Effect of Alteration of Antioxidant by UV Treatment on the Dielectric Strength of BOPP Capacitor Film

Janet Ho, Ramamurthy Ramprasad, and Steven Boggs
Institute of Materials Science
University of Connecticut U-3136, USA
97 North Eagleville Road
Storrs, CT 06269-3136

ABSTRACT

The effect of altered products of the antioxidant by UV irradiation on dielectric breakdown strength of metallized biaxially-oriented polypropylene (BOPP) capacitor film was studied. Results from Weibull statistical analysis show that an optimal UV exposure level of ~3.4 J/cm^2 (200-400 nm) increases the breakdown strength at 5% probability by ~20%. As indicated by UV spectroscopic analysis, the antioxidant in BOPP was consumed almost entirely to form 2,6-di-tert-butyl-p-benzoquinone (2,6-DTB-PBQ). Density functional theory computations indicate that this compound is electronegative, which probably accounts for the increased dielectric strength.

Index Terms — **metallized film capacitor, irradiation, antioxidant, breakdown.**

1 INTRODUCTION

OVER the past two decades, the energy density of capacitors has been improved from ~0.05 J/cm^3 for the film/foil construction [1] to about 1 J/cm^3 for pulse discharge metallized film capacitors. Demand for ever higher energy density continues. As the energy density scales with the square of the electric field in the dielectric, significant improvement of energy density for a given dielectric can only be achieved by increasing the dielectric breakdown strength. Schneuwly et al [2] have shown that impregnation with rape-seed oil increased the breakdown strength of metallized polypropylene film by as much as 25%, but a complete impregnation required 20 days. Our original approach was to impregnate unmetallized biaxially-oriented polypropylene (BOPP) film with a monomer, followed by polymerization of the monomer by UV light, but we discovered that improvement of the breakdown strength was achieved by UV treatment alone. In this work, we investigated the effect of altering the antioxidant by UV irradiation on the breakdown strength of metallized BOPP capacitor film, both experimentally and through density functional theory computations. We find that conversion of the ~0.3% anti-oxidant, Irganox 1010, present in the as-received film to benzoquinone, an electronegative species as indicated by density functional theory computations, improves the dielectric strength of the film appreciably. As the antioxidant is not required in a capacitor winding which operates in an anaerobic environment, as is typical of high energy density capacitor windings, such UV treatment of the BOPP film is a practical option.

2 EXPERIMENTAL

2.1 MATERIALS

The material studied was commercially available metallized BOPP capacitor film with a nominal thickness of 7 µm. The film was metallized on one side with a layer of Zn/Al alloy, the thickness of which is approximately 10 nm (7 Ω/sq). The BOPP film was used as-is. Reference chemicals used for comparison purpose in UV analysis include penta-erythritol tetrakis (3,5-di-*tert*-butyl-4-hydroxyhydrocinnamate) or Irganox 1010 from Aldrich and calcium stearate from TCI America. Acetonitrile (HPLC grade) was used as solvent, the UV cutoff of which is 190 nm.

2.2 BREAKDOWN STRENGTH MEASUREMENT

Figure 1 shows the breakdown measurement configuration for a metallized film sample. The bottom electrode is the metallization of the film under test, while the top electrode is a second metallized film with the metallization facing the unmetallized side of the test sample. A piece of metallized film with the metallization facing up is used below the test sample to make connection to its metallization. To protect against discharge from the top electrode to the edge of the metallization of the test sample and to define the active area for the breakdown measurement, a 100 µm polyimide mask with a window is placed in between the top electrode (metallized film) and the film under test. The active break-down area was 2 cm^2, roughly in the form of a square. When a ramp DC voltage was applied, the layers were drawn together by the electrostatic force, creating smooth interfaces without trapped air bubbles. Near the typical breakdown field of 700 V/µm, the pressure generated by the electrostatic force is ~5 MPa. Breakdown measurements were performed using

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a linear voltage ramp of 300 V/s at ambient conditions. A new set of film electrodes was used for each measurement.

