Toxics Use Reduction Institute

ASSESSING THE ELECTRICAL PROPERTIES OF ALTERNATIVE WIRE AND CABLE COATINGS: METALLOCENE EPDM

TOXICS USE REDUCTION INSTITUTE

UNIVERSITY RESEARCH IN SUSTAINABLE TECHNOLOGIES PROGRAM

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Assessing the Electrical Properties of Alternative Wire and Cable Coatings: Metallocene EPDM

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The Toxics Use Reduction Institute
University Research in Sustainable Technologies Program

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Preface

The University Research in Sustainable Technologies program taps the research capabilities of the University of Massachusetts to advance the investigation, development and evaluation of sustainable technologies that are environmentally, occupationally and economically sound.

The Toxics Use Reduction Institute at the University of Massachusetts Lowell (UML) has actively worked with industries in Massachusetts to encourage their use of technologies that minimize toxic material usage. At the same time, the Institute has been instrumental in creating partnerships between industry and academia through its University Research in Sustainable Technologies program, among others, to link the scientific research needs of industry to the expertise of Massachusetts universities. A recent example of this kind of partnership is in the coated wire and cable industry, which has a strong presence in Massachusetts. The Institute developed a supply chain sector initiative for this industry[1], linking the coating producers and extruders to their original equipment manufacturers and suppliers for dialogue about how to plan for and address the impact of emerging international materials restrictions. For example, the use of lead stabilizers in wire coatings will be phased out under the European WEEE and RoHS electronics directives[2]. Faculty at UML have conducted relevant research to assist this industry sector in finding alternatives to existing products in order to maintain market share in Europe and around the world[3].

The Institute provided funding to the UML Plastics Engineering Department to conduct research into alternative wire and cable coatings that eliminate the need for lead. The following report, which is based on a presentation, provided by the authors at the Rubber Division, American Chemical Society conference in Grand Rapids, Michigan on May 17, 2004, discusses the research conducted and the findings.

Abstract

New regulations have led to the need for non-lead compounds to be used in wire and cable applications. There are two approaches for lead free EPDM wire and cable compounds. One is to find alternative stabilizers, and the other is to change resins. In a previous study, hydrotalcite was examined as a replacement for lead additives in applications using Ziegler-Natta based EPDM, and was very effective as an alternative stabilizer. In this research, the metallocene based EPDM was investigated as a potential replacement elastomer, which would not require lead stabilization.

The effect of hydrotalcite and metallocene EPDM on the behavior of a representative wire and cable was studied. Changes in properties, such as water absorption, volume resistivity (aged and unaged), dielectric constant (aged and unaged), and heat resistance were measured and indicated that metallocene EPDM compounds may be effective replacements for Ziegler-Natta based EPDM compounds without requiring lead stabilization.

Introduction

Basic cable consists of a metallic conductor encased in a dielectric. This system is surrounded by a shield, and finally a jacket material to provide environmental protection.[4] Polymeric materials can be used in many of the components, particularly insulation and jackets. Insulation compounds are designed to meet the electrical requirements of the application. The requirements for jacketing materials depend greatly on the specific application, but resistance to environmental conditions (such as moisture) is typically required.

As current is put through a conducting material, the temperature of the conductor will increase from resistive heating. Insulating materials must be able to withstand the heat generated by the flow of current through the conductor. To a certain extent, the ability of the insulation to withstand the heat generated is a limitation to the amount of current that may be put through the conducting wire. Therefore, the effect of temperature on material properties should be evaluated for wire and cable materials. Electrical properties that are critical to wire and cable applications include resistivity, dielectric strength, dielectric constant, and dielectric loss. In certain applications, fire resistance properties are also important.

Chlorine containing polymers, such as polyvinyl chloride (PVC) resins and neoprene, are used in the wire and cable industry as an insulation material. They are, however, susceptible to degradation reactions from elevated temperatures and UV radiation, leading to the production of hydrogen chloride (HCl) as illustrated in Figure 1. The presence of HCl in the compound causes an acceleration in the degradation rate, as well as increased moisture uptake, and reduced electrical properties[5,6]. In order to provide resistance to degradation, stabilizing additives are required. Lead additives are known to be particularly effective stabilizers[7], reacting with the HCl evolved during decomposition, preventing it from causing further degradation, and yielding reaction products that do not further destabilize the system[8]. Lead additives are also used for EPDM elastomers. Although the EPDM backbone does not contain chlorine groups, EPDM may have chloride ion content in the range of 100 to 800 ppm from the presence of residual catalyst[9], causing poor electrical properties[10].

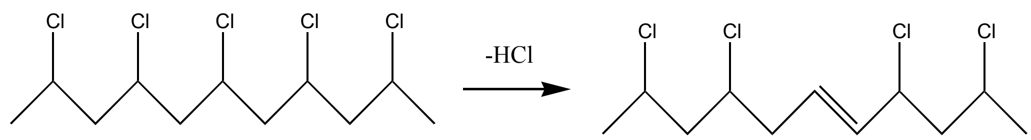


Figure 1. Degradation reactions leading to the production of hydrogen chloride.

In the 1990s, regulations, such as California's Proposition 65, and public reaction to the issue of lead content in consumer products, put pressure on wire and cable manufacturers to reduce the lead content in cable applications where there may be human contact[11]. A number of alternative stabilizers have been developed, but typically they are not as effective as lead stabilizers. Furthermore, the lead chloride reaction products, do not destabilize the system, as do the reaction products from other stabilizer systems, for example, zinc chloride[12].

Although EPDM prepared by Ziegler-Natta (Z/N) catalysts has no chlorine groups in the backbone, to obtain good electrical properties anion scavengers, such as lead additives, are required in the formulation. This is a result of residual Z/N catalyst in wire and cable compounds. The residual Z/N catalyst in the compound leads to an acceleration in the degradation rate, as well as increased moisture uptake and reduced electrical properties, and as well as susceptibility to degradation reactions from elevated temperatures and UV radiation, leading to the production of hydrogen chloride (HCl)[13,14]. In order to provide resistance to degradation, stabilizing additives are required such as lead additives. New regulations have led to the need for non-lead compounds to be used in wire and cable applications[15].

