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The effect of thermal ageing on electrical and mechanical properties of thermoplastic nanocomposite insulation of power high-voltage cables

This research explores the thermal ageing influence on the Low Density Polyethylene (LDPE) dielectric properties, which is utilised as electrical insulation in high-voltage cables. An accelerated thermal ageing test was done at four temperature ranges ranging from 25 °C to 120 °C to define the degree of material deterioration under thermal ageing and to prevent its failure. LDPE composite samples were made by adding aluminium oxide (Al_2O_3) inorganic filler in two different grain sizes (nano and micro) with various concentrations. The effect of adding inorganic filler on the acceleration of the thermal ageing of the polymer was studied by heating the samples for different periods of time and measuring the dielectric strength of the samples. The obtained results show that thermal ageing considerably affects the electrical properties of the material. The LDPE/Al_2O_3 nanofiller sample has the highest dielectric strength value at different temperatures. Thermogravimetric analysis was used to investigate the thermal characteristics of materials. The mechanical characteristics of LDPE polymer are studied using tensile strength and elongation at break tests. References 27, table 4, figures 6.

Key words: **low density polyethylene, nano filler, micro filler, dielectric strength, thermal ageing, thermogravimetric analysis.**

У цьому дослідженні вивчається вплив термічного старіння на діелектричні властивості поліетилену низької щільності (LDPE), який використовується як електрична ізоляція у високовольтних кабелях. Випробування на прискорене термічне старіння було проведено в чотирьох температурних діапазонах від 25 до 120 °C, щоб визначити ступінь руйнування матеріалу при термічному старінні і запобігти його виходу з ладу. Композитні зразки LDPE були виготовлені шляхом додавання неорганічного наповнювача з оксиду алюмінію (Al_2O_3) з двома різними розмірами зерен (нано та мікро) у різних концентраціях. Вплив додавання неорганічного наповнювача на прискорення термічного старіння полімеру вивчали шляхом нагрівання зразків протягом різних періодів часу та вимірювання діелектричної міцності зразків. Отримані результати показують, що термічне старіння істотно впливає на електричні властивості матеріалу. Зразок нанонаповнювача LDPE/Al_2O_3 має найбільше значення діелектричної міцності за різних температур. Термогравіметричний аналіз використовувався для дослідження термічних характеристик матеріалів. Механічні характеристики полімеру LDPE вивчаються з використанням випробувань на міцність на розрив та подовження при розриві. Бібл. 27, табл. 4, рис. 6.

Ключові слова: **поліетилен низької щільності, нанонаповнювач, мікронаповнювач, діелектрична міцність, термічне старіння, термогравіметричний аналіз.**

Introduction. Super insulating polymers are commonly employed in high voltage insulators, particularly in high voltage cables. Thermal oxidation processes may occur for the insulation layers in contact with the cable core due to the high working temperature of the cable (around 90 °C) because of loading or overloading for short durations, resulting to insulation degradation and even failure. As a result, many researchers have been interested in the ageing and insulating properties of polymers in this environment [1, 2]. To create materials with better electrical and thermal properties, nanofillers were chosen to be added to polyethylene due to the high surface area presented to the matrix [3, 4]. Interestingly, with several weight percents of nanoparticles, PE-based nanocomposite can promote insulation properties effectively, which could be attributed to the nanoparticle-matrix interface [5, 6].

According to existing research on polyethylene insulating materials, filling nanoparticles can reduce the creation of space charge and enhance the dielectric, mechanical, and thermal properties of polyethylene [7-9]. Numerous studies make use of inorganic filler oxides like MgO, SiO_2, TiO_2, BN, etc., as well as how the improvement of polymer properties is impacted by the grain size of the filler (nano or micro) [10-12]. When evaluating the future application of polyethylene nanocomposites, the extended service life of insulating materials cannot be overlooked. Thermal ageing has been shown to have a major impact on the qualities of polyethylene materials, with numerous modifications possible, including variations in physicochemical parameters and microstructure [13]. During thermal ageing, several oxygenated compounds of low molecular weight may form in polyethylene, which may have a major influence on the space charge behavior of polyethylene insulating material [14, 15].

The purpose of this paper was to determine whether Al_2O_3 nano- and micro-particles, which have been shown to improve the dielectric strength of LDPE composites, can maintain these electrical properties after thermal ageing. To conduct thermal ageing tests, we chose four different percentages of nano composites and four different percentages of micro composites. The thermal properties of composites after thermal ageing were investigated using the thermogravimetric analysis (TGA) test. The dielectric strength test was used to evaluate the electrical characteristics of LDPE/Al_2O_3 composites after thermal ageing and the anti-thermal ageing mechanism offered by nanoparticles.

