

# **CABLE AGING AND CONDITION MONITORING OF RADIATION RESISTANT NANO-DIELECTRICS IN ADVANCED REACTOR APPLICATIONS**

**Robert C Duckworth\*, M. Parans Paranthaman, Tolga Aytug, Michelle K. Kidder, Georgios Polizos, and Keith J. Leonard**  
Oak Ridge National Laboratory  
PO Box 2008, One Bethel Valley Road, Oak Ridge, TN 37831

## **ABSTRACT**

Cross-linked polyethylene (XLPE) nanocomposites have been developed in an effort to improve cable insulation lifetime to serve in both instrument cables and auxiliary power systems in advanced reactor applications as well as to provide an alternative for new or retro-fit cable insulation installations. Nano-dielectrics composed of different weight percentages of MgO & SiO<sub>2</sub> have been subjected to radiation at accumulated doses approaching 20 MRad and thermal aging temperatures exceeding 100°C. Depending on the composition, the performance of the nanodielectric insulation was influenced, both positively and negatively, when quantified with respect to its electrical and mechanical properties. For virgin (unradiated) or thermally aged-samples, XLPE nanocomposites with 1wt.% SiO<sub>2</sub> showed improvement in breakdown strength and reduction in dissipation factor when compared to pure (undoped) XLPE, while XLPE 3wt.% SiO<sub>2</sub> resulted in lower breakdown strength. When aged in air at 120°C, retention of electrical breakdown strength and dissipation factor was observed for XLPE 3wt.% MgO nanocomposites. Irrespective of the nanoparticle species, XLPE nanocomposites that were gamma irradiated up to the accumulated dose of 18 MRad showed a significant drop in breakdown strength especially for particle concentrations greater than 3 wt.%. Additional Fourier transform infrared (FTIR) spectroscopy measurements suggest changes in the structure of the XLPE SiO<sub>2</sub> nanocomposites associated with the interaction of silicon and oxygen. Discussion on the relevance of property changes with respect to cable aging and condition monitoring is presented.

*Key words:* cable aging, cable insulation, nanocomposites, cross-linked polyethylene.

## **1 INTRODUCTION**

The quality and performance of cable insulation can impact the ability of operators to control instruments and activate safety controls and auxiliary power systems during daily operations and off-normal emergency events over the lifetime of a nuclear power plant (NPP). Cable insulations in nuclear reactors are deployed in a unique environment compared to other applications in that these materials must remain mechanically and electrically sound over a 40-year or greater lifetime as they are exposed to the deleterious combined effects of radiation, mechanical stresses, temperatures exceeding 100°C, and humid environments. While cable insulations such as cross-linked polyethylene (XLPE), ethylene propylene

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rubber (EPR), polyvinyl chloride (PVC), neoprene, and chlorosulfonated polyethylene have shown suitable radiation tolerance in laboratory tests [1-2] and met the requirements for cable insulations in current nuclear environments [3-4], a number of cable failures have been observed within a 20-30 year time frame under normal service conditions [5]. Investment in cable aging management programs by DOE, EPRI, and the NRC has furthered understanding of cable aging performance in materials currently deployed in NPPs as evidenced by NUREG/CR-7153 [6]. However, these insulations may not be able to completely meet the future and/or next generation nuclear reactor requirements that will operate for longer time periods (60-80 years) under higher thermal and radiation operating loads or exposure to abnormal conditions [7-8].

Of the cable insulations, XLPE is used in nearly 36% of cable installations in current NPPs [9-10]. The benefits from XLPE originate from good dielectric properties along with improved mechanical and thermal behavior [11-13]. The cross-linking that is achieved through chemical or radiation processing [11, 13] and/or antioxidant addition [14-16] prevents the formation of radicals within the XLPE once the oxidation occurs during radiation, thermal, and/or electrical aging. While antioxidants function with cross-linking to prevent degradation due to oxidation under radiation and/or thermal exposures, a lack of consistent improvement in the electrical and/or the mechanical performance has been observed and is dependent on the aging conditions, the formulation of cross-linking, and antioxidant chemistry [14-16]. While XLPE has shown suitable radiation tolerance in laboratory tests, failures before their useful lifetime have occurred in the field due to the combined environmental effects of radiation, temperature and moisture, as well as abnormal operating conditions [17-18].

