators treated as described in the section Determination of Volume Resistivity.

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<sup>4</sup>Margaret Reid of Lewis, unpublished data, 1977.



The X47W point in figure 4, even though it is outside the range of the other points, is apparently consistent. The material (table II) has a small calculated surface area ( $183 \text{ m}^2/\text{g}$ ) and would therefore be associated with less plasticizer. The calculated plasticizer layer is 3.2 nanometers thick, which could account for the high resistivity ( $445 \text{ } \Omega\text{-cm}$ ) that was measured. The other outside point (X37) indicates a greater adsorption of plasticizer on the particles than for the other cases. There is no obvious or plausible explanation for it at this time.

B.E.T. measurements of separator pore size show a fairly sharp peak in the 2.5-nanometer region and a less well-defined region from 5 to 25 nanometers, the limit of the method. The smaller, 2.5-nanometer pores presumably are due to the pathway left by the evaporating solvent. Hence, it appears that ionic conduction through the separator depends primarily on the thickness of the ionic conduction pathway through and around the filler particles. This pathway is created by reaction of KOH with the adsorbed organic additive. A model of this pathway concept is shown in figure 5. Ion conduction is improved because pores are created by the reaction of fine-particle fillers with the KOH electrolyte. The pathways created by solvent evaporation probably also contribute. The reaction of the polymeric polyester (organic additive) with the KOH produces 1,2 propanediol and the potassium salt of azelaic acid. This salt presumably remains in the pathway as insoluble potassium azelate and further restricts the average diameter of the pathway.

#### Effect of Filler Surface Area on Volume Resistivity

The volume resistivity data in figure 3 show a marked dependence on the amount of fine-particle silica added to the coating formula. Figure 6 shows volume resistivity as a function of total filler surface area, in  $\text{m}^2/47 \text{ g}$  of PPO (with 47 g of P-9750). The symbols are identical to those in figure 3 and represent the same materials as well as X47 (circle) from table II.

The two triangular points determine the line passing through the data points. This line was predicted from coefficients derived from a linear regression analysis of  $\log (\Omega\text{-cm})$  as a function of total filler surface area in 47 grams of PPO (with 47 g of P-9750). The correlation coefficient is  $-0.88$ . The fit is good considering that the data were taken from coating formulas containing from one to three fillers with varying surface areas. The two data points in figure 6 labeled X47W2 and X37 (table II) demonstrate the proportional relationship of volume resistivity to the surface area of the fillers. The volume of Cab-O-Sil in X47W2 ( $1.9 \text{ cm}^3$ ) is 84 percent of that in X37 ( $2.28 \text{ cm}^3$ ). The surface area of X47W2 ( $1025 \text{ m}^2/\text{g}$ ) is 81 percent of that of X37 ( $1258 \text{ m}^2/\text{g}$ ), and the reciprocal volume resistivity of X47W2 ( $1/21 \text{ } \Omega\text{-cm}$ ) is 84 percent of that of X37 ( $1/17.7 \text{ } \Omega\text{-cm}$ ).

## Effect of Volume Percentage of Fillers in Kraton G on Volume Resistivity

A more flexible separator system than could be obtained with PPO was desired for Ni-Zn cells. Based on results of 500 hours of immersion in 45-percent KOH at 105<sup>0</sup> to 110<sup>0</sup> C, a Kraton G block copolymer was selected as the coating resin for this application. Tests were performed to determine if the control of volume resistivity in PPO was more generally applicable to other polymer systems. An epoxidized soya bean oil plasticizer, Paraplex G-62 (a polar compound), was used with the Kraton G. One of its reaction products with KOH was ethylene glycol (ref. 4, table IV).

The effect on volume resistivity of the volume percentage of fillers in Kraton G (with no plasticizer) was studied. Table IV shows the effect of Silene EF and Cab-O-Sil on volume resistivity. These data indicate that 10.1 volume percent of Cab-O-Sil is equivalent to 39.1 volume percent of Silene EF. The effect is presumably due to the greater surface area of Cab-O-Sil. Table V shows the effect of adding the plasticizer to the polymer along with fillers; compare formulas K52, K53, and K51. The addition of a small volume percentage of Cab-O-Sil (formula K53, table V) to the Silene EF mixture (formula K51) produced a substantial drop in volume resistivity.