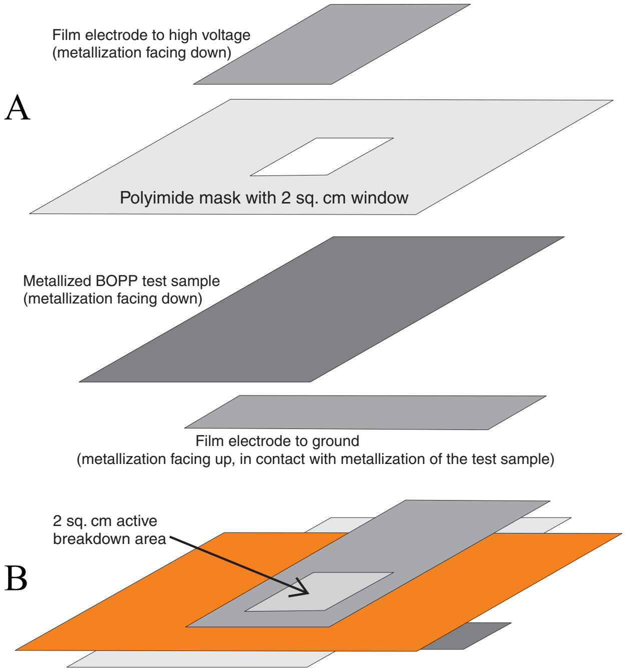


Figure 1. Schematic of the breakdown strength measurement set-up (a) the four individual layers separated, (b) during measurement

2.3 UV TREATMENT

Treatment of samples was conducted using a UV-curing chamber (Model Zeta 7200) from Loctite Corporation. The UV lamp system was a five-inch, 300 Watt/inch medium pressure mercury arc lamp with a parabolic reflector. In the case of breakdown strength measurement, a sample of the metallized BOPP film with metallization facing down was fixed on a metal frame that has a window of 72 mm x 90 mm. The assembly was then placed inside the UV curing chamber. UV irradiation of the film was carried out at a distance of 26.5 cm from the light source at ambient temperature and in the presence of air. Breakdown tests were carried out for exposure durations 5, 15, 25, 35, and 45 s. The 15 s and 45 s measurements were repeated with the addition of an infrared (water) filter in the exposure path to reduce the amount of heat to which the test specimens were subjected. Immediately after treatment, the assembly holding the exposed sample was transferred to a dust-free container to allow the sample to cool for ten minutes before the sample was removed from the frame for breakdown measurement. An untreated sample, taken from section of the BOPP film roll immediately adjacent to the corresponding treated sample was used as a control for all breakdown measurements. Variations in dielectric strength have been observed along the roll, and therefore individual control groups were used. To condition unmetallized BOPP samples for UV analysis, unmetallized BOPP film was placed on a piece of aluminum foil during irradiation to achieve a similar UV dosage as for a metallized sample.

2.4 UV LAMP INTENSITY MEASUREMENT

The intensity of the lamp in the UV-curing chamber was characterized using two different types of UV-enhanced PIN photodiodes, one silicon (Model F53-375, Edmund Optics) used with a visible/IR blocking filter (Model U-330, Hoya) and the other silicon carbide (Model PDU-S101, Photonic Detectors Inc.), which is only sensitive to the region from 200 nm to 400 nm, with peak response at 280 nm. Intensity of the lamp as a function of distance from light source to sample is shown in Figure 2. At 26.5 cm, the intensity was 0.2 to 0.25 W/cm² over the UV range from 200 to 380 nm.

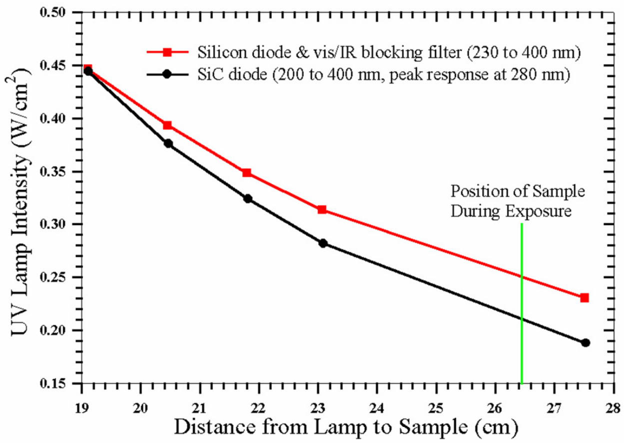


Figure 2. Intensity of the UV light source in the UV-curing chamber (Model Zeta 7200, Loctite) at various distances from sample.

2.5 UV ANALYSIS

All UV absorption spectra were obtained using Perkin-Elmer Lambda 900 UV/Vis spectrometer. A spectrum was taken before and after each UV exposure.

