

Effect of nanoparticles on dielectric, mechanical and thermal characteristics of XLPE/TiO₂ nanocomposites

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Abstract

Insulators are required in a power system network to provide ground isolation and mechanical support for line conductors. Different kinds of insulators are being utilized in transmission lines and substations. Many power utilities are now using non-ceramic insulators like Cross-Linked Polyethylene (XLPE) composite insulators. This research aims to develop XLPE nanocomposites for use as power cable insulation in industrial applications. To attain this, XLPE nanocomposites were made with five different loadings of Titanium Dioxide (TiO₂) nanoparticles: 0, 1, 3, 5 and 7 weight (wt)% in the presence of a Dicumyl Peroxide agent, which is used to minimize nanoparticle agglomeration and enhance compatibility within the polymer matrix. A Scanning Electron Microscope (SEM) was utilized to investigate the structure and distribution of nanoparticles inside the XLPE. The dielectric properties of these developed XLPE/TiO₂ nanocomposites were studied by measuring the AC dielectric strength with a regulated high voltage testing transformer (50Hz). The mechanical properties such as tensile strength and elongation at break were also assessed. The thermal properties of nanocomposites were examined using Thermo Gravimetric Analysis (TGA). When TiO₂ nanoparticles are included in the XLPE matrix, the dielectric strength of XLPE/TiO₂ is shown to be higher than pure XLPE. This indicates that XLPE/TiO₂ nanocomposites have higher dielectric characteristics, with a TiO₂ filler loading of 5 wt% being the best. This might be because TiO₂ nanoparticles have a low surface energy, which prevents them from clustering together. Also, the addition of nano TiO₂ improves the mechanical and thermal characteristics of XLPE nanocomposites. XLPE with 5 wt% nano TiO₂ showed the best improvement in different properties.

Keywords

Nanocomposites, XLPE, Titanium dioxide filler, Dielectric strength, Tensile strength, TGA.

1. Introduction

Because of its high flexibility and mechanical strength, superior dielectric characteristics, strong chemical resistance, ease of processing, and low cost, Cross-Linked Polyethylene (XLPE) has been widely used as an insulating material in high-voltage underground cables [1–3]. Composite materials are made up of two or more components with drastically different physical and chemical characteristics. New materials with properties distinct from the individual components are created as a result of the controlled mixing of the components [4]. Nanocomposites are materials that have at least one component with nanometric dimensions [5].

Traditional composites differ from nanocomposites in three ways: As a result, nanocomposites have unique properties such as homogeneous structure, optical transparency, and increased or unaffected processability [6]. Ceramic nanocomposites, metal nanocomposites, and polymer nanocomposites [7] are the three types of nanocomposites classified by matrix material.

According to Reference [6], polymer nanocomposites are polymers with a small number of nano-sized fillers (less than 10 weight (wt)%) dispersed homogeneously. Due to their superior material performance, composite materials are frequently chosen over traditional materials. Lightweight materials with higher mechanical strength are chosen for a variety of applications in the automotive, aircraft, and maritime industries. The current review focuses on micro and nanocomposites utilized in high voltage applications.

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Although Lewis [8] developed the term "nanometric dielectrics" [8] or "nanodielectrics" [9] in 1994, it was unclear how the anticipated property changes due to nano-sized filler additions would help electrical insulation. Different properties, such as electrical, mechanical, physical, and thermal, have all been studied extensively [8, 10]. In 1988, Guastavino et al. [11] demonstrated that mica-based methods for groundwall insulation of form-wound generators could provide some advantages. Similar studies on nano SiO₂ filler when dispersed in polymers that improve the electrical properties of polymer insulation were conducted by Henk et al. [12] in 1999.

Singha and Thomas [13], Nelson and Fothergill [14] studied the effect of nanodielectrics on power and high voltage engineering, which attracted limited attraction from researchers. Many experiments were carried out in order to gain a basic understanding of how nanoparticles interact with a polymer matrix to change the dielectric characteristics.

The purpose of this research is to examine the impact of introducing nano Titanium Dioxide (TiO₂) into the XLPE matrix. The dielectric properties such as dielectric strength, mechanical and thermal characteristics will be measured.

Many investigations have shown that traditional microfilled materials can decrease the dielectric strength of insulating polymers, which could be related to bulk charge accumulation [15]. Furthermore, the nanofiller materials cause extensive interfacial areas, which are defined as "an interaction zone between the polymer matrix" and the filler. As a result, the particle type, size, surface, and weight of the nanoparticles inside the polymer nanocomposites play a significant role in their fundamental properties [16, 17].

The effect of high temperatures and thermal aging at high temperatures for 24 hrs on the performance of XLPE nanocomposite insulators has been determined and served to decrease the dielectric strength of XLPE composites. By nano TiO₂ addition, dielectric, mechanical and thermal properties are enhanced.

2.Literature review

Clay, Acrylic (PA₄₀), Silica (silicon dioxide) (SiO₂), Alumina (Al₂O₃), TiO₂, Calcium Carbonate (CaCO₃) and other metal oxides like Magnesium Oxide (MgO), Zinc Oxide (ZnO), et cetera) have all been used in the power industry to improve the performance of insulating materials. To attain good electrical,

mechanical, and thermal properties, these fillers are frequently included in electrical insulating polymers.

Previous studies have found that adding ZnO, Al₂O₃, SiO₂, and Al (OH)₃ nanofillers to XLPE base polymer enhanced electrical characteristics slightly at low filler loading [18–21]. Furthermore, because of its unique properties such as high catalysis activity, nontoxic nature, high mechanical stability, and so on, TiO₂ nanocomposites were studied in this work [22–26].

Abd Rahman et al. [21] reported the influence of Zirconium Oxide (ZrO₂) and SiO₂ nanocomposites on the AC breakdown strength of XLPE. Extrusion and crosslinking methods are used to produce pure XLPE and XLPE composites with (1, 3, 5, and 10 wt%) nanofillers. The AC breakdown strength of XLPE nanocomposite samples was carried out. The findings demonstrated that when nanofillers were used in small amounts, AC breakdown strength performance improved. Different behaviours were seen when ZrO₂ and SiO₂ were compared.