This effect was then tested in actual separator coatings that applied the principle demonstrated in figure 3, that of increasing the amount of the fine-particle reactive silica in a coating mixture containing the Kraton G polymer, a plasticizer, and two unreactive fillers - lead titanate and natural calcium silicate (wollastonite).

These results are shown in table V and figure 7. Formula K19W contains no Cab-O-Sil. Formula K19W2 contains twice as much as K19W1. The K19W/2 is a mixture of equal volumes of Silene EF and wollastonite, the total volume of which is equal to the volume percentage of wollastonite in the other formulas. The square points in figure 7 used to draw the curve represent two points predicted by the linear regression analysis of volume resistivity as a function of the surface area of fillers in 40 grams of Kraton G. The linear correlation coefficient for the regression analysis is -0.97.

These data show an effect similar to that observed with fillers and an organic additive to PPO. This method of resistivity control is therefore also useful and predictable in at least one other resin-plasticizer system and may be generally applicable.

## CONCLUDING REMARKS

Small percentages of high-surface-area, fine-particle silica incorporated in coating systems where the filler content is below the critical pigment volume concentration have been shown to be a predictable means of controlling the volume resistivity of the

coated substrate. Many of the formulas described in this paper have also been evaluated as separators in nickel-zinc and silver-zinc cells. Some of these cells have shown acceptable performance.

Lewis Research Center,  
National Aeronautics and Space Administration,  
Cleveland, Ohio, January 17, 1979,  
506-23.

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TABLE I. - FILLERS AND ORGANIC ADDITIVES USED IN COATINGS

Filler	Trade name	Manufacturer
Magnesium silicate	Microtalc MP-12-50	Pfizer Minerals, Pigments and Metals Division, New York, N. Y. 10017
Aluminum silicate	Kaolinite (Hydrite UF)	Georgia Kaolin Co., Elizabeth, N. J. 07207
Synthetic magnesium aluminosilicate	Barasym NAS-100	NL Industries, Baroid Division, Houston, Tex. 77001
Magnesium titanate	TICON MT(C)	NL Industries, TAM Division, Hightstown, N. J. 08520
Natural calcium silicate	Wollastonite P-15	Interpace Corp., Willsboro, N. Y. 12996
Lead titanate	TICON PT	NL Industries, TAM Division, Hightstown, N. J. 08520
Reprecipitated calcium silicate	Silene EF	PPG Industries, Pittsburgh, Pa. 15219
Fumed silicon dioxide	Cab-O-Sil, grade M-5	Cabot Corp., Boston, Mass. 02110
Polymeric polyester	P-9750	Emery Industries, Cincinnati, Ohio 45202
Epoxidized soya bean oil	Paraplex G-62	Rohm and Haas Co., Philadelphia, Pa. 19105

TABLE II. - PPO COATING COMPOSITIONS

	Formula									
	X47NF	X47	X37NF	X37	X39NF	X39	X31	X47W	X47W1	X47W2
Weight, g:										
PPO	47	47	47	47	47	47	47	47	47	47
P-9750	47	47	47	47	47	47	47	47	47	47
Magnesium titanate	36	36	71	71	142	142	36	36	36	36
Wollastonite P-15	0	0	0	0	0	0	0	26.2	26.2	26.2
Cab-O-Sil	0	0	0	4.8	0	4.8	4.8	0	2	4
Silene EF	0	19	0	0	0	0	0	0	0	0
Volume resistivity, $\Omega$ -cm	243	54.6	141	17.7	28.5	17.8	19	445	46	21
Total filler surface area, $m^2/g$ (47 g of PPO)	125	1207	247	1258	494	1505	1137	183	604	1025
Ratio of P-9750 to PPO (peak heights, fig. 4)	1.73	1.69	1.63	1.40	1.77	1.70	----	1.93	----	----