3 RESULTS AND DISCUSSION

2-parameter Weibull statistical analysis of the breakdown strength of metallized BOPP film was carried out for the various UV exposure durations. Figure 3 shows the breakdown strength and 90% confidence intervals at the cumulative probability of 63%, which is the Weibull characteristic value, and at the 5% level, which is more relevant to capacitor design. The active breakdown area was 2 cm^2. Data for the individual control groups and for all controls combined together are included for comparison. Figure 4 shows similar data with the addition of an infrared (water) filter in the optical path during exposure to reduce heating of the sample. Tables 1 and 2 provide summaries of the Weibull parameters for the various levels of UV exposure. The data plotted in Figures 3 and 4 suggest an optimal exposure of 5 to 25 s, at which the breakdown strength at 63% probability was increased by about 7% without the IR filter and by 12% with the IR filter, while at 5% breakdown probability, the breakdown strength was increased by 13% without the IR filter and by 10% with the IR filter relative to the untreated samples. At exposure levels above 35 s without the IR filter, the breakdown strength at 63% probability decreased to below that of the untreated samples. However with the IR filter, the Weibull characteristic breakdown strength changed little from 15 to 45 s exposure.

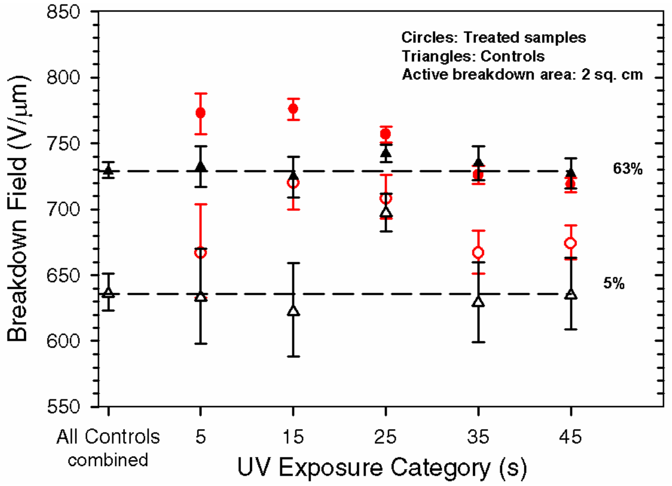


Figure 3. Breakdown strength at 63% (solid symbols) and 5% (hollow symbols) cumulative probabilities with 90% confidence limits for 7 µm thick metallized BOPP film at various UV exposure levels without an infrared (water) filter in the optical path.

Table 1. Summary of the 2-parameter Weibull statistics of metallized BOPP film at various UV exposure levels without an IR filter.

UV Exposure Level (S)	Weibull Characteristic Breakdown Strength (V/µm)	Breakdown Strength at 5% Probability (V/µm)	Slope	Number of Samples	Quality of Fit
0	729	636	21.9	101	0.96
5	773	667	20.2	18	0.96
15	776	720	38.8	18	0.98
25	757	708	45.3	20	0.97
35	726	667	35.2	26	0.86
45	719	674	46.8	27	0.96

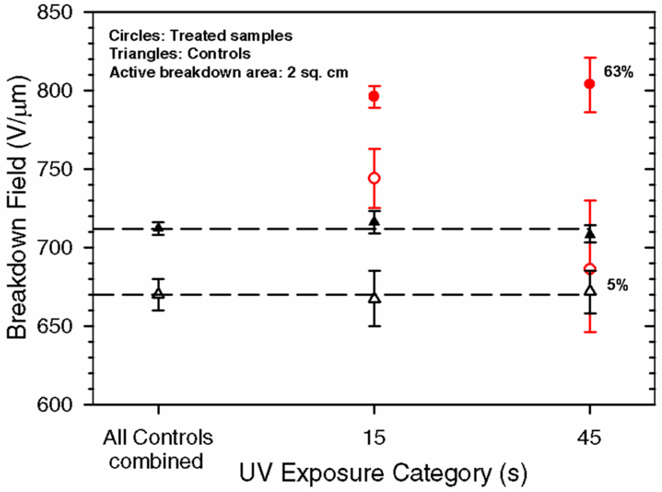


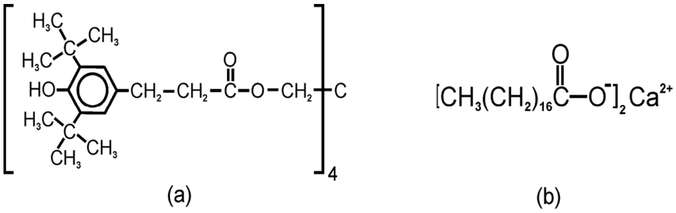
Figure 4. Breakdown strength at 63% (solid symbols) and 5% (hollow symbols) cumulative probabilities with 90% confidence limits for 7 µm thick metallized BOPP film at various UV exposure levels with an infrared (water) filter in the optical path.