Abd Rahman et al. [27] investigated the addition of TiO₂ and Barium Titanate (BaTiO₃) to the dielectric strength and space charge of XLPE. According to American Society for Testing and Materials (ASTM) D-149, the dielectric strength performance has been tested. In order to explore charge formation inside the composite samples, the space charge formation was also measured. The results showed that space charge and dielectric strength were both positive. However, in order to achieve improvement, the optimal amount of nanofillers must be carefully assessed. In a study of BaTiO₃ and TiO₂, distinct behaviours were seen for both properties. The nanofillers in the XLPE matrix were proved to have an excellent effect on this investigation.

Eldesoky et al. [28] studied high voltage XLPE properties improvement using functionalized ZnO nanoparticles, to accomplish this, XLPE/ZnO nanocomposites were made with four different loadings of ZnO nanoparticles: 0.5, 2, 3.5, and 5 wt%, in the presence of a suitable coupling agent that reduced nanoparticle aggregation and improved compatibility inside the polymer matrix. SEM was used to investigate the shape and distribution of nanoparticles within the XLPE polymer. The mechanical properties of the material were also assessed. The thermal characteristics of nanocomposites were examined using Differential Scanning Calorimetry (DSC) and Thermo Gravimetric Analysis (TGA). The loss tangent and the dielectric

constant of these produced XLPE/ZnO nanocomposites were measured at frequencies ranging from 1 Hz to 1 MHz to investigate their dielectric characteristics. The AC Breakdown voltage was also measured in the sphere-to-sphere field using a regulated high voltage testing transformer. The addition of ZnO nanoparticles to the XLPE matrix improved the thermal and mechanical properties of the polymer, and the functionalization nanoparticles resulted in efficient dispersion within the polymer matrix, according to the findings.

3.Methods

The study paper's testing mechanism is displayed in *Figure 1*.

The testing mechanism employed in this investigation is depicted in *Figure 1*. To begin, different nanofiller concentrations were used to prepare the XLPE/TiO₂ 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 TiO₂ filler for improving electrical, mechanical, and thermal properties.

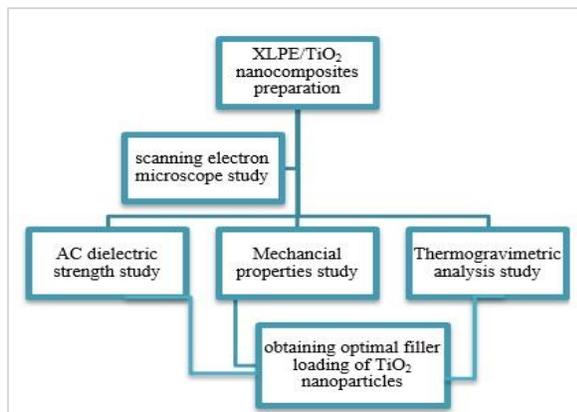


Figure 1 Testing mechanism of research paper

3.1Materials

SABIC KSA provided pure cross-linked low density polyethylene pellets with a density of 0.924 g/cm³ and a melt flow rate of 2.00 g/10 min at 2.16 kg at 190°C and a melt flow rate of 2.00 g/10 min at 2.16 kg at 190°C. To make compounding easier, it was crushed into smaller particles with a diameter of less than 0.2 mm. Nano size TiO₂ for industrial applications was purchased from Nanotech Egypt.

3.2Preparation of XLPE composite samples

According to ASTM F876- 10e1, LDPE was mixed with dicumyl peroxide (3 wt%) as a cross linking agent

without any filler to make XLPE. The nanocomposites that were made are XLPE loaded with nano fillers at various percentages (1, 3, 5, and 7) wt%. *Table 1* lists all of the formulations of XLPE/TiO₂ nanocomposites. All nanocomposites were combined in a Barbender Plasticoder type electrically heated chamber (C.W. Pra instrument, INC.50 Hackensack, 230 Volt, 40 Amp). Mixing was performed at 160°C and 100 RPM for 10 min to allow the torque to approach equilibrium. After the mixing was finished, it was pressed at 4 MPa and molded at the same temperature as before. According to ASTM, the sample was cut and prepared with dimensions that best matched each testing technique. Finally, 20 × 20 cm square sheets with a thickness of around 1 mm are produced. *Figure 2* shows the different types of nanocomposite samples for XLPE nanocomposites.

Many sheets different composites and bearing varying loads, were purchased in order to carry out the specified testing on them.

Table 1 Mixing formulations of XLPE/TiO₂ nanocomposite samples

Sample	Acronym	XLPE (wt %)	Nano TiO ₂ (wt %)
XLPE	B	100	0
XLPE + 1% TiO ₂	T1	99	1
XLPE + 3% TiO ₂	T3	97	3
XLPE + 5% TiO ₂	T5	95	5
XLPE + 7% TiO ₂	T7	93	7

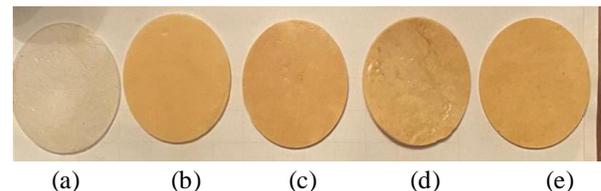


Figure 2 Different types of nanocomposite samples for XLPE (a) sample B (b) sample T1, (c) sample T3, (d) sample T5 and (e) sample T7

3.3 Scanning electron microscope

Scanning Electron Microscopy (SEM) was utilized to investigate the surface morphology of XLPE/TiO₂ nanocomposites. The micrographs were gathered at an accelerating voltage of 10–15 kV, and the microscope was connected to the energy dispersive X-ray analysis unit.