Literature review. Thermal deterioration of LDPE has been investigated. Chemical and electrical testings were performed on LDPE plaques that had been thermally stressed at high temperature (110 °C). Changes in the imaginary component of the dielectric constant have been connected to contributions from oxidation and morphological changes inside polymers. This comparison may serve as the starting point for the creation of non-destructive methods for electrical measurements-based polymer diagnostics [16, 17].

Nanoparticles improve the anti-thermal ageing capability of PE-based nanocomposites. The three metal oxides – magnesium oxide (MgO), zinc oxide (ZnO), and silicon dioxide were combined to form nanocomposites with a 1 wt.% concentration in each. Fourier-transform infrared spectra revealed that LDPE/MgO nano filler composites had the best anti-thermal ageing performance when compared to LDPE/SiO_2 nanocomposites, which had the worst using dielectric characteristics and space charge dispersion. The capacity of nanocomposites to maintain electrical properties was then investigated [18].

The zeolite/LDPE nano filler samples were produced and thermally aged to produce samples with varying ageing times. It was demonstrated that nano-zeolite doping may be an efficient way to stop the internal structure of the nanocomposite from being damaged by thermal ageing; during thermal ageing, carbonyl and hydroxyl levels considerably decreased and crystallinity greatly increased. The nanocomposite's shape and ageing resistance were greatly enhanced by nano-zeolite doping. It was discovered during the dielectric strength test that nano doping may significantly increase DC and AC breakdown field strength and stability during thermal ageing. Nanocomposite's dielectric constant can be decreased, and the rate of dielectric loss did not alter noticeably as the material aged [19].

During thermal ageing, the crystallinity and space charge accumulation characteristics of pure LDPE and LDPE/TiO_2 samples were assessed using a pulse electro-acoustic method system and a differential scanning calorimeter. It was determined that TiO_2 nano filler may increase LDPE crystallinity, and the capacity of LDPE/TiO_2 to reduce space charge was substantial at a TiO_2 mass concentration of 1 %. Furthermore, thermal ageing can degrade the microstructure and impair material crystallinity, increasing the sources of space charge [20].

Methods. Sample preparation. The basic polymer utilised was additive-free LDPE with a particle diameter of less than 0.2 mm on average, a melt flow index of 2 g/10 min at 190 °C, and a density of 0.91-0.925 mg/cm^3. It was purchased from SABIC, KSA, in the form of granules. The nano particles used were Al_2O_3 inorganic fillers with two different grain sizes (micro filler with a 60 µm particle size and nano filler with a 50 nm particle size). In a twin-screw extruder at 448 K, LDPE and filler particles were melt-blended. Nanocomposites were made in concentrations of 1 wt.%, 3 wt.%, 5 wt.%, and 7 wt.%, and micro composites were made in concentrations of 10 wt.%, 20 wt.%, 30 wt.%, and 40 wt.%. Composite samples were press-moulded at 433 K and at a pressure of 10 MPa to produce sheets with dimensions of 150 mm by 150 mm and a thickness of about 1 mm.

Samples were thermally aged in a fixed-temperature vacuum oven at 25 °C, 60 °C, 100 °C, and 120 °C at regular intervals (0, 10, 20, and 30 min). The aged samples were subjected to dielectric strength testing.

Table 1 shows the weight of the LDPE composite mixture (g) used in the manufacture of samples. It also shows the added weight of the LDPE polymer and the added weight of the Al_2O_3 filler at each mixing ratio.

Table 1

Different samples compositions

Sample		Symbol	LDPE, g	Al_2O_3, g
LDPE		B	150	0
LDPE + micro Al_2O_3	10 %	M10	135	15
	20 %	M20	120	30
	30 %	M30	105	45
	40 %	M40	90	60
LDPE + nano Al_2O_3	1 %	N1	148.5	1.5
	3 %	N3	145.5	4.5
	5 %	N5	142.5	7.5
	7 %	N7	139.5	10.5

Thermal ageing test. Heat causes some physical and chemical changes in polymers. These variations are determined by the severity of the temperature and the length of exposure. High temperatures do not always cause the polymer material to decompose. Prolonged exposure to high temperatures, on the other hand, will cause gradual changes in physical properties, eventually leading to collapse. The cable is subjected to overloads and short-circuit currents. When the current exceeds the rated value, it raises the temperature of the core. The electrical performance of the cable is affected by repeatedly exposing the insulation layers adjacent to the core to high temperatures. Thermal ageing of the samples is performed to determine the effect of temperature increases on the dielectric strength value. Thermal ageing was tested according to [21].