Improvements in mechanical and electrical properties have been realized from the inclusion of nanoparticles in low-density non-cross linked polyethylene [19-20]. However, the influence of radiation on these nanocomposites [21] resulted in mixed changes in the dielectric properties of the materials. Therefore, a systematic study assessing the radiation and thermal aging tolerances of these nanocomposites is needed in order to determine whether nanocomposite polyethylene and/or other nanocomposite dielectrics have merit in future nuclear reactors. This paper reports on the recent efforts to develop XLPE-based nanocomposite dielectrics for nuclear reactor applications and their performance when aged by radiation and temperature.

## **2 XLPE NANOCOMPOSITE DIELECTRICS FORMULATION**

While a detailed description of XLPE-based nanocomposite dielectric processing is presented elsewhere [22], a summary is given here for reference purposes. Two pathways were developed for the fabrication of these nano-composite materials. The first pathway employed an in-situ method where the nano-particles are formed within the XLPE, which is intended to allow for uniform dispersal of particles through a commercially scalable method with minimal modification to standard XLPE processing conditions. The second pathway utilized an ex-situ method, similar to those reported in Refs. 19-21, except that cross-linking chemical agents are added to the polyethylene during twin-screw driven mixing with a known concentration of the nanoparticles to produce the final XLPE composite.

For the incorporation of SiO<sub>2</sub> nanoparticles using the in-situ method, a silicic acid-based precursor solution was mixed with high-density polyethylene (HDPE), 1,2,4-Trichlorobenzene (TCB), ~ 4.8% by weight vinyltriethoxysilane (VTES) and 1.7% by weight Tert-butyl peroxide (TBP) in a beaker. VTES was utilized as a surface modifier for the SiO<sub>2</sub> and the TBP served to cross-link the polyethylene and initiate chain formation. The beaker was then placed in a hot oil bath and magnetically stirred until the bath temperature reached 125°C. After additional TCB (20% of the original amount) was added to the solution, heating continued until a temperature of 130-135°C was reached. The solution was then removed and cast into a Teflon® evaporating dish, which was left in a heated oil bath in a laboratory hood up to 6 h to evaporate the solvent.

A faster, ex-situ fabrication was developed in order to allow for different nanoparticles to be examined given the time consuming nature of the in-situ process for new nanoparticle formulations. This enabled performance optimization while minimizing processing time. For ex-situ processed XLPE-based

SiO<sub>2</sub> and MgO nano-composite dielectrics, either fumed silica or fumed magnesia along with desired amounts of HDPE, VTES and TBP, were fed into a DSM Xplore 5 cm<sup>3</sup> twin-screw Micro-Compounder and mixed at 200<sup>0</sup>C for 10 min before being extruded as filaments. The filaments were then placed between two 6" x 6" Teflon® sheets. These sheets were sandwiched between two metal plates and then placed in a Carver Auto Hot Press with the temperature set at 365<sup>0</sup>F (185<sup>0</sup>C), force of 11,000 lbs. (48.9 KN), and dwell time of 5 min. The Teflon® sheets were then removed from the press and set aside to cool for 30 min. Finally, the Teflon® sheets were pulled apart to remove the XLPE/SiO<sub>2</sub> or XLPE/MgO films.