There are two possible approaches to remove the lead additives from EPDM compounds for wire and cable applications. The first is to identify alternative stabilizers, and the second is use an EPDM with reduced levels of catalyst, such as a metallocene EPDM.

Exxon marketed metallocene-LLDPE in 1992 for the first time. Since the first development of metallocene based polymers, demand for metallocene and single site polymers in the US has increased sharply, with growth rates expected to average roughly 7.3% through 2007[16,17]. Polymers produced by metallocene catalysts have different microstructure compared to polymers synthesized by Ziegler–Natta catalysts. Metallocene based polymers have unique properties, such as narrow molecular weight and uniform co-monomer distributions. Therefore, metallocene polymers are now typically used in numerous applications due to their improved barrier properties (e.g., reduced oxygen and water vapor permeability) as well as superior mechanical and optical properties[18].

In view of dielectric properties, most polymers are good insulators because of the low availability of free electrons[19]. Although the Z/N EPDM backbone does not contain chlorine groups, the EPDM may have chloride ion content in the range of 100 to 800 ppm from the presence of residual catalyst[20]. The presence of residual chloride ion in EPDM can be responsible for poor electrical properties, as well as causing corrosion problems[21]. The presence of mobile ions will result in decreased insulating ability (increased conductivity)[22]. In addition, the reaction of the chloride ions with zinc oxide, which is commonly used in rubber compounds, results in the formation of zinc chloride[23].

In our previous study[24], the use of hydrotalcite as an anion scavenger was examined as an alternative to the use of lead stabilizers for Z/N based EPDM. Hydrotalcite was shown to be an effective alternative stabilizer for Z/N EPDM elastomers. In this paper, we investigate the use of metallocene based EPDM as an alternative to Z/N based EPDMs. The effect of replacement of the Z/N EPDM with metallocene EPDM and the addition of hydrotalcite stabilizers on the water absorption, volume resistivity (aged and unaged), dielectric constant (aged and unaged), and heat resistance will be discussed.

Experimental

Materials

Two different grades of metallocene EPDM, Nordel IP 4725 and Nordel IP 3720, were supplied by Dow-Dupont elastomers. Both exhibit low Mooney viscosities, but differed in the diene level (Nordel IP 4725 contained a high diene level, while Nordel IP 3722 was low diene level). A standard wire and cable Z/N EPDM compound without stabilizers was supplied by BIW Cable Systems, Inc., and was prepared as a control compound by adding lead stabilizer. The basic rubber formulation is shown in Table I (values expressed in parts per hundred, phr), for each different compound the type of EPDM and stabilizer was changed. Table II lists the formulations with the type of EPDM and the quantity of hydrotalcite used for each of the compounds.

Table I - Base Rubber Formulation

Ingredients	Parts per Hundred Resin (phr)
EPDM	100
Fillers (non black)	113
Antioxidant	1
Coupling agent	1
Internal lubricant	15
Stabilizer	Various
Curing agent	8

Table II - EPDM Rubber Formulations with Stabilizers

Compound	Stabilizer(g)	
	Lead Stabilizer	Hydrotalcite
Z/N EPDM-Pb	10 phr	---
Nor-4725-0	---	0 phr
Nor-4725-10	---	10 phr
Nor-4725-20	---	20 phr
Nor-3722-0	---	0 phr
Nor-3722-10	---	10 phr
Nor-3722-20	---	20 phr

The base EPDM was first mixed with 20% of the total amount of clay and talc, zinc oxide, antioxidant, and stearic acid in a torque rheometer (Haake) at 50°C for 10 minutes. After the mixing, the rest of ingredients were added and mixed for an additional 15 minutes at the same speed, until the temperature reached approximately 80°C. After mixing in the torque rheometer, the EPDM rubbers were cured into sheets using a hot press at 177°C and 10 MPa for 20 minutes.

Water and Toluene Absorption

For the water resistance tests, 1 cm x 2 cm samples were placed for 2 hours in a boiling water bath or 24 hours in a room temperature water bath. After removal from the liquid, they were wiped with a dry cloth and weighed. In addition, the water absorption after aging for seven days in 100°C air oven was measured by immersing the aged samples in boiling water for two hours or for 24 hours in a room temperature water bath. The liquid absorption was measured as described above. The toluene absorption was measured by immersing the samples in toluene for three days and two months at room temperature. After the specified time period the samples were removed, wiped dry, and weighed.

Electrical Properties Testing

The dielectric constants were measured using Boonton capacitance. The dielectric constant of the unaged rubber sheets was measured. The effect of aging on the electrical properties was determined by aging the rubber sheets at 100°C (to accelerate the aging) in an air oven for about 7 days, followed by measurement of the electrical properties. The effect of aging and moisture was evaluated by measuring the electrical properties of rubber sheets which were aged at 100°C and immersed in boiling water. Prior to electrical testing the samples were removed from the water, wiped, and allowed to dry for one hour prior to measuring the electrical properties. Volume resistivity using an HP 4210 ohmmeter was also measured. To see the dependence of temperature on volume resistivity, the volume resistivity at different temperatures was measured.

Mechanical Properties

For mechanical properties, rubber sheets were cut into dogbone shaped specimens using an ASTM D412 type D die. The mechanical properties were measured using a universal testing machine (Instron 4400R) with a crosshead speed of 50 mm/min. Crosshead displacement was used to measure the strain. The load and displacement data were recorded using data acquisition software (Instron Series IX) with a sampling rate of 20 points per second.

Hardness and Resilience Tests

EPDM compounds were measured with a Shore A scale durometer. The test specimens were generally 6.4mm thick. The resilience of elastomers was measured with a rebound tester (Bashore).