Table 2. Summary of the 2-parameter Weibull Statistics of Metallized BOPP film at various exposure levels with IR filter.

UV Exposure Level (S)	Weibull Characteristic Breakdown Strength (V/µm)	Breakdown Strength at 5% Probability (V/µm)	Slope	Number of Samples	Quality of Fit
0	712	670	49.3	34	0.95
15	796	725	43.8	18	0.94
45	804	646	18.8	18	0.98

In order to understand the observed breakdown behavior, we investigated Irganox 1010 and calcium stearate, which are the only two additives present in the as-received BOPP film. Irganox 1010 is a sterically-hindered phenolic antioxidant for removing unstable radicals, while calcium stearate is a lubricant and an acid scavenger to neutralize any hydrochloric acid generated from reactions between the hindered phenol and residual chloride-containing catalyst [3]. The chemical structures of the two additives are shown in Figure 5. Irganox 1010 is an aromatic compound in which the phenyl group is an UV chromophore. Both calcium stearate, which is a long-chain aliphatic ester, and polypropylene do not have any absorption in the near UV region [4]. Therefore, UV spectroscopy was employed to study the behavior of Irganox 1010 under UV treatment.

Figure 6 shows the UV absorption spectrum of unmetallized as-received BOPP film. For comparison, the spectra of Irganox 1010 and calcium stearate in acetonitrile are also included. Two absorption peaks are apparent for the BOPP sample and the antioxidant reference sample, a narrow peak at around 204 nm with a shoulder, and a broad peak centered around 280 nm. An absorbance of about 0.05 is caused by the BOPP sample opacity. Since polypropylene and calcium stearate do not show significant UV absorption in the range of wavelengths studied, the two peaks shown in the BOPP film sample are attributed to the antioxidant. The concentration of Irganox 1010 present in BOPP film can be calculated using the Beer-Lambert law $A=acl$ [5], where A is the absorbance of a transparent medium with thickness l in centimeters, a the molar extinction coefficient in liters/mole-cm, and c the concentration in moles per liter. a is an intrinsic property of an absorbing molecule at a given wavelength in a given solvent and is independent of the concentration and thickness of the sample. By comparing the absorbance of BOPP film at 204 nm after baseline correction to that of 0.0003% Irganox 1010 in acetonitrile, we estimated the amount of Irganox 1010 remaining in BOPP film as about 0.3% by weight.



$$[CH_3(CH_2)_{16}C(=O)-O^-]_2Ca^{2+}$$

Figure 5. Chemical structure of (a) Irganox 1010, (b) Calcium stearate

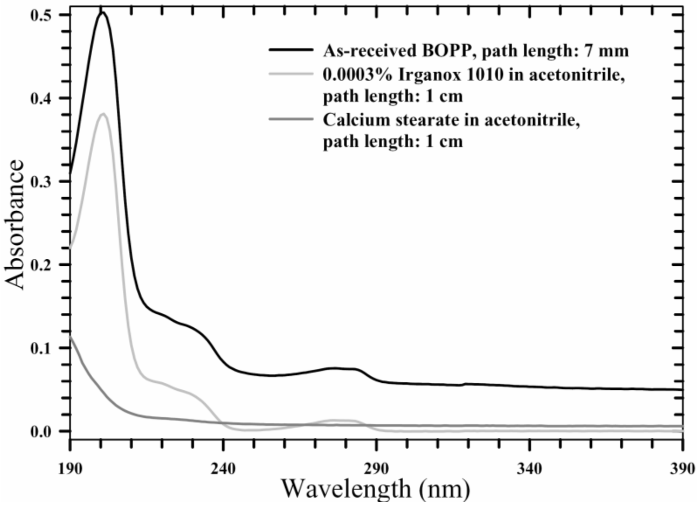


Figure 6. UV absorption spectra of an as-received BOPP film, Irganox 1010, and calcium stearate.