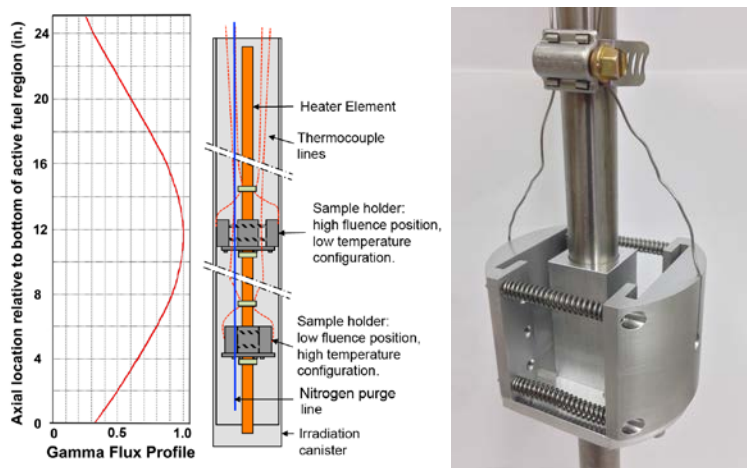
FTIR analysis was conducted on a Digilab FTS 7000 equipped with DTGS detector and a PIKE MIRacle ATR accessory with a diamond crystal. Sixty-four scans were taken at a resolution of 4 cm<sup>-1</sup>.

### 3 EXPOSURE OF XLPE NANOCOMPOSITES

#### 3.1 Irradiation of XLPE Nanocomposites

Irradiation of the nano-composite dielectrics was done in the Gamma Irradiation Facility (GIF) at the High Flux Isotope Reactor (HFIR) at ORNL. The GIF, located in the reactor bay pool, allows the selection of spent annular fuel assemblies from HFIR to be used for the source of the gamma rays. Depending on when the assembly was removed from HFIR, different gamma flux levels can be achieved (0.03 Mrad/h to 10 Mrad/h (30 Gy/h to 100 kGy/h). With respect to the measurements of the samples that are presented in this paper, the selected fuel assembly produced an average dose rate of 0.56 MRad/hr. (5.6 kGy/hr.) for the time in which the measurements took place. The samples are placed in a specialized variable position holder that is inserted into a steel irradiation canister and lowered into position within the flux trap of the spent fuel core. A gamma flux profile along the longitudinal direction of the flux trap is present and was utilized in this work to allow for testing to different accumulated dose levels within the same exposure run.

An example of the sample holder configured for the study of the nanocomposite polymers is provided in Fig. 1. The aluminum holders allow the polymer films to be held in the vertical position. The nanocomposite films are separated by 0.05 mm thick high purity aluminum foil with the stack of films



**Figure 1. Longitudinal cross-section view of gamma profile in the HFIR GIF (left) with a schematic of the sample fixture utilized and (right) an image of the sample holder without polymer films loaded into the vertical slots on each side of the holder.**

further supported in the fixture by a thicker high purity aluminum backing plate for support. The sample holders can either be set in the extended position or retracted back against a thermal block connected to a rod heater. In the extended position, steel springs press the holders against the inside radial wall of the

steel irradiation canister. In this configuration, maximum sample cooling is achieved with temperatures averaging around 38°C, which is roughly the outside water temperature in the spent fuel core (which can either increase or decrease pending on the age of the assembly). In the retracted configuration, the samples are connected to the internal rod heater. However, for the work presented in this paper, the samples holders were placed in the low temperature configuration, with only a surrogate steel tube used for the support column instead of a rod heater. The holders are machined to allow thermocouple placement next to the outermost (relative to the radial direction) sample. The alloy 600 sheathed type K thermocouples lines, heater supply and gas purge inlet and outlet lines exit the irradiation canister via umbilical connection through the spent fuel pool into the HFIR instrument room where temperatures, gas pressure and flow are monitored. For these tests argon sweep gas was used. Radiographic dosimetry films were placed next to the nanocomposite films to compliment calculated dose rates, with the dosimetry results used in the reporting of the data.

### 3.2 Thermal Aging of XLPE nano-composites

In addition to irradiation, XLPE nano-composites were also exposed to aging in air at elevated temperatures in the industrial furnace shown in Fig. 2. Twenty-seven samples of pure XLPE and XLPE nanocomposites (3 wt.% SiO<sub>2</sub> and 3 wt.% MgO) were arranged within the furnace to allow for exposure of each film in air at 120°C. Samples were removed over a five week period with nine samples removed after one week, another nine samples after three weeks, and the remaining nine samples removed after five weeks. With three samples of each type, pure, 3 wt.% SiO<sub>2</sub> and 3 wt.% MgO - electrical, mechanical, and chemical characterizations were carried out in order to understand the impact of the nanoparticles on performance. While the samples that were removed at one week and three week intervals did not show any change in appearance that would suggest degradation, a dramatic change in appearance was observed for the five week aging period, as shown in Fig. 3.



