Improvement of dielectric strength and properties of cross-linked polyethylene using nano filler

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ABSTRACT
Power cables insulated with cross-linked polyethylene (XLPE) have been utilized worldwide for distribution and transmission networks. There are several advantages for this type of insulation; it has better electrical, thermal, and mechanical properties compared to other types of insulation in medium and high voltage networks. Many studies aimed to improve the XLPE characteristics through introducing nano fillers to the XLPE matrix. Therefore, this paper investigates the AC (HV) breakdown voltage (dielectric strength) of XLPE after adding nano-sized zeolite (Z) fillers with various concentrations of 1 wt%, 3 wt%, 5 wt% and 7 wt%. The dielectric strength is tested in different temperatures of 30°C and 250°C. Additionally, it was tested in low and high salty wet conditions. The dielectric strength of the XLPE has been enhanced by inducing the Z nano filler. The results of the tests were used to train the artificial neural network (ANN) to calculate the dielectric strength of XLPE composites with different concentrations of nano Z filler under different weathering conditions. Thermogravimetric analysis, tensile strength, and elongation at break tests were applied to check the thermal and mechanical characteristics of the samples. Experimental findings show that the optimum concentration of nano Z is 3.64 wt% to enhance the electrical, thermal, and mechanical properties.

Keywords:
Cross-linked polyethylene
Dielectric strength
Nanocomposites
Tensile strength
Thermogravimetric analysis

1. INTRODUCTION
Polymer composites and blends in dielectric applications have recently attracted researchers’ interest [1]–[3]. The cross-linked polyethylene (XLPE) power cables must be used with a high degree of reliability and safety over a very long period and in different environmental conditions. This insulation has very good electrical, thermal, and mechanical properties for medium and high voltage networks. Because of its various advantages, the XLPE insulated power cables have widely replaced the traditional classic paper insulated cables [4], [5].

Many studies have been carried out to improve the electrical characteristics of the XLPE represented by the dielectric strength, which is the maximum electric field intensity that can resist without encountering failure due to its insulating properties [6]–[13]. In those methods, nano-sized fillers were proposed to improve the XLPE insulation properties of the cables. Reddy and Ramu [14] explained the advantage of nano-particle fillers in improving insulating materials. Another example of nano-fillers injection is the SiO₂...
that is used by Wahab et al. in [7] to improve the dielectric strength. The filling injection to the XPLE was further extended to the micro scale when Essawi et al. [15] studied the effect of adding the micro fillers of CaCO$_3$ to the XLPE to improve the dielectric strength. Okuzumi et al. [16] compared the effect of adding nano-filler or micro-filler of magnesium oxide (MgO) on the breakdown strength of low-density polyethylene (LDPE). The drawn conclusion is that the nano filler was better than the micro-sized filler in improving the dielectric strength.

In addition to improving the electrical properties, several studies have been conducted to improve the thermal and mechanical properties of the XPLE. This is due to the fact that the XPLE cables are used in various environmental conditions and the insulating materials are supposed to endure all these conditions and still perform efficiently. For instance, the studies [17], [18] investigated the thermal and mechanical characteristics of XLPE when it is loaded with inorganic fillers. However, recent studies have shown that the characteristics of the XLPE in terms of mechanical, thermal, and electrical properties cannot be decoupled. For instance, Mecheri et al. [19] concluded that although the dielectric breakdown strength was improved, the mechanical properties were degraded. Hence, achieving a good balance between the three properties is challenging.

Thus, this research aims to investigate the dielectric strength of different samples of XLPE loaded with nano-sized zeolite filler in different weight percentage concentrations (wt%). It focuses on interpreting the optimum (wt%) of such a blend in order to enhance the dielectric strength. Also, tensile strength and elongation at break tests were carried out to check the mechanical properties of the samples. Finally, thermogravimetric analysis (TGA) for the samples was applied as well. The testing of the samples is done at different operating conditions, such as different temperatures, salty and wet conditions. Also, because of the shortage of empirical results due to the high cost of raw materials and equipment used in the experiments, as well as the difficulties encountered in the labs, a technique for constructing an artificial neural network was developed to be able to estimate the dielectric strength of composites with any filler concentration.