3.4Dielectric properties

The dielectric properties are characterized by the AC dielectric strength. A test cell electrified by a test transformer (220V/100kV) was used to determine the

breakdown strength. The ASTM (D149-09) standard is followed when filling the test cell with transformer oil. All of the samples' breakdown strength has been determined. The dielectric strength test has been studied in two different cases as summarized in *Table 2*:

- i. The first case required placing the nanocomposite samples at two different temperatures. The first testing temperature is set at 30°C in order to simulate operating conditions at room temperature. The second testing temperature is set at 250°C which can simulate the highest short circuit working condition for a cable operating above 30 kV. By exposing the nanocomposite samples to this temperature in the laboratory oven, the temperature of the nanocomposite samples reached 250°C. Furthermore, the samples were immersed in oil to limit the possibility of any flashover. The nanocomposite samples should be clean and dry for those two testing conditions before beginning the high voltage test. The voltage was steadily raised at a rate of 2 kV/s till the breakdown voltage occurred.
- ii. The second case required exposing the nanocomposite samples to thermal aging for 24 hrs in different high temperatures (120°C and 160°C).

Table 2 Testing conditions for AC dielectric strength test

Different conditions for dielectric strength test	Discription
Condition 1	30°C (at room temperature) 250°C (to simulate high short circuit)
Condition 2	Thermal aging at 120°C for 24hrs Thermal aging at 160°C for 24hrs

3.5 Mechanical properties

Mechanical tests such as tensile strength and elongation at break were carried out to show the ability of nanocomposite samples to withstand mechanical forces. The samples should be in the form of a dumbbell with a thickness of 0.2 cm and a length of 5 cm. The test was performed using the Zwick Roell LTM electrodynamic testing machine in accordance with ASTM D-412.

3.6 Thermo gravimetric analysis (TGA)

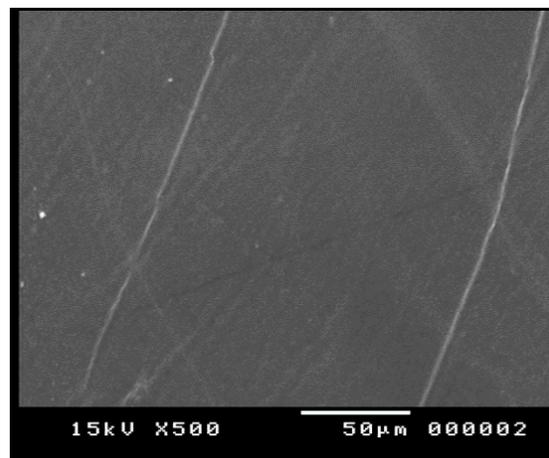
STA 6000 Perkin Elmer Analyzer and thermogravimetric analysis (TGA, Shimadzu DTG-60, Japan) were used to investigate the thermal

stability of XLPE/TiO₂ nanocomposites from 30°C to 600°C at a heating rate of 10°C/min.

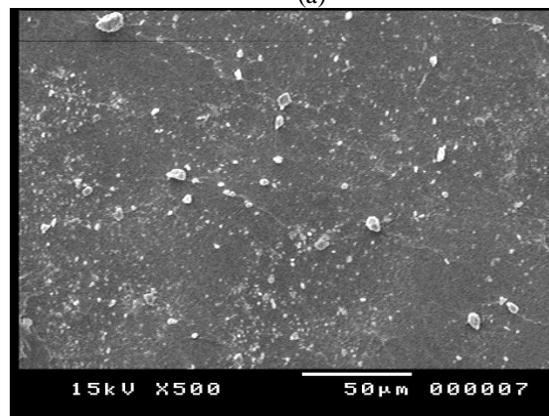
4. Results

4.1 Morphological analysis

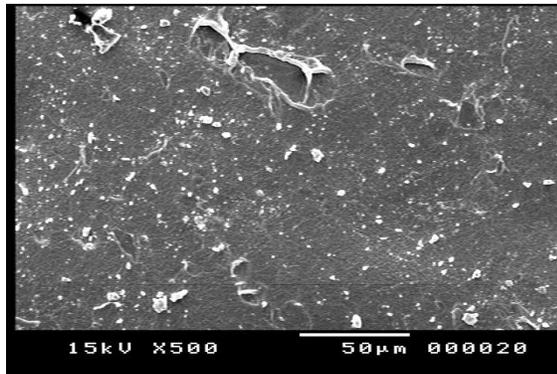
Electron microscopy is an effective method for determining the morphology of TiO₂ nanoparticles that have been generated. Using a Scanning Electron Microscope (SEM), the structure of the pure XLPE and the dispersion of TiO₂ nanoparticles into the polymer matrix were investigated. *Figures 3a, 3b, 3c, 3d* and *3e* show SEM images. The surface morphology of the composite was modified by embedding TiO₂ onto XLPE (*Figures 3b, 3c, 3d* and *3e*). Within the cross-section area and surface of the sample T5 composite, there is good dispersion without the formation of agglomeration (*Figure 3d*). While aggregates of TiO₂ emerged as white dots within the surface and cross-section area of the TiO₂/XLPE composite (*Figures 3c* and *3e*), the white light region is the XLPE filler, and the dark region is the base material.