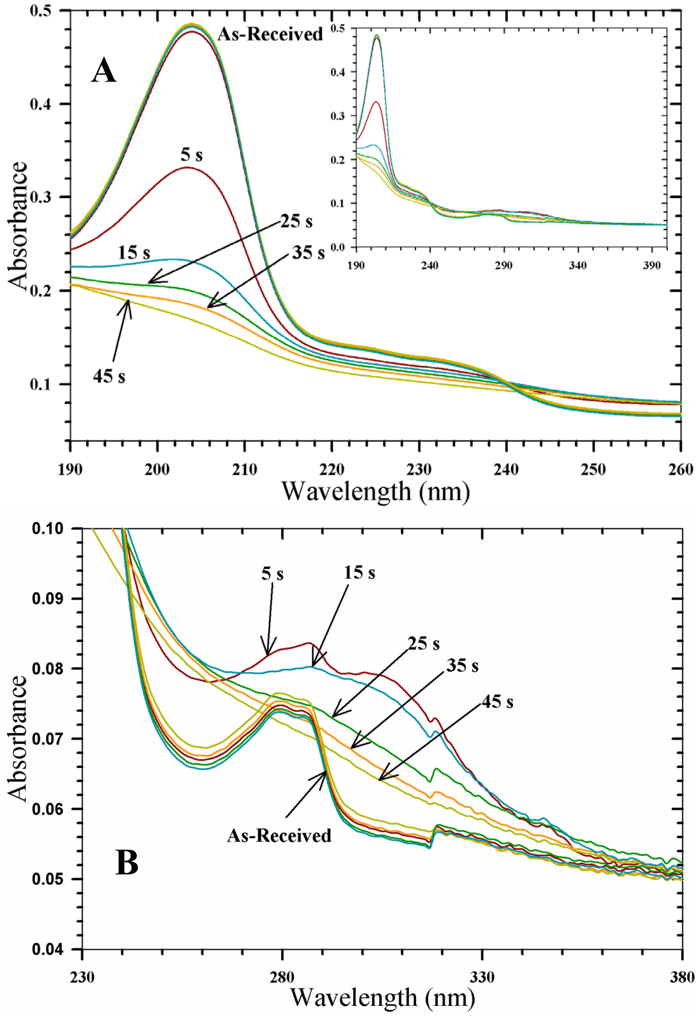


Figure 7. UV absorption spectra of BOPP film before and after UV exposure at various exposure levels. The spectral region is divided for clarity. (a) Wavelength from 190 to 260 nm (b) Wavelength from 230 to 380 nm. Inset shows the complete region.

Figure 7 illustrates the effect of UV irradiation on Irganox 1010 in BOPP. The decrease in absorbance at 204 nm suggests that the antioxidant disappeared gradually, but by-products were created, giving rise to the broad and structureless peak between 250 and 350 nm, which subsequently disappeared. The broad structureless peak has been observed previously in the extract of polypropylene pellets by ultrasonication in chloroform and also in polypropylene that had undergone gamma ray or pulse electron irradiation [6]. The transformation products of Irganox 1010 and other similar sterically-hindered phenolic antioxidants have been identified by a number of investigators [7-11] as 2,6-di-tert-butyl-p-benzoquinone (2,6-DTB-PBQ) and stilbenequinone which are olefinic ketones that usually display absorption around 220-250 nm and 290-330 nm [4]. Figure 8 shows the generalized radical-scavenging mechanism for typical hindered-phenol type of antioxidants [12].

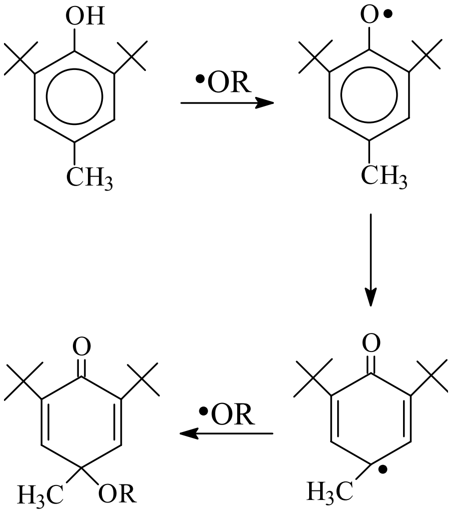


Figure 8. A generalized radical-scavenging mechanism for hindered phenols.

Figure 7 indicates that most of the Irganox 1010 was converted to 2,6-DTB-PBQ after 15 s of UV irradiation, while Figures 3 and 4 are consistent in indicating a substantial increase in the breakdown strength after 15 s of UV irradiation at both 63% and 5% breakdown probability. With further UV irradiation, Figure 7 indicates that the 2,6-DTB-PBQ degrades or is lost. With the IR filter, this loss will be caused by UV degradation, while without the IR filter loss will occur both through UV degradation and the vapor pressure of the 2,6-DTB-PBQ which becomes significant at about 80 °C. The degradation of the 2,6-DTB-PBQ is accompanied by a reduction in the breakdown strength, although with the IR filter, the Weibull characteristic breakdown strength does not degrade significantly from 15 s to 45 s exposure for unknown reasons.