2. TESTING MECHANISM

In this section, the preparation of samples and testing procedures for the dielectric strength, thermal and mechanical properties are discussed in detail. Figure 1 shows the testing mechanism of the research paper. Firstly, different concentrations of nanofiller were used to prepare the XLPE/zeolite nanocomposite samples. Second, various tests such as dielectric strength, tensile strength, elongation at break, and TGA will be conducted in order to determine the optimal concentration of nano zeolite filler for improving electrical, mechanical, and thermal properties. Finally, artificial neural network (ANN) has been used to estimate the dielectric strength of XLPE/zeolite at different weather condition.

![Figure 1. Testing mechanism of the manuscript](image-url)
2.1. Samples preparation

The XLPE preparation was carried out in the National Research Centre, Polymers and Pigments Dept. in Cairo, Egypt. In order to prepare the XLPE according to the standard ASTM F876-10e1, high density polyethylene (HDPE) was mixed with dicumyl peroxide 4-methoxy phenol as cross-linked agent in without adding any filler. Table 1 presents the XLPE samples loaded with various nano Z wt%. The nano Z particle size is 100±50 nm. The concentrations targeted in this paper are 1 wt%, 3 wt%, 5 wt%, and 7 wt%. All of the formulations listed in Table 1 were mixed in an electric heat chamber of the Barbender Plasticoder (C.W. Pra, INC.50 Hackensack model) with voltage and current ratings of 220 V and 40 Amp, respectively to make XLPE composites with varied amounts of nano Z filler. Zeolite is a type of clay. The use of zeolite in this research is due to its unique physical and chemical properties (high sorption capacity and ion-exchange, ion-exchange selectivity, and structural thermal stability). Zeolite nano particles were prepared by ball milling machine [19]. The nano Z particle size is 100±50 nm. The prepared XLPE composites were in the form of a sheet with dimensions of 20×20 cm when the mixing was completed. According to ASTM, the specimens were cut to the dimensions that best matched each testing procedure.

<table>
<thead>
<tr>
<th>XPLE sample Symbol</th>
<th>Concentration of Nano Z (wt%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>B (XLPE without filler)</td>
<td>0</td>
</tr>
<tr>
<td>Z₁</td>
<td>1</td>
</tr>
<tr>
<td>Z₂</td>
<td>3</td>
</tr>
<tr>
<td>Z₄</td>
<td>5</td>
</tr>
<tr>
<td>Z₇</td>
<td>7</td>
</tr>
</tbody>
</table>

2.2. Dielectric strength test

The dielectric strength is the major test for the electrical properties of an insulating material and it can be expressed in voltage per unit length (kV/mm). It demonstrates how the insulation material XLPE can withstand the electric field intensity without reaching a state of failure to its insulating properties. The samples are in the shape of a disc with 1 mm thickness and a diameter equals to 5 cm.

It is worth mentioning that the voltage applied to the specimen should be varied smoothly and gradually in order to avoid surges on the HV side of the transformer and to determine the breakdown voltage of the specimen more accurately. Accordingly, a voltage regulating method is used on the LV side of the transformer for smoothly varying the voltage. The voltage regulating method used in this paper is an inductor voltage divider to avoid the distortion in the voltage waveform compared to the methods that depend on using power electronics devices. Another advantage for the inductor voltage divider is the low power loss compared to the resistive voltage dividers. Figure 2 presents the laboratory circuit utilized for the dielectric breakdown strength test.

The voltage in kV is applied gradually on the samples sets of XPLE mixed with nano Z in different concentrations that have been prepared. These samples were tested using high voltage (HV) AC at different conditions that will be explained later. In order to ensure high accuracy of the recorded results, the average value of five tested samples was calculated and recorded.

The dielectric strength has been studied under two different scenarios:

- The first scenario is based on testing the samples in 2 different temperatures. The first testing temperature is 30 °C so that it can simulate the operating conditions at normal ambient temperature. The second testing temperature is 250 °C, hence, it can simulate the operating conditions of the maximum short circuit for a cable operating above 30 kV. The temperature of the samples reached 250 °C by exposing them to this temperature in the laboratory oven. Also, the samples were submerged in oil to reduce the probability of occurrence of any flashover. For those two testing conditions, the composites should be dry and clean, i.e., removing dust and other particles on the surface of composites before starting the high voltage test. The voltage was gradually escalated at a constant rate of 2 kV/s until the voltage breakdown occurs. The test was completed according to ASTM-D149.