Surface Characterization

Scanning Electron Microscopy (SEM, Amray 1400) with energy dispersive X-Ray Spectroscopy (EDXS) analysis was used to study the surface morphology and chemical analysis of the various samples. When electrons hit the specimen, characteristic X-rays are emitted for each element present on the surface, which when detected by the EDXS detector gives the characteristic X-ray peak for that element on the spectrum. Using this information, it was possible to perform elemental analysis of the surface and determine the presence of fillers and various additives on the sample surface. SEM samples were prepared by cutting fresh slices of the sample surface using a sharp knife and then sputter coating the samples using graphite rods to prevent charging during SEM imaging.

Roughness Test Using AFM

To measure the surface roughness of the samples, atomic force microscopy (AFM) was used. XE- 150 AFM (PSIA Inc.) was used in the non-contact mode with non-contact silicon cantilevers (NSC-15) and a force constant of 40 N/m at scan size of 50*50 µm. The roughness values were calculated using the AFM image processing software XEI 1.2.

Results and Discussion

Water Absorption

Table III shows the water uptake data for the metallocene EPDM compounds and the control lead stabilized Z/N EPDM compound. As seen in Table III, all of the compounds showed very little moisture uptake in either boiling water or room temperature water, regardless of the type of EPDM and or the amount of stabilizer. EPDM rubbers are known for their resistance to polar fluids, such as alcohols, ketones, certain esters and acetates, water, and a variety of aqueous solutions. The highly saturated polymer backbone of EPDM is key to providing oxidative stability and excellent weathering resistance. It was therefore important that any additives preserved these excellent characteristics. All the compounds showed no increase in moisture uptake, indicating that the alternative systems were comparable to the lead stabilized system in terms of moisture absorption.

Table III - Water Absorption of Wire and Cable Compounds

Compound	Boiling Water (Weight change %) 2 hours	Room Temperature Water (Weight change %)	
		24 hours	120 hours
Z/N EPDM-Pb	0.09	Negligible	Negligible
Nor-4725-0	Negligible	Negligible	Negligible
Nor-4725-10	Negligible	Negligible	Negligible
Nor-4725-20	Negligible	Negligible	Negligible
Nor-3722-0	Negligible	Negligible	Negligible
Nor-3722-10	Negligible	Negligible	Negligible
Nor-3722-20	Negligible	Negligible	Negligible

In Table IV, the percentage of toluene absorbed for all the samples is reported. The Nor 3722 compounds had the highest toluene absorption of all the compounds. The Nor 4725 compounds had intermediate swelling values, while the Z/N EPDM lead compounds had the lowest swelling values. Within a grade, the amount of toluene absorbed increased as the amount of hydrotalcite increased. After immersion for two months, all the materials showed an increase in toluene uptake. This may be due to degradation or leaching of material from the compound.

Table IV - Toluene Absorption of Wire and Cable Compounds

Compound	Percent Swelling due to Toluene Absorption	
	3 days	2 months
Z/N EPDM-Pb	28.2	108.3
Nor-4725-0	45.2	94.7
Nor-4725-10	52.6	96.5
Nor-4725-20	58.8	96.3
Nor-3722-0	64.4	121.1
Nor-3722-10	72.4	109.8
Nor-3722-20	80.0	130.5

Heat Stability

Figure 2 shows the weight loss data for rubber specimens aged in an air oven at 100^{o}C. As seen in Figure 2, the addition of a stabilizer had negligible effects on the aging resistance for metallocene EPDM, while it was found to significantly affect the aging resistance for Z/N EPDM in the previous study[24]. The Z/N EPDM compounds with lead stabilizer showed the highest weight loss values of all the compounds, while overall the metallocene EPDM compounds gave better heat resistance.