4 DENSITY FUNCTIONAL THEORY

4.1 INTRODUCTION

To understand why the breakdown strength was improved, the effect of electric field on Irganox 1010 and 2,6-DTB-PBQ was studied. An electric field can cause dissociation and ionization of molecules, which results in formation of free electrons and ions that facilitate breakdown by increasing the conduction current [13]. In this preliminary work, electron affinities and ionization energies of the simplified Irganox 1010, 2,6-DTB-PBQ, and a 12-C polymer chain were computed as a function of electric field in vacuum by means of quantum mechanical computations based on density-function theory (DFT) [14]. Such DFT computations have been performed earlier by Quirke *et al* [15-17] to study the class of physical and chemical defects (traps) intrinsic to XLPE. However, traps arising due to additives, such as Irganox, to XLPE have not been considered earlier. Our goal was to obtain qualitative trends and insights into the effect of the two molecules representing additives on the breakdown strength of BOPP. DFT has been used to study dissociation of molecules in electric fields [18] and to calculate electron affinity of p-benzoquinone [19], but to our knowledge, the effect of electric fields on the two molecules under study has not been explored.

DFT is known to predict structure with a high level of accuracy (within 1% of experiments), but has the well known deficiency of underestimating bandgaps in semiconductors and HOMO-LUMO gaps in molecules by up to 50%. Nevertheless, *relative* energies and energetic trends, such as electron affinities [16,17] and *changes* in electron affinities, are well captured by DFT.

4.2 METHOD AND MODELS

The local density approximation within DFT as implemented in the local orbital SIESTA code [20] was employed for all calculations. The code was first tested on two smaller molecules, p-benzoquinone (PBQ) and 2,6-dimethy-p-benzoquinone (2,6-DM-PBQ). The calculated electron affinities for the two molecules are 1.93 and 1.82 eV, respectively, which is in good agreement with the experimental values (1.91 eV for PBQ and 1.77 eV for 2,6-DM-PBQ) [19]. To our knowledge an experimental electron affinity for 2,6-DTB-PBQ is not available. The bond lengths and angles for PBQ are shown in Table 3. The system used for all calculations was the molecule of interest in vacuum environment. A $C_{12}H_{26}$ hydrocarbon chain was used to model the polymer, and for Irganox 1010, only one of the four identical branches was modeled. Figure 9 shows the chemical structures of 2,6-DTB-PBQ and the model compound for Irganox 1010. The equilibrium atomic positions of each neutral molecule were first determined in the absence of external electric field by constraining the forces on each atom to be less than 0.04 eV/Å. A ±1 electron charge was then added to the optimized structure, and the equilibrium atomic positions of the two charged states were determined, after which electric field up to 3000 V/μm were applied to all three cases. The ionization energy and electron affinity were calculated as energy differences of the +1 charge state and of the -1 charge state relative to the ground state.

Table 3. Comparison of Bond Lengths and Bond Angles of p-Benzoquinone Calculated by DFT and Experimental Values [21].

	Calculated	Experimental
C=O (Å)	1.234	1.225
C=C (Å)	1.356	1.344
C-C (Å)	1.480	1.481
C-H (Å)	1.114	1.089
ŚO=C-C	120.9	121.0
ŚC=C-C	120.9	121.0
ŚC-C-C	118.2	118.1

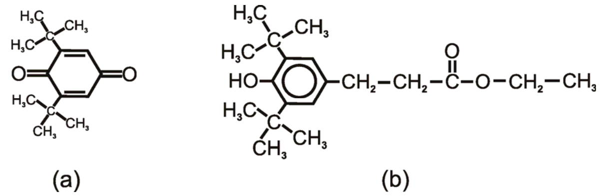


Figure 9. Chemical structures of (a) 2,6-di-tert-butyl-1,4-benzoquinone (b) the model compound of Irganox 1010.

4.3 RESULTS AND DISCUSSION

4.3.1 IRGANOX 1010

The first ionization energy and electron affinity of Irganox 1010 is shown in Figure 10. The relative stability of the molecule in various charge states was determined for several values of the external electric field in the direction which results in stretching of the OH group, as shown in Figure 10. Our results indicate that Irganox 1010 is unlikely to ionize or accept an electron in the range of electric fields studied since the ground state has the lowest energy. The tendency of the molecule to dissociate due to an electric field was studied by monitoring the bond lengths as a function of fields. The OH bond in Irganox 1010 is of special interest because the H atom is more weakly bound than the others due to the high electronegativity of the O atom. Figure 11 shows the bond lengths of the OH bond as a function of fields. The results shown in Figure 10 and 11 indicate negligible change in the energies or bond length due to electric field, indicating that the molecule is relatively stable with respect to electric fields.