Figure 2. Furnace used to thermal age XLPE nanocomposites at a temperature of 120°C.

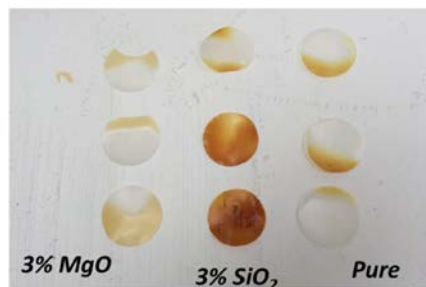


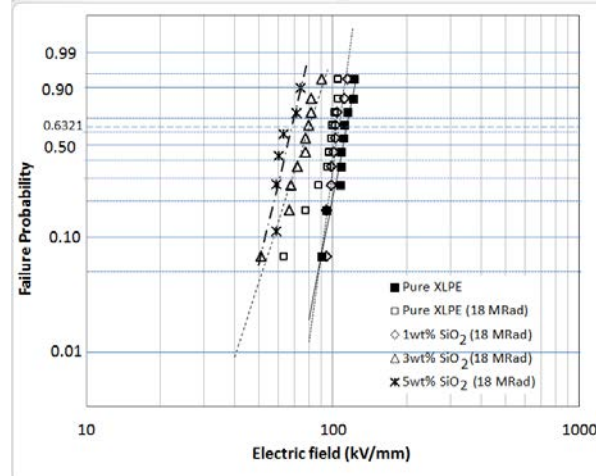
Figure 3. XLPE nanocomposites with different nanoparticle compositions after five weeks of exposure to air at 120°C.

## 4 RESULTS AND ANALYSIS

### 4.1 Breakdown Voltages of Irradiated XLPE Nanocomposites

The electrical performance as a function of electrical breakdown of the XLPE nano-composites that were irradiated to a total accumulated dose of 18 MRad is shown in Fig. 4 [22]. The electrical performance is quantified with respect to the AC electrical breakdown of the films when a 60 Hz high voltage is applied at a rate of 500 V/s in a LD60 breakdown tester. Given the small sample size of breakdown voltages for each film, Weibull analysis [23] was carried out to determine the performance,  $E_0$ , and uniformity,  $\beta$ , of each film. These values are shown in Table I.

The addition of 1 wt. %  $\text{SiO}_2$  to XLPE showed improvement in high voltage breakdown when compared to the pure XLPE after exposure to 18 Mrad. However, there is a systematic decrease in performance with further increase in  $\text{SiO}_2$  concentration. The decrease in performance appears to accelerate with increasing the accumulated dose. Given that the irradiation was done in argon and not in air, the observed degradation due to irradiation could have resulted from polymer chain interaction with the nanoparticles within the film instead of chemical interaction of the broken polymer chains with



**Figure 4. Breakdown strength as measured with respect to electric field with respect to failure probability for XLPE nanocomposites with different concentrations of  $\text{SiO}_2$  nanoparticles after gamma exposure in argon atmosphere at  $38^\circ\text{C}$  at an accumulated dose of 18 Mrad.**