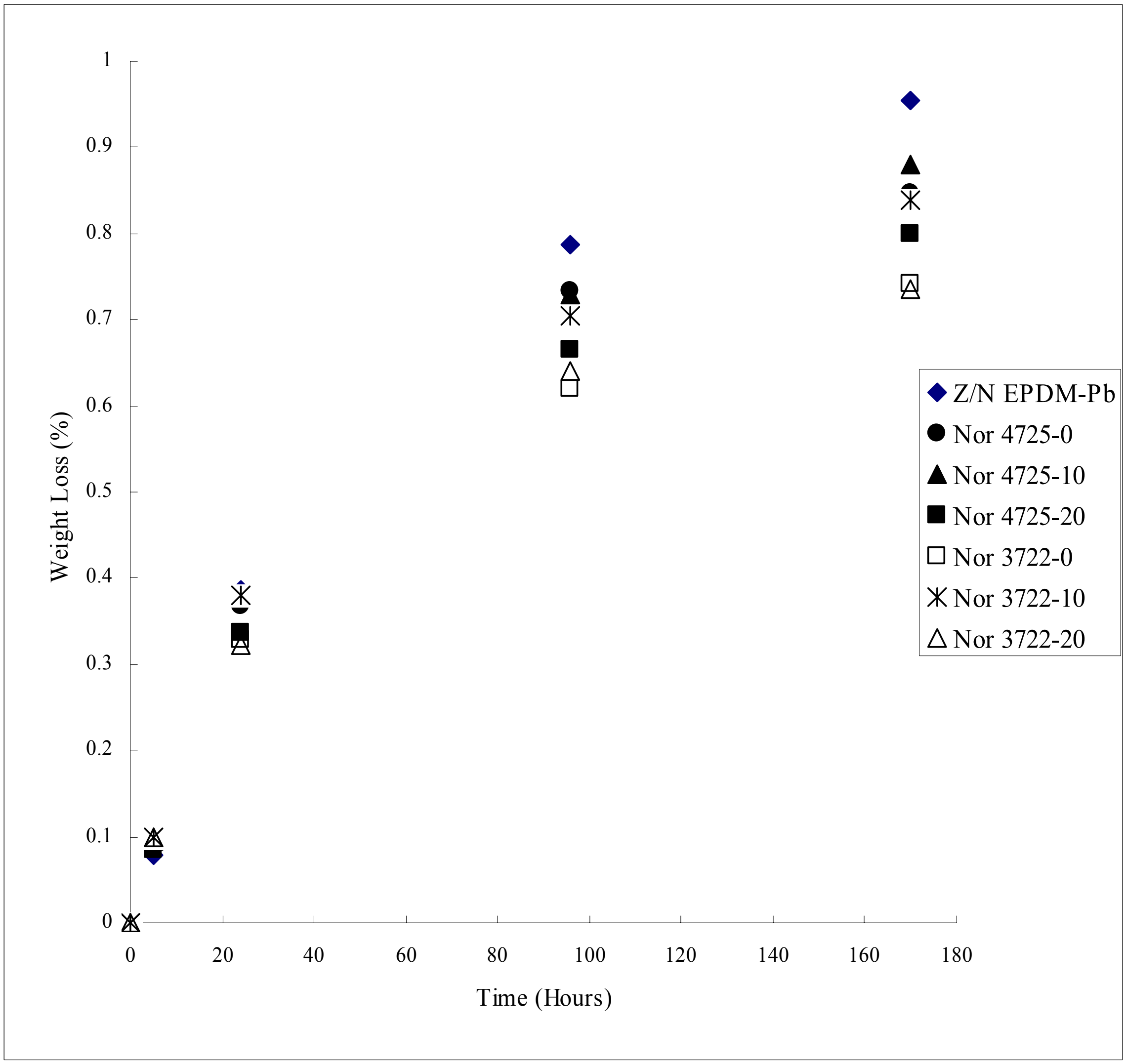


Figure 2 - Percent weight loss in 100^{o}C oven.

Electrical Properties

Dielectric constant measurements are used to determine the ability of an insulator to store electrical energy. The dielectric constant is the ratio of the capacitance induced by two metallic plates with an insulator between them to the capacitance of the same plates with air between them. When polymer compounds are placed in an electric field, the polar groups in the polymer tend to orient in that field. For electrical applications a low dielectric constant is desirable.

Table V shows the dielectric constants for all of the compounds in this study. The dielectric constant values of EPDM compounds are generally very low, as a result of the lack of polar groups in the backbone. The dielectric constant values for unaged compounds ranged from 2.9 to 3.1. Although the metallocene EPDM showed slightly higher dielectric constants than the Z/N EPDM compounds initially, the dielectric constants were remarkably similar after aging. After aging in the 100°C air oven for seven days, the values of the dielectric constant increased for all compounds. This may be the result of oxidation reactions, generating polar groups in the molecules. For samples immersed in 100°C water for 2 hours after aging at 100°C in an air oven for seven days, (see Table V) the dielectric constant shows very little increase and all compounds behaved similarly. This may be the result of the low water absorption of these compounds. Addition of hydrotalcite showed no effect on the performance of the compounds.

Table V - Dielectric Constant After Oven Aging

Compound Number	Unaged	Aged at 100°C in Oven for 10 hours	Aged 100°C Oven for 7 Days and Immersed in 100°C Water for 2 Hours
Z/N EPDM-Pb	2.9	3.1	3.2
Nor-4725-0	3.1	3.2	3.2
Nor-4725-10	3.1	3.3	3.3
Nor-4725-20	3.0	3.1	3.3
Nor-3722-0	3.1	3.2	3.3
Nor-3722-10	3.1	3.2	3.3
Nor-3722-20	3.0	3.3	3.3

Volume resistivity is the resistance to leakage of current through the body of an insulating material. Table VI shows the volume resistivity of all the compounds. A decrease in electrical resistivity is generally the result of both ionic impurties and mobility. Ionic impurities typically provide the means for conduction of electricity through polymeric materials[25]. As a result, both mobility and ion content will affect the electrical properties. As seen in Table VI, Z/N EPDM had higher volume resistivity than that of the metallocene based EPDM compounds. Compounds with the low diene content metallocene EPDM had higher resistivity compared with the high diene content metallocene EPDM. These results indicate that the low diene content metallocene materials offer promise for electrical insulating applications. The addition of the hydrotalcite stabilizer appeared to have no effect on volume resistivity behavior.

Table VI - Volume Resistivity

Compound Number	Unaged (10^{14} Ohm cm)	Aged at 100°C in Oven for 10 Hours (10^{14} Ohm cm)	Immersing Boiling Water for 2 Hours After Aged 100°C Oven for 7 Days (10^{14} Ohm cm)
Z/N EPDM-Pb	13.0	12.9	0.26
Nor-4725-0	5.5	5.5	0.050
Nor-4725-10	2.6	2.0	0.051
Nor-4725-20	3.5	2.8	0.075
Nor-3722-0	8.1	6.9	0.077
Nor-3722-10	8.9	6.5	0.089
Nor-3722-20	8.8	6.7	0.088

Table VI also show the results of immersing oven aged samples in the boiling water for two hours. A significant decrease in electrical resistivity was found for all compounds, the result of both water absorption and degradation. Z/N EPDM had still higher volume resistivity than that of metallocene EPDM. Figure 3 shows the dependence of volume resistivity on temperature for the control compound (Z/N EPDM with lead) and the Nor 4725-10 metallocene EPDM compound. Both materials show a decrease in resistivity with temperature. Overall, the volume resistivity of the Z/N EPDM compound was greater. At higher temperatures, the ionic mobility would be increased and the volume resistivity would be expected to decrease.