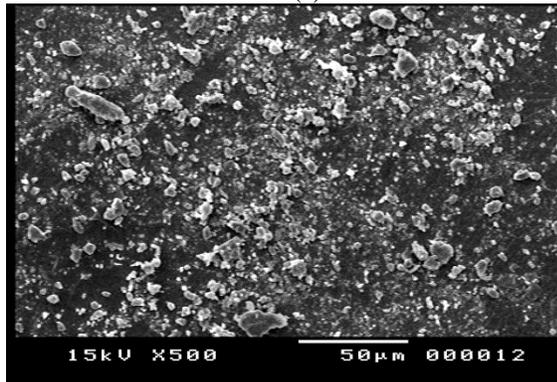
(a)



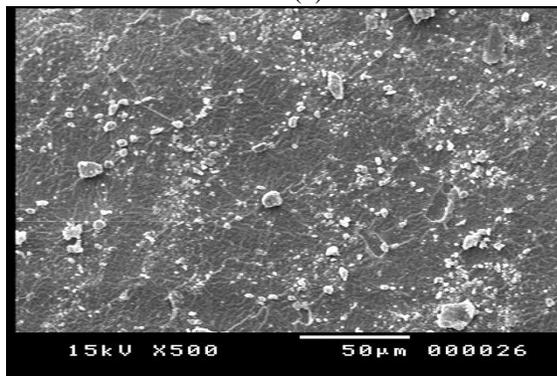
(b)



(c)



(d)



(e)

Figure 3 SEM images of XLPE/TiO₂ nanocomposite samples (a) sample B (b) sample T1, (c) sample T3, (d) sample T5 and (e) sample T7

4.2AC Dielectric strength test results

The ASTM D149-09 standard is used to determine the AC dielectric strength of XLPE/TiO₂ nanocomposites. A test transformer (50Hz, 220V/100kV) is connected to the test cell. The measured values of AC dielectric strength are shown in *Figure 4*. The formation of a strong interfacial area between the polymer matrix and the nanoparticles may be responsible for this. Furthermore, by increasing the concentration of TiO₂ nanoparticles, the dielectric strength was increased up to 5wt% of TiO₂ nano filler, and after that, the dielectric strength decreased.

It can be seen from *Figure 4* that a significant rise in the breakdown voltage can only be obtained at low loading percentages. The composite with the highest breakdown voltage has roughly 5 wt% filler weight. This is followed by a high rate of discrimination with increasing concentration levels.

It can be investigated from *Figure 4* that:

- At room temperature (30°C) condition, the AC dielectric strength of XLPE loaded with TiO₂ (1, 3, 5 and 7 wt%) was improved by 6%, 16%, 35% and 24% to reach 39.98, 43.62, 50.89 and 46.64kV/mm, respectively, compared to pure XLPE (37.58kV/mm).
- At high temperature (250°C) condition, the AC dielectric strength of XLPE loaded with TiO₂ (1, 3, 5 and 7 wt%) was improved by 7%, 12%, 36% and 19% to reach 30.98, 32.47, 39.29 and 34.46kV/mm, respectively, compared to pure XLPE (28.89kV/mm).
- When samples are exposed to thermal aging for 24hrs at 120°C, the AC dielectric strength of XLPE loaded with TiO₂ (1, 3, 5 and 7 wt%) was improved by 7%, 9%, 23% and 21% to reach 24.98, 25.47, 28.76 and 28.26kV/mm, respectively, compared to pure XLPE (23.38kV/mm).
- Finally, when samples are exposed to thermal aging for 24hrs at 160°C, the AC dielectric strength of XLPE loaded with TiO₂ (1, 3, 5 and 7 wt%) was improved by 7%, 15%, 24% and 16% to reach 16.98, 18.29, 19.68 and 18.47kV/mm, respectively, compared to pure XLPE (15.89kV/mm).

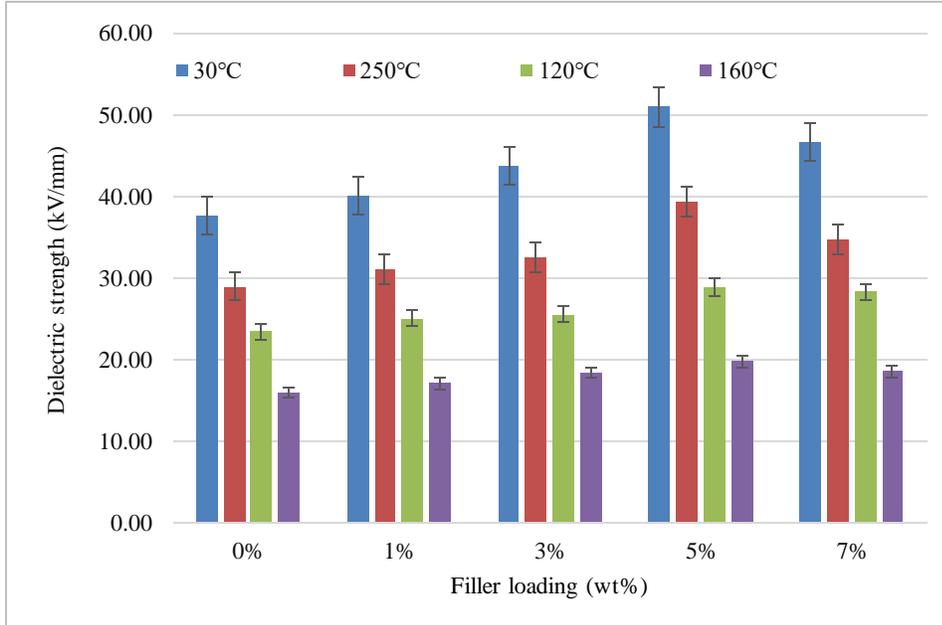


Figure 4 Dielectric strength (kV/mm) of XLPE/TiO₂ nanocomposite samples at different conditions

4.3 Mechanical results

4.3.1 Tensile strength

The dependency of tensile strength on the percentage of nano TiO₂ filler added to XLPE is shown in *Figure 5*.

It is shown in *Figure 5* that XLPE has an initial value of tensile strength of 8.18 MPa. The addition of nano TiO₂ with a concentration up to 5 wt% causes an increase in the tensile strength up to 10.90 MPa. The TiO₂ concentration of 7 wt% causes a decrease in the tensile strength to a value of 9.41 MPa.