TABLE III. - PARTICLE SIZES AND SURFACE AREAS OF  
REACTIVE AND INERT FILLERS

	Particle size, $\mu\text{m}$	Surface area, $\text{m}^2/\text{g}$
Reactive fillers:		
Reprecipitated calcium silicate (Silene EF)	0.03	57
Fumed silicon dioxide (Cab-O-Sil)	.015	210
Inert fillers:		
Lead titanate (TICON PT)	1.14	1.88
Natural calcium silicate (wollastonite P-15)	4.6	2.21
Magnesium titanate (TICON MT(C))	1.7	3.48
Magnesium aluminosilicate (NAS-100)	-----	281
Magnesium silicate (Microtalc)	-----	3

TABLE IV. - EFFECT OF REACTIVE FILLERS IN  
KRATON G (NO PLASTICIZER)

Reactive filler	Content, vol%	Surface area, $\text{m}^2/40 \text{ g of Kraton G}$	Volume resistivity, $\Omega\text{-cm}$
Silene EF	10	524	6717
	29.9	2278	182
	39.1	3417	63
	50	5308	18
Cab-O-Sil	2.2	463	4071
	6.3	1361	3473
	10.1	2317	61

TABLE V. - KRATON G COATING COMPOSITIONS

	Formula						
	K52	K53	K51	K19W	K19W1	K19W/2	K19W2
Weight, g:							
Kraton G	40	40	40	40	40	40	40
Paraplex G-62	16	16	16	16	16	16	16
Wollastonite P-15	0	0	0	55.2	55.2	27.6	55.2
Lead titanate	0	0	0	20	20	20	20
Cab-O-Sil	0	1.6	0	1.6	3.2	1.6	6.4
Silene EF	0	40	40	0	0	-20	0
Volume resistivity, $\Omega$ -cm	8175	15.3	35.7	37.6	30	18.2	17.4
Total filler surface area, $m^2/40$ g of Kraton G	0	2606	2278	535	833	1208	1507

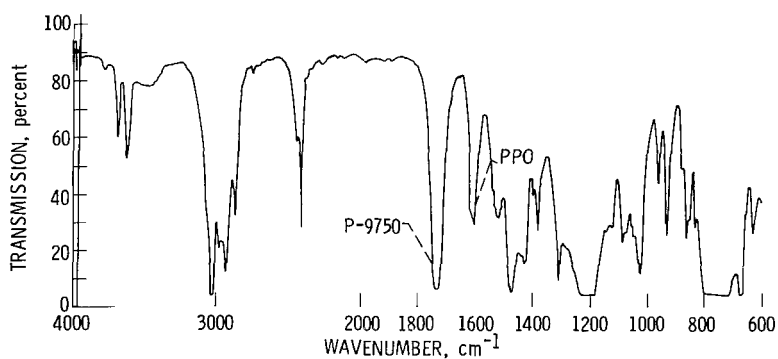


Figure 1. - Infrared spectrum of supernatant from formula X39NF (table II).

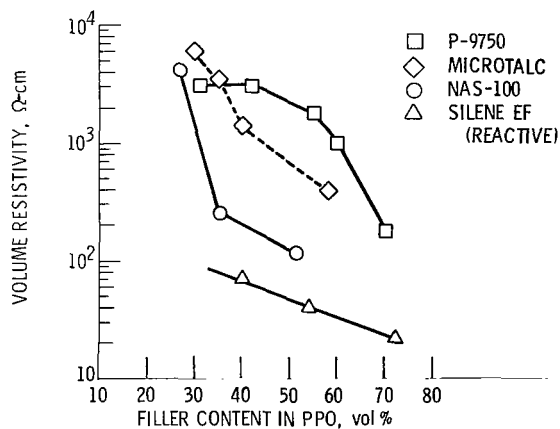


Figure 2. - Effect of filler volume content on volume resistivity - no plasticizer.