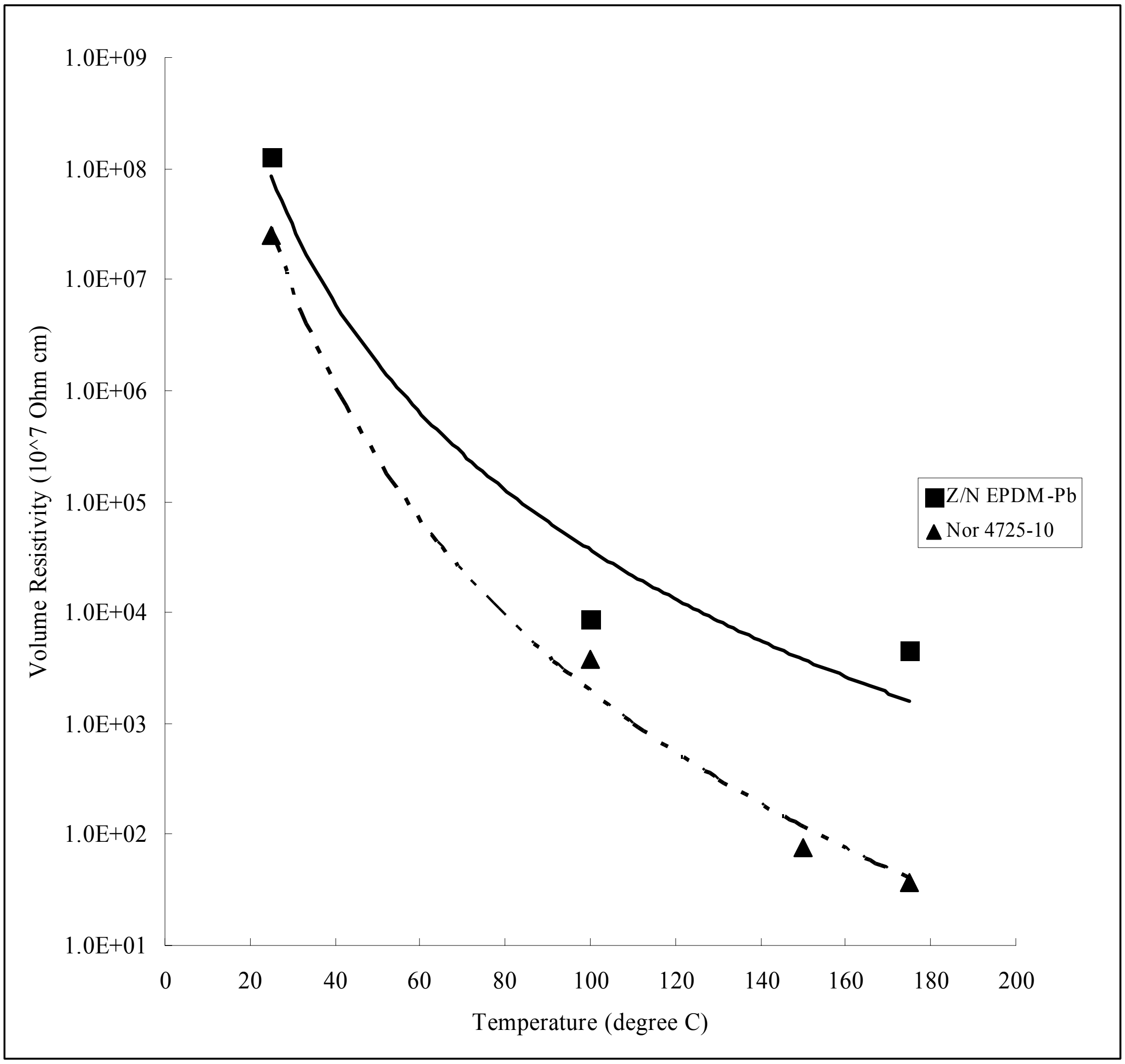


Figure 3 - The dependence of volume resistivity on temperature

Mechanical Properties

Table VII shows the tensile properties for all compounds investigated. Figure 4 compares the tensile moduli for all compounds and Figure 5 compares the elongation at break. The 100% modulus, 300% modulus, and tensile strength values of the control compound (Z/N EPDM with lead) were roughly one half that of the metallocene EPDM. The elongation at break of the control (Z/N EPDM with lead) was higher, however, than any of the other compounds. The compound formulation was not adjusted for the metallocene EPDM and these results show that a direct substitution into a formulation would not be advisable. Comparing the metallocene EPDM compounds with different levels of hydrotalcite, we find that increasing the amount of additive reduces the 100% modulus, 300% modulus, tensile strength, and elongation at break. The results indicate that the formulation should be optimized for the specific elastomer grade.

Table VII - Tensile Strength and Elongation of Unaged Compounds

Compounds	100 % Modulus (MPa)	300 % Modulus (MPa)	Tensile Strength (MPa)	Elongation at Break (%)
Z/N EPDM-Pb	2.4	1.4	6.1	590
Nor-4725-0	6.2	3.7	15.1	522
Nor-4725-10	5.6	3.4	13.0	476
Nor-4725-20	5.5	3.1	12.0	453
Nor-3722-0	6.4	3.5	11.7	432
Nor-3722-10	6.3	3.4	11.3	407
Nor-3722-20	5.8	3.1	10.3	401