- In the second scenario, the samples are tested in salty wet conditions to simulate the effect of salty water condition on cable insulation. Two different salient solutions of sodium chloride and distilled water are prepared, with two different salinity amounts of 20000 μs/cm and 50000 μs/cm which are considered as low and high salinity conditions, respectively. The samples were immersed in these solutions for 24 hours, then, they were removed and cleaned. Finally, the samples were immersed in oil and tested according to the ASTM D570 standard. The smooth variation of voltage was carried out same as previous scenario.
2.3. Artificial neural network

An Artificial Neural Network is used to determine the dielectric strength level of XLPE/Z composites with any nano Z filler concentration and deployed under various weather situations. The feed forward neural network was chosen due of its simplicity, popularity, and accuracy. The filler concentration and various weather circumstances are the two inputs to the proposed network, with the projected dielectric strength in kV/mm being the output.

2.4. Thermal test

Thermogravimetric analysis test (TGA) is one of the major techniques that can analyze the thermal properties of the insulating material. TGA measures the rate of weight loss in the material while increasing the temperature in a controlled atmosphere. Moreover, TGA provides information about the thermal stability of the material and its decomposition rate at higher temperatures up to 800 °C in some cases. By using micro/macro thermogravimetric analyzers (TGA 50), this test was carried according to the ASTM D3850-12 standard in temperature range from 30 °C up to 600 °C with 10 °C/min rate.

2.5. Mechanical test

Tensile strength and elongation at break tests are carried out to explain the mechanical properties of the samples that were mentioned in Table 1. The tensile strength is the force that the sample can withstand before necking divided by the cross-section area. The elongation at break measures the percentage increase in length of the samples after being stretched. The shape of the sample was in the form of dumbbell of 5 cm length and 1mm thickness. The average of the three samples that are tested is calculated and presented in this study. Both tests were performed according to the ASTM D638-14 standard using Zwick Roell testing machine.

3. RESULTS AND DISCUSSION

3.1. Dielectric strength results

The results of the dielectric strength for the composites are shown in Figure 3. The X axis represents the different testing conditions as mentioned before and the Y axis represents the dielectric strength in terms of the dielectric strength (kV/mm). It can be noticed that the dielectric strength of the XLPE is improved by adding nano Z in all testing conditions.

At 30°C testing condition, the dielectric strength of XLPE loaded by Z₁, Z₃, Z₅, and Z₇, is improved to reach 40.25 kV/mm, 41 kV/mm, 43 kV/mm, and 42 kV/mm, respectively, which are higher than the breakdown voltage, 35 kV/mm, of XLPE without filler. Similar improvements at 250°C, the break down voltage is increased to 31.64 kV/mm, 33.62 kV/mm, and 32.76 kV/mm for the XLPE loaded by Z₁, Z₃, Z₅, and Z₇, respectively as shown in Figure 3. These values are higher than 28.3 kV/mm which is the breakdown voltage of the XLPE without adding any fillers.

At low salty conditions, the dielectric strength of XLPE is also improved to 34.02 kV/mm, 35 kV/mm, 36.86 kV/mm, and 35.65 kV/mm when the XLPE is loaded by Z₁, Z₃, Z₅, and Z₇, respectively, compared to 31.5 kV/mm when the XLPE is not loaded by any filler. The same conclusion at high salty conditions. The dielectric strength of the XLPE is increased by 3.5%, 6.5%, 15.4%, and 12.6% when it is loaded by Z₁, Z₃, Z₅, and Z₇, respectively.
It can also be noticed from Figure 3 that the dielectric strength increased up to Z₅, then it began to decline at higher loading wt%, such as in the case of Z₇. This means that the dielectric strength of the XLPE is improved by adding nano-fillers up to a certain concentration level, then it starts to decrease [20]–[22]. This certain concentration level is defined by the optimum weight percent and in this study it is the case when the XLPE is loaded by Z₅. The dielectric strength enhancement could be attributed to the effect of interaction zones. By adding the nano-filler to the matrix of the XLPE, the surface area of the particles and the interfacial area are increased. Therefore, a high volume of interaction zones is formed [23], [24], which in turn escalates the trapping of charges such as electrons inside the interaction zones.