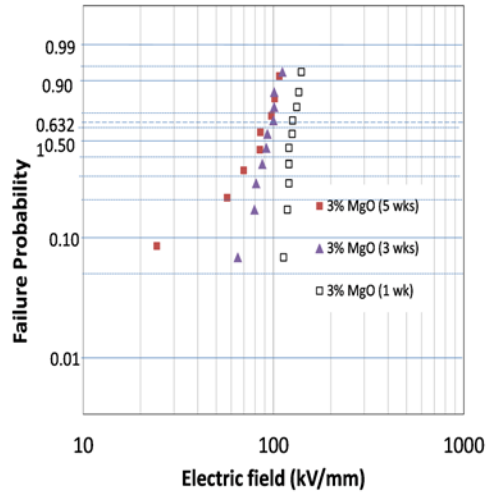
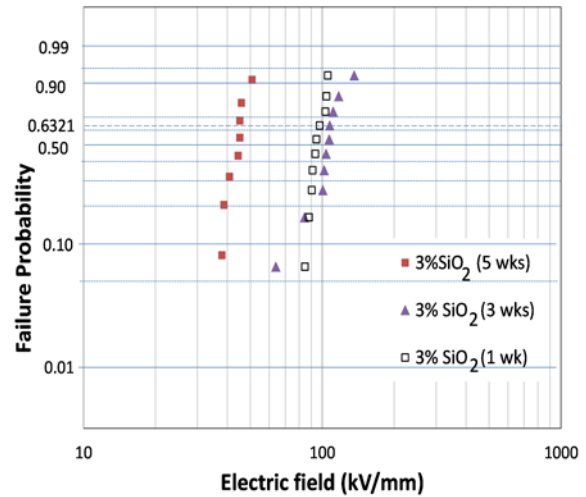
oxygen. Given that the XLPE nano-composites with 3wt.% and 5wt.%  $\text{SiO}_2$  did show measureable change in performance at 18 Mrad, irradiation could have resulted in a negative interaction between polymer and particles, or is indicative of nanoparticle agglomeration in the film reducing their effectiveness in limiting damage to the polymer. As the non-irradiated  $E_o$  values are slightly lower in the 3 and 5 wt.% loaded film, the increased agglomeration of the particles may limit the uniformity in distribution of the particles through the polymer film matrix, making them less efficient in neutralizing free radicals or broken polymer chains. Additional chemical and mechanical characterizations of XLPE MgO-based nanocomposites and irradiation experiments in air of both XLPE MgO and  $\text{SiO}_2$ -based nanocomposites are underway to better understand the nature of degradation observed in these first set of samples.

## 4.2 Breakdown Voltages of Thermal-Aged XLPE Nanocomposites

The electrical performance with respect to electrical breakdown of the thermally aged XLPE nanocomposites, in air at a temperature of  $120^\circ\text{C}$ , is shown in Figs. 5 & 6. From these figures and from Weibull distribution analysis given in Table II for the different XLPE nanocomposites, there is a significant change in electrical properties that is consistent with the changes in appearance observed in Fig. 3. It is worthy to note that the dissipation factor,  $\tan \delta$ , significantly increased for the pure XLPE and the XLPE 3wt.%  $\text{SiO}_2$  nanocomposites, but there was little change for the XLPE with 3wt.% MgO despite the physical change in the film's appearance. The AC breakdown performance of the MgO also did not degrade like the other films. This would suggest that the change in the XLPE MgO film was localized and not as uniform as the other materials.