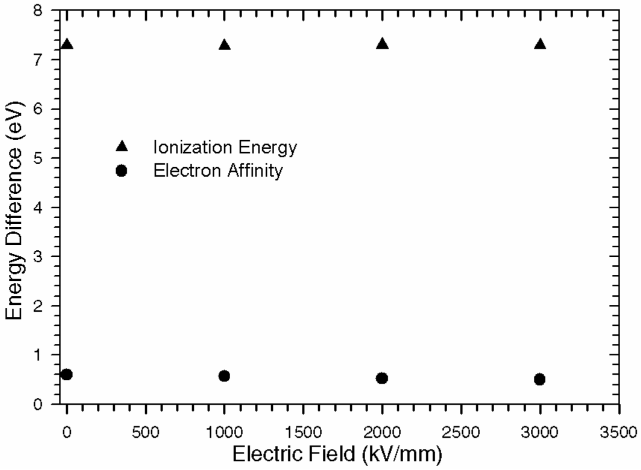


Figure 10. The first ionization energy and electron affinity of the model compound of Irganox 1010.

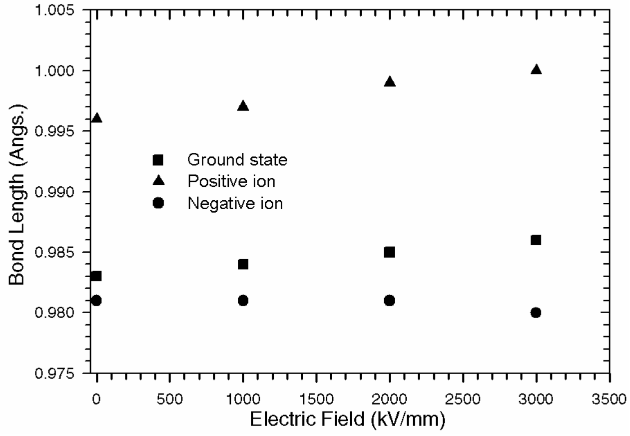


Figure 11. Bond length of the OH group in the model compound of Irganox 1010.

4.3.2 2,6-DI-TERT-BUTYL-p-BENZOQUINONE (2,6-DTB-PBQ)

Figure 12 shows the first ionization energy and electron affinity of 2,6-DTB-PBQ. The result suggests that while ionization is not likely, the molecule tends to accept an electron and that it is stable when subjected to electric field. As a comparison, the energies of a 12-carbon polymer chain were also determined and are illustrated in Figure 13.

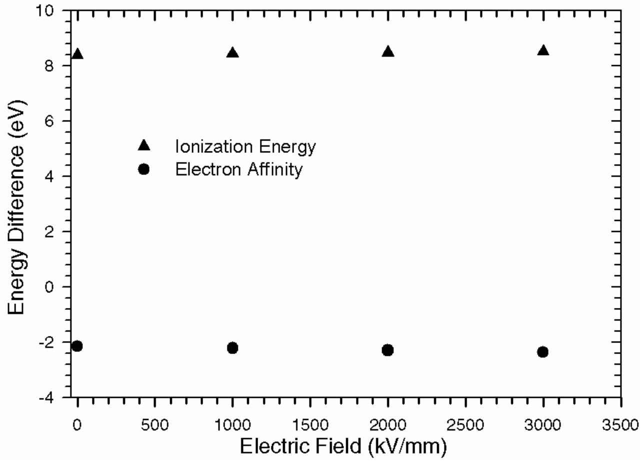


Figure 12. The first ionization energy and electron affinity of 2,6-di-tert-butyl-1,4-benzoquinone.