The reason for the reduction in the dielectric strength when the wt% is increased further than Z₅ is that the number of particles is increased and, hence, the distance between the particles is decreased. Therefore, the overlapping of the interfaces between the nano Z and the XLPE matrix is created. Moreover, the accumulation of nano-fillers occurring at higher wt% causes defects such as macro or nanovoids at the interface [25].

![Figure 3. Dielectric strength of XLPE loaded with various wt% of nano (Z) in different conditions](image)

### 3.2. Artificial neural network results

The proposed feed forward artificial neural network practice has shown to be a successful approach. The network has trained to anticipate the value of dielectric breakdown as an output over the course of the iterations. The ANN's result error was calculated. The checker point for each evaluated condition was removed from the training process as a check; instead, those saved assets were used as inputs by the network to validate the ANN approach. Table 2 shows the two inputs in the second and the third columns as well as the experimental results of dielectric strength value, predicted values obtained by the ANN, and calculated error percentages. It can be noted from Table 2 that the artificial neural network is a good estimator for dielectric strength values of XLPE/zeolite composites. The results demonstrate an acceptable error percentage, with the maximum error value of all concentrations in all weather conditions not exceeding 2.5185%, indicating that the intended goal was met.

### 3.3. TGA results

The thermal stability of XLPE and its samples with different concentrations (1 wt%, 3 wt%, 5 wt%, and 7 wt%) was determined in terms of the weight loss percentage at various temperatures, which is represented in Figure 4. The results show that the increase of nano Z improves the thermal stability as per TGA test. The measurements were carried out from 30 °C to 600 °C in nitrogen atmosphere. It can be seen from Figure 4 that:

- The weight loss increased by temperature rise for all composites.
b. The weight loss is inversely proportional to the loading of nano zeolite into XLPE.
c. As nano zeolite filler content increased from 1 to 7 wt%, the rate of weight loss decreased.
d. The thermal stability of XLPE loaded with nano zeolite (7 wt%) is better than other XLPE/zeolite composite samples.
e. XLPE filled with 7 wt% nano zeolite gave the best thermal stability compared with all composite samples and with pure one.

<table>
<thead>
<tr>
<th>Sample symbol</th>
<th>Weathering condition (i/p1)</th>
<th>Dielectric strength test results (i/p2)</th>
<th>ANN results (o/p)</th>
<th>Percentage of error (%)</th>
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<tr>
<td>B</td>
<td>30℃</td>
<td>35.0000</td>
<td>35.5516</td>
<td>1.5760</td>
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<tr>
<td>Z₁</td>
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<td>40.1616</td>
<td>0.2196</td>
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<tr>
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<td>41.7244</td>
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<td>Z₅</td>
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<td>35.9268</td>
<td>2.5185</td>
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<tr>
<td>Z₇</td>
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<td>36.1099</td>
<td>0.2071</td>
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<tr>
<td>B</td>
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<td>33.8561</td>
<td>0.1364</td>
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<tr>
<td>Z₁</td>
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<tr>
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<td>33.8561</td>
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<tr>
<td>Z₅</td>
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<td>34.7111</td>
<td>0.7492</td>
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</tr>
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</table>

Table 2. ANN inputs, output, and error percentage

Figure 4. TGA of XLPE with different nano Z wt% concentrations

3.4. Mechanical results

Table 3 shows the relation between the loaded added Zeolite and tensile strength (MPa) and elongation at break (%) of XLPE composites. The presence of Zeolite incorporated with XLPE slightly enhanced the tensile strength and elongation at break with optimum values appeared with 5 wt% zeolite. Table 3 shows that including nano zeolite into XLPE enhances the tensile strength of the composites up to 5 wt% nano zeolite. The tensile strength of the neat XLPE is 10.56 MPa. XLPE filled with 3 wt% nano zeolite has a maximum tensile strength (11.71 MPa) compared to pure XLPE and other concentrations of XLPE/zeolite composites samples.
Table 3. Tensile strength and elongation at break tests results