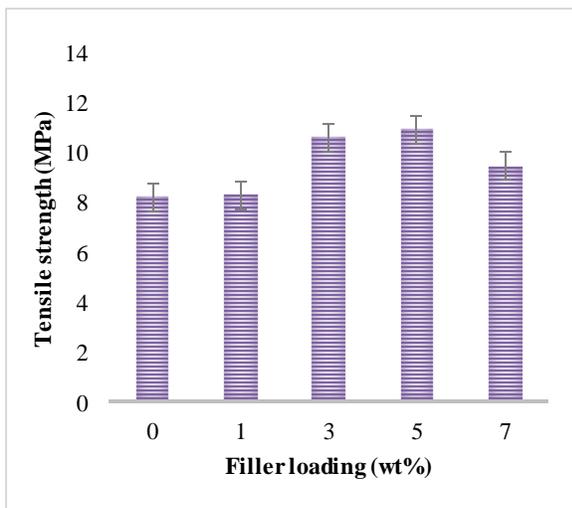


Figure 5 Tensile strength (MPa) for XLPE/TiO₂ composite samples

4.3.2 Elongation at break

Figure 6 shows the relation between the loaded added TiO₂ and elongation at break of XLPE composites. The presence of TiO₂ incorporated with XLPE slightly enhanced the tensile strength with optimum strength appeared with 5 wt% TiO₂.

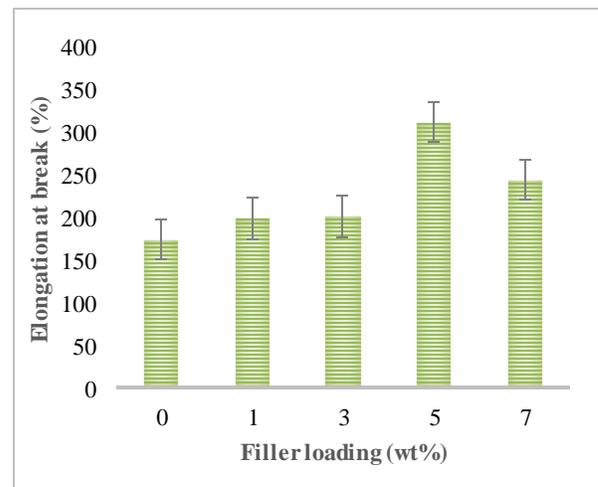


Figure 6 Elongation at break (MPa) for XLPE/TiO₂ composite samples

Figure 6 shows that the addition of TiO₂ nanoparticles increased the elongation at break of XLPE. The high specific surface accessible for interactions with the polymer chains has resulted in this behavior in mechanical characteristics and a rise in the elongation at break value with minimal loading of nanoparticles.

Also, the addition of nanofiller to most polymer nanocomposites is expected to reduce elongation values, which relates to the restriction of chain mobility caused by the connection between the nanoparticles and the polymer matrix [29].

It can be investigated from *Figures 5 and 6* that:

- The addition of nano TiO₂ to the XLPE matrix increases the tensile strength and elongation at break up to 5 wt% and after that, the elongation at break decreases.
- XLPE with added 5 wt% of nano TiO₂ filler has a maximum value of tensile strength (10.89 MPa) and elongation at break (310.91%) compared to pure XLPE and other nanocomposites concentrations.

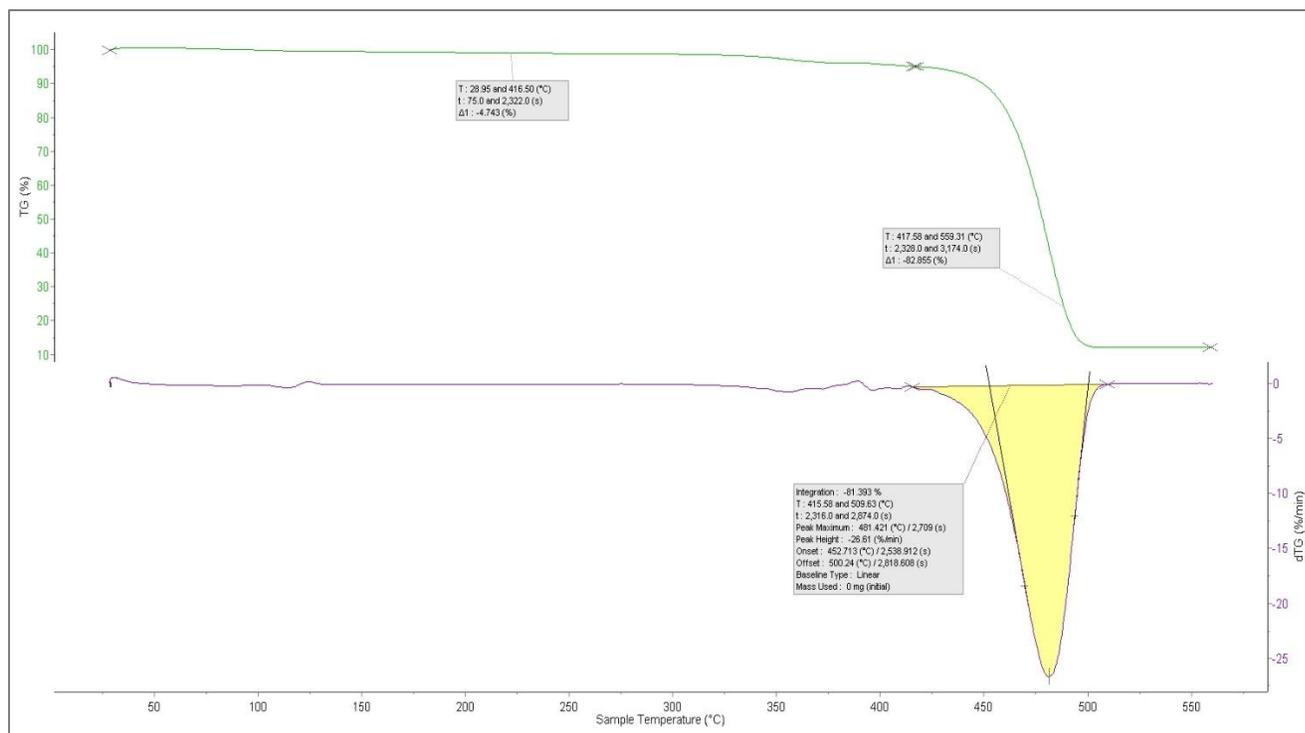
4.4 TGA results

The rate of degradation in polymer samples with the relevant independent variable is determined by the initial experimental setup and the basic percentage weight loss vs temperature. TGA data as a function of temperature can also be used to track the thermal recovery of mechanical deformation [30]. Thermal degradation could be indicated by irreversible weight