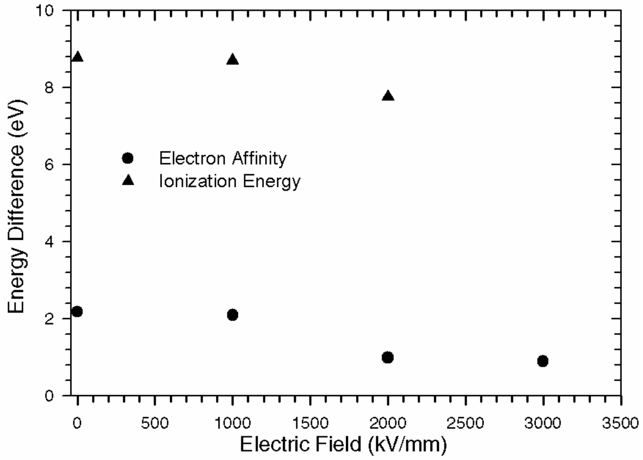


Figure 13. The first ionization energy and electron affinity of a 12-carbon polymer chain.

p-benzoquinones are well known as electron acceptors [19, 22-24]. The electron affinity of 2,6-DTB-PBQ shown in Figure 12 is about 2 eV, as compared to 1.05 for SF_6, which is an important gaseous dielectric and electron scavenger [25]. As breakdown in dielectrics is initiated by free electrons colliding with trapped or bound electrons leading to avalanches [26], abstracting the free electrons by electron scavengers, thus inhibiting or delaying the on-set of avalanches, should improve the breakdown strength. In conclusion, the results shown in Figure 10, 11, and 12 suggest that Irganox 1010 might not be damaging to the breakdown strength, but its altered product, 2,6-DTB-PBQ, appears to be beneficial as a result of its highly electronegative nature.

5 CONCLUSIONS

With a UV exposure level of about 3.4 J/cm², the dielectric breakdown strength of metallized BOPP capacitor film at 63% probability and at the 5% probability of breakdown was increased by about 7% and 13%, respectively without an IR filter and by 12% and 8% with an IR filter. UV analysis of the additives in BOPP revealed that the antioxidant, Irganox 1010, was consumed almost completely by the UV exposure, and altered products, such as 2,6-di-tert-butyl-1,4-benzoquinone, were created. As the benzoquinone has a relatively high vapor pressure above 80 °C, introduction of the IR filter probably reduces volatilization during exposure.

DFT computations suggest that all molecules studied are stable with respect to the electric fields imposed, and that 2,6-di-tert-butyl-1,4-benzoquinone is highly electronegative . The improvement in dielectric strength of BOPP caused by UV treatment appears to be the result of conversion of the Irganox 1010, which is probably relatively benign, to 2,6-di-tert-butyl-1,4-benzoquinone, which is electronegative as indicated by the DFT results and beneficial to the dielectric strength of the BOPP. As the melting point of 2,6-di-tert-butyl-1,4-benzoquinone is well below 100 °C, impregnation of BOPP with 2,6-di-tert-butyl-1,4-benzoquinone may be studied for improvement of the dielectric strength. DFT calculations should also be performed on a polymer-Irganox 1010 system and a polymer-2,6-BTBBQ system to compare the results with the individual systems.

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Janet Ho was graduated with a B.S. in chemical engineering from the University of California, Berkeley in 1995. She spent five years working in the field of polymeric opthalmic lenses. In 2000, she joined the Polymer Science Program at the University of Connecticut in pursuit of a doctorate, which she completed in 2006. She is presently with the Army Research Laboratory in Adelphi, MD working in the area of capacitor technology.

Rampi Ramprasad was graduated from the Indian Institute of Technology, Madras, India, in 1990 with a B.Tech. in metallurgical engineering, and from the University of Illinois, Urbana-Champaign, in 1997 with a Ph.D. in materials science and engineering. After a six-years at Motorola, Inc., (Semiconductor Product Sector), where he worked on problems related to field emission for flat panel display applications, advanced dielectric and magnetic materials, and metamaterials for RF and microwave applications, he joined the Department of Materials Science and Engineering at the University of Connecticut as a faculty member. Dr. Ramprasad is the author of about 30 peer-reviewed journal papers, numerous conference papers, and one patent (and several pending).

Steven A. Boggs (F '92) was graduated with a B.A. from Reed College in 1968 and received his Ph.D. and MBA degrees from the University of Toronto in 1972 and 1987, respectively. He spent 12 years with the Research Division of Ontario Hydro. He was elected an IEEE Fellow for his contributions to understanding of SF_6-insulated systems. From 1987 to 1993, he was Director of Research and Engineering at Underground Systems, Inc. where he was responsible for the USi subsidiary Chicago Condenser Corporation. He is presently Director of the Electrical Insulation Research Center and Research Professor of Materials Science, Electrical Engineering, and Physics at the University of Connecticut as well as an Adjunct Professor of Electrical and Computer Engineering at the University of Toronto.