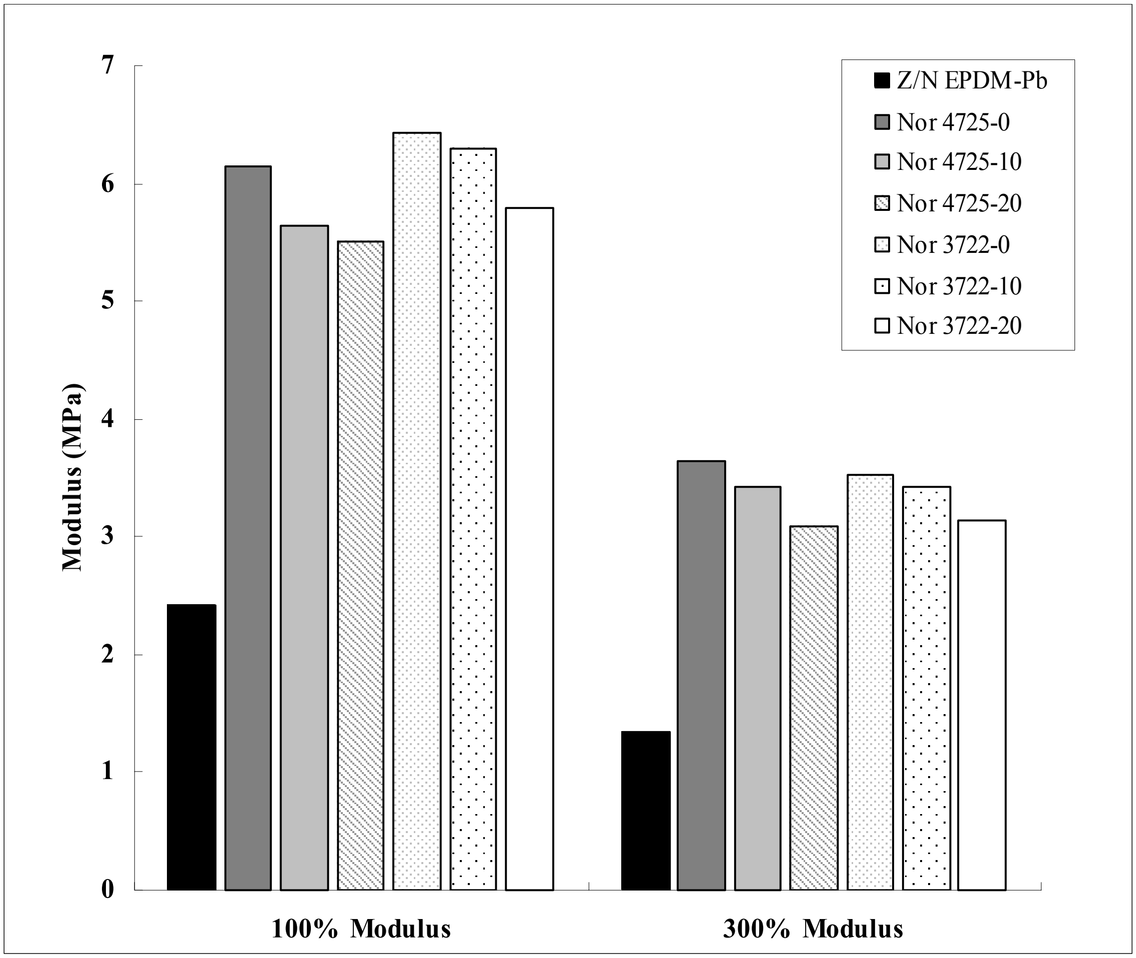


Figure 4 - Comparison of tensile moduli

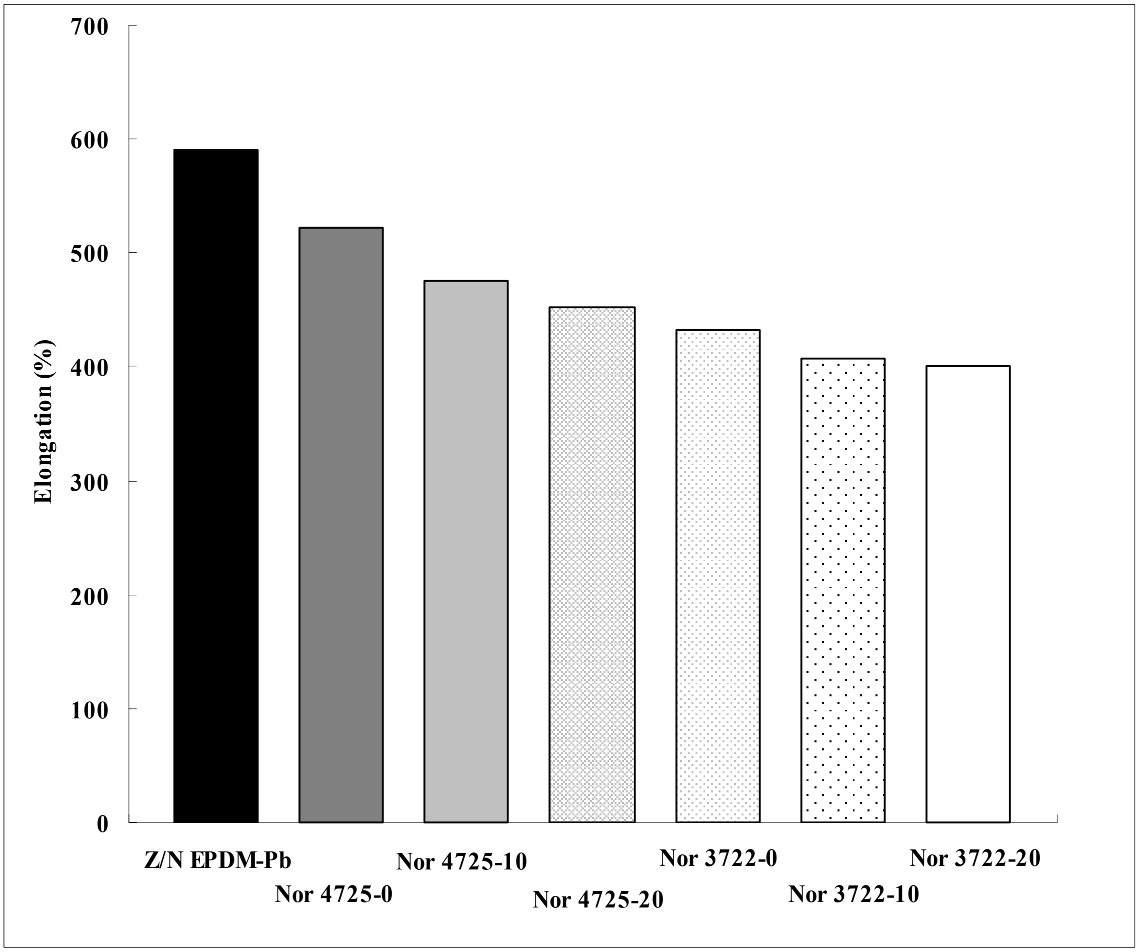


Figure 5 - Comparison of elongation at break

Hardness and Resilience

Table VIII shows the hardness and resilience test results for all compounds investigated. As expected, the metallocene based compounds showed increased hardness compared to the Z/N EPDM compounds. The resilience of the control (Z/N EPDM) and one of the metallocene (Nor 4725) based compounds were similar, while the other metallocene based EPDM showed lower resilience compared to the Z/N compound.