**Table I. Weibull scale and shape parameters for irradiated XLPE SiO<sub>2</sub> nanocomposites.**

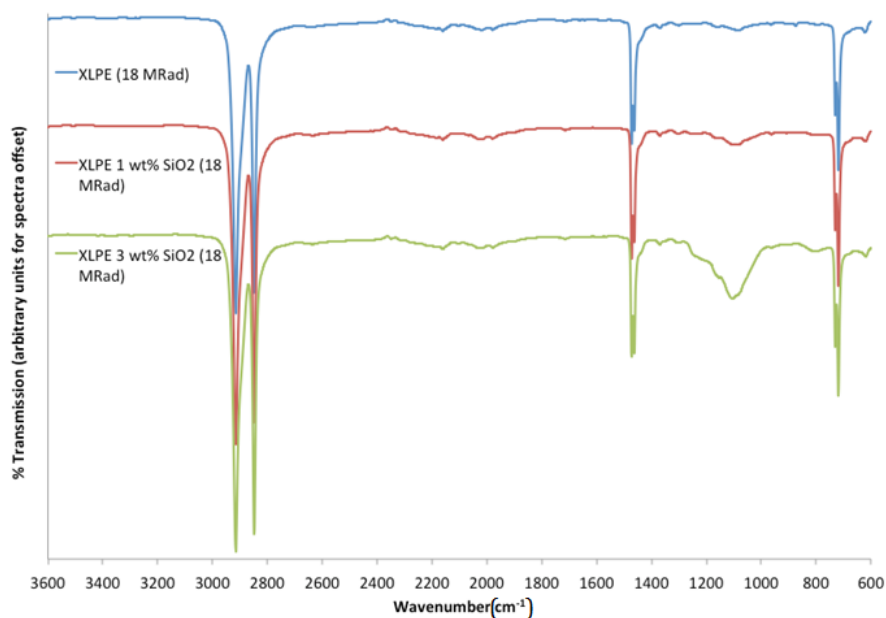
Composition	Accumulated Dose [MRad]	Weibull Parameter $\alpha$ [kV/mm]	Weibull Parameter $\beta$ [-]
Pure	0 MRad	113.6	11.26
	10 MRad	97.6	15.42
	18 MRad	99.8	6.63
1 wt.% SiO <sub>2</sub>	0 MRad	146.0	6.35
	10 MRad	96.2	7.37
	18 MRad	105.6	15.90
3 wt.% SiO <sub>2</sub>	0 MRad	100.0	6.82
	10 MRad	101.9	10.37
	18 MRad	79.9	6.83
5 wt.% SiO <sub>2</sub>	0 MRad	n/a	n/a
	10 MRad	90.2	22.63
	18 MRad	68.0	9.20

**Figure 5. AC dielectric strength with respect to electric field of 3 wt.% MgO incorporated XLPE composites after different amounts of thermal aging at 120°C.****Figure 6. AC dielectric strength with respect to electric field of 3 wt.% SiO<sub>2</sub> incorporated XLPE composites after different amounts of thermal aging at 120°C.****Table II. Comparison of Weibull parameters and dissipation factor ( $\tan \delta$ ) for XLPE nanocomposites that have been thermally aged at 120°C for different periods of time at a relative humidity between 45% and 55%.**

Composition	Time [weeks]	Weibull Parameter $\alpha$ [kV/mm]	Weibull parameter B [-]	$\tan \delta$ @ 1 kHz
Pure	1	126.4	7.82	0.0003
	3	92.6	12.86	0.0002
	5	55.0	5.64	0.0193
3 wt.% SiO <sub>2</sub>	1	98.5	14.40	0.0004
	3	112.2	5.26	0.0003
	5	45.6	10.76	0.0338
3 wt.% MgO	1	129.3	15.71	0.0003
	3	97.0	7.08	0.0014
	5	92.3	2.10	0.0003

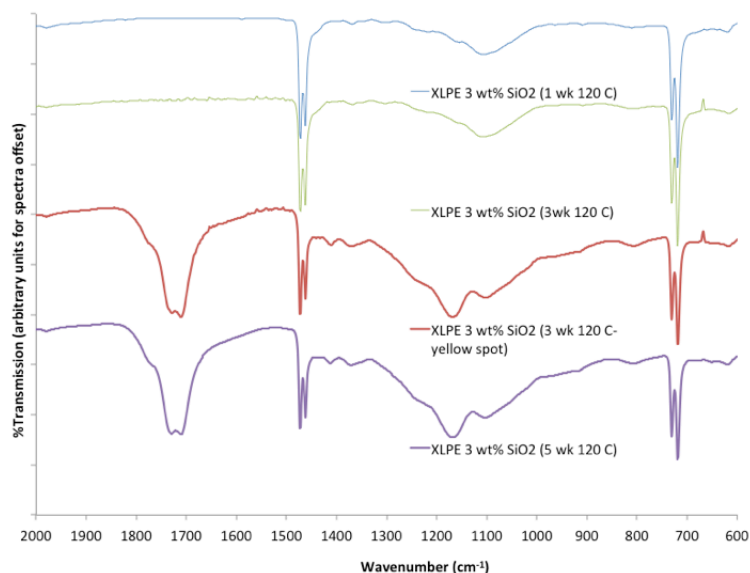
### 4.3 FTIR Spectroscopy of XLPE nanocomposites