<table>
<thead>
<tr>
<th>Samples symbol</th>
<th>Tensile strength (MPa)</th>
<th>Elongation at break %</th>
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</thead>
<tbody>
<tr>
<td>B</td>
<td>10.56</td>
<td>468.84</td>
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<td>Z₁</td>
<td>10.92</td>
<td>473.98</td>
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<td>Z₃</td>
<td>11.71</td>
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<td>Z₅</td>
<td>10.76</td>
<td>446.10</td>
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<tr>
<td>Z₇</td>
<td>9.78</td>
<td>436.20</td>
</tr>
</tbody>
</table>

Table 3 further shows that Sample Z₇ has the smallest elongation at break (436.20%). The elongation at break rises from 9.78% to 11.71%. A small amount of nano zeolite added into XLPE improves the polymer bonds of the XLPE when it is loaded by Z₁ and Z₃, meanwhile increase the tensile strength and elongation at break otherwise the two other concentrations of Z₅ and Z₇ decreased the mechanical characteristics of the samples, as the samples became more brittle (weak polymer bonds) due to higher filler loading in XLPE matrix.

To describe both electrical and mechanical properties and plot them simultaneously. This plot would help us in selecting an optimum concentration of ATH filler which gives the best composite when mixing XLPE with zeolite based on both electrical and mechanical properties. The best curve fitting for the dielectric strength and tensile strength is shown in Figure 5.

The intersection point of the blue dielectric strength curve with the red tensile strength curve indicates the optimum value of zeolite filler in the composite under at room temperature is approximately 3.64 wt% as shown in Figure 5. The curve fitting using MATLAB has been used to estimate the optimal concentration of nano Zeolite to improve dielectric and mechanical properties of XLPE composites. The intersection point of the blue dielectric strength curve with the red tensile strength curve indicates the optimum value of zeolite filler in the composite under at room temperature is approximately 3.64 wt% as shown in Figure 5.

## 4. CONCLUSION

The optimum concentration of zeolite nano filler is 3 wt % loaded to XLPE to improve the dielectric strength by almost 18%, 10%, 9.5% and 8% in room temperature, 250 °C, low and high salinity conditions respectively. Mechanical properties increased till Z₃ to reach 11.71 MPa for tensile strength and 496.79% for elongation and break. The thermal stability also was enhanced by adding nano zeolite filler to the XLPE matrix. It may be recommended the 3.64 wt% of nano zeolite to XLPE insulation to enhance all its characteristics (electrical, thermal, mechanical). The results of the experiments were used to train the ANN, which was able to evaluate and predict dielectric strength values with excellent accuracy.
REFERENCES


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Improvement of dielectric strength and properties of cross-linked polyethylene using ... (Sherif Essawi)
BIOGRAPHIES OF AUTHORS

Sherif Essawi was born in Cairo, Egypt on 1983. He received his B. Sc from Faculty of Engineering, Ain shams University, Electrical Power and Machine department in 2005. He works as design engineer for Low and Medium voltage Networks. Also he worked in the planning of the high Voltage network in Egypt. He received his Msc in 2012 from faculty of Engineering, Ain shams university, Electrical and high voltage department. His researches focuses on the improvement of High voltage insulation in cables. He can be contacted at email: eng.sherifessawi@gmail.com.

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Jeannette Asaad is a professor at National Research Center (NRC), polymers and pigments department. She joined the department in 1985. She was born in Egypt and graduated from Ain Shams University in 1981, she also received her M.Sc and Ph.D. in 1995 and 2002 from Cairo University, respectively. She has been appointed at NRC as Researcher (2002), as Associate Prof. (2008) and Professor (2014). Dr. Jeannette granted postdoctoral fellowship to Germany through DFG in 2011 at the Institute of Polymer Materials and Plastics Engineering, Clausthal University of Technology. Where, she carried out some work on the influences of proton-donators onto epoxy resins leading to reaction kinetic. She supervised M.sc and Ph.D student to completion. These studies dealt with polymer composites and rubber blends as high voltage insulators and cables. She published more than 40 peer–reviewed papers in national, international journals and conferences. Reviewed papers for international Journals: Polymer Bulletin, Journal of applied polymer science, Polymer engineering and science and Journal of Elastomers and Plastics. She can be contacted at email: najjeannette@yahoo.com.