Table VIII - Hardness and Resilience

Compound	Hardness	Resilience
Z/N EPDM-Pb	55	47
Nor-4725-0	79	42
Nor-4725-10	77	44
Nor-4725-20	74	47
Nor-3722-0	83	22
Nor-3722-10	82	25
Nor-3722-20	81	25

Surface Characterization and Roughness Measurement

SEM/EDXS analysis was performed on the samples after the water aging test. Table IX shows the major and minor elements found on the surface of the various samples. The major elements constitute the elements that are present all over the surface, whereas the minor elements are the ones that are found in only particles or pits present on the sample surface and in limited locations. Figure 6 shows a representative SEM image along with EDXS maps for the Z/N EPDM sample. The surface analysis of the samples revealed that all samples showed similar microstructure with presence of pits and particles, however in samples with higher parts per hundred of resin (phr) hydrotalcite, calcium was present more prominently on the surface. More titanium peaks were found in the particles on the surface of Nor 3722 samples as compared to Nor 4725.

Table IX - Major and Minor Elements Present on the Surface as Determined By EDXS Analysis

Compound	Major Element	Minor Element
Nor-4725-0	Si, Al, Zn	Na, Mg , Fe, Cl, K,
Nor-4725-10	Si, Al, Mg, Zn, Ca	Cl, K, Fe
Nor-4725-20	Si, Al, Mg, Zn, Ca	Ti , Fe
Nor-3722-0	Si, Al, Zn	Mg, Ca, Ti
Nor-3722-10	Si, Al, Zn, Ca	Ti, Mg, Fe
Nor-3722-20	Si, Al, Zn, Ca, Cl	Ti
Z/N EPDM-Pb	Si, Al, Mg, Zn	Cl, Ti, Pb

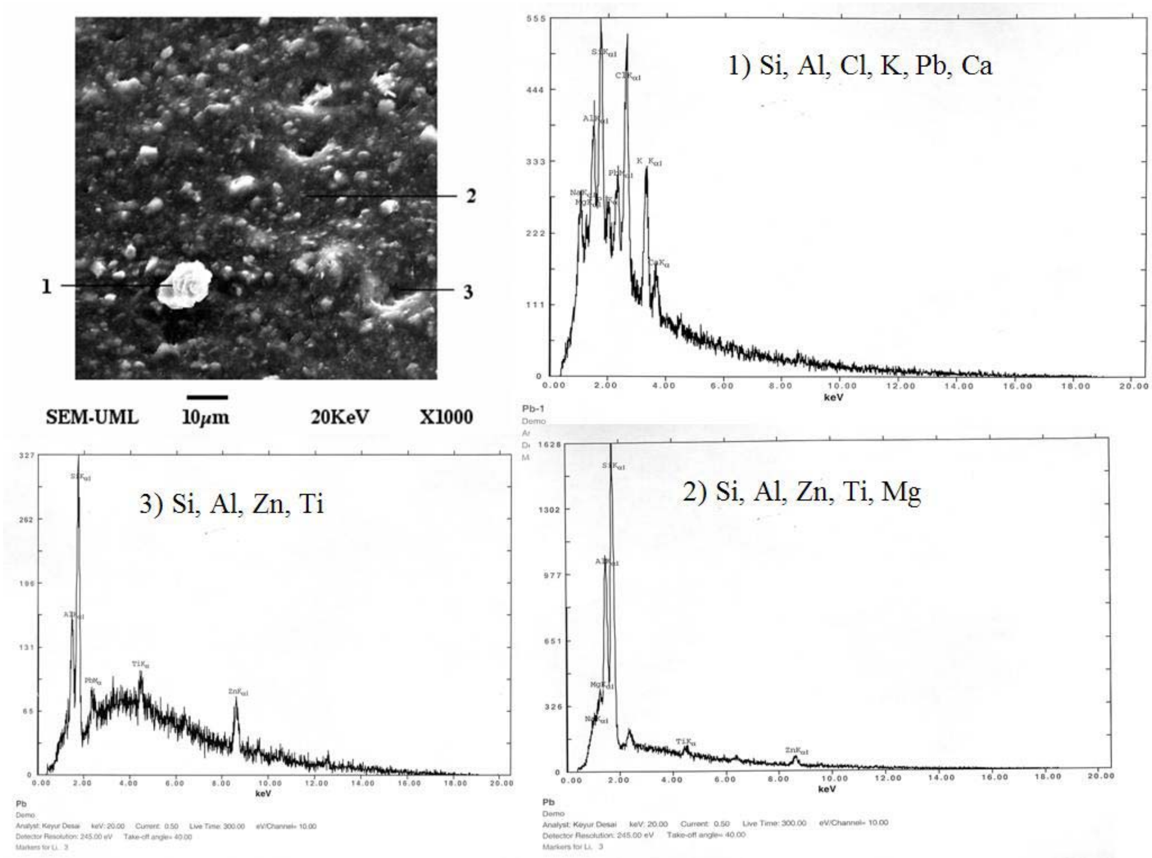
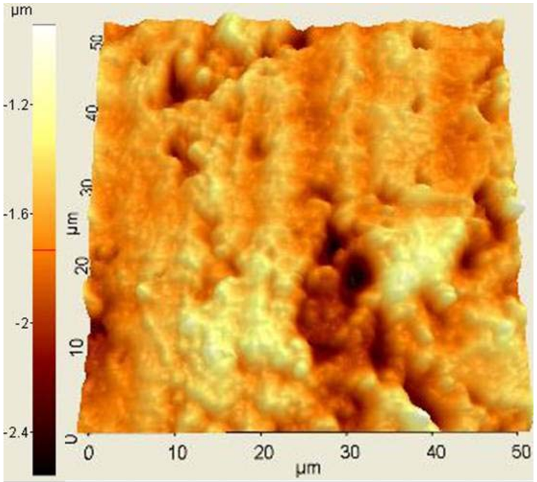
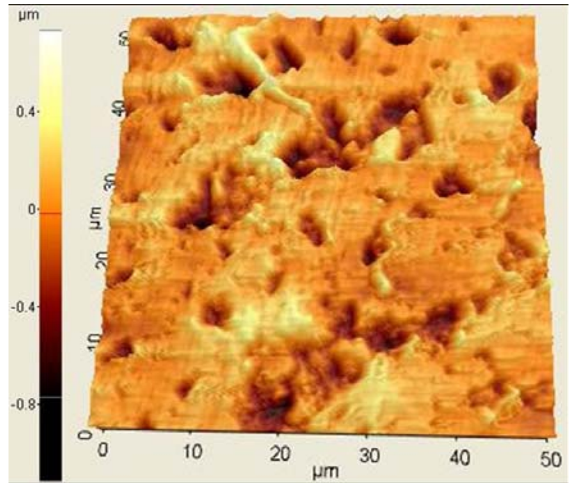


Figure 6 - SEM/EDXS analysis of Z/N EPDM sample containing lead the EDXS maps 1, 2 and 3 are from areas marked 1, 2 and 3 on the sample.