Fourier transform infrared spectroscopy (ATR-FTIR) was carried out on a section of each film to attempt to quantify the nature of the chemical change in the XLPE nanocomposites. Figs. 7 and 8 show the FTIR spectrum for irradiated XLPE SiO<sub>2</sub> nanocomposites and thermally aged XLPE 3wt.%SiO<sub>2</sub> nanocomposites respectively. Given that the irradiation of XLPE SiO<sub>2</sub> nanocomposites was conducted under an argon environment, the increase in the broad peak intensity observed at 1105 cm<sup>-1</sup> likely corresponds to the increase in SiO<sub>2</sub> concentration in the films and is not necessarily associated with the damage from the irradiation itself. From Fig. 8, it is clear that once the XLPE 3wt.% SiO<sub>2</sub> nanocomposite is aged for five weeks at 120°C, a new intense peak at 1715 cm<sup>-1</sup> and 1728 cm<sup>-1</sup> (C=O) grows due to oxidation as well as an increase in intensity between 1400-1363 cm<sup>-1</sup> attributed to many Si-O-Si vibrational modes. Specifically the peak at 1168 cm<sup>-1</sup> has significant growth which may indicate vibrational modes of Si-O-C and/or Si-C<sub>2</sub>H<sub>5</sub> are enhanced. It was also noted that samples aged at one and three weeks did not exhibit significant visible color compared to the 5 week samples, which turned completely yellow. However, the edge of the three week sample had some discoloration. The IR of this discoloration spot compares to the 5 week sample (Fig. 8 denoted as “yellow spot”) showing that oxidation starting at the edges is probable. These changes seen in the aged samples Fig. 8 suggest possible changes in the bonds of the Si and oxygen that require further investigation. The spectrum for both the 3 week aged and 18 Mrad exposure samples are very similar for the 3 wt.% SiO<sub>2</sub> XLPE. Measurement of the cross-linking concentration and oxidation time for each sample might reveal the nature of the SiO<sub>2</sub> interactions. Further FTIR measurements are planned to refine understanding of these XLPE systems.



**Figure 7. ATR FTIR spectra for XLPE nanocomposites with different concentrations of SiO<sub>2</sub> that have experienced gamma radiation for accumulated dose of 18 MRad.**





**Figure 8. ATR FTIR spectra for XLPE 3wt.% SiO<sub>2</sub> nanocomposites that have thermally aged at 120°C.**

## **5 CONCLUSIONS AND IMPLICATIONS FOR CABLE AGING AND CONDITION MONITORING**

The XLPE nanocomposites with the addition of SiO<sub>2</sub> and MgO have been successfully developed and their electrical performance after irradiation and thermal aging has been studied. For un-irradiated samples, XLPE 1wt.% SiO<sub>2</sub> nanocomposites showed improvement in breakdown strength and reduction in dissipation factor when compared to pure XLPE and XLPE 3wt.% SiO<sub>2</sub> nanocomposites. When XLPE nanocomposites were irradiated to 18 MRad, the differences between irradiated and non-irradiated XLPE nanocomposites became larger with respect to breakdown strength, especially with concentrations greater than 3 wt.%. ATR-FTIR measurements did show changes in the IR spectrum with increasing concentration of SiO<sub>2</sub> particles and increasing duration of thermal aging. However, additional testing is needed to optimize the nanoparticle concentration for a given temperature and radiation dose.

With respect to cable aging, XLPE nanocomposites could be advantageous when compared to pure XLPE if the electrical responses of the samples are considered. Nondestructive cable monitoring methods such as line impedance resonance analyzers and time or frequency domain reflectometry methods rely upon changes in impedance in order to detect defects or areas of degradation along a cable for lengths greater than 10 m. If one of these methods is applied to a XLPE nanocomposite that has a different impedance performance like that shown in Table II in the dissipation factor,  $\tan \delta$ , it is conceivable that defects or degradation could be detected. Additional modeling and measurements of the impedance are needed to verify this potential benefit. Future work on these materials across a broader spectrum of temperatures, air exposures, and radiation conditions would help identify key aging mechanisms with XLPE nanocomposite formulations that would, in general, benefit implementation of cable insulation in a variety of applications.

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