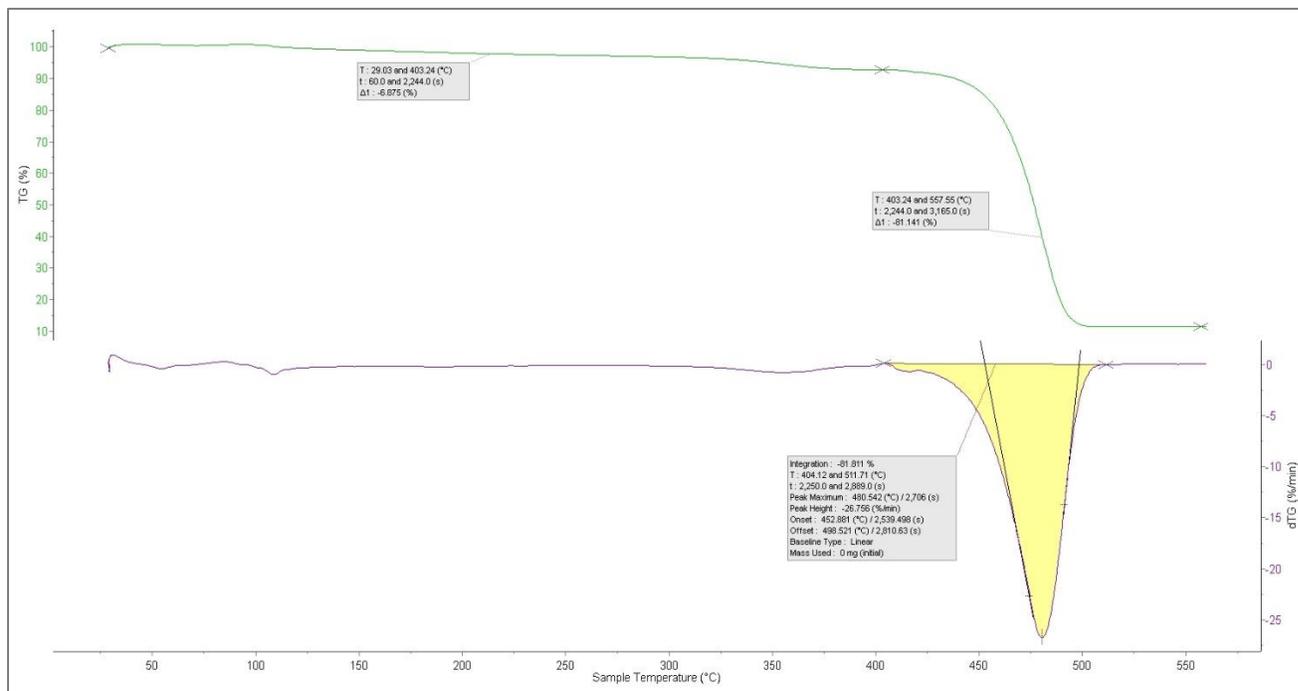
loss. The thermal stability of nanocomposites was determined in this study using weight loss at 300°C, 400°C, and 480°C (decomposition temperatures), as shown in *Figure 7*. The thermal stability of XLPE/TiO₂ nanocomposites was determined by the higher values of decomposition temperatures [31]. Weight loss was reduced when TiO₂ nanoparticles were added. This indicates that TiO₂ nanoparticles added to the XLPE polymer increased thermal stability. This could be owing to the inorganic nanomaterial's high thermal resistance, with an onset temperature of 560°C [32–35], or to the matrix chains' decreased mobility as a result of the good bond between the nanoparticles of TiO₂ and the XLPE polymer.

It can be also noted from *Figure 7* that:

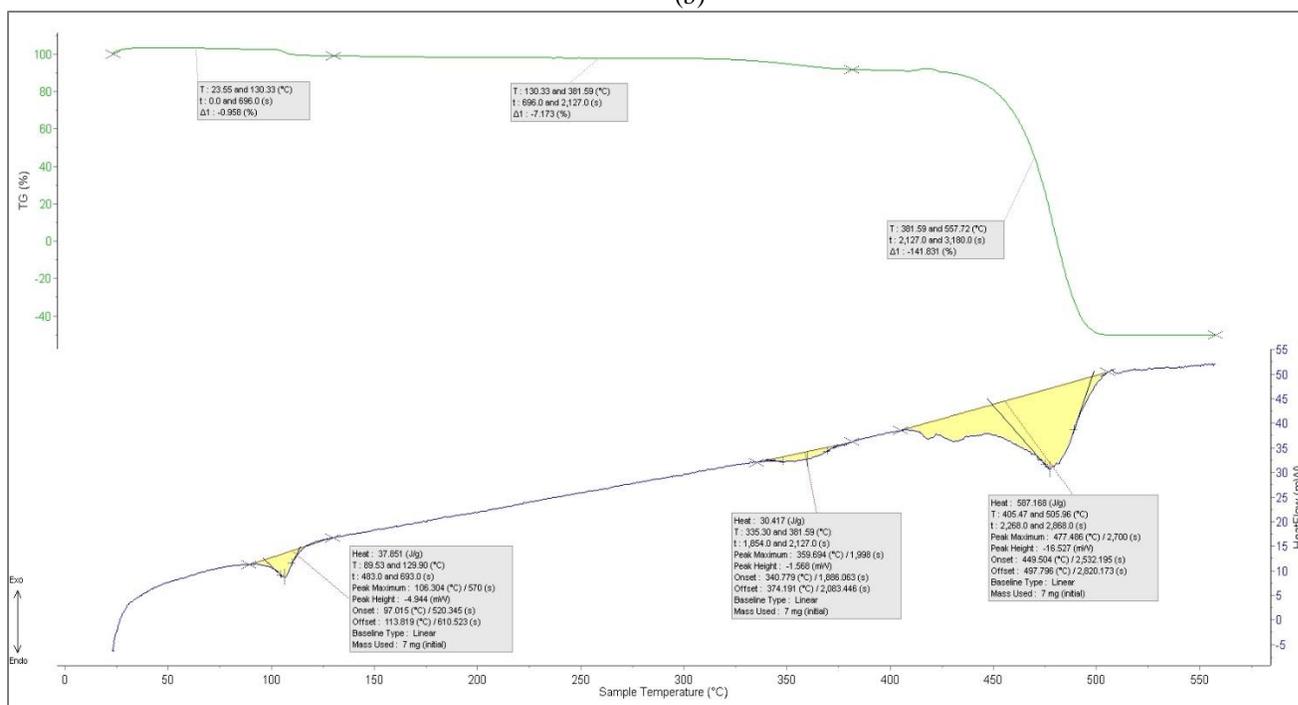
- The weight loss was increased by the temperature rise for all nanocomposites.
- The weight loss is inversely proportional to the loading concentration of nano TiO₂.
- The thermal stability of XLPE filled with nano TiO₂ (5 wt%) is better than pure XLPE and other nanocomposites concentrations.



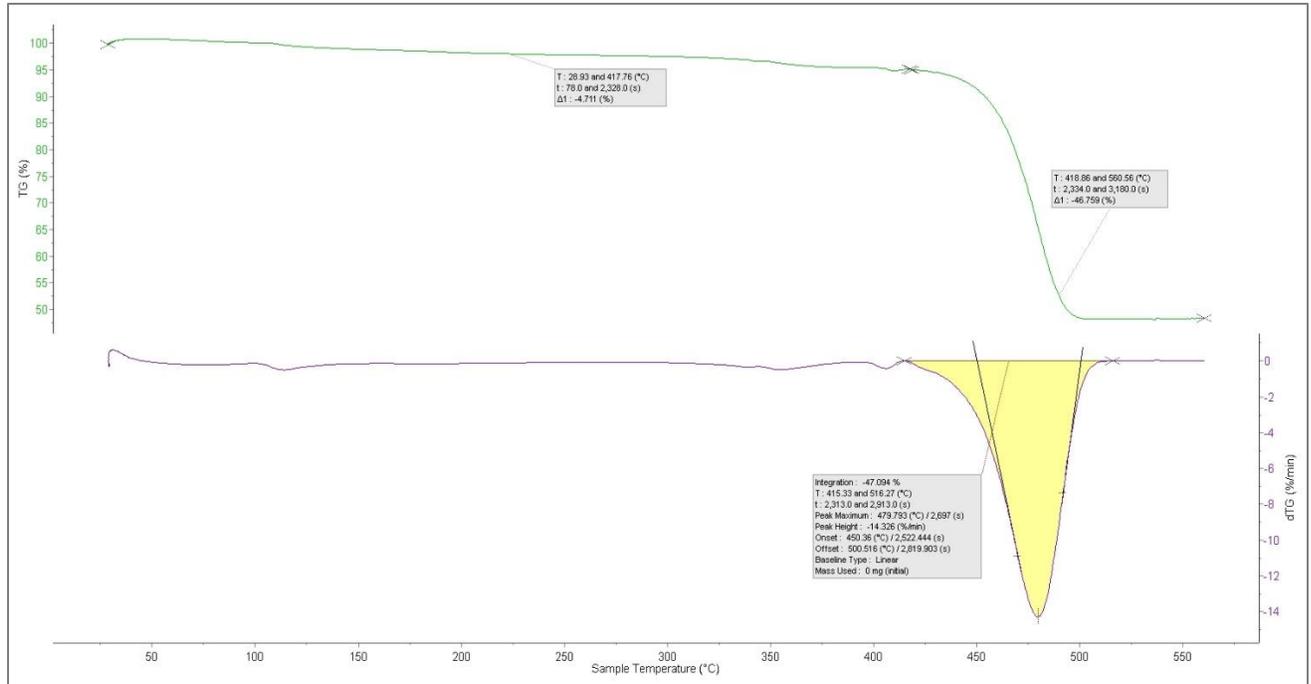
(a)