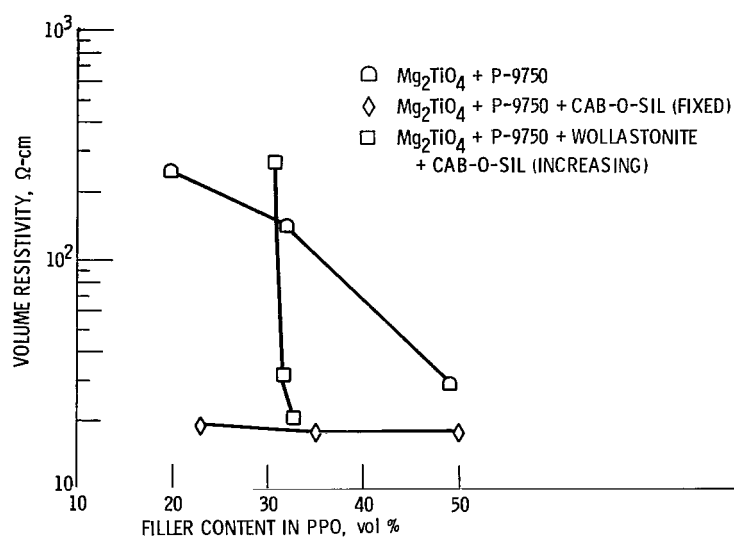


Figure 3. - Effect of volume content of filler plus organic additive (plasticizer) on volume resistivity.

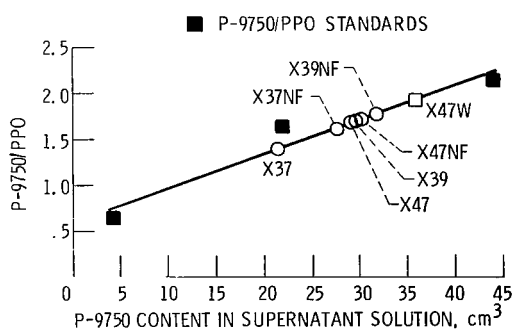


Figure 4. - Ratio of P-9750 to PPO as related to amount of P-9750 in supernatant solution.

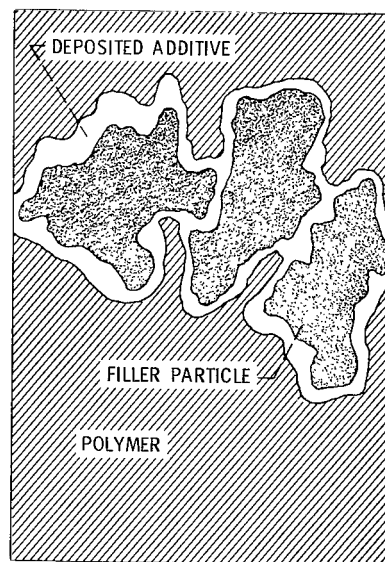


Figure 5. - Simplified ionic conduction model.

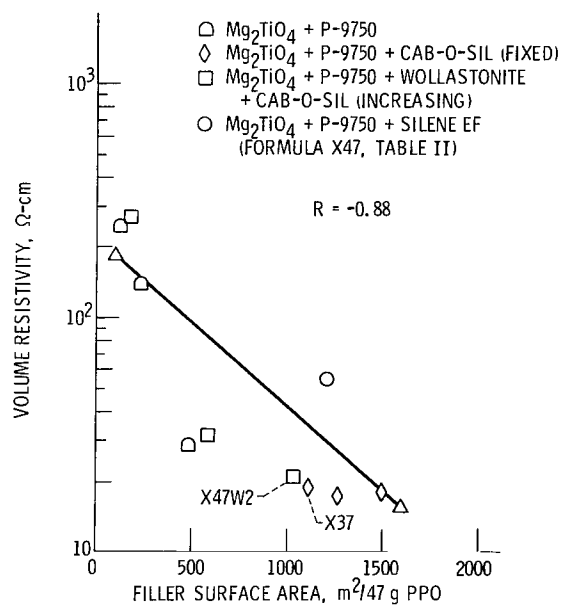


Figure 6. - Effect of total filler surface area on volume resistivity of PPO coatings.

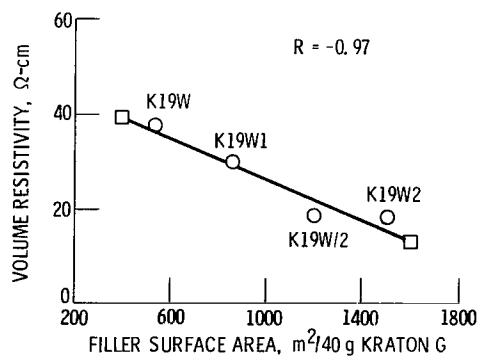


Figure 7. - Effect of total filler surface area on volume resistivity of Kraton G coatings.



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