The effects of water aging on the surface characteristics were evaluated using AFM. Figure 7 shows a representative AFM image of the surface before and after water aging tests. The surface roughness values before and after the water aging for all the samples are shown in Figure 8. Nor-4725-0 had the roughest surface amongst all the samples, while the other samples showed roughness similar to the Z/N EPDM sample. There is a slight increase in surface roughness after water testing due the development of cracks and pits on the surface. The addition of hydrotalcite appears to reduce the change in surface roughness with water aging.





(A) (B)

Figure 7 - 3-D AFM topography image of NOR 4725 0 (0% phr hydrotalcite) - A is virgin sample, B is the same material after water aging.

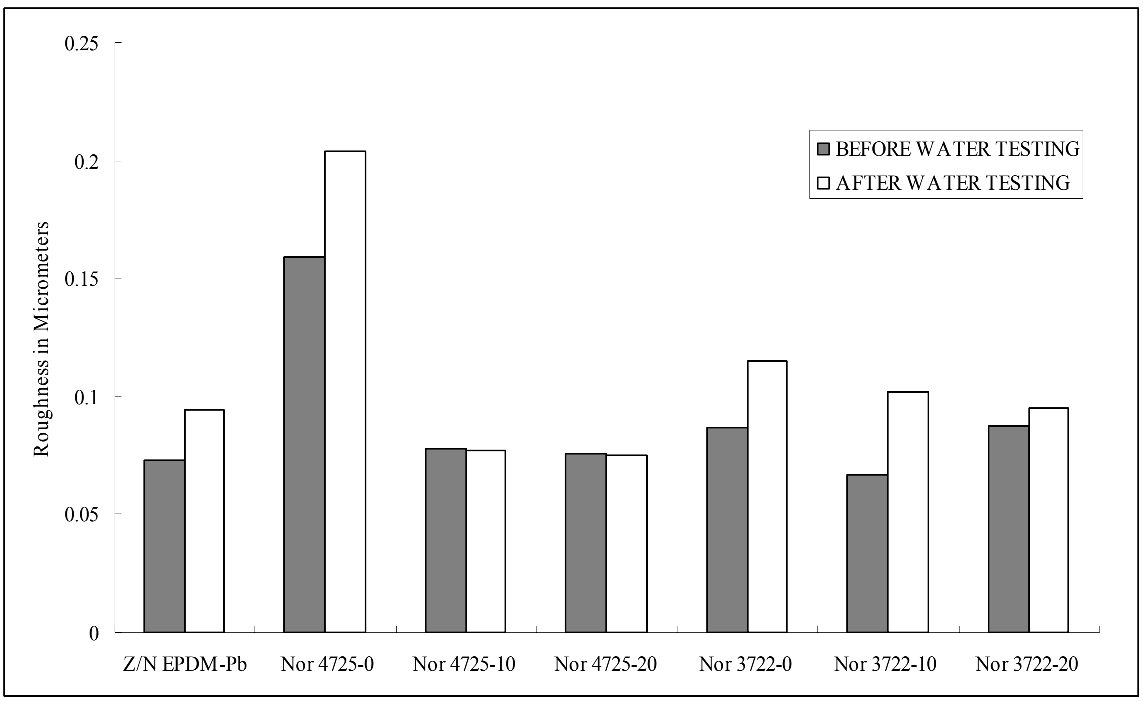


Figure 8 - Surface roughness values of various samples before and after water aging.

Conclusions

The use of metallocene EPDMs with hydrotalcite stabilizer for wire and cable compounds was investigated as an alternative to lead stabilized compounds. Metallocene based EPDM with various percentages of hydrotalcite stabilizers (no lead stabilizer) were compared to a lead stabilized Ziegler-Natta based EPDM compound.

The metallocene based EPDM compounds showed similar moisture uptake to the Z/N EPDM compounds without the presence of the lead stabilizer. Metallocene based EPDM compounds showed improved heat stability over the Z/N based compounds. Dielectric constant values were similar for all compounds, with values increasing after heat aging. Addition of hydrotalcite to the metallocene based EPDMs showed little effect on the dielectric constant. The volume resistivity measurements showed that the Z/N EPDM had higher resistivity than the metallocene based EPDM compounds. Hydrotalcite addition appeared to have little effect on volume resistivity.

The 100% modulus, 300% modulus, and tensile strength values of the metallocene EPDM based compounds were nearly double those of the control compound (Z/N EPDM with lead), while the elongation at break of the control (Z/N EPDM with lead) was highest. Addition of hydrotalcite was found to decrease the 100% modulus, 300% modulus, tensile strength, and elongation at break within an elastomer grade. These results show that further work is required to optimize the formulation.

The results indicate that metallocene based EPDM compounds offer promise for electrical applications without the need for the addition of lead stabilizers as is required for Ziegler-Natta based EPDM compounds. Further compound development is necessary to optimize the performance characteristics of the metallocene based EPDM materials.

Acknowledgements

The authors would like to acknowledge the financial support of the Toxic Use Reduction Institute (TURI), the material support of Dow-Dupont elastomers, and the technical support of Draka USA (BIW) Company.

Reference

[1] Visit the Toxics Use Reduction Institute website at www.turi.org for more information about our supply chain work

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