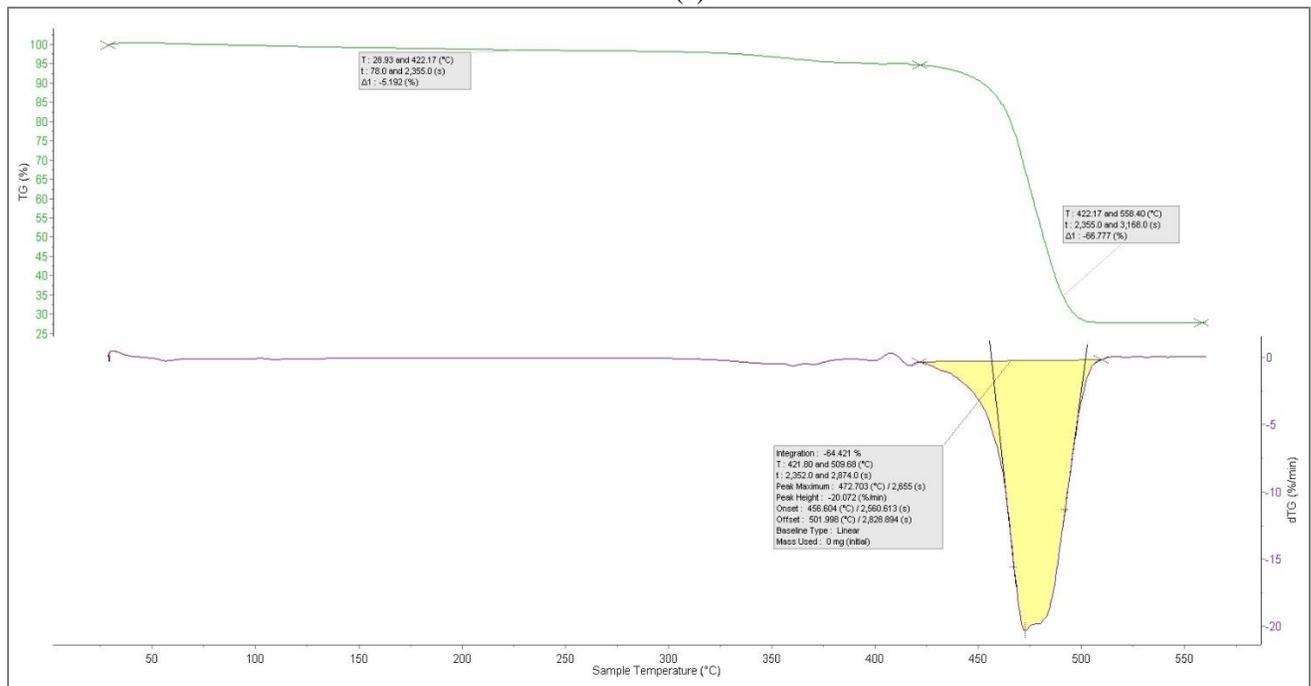
(b)



(c)



(d)



(e)

Figure 7 TGA of (a) sample B (b) sample T1, (c) sample T3, (d) sample T5 and (e) sample T7

5. Discussion

In general, doping a polymer with an inorganic filler creates traps that can capture charge carriers. TiO₂ nanoparticles have a uniform distribution in the matrix at low filler loadings, and the density of the emerging

traps is sufficient to marginally suppress charge mobility and increase breakdown voltage [36].

To get surface-modified nano TiO₂, dicumyl peroxide was used as the coupling agent in this study. On one

end, the coupling agent is chemically connected to nanoparticles, and on the other, the coupling agent is an alkyl chain that is compatible with the XLPE matrix. The dielectric strength of all polymers reduces with increasing temperature in a clear tendency. The reason for this is that when the temperature rises, the secondary breakdown processes become more essential [20].

The increase in mechanical properties of composites in the concentration range of 1–5 wt% can be attributed to the ease of homogeneously dispersed nano TiO₂ in the polymer matrix. Thus, there is an increase in mechanical properties for composites compared with pure XLPE. However, the decrease in composite mechanical properties with further addition of TiO₂ over 5 wt% may be explained by the agglomeration of nano TiO₂. The agglomeration of nano TiO₂ is strictly associated with inhomogeneous distribution of nano TiO₂ in the matrix and reduced interaction between nano TiO₂ and the XLPE matrix. Another possibility is due to the high viscosity of the mixture at high concentrations of nano TiO₂, which causes an improper mixing process. TGA test illustrates that XLPE with 5 wt% TiO₂ nano filler represents the best sample from stability point and pure XLPE represents the worst sample as shown in *Figure 7*. It indicates that nano TiO₂ filler plays an important role in the thermal stability improvement. A complete list of abbreviations is shown in *Appendix I*.

6. Conclusion and future work

The primary insulation in power cables is cross-linked polyethylene. Their electrical, physical, and chemical properties are all significant. This paper is interested in improving these characteristics, particularly its electrical characteristics, by adding TiO₂ nanoparticles, which are limited in terms of publication issues because most researchers are focused on high- and low-density polyethylene. In this research, industrial-scale XLPE/TiO₂ nanocomposites with various titanium dioxide nanoparticle loading ratios were manufactured. The thermal, mechanical, and dielectric characteristics represented in the dielectric strength of the nanocomposites were measured. The dispersion of nano TiO₂ filler into the XLPE matrix improved the electrical, mechanical, and thermal properties of the polymers, according to the results. The dielectric strength of TiO₂ nanocomposites was higher than that of XLPE. The interparticle distance reduces as the percentage weight of nanoparticles increases, causing a barrier to charge carriers transferring and forming a strong bond, resulting in higher dielectric strength. So, it was shown

that increasing the amount of TiO₂ nanoparticles reduced the dielectric strength, which could be because the high concentration of nanoparticles causes greater agglomeration, resulting in a significant reduction in dielectric strength.

However, there are many aspects which may affect the performance of XLPE and further research is suggested to improve the performance of XLPE.

- Studying insulation systems from the point of aging for XLPE composites on the practical site.
- It is suggested to combine two or three nanofillers with XLPE and study their electrical, mechanical, physical and thermal properties.
- Computer software can be used to calculate the average leakage current, aging time, etc. by using a data acquisition system.

Acknowledgment

None.

Conflicts of interest

The authors have no conflicts of interest to declare.

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Appendix I

S.N.	Abbreviation	Description
1	Al ₂ O ₃	Aluminum Oxide
2	Al(OH) ₃	Aluminum Hydroxide
3	ASTM	American Society for Testing and Materials
4	BaTiO ₃	Barium Titanate
5	CaCO ₃	Calcium Carbonate
6	DSC	Differential Scanning Calorimetry
7	MgO	Magnesium Oxide
8	PA ₄₀	Acrylic
9	SEM	Scanning Electron Microscope
10	SiO ₂	Silicon Dioxide
11	TGA	Thermo Gravimetric Analysis
12	TiO ₂	Titanium Dioxide
13	wt%	Weight Percentage
14	XLPE	Cross Linked Polyethylene
15	ZnO	Zinc Oxide
16	ZrO ₂	Zirconium Oxide