The required procedures and precautions to obtain highly accurate readings for each sample during the thermal ageing test are as follows:

- Before beginning thermal ageing tests, clean and dry the samples.
- The samples were heated to different temperatures (25 °C, 60 °C, 100 °C, and 120 °C) during specified periods of time (0, 10, 20, and 30 min) using a fixed-temperature oven, as shown in Fig. 1.
- As shown in Fig. 2, the dielectric strength of the aged samples was measured. The measurement was repeated ten times with three samples exposed to the same conditions, and an average value was taken for the experimental readings.
- The time intervals between successive tests of each sample should be suitable and sufficient.
- To apply electrical safety requirements, all testing circuit linkages must be correct.
- The voltage was gradually raised until the voltage breakdown occurred at a nearly constant rate of 2 kV/s. The dielectric strength was recorded.

Fig. 1. Fixed temperature oven

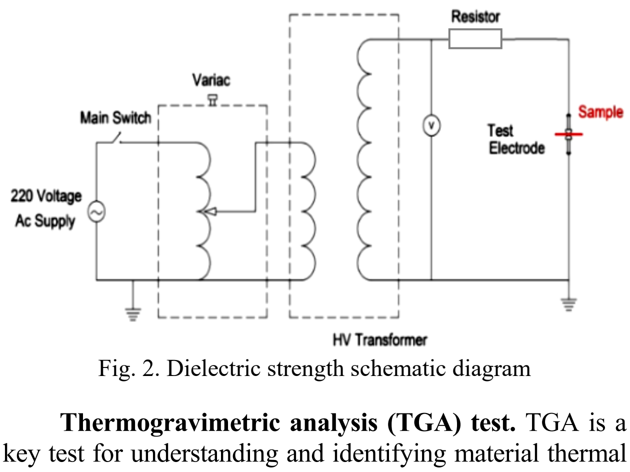

Fig. 2. Dielectric strength schematic diagram

Thermogravimetric analysis (TGA) test. TGA is a key test for understanding and identifying material thermal characteristics. TGA is a thermal test that calculates the weight loss of volatile components with temperature rise uniformly. According to [22], based on weight loss at high temperatures as well as thermal stability in a brief period, it is a great approach for figuring out the filler and polymer content. The TGA test was carried out in the NSI nitrogen environment using Perkin-Elmer equipment [23-26]. Approximately 10 mg of Al_2O_3/LDPE samples were sliced and heated from 35 °C to 700 °C while the samples were weighed and shown on the computer screen.

Mechanical analysis. Tensile strength (MPa) and elongation at break (%) are critical metrics for characterizing polymer mechanical performance and determining the influence of inorganic filler. A Zwick Roell LTM electrodynamic testing device was used to assess the tensile strength (MPa) and elongation at break (%) of the composite specimens. A schematic representation is shown in Fig. 3. The test results were evaluated using [27].

Results and analysis. Thermal ageing measurements. Table 2 shows average results for the dielectric strength of LDPE loaded with varied percentages of Al_2O_3 inorganic filler thermally strained over different time intervals at different temperatures.

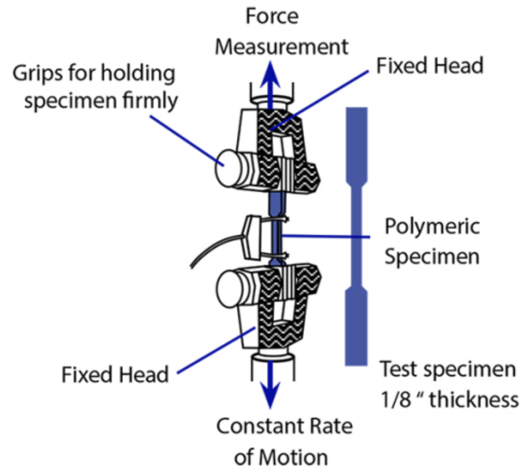

Fig. 3. Schematic diagram for measuring the tensile strength of LDPE composite samples

Table 2 shows that, when compared to neat and micro filled with LDPE, all LDPE micro composites have increased dielectric strength. When compared to other concentrations of the same particle size of filler, the micro Al_2O_3 composite of 30 % has the highest dielectric strength value.

Table 2

Average dielectric strength for micro- and nano-Al_2O_3 composite samples thermal stressed at 25 °C, 60 °C, 100 °C, 120 °C for different times 10 min, 20 min, 30 min

Sample	Average dielectric strength, kV/mm											
	at 25 °C			at 60 °C			at 100 °C			at 120 °C		
	10 min	20min	30min	10 min	20min	30min	10 min	20min	30min	10 min	20min	30min
B	20.45	18.28	17.08	18.33	16.02	15.11	14.61	12.08	11.21	12.77	11	9.89
M10	25.81	25.27	24.71	22.83	22.29	21.74	20.38	19.85	19.29	16.55	16.02	15.46
M20	28.59	28.17	27.64	25.66	25.21	24.69	22.46	22.01	21.49	18.04	17.59	17.07
M30	32.67	32.25	31.74	30.54	20.09	29.58	27.34	26.91	26.39	23.94	23.49	22.95
M40	29.77	29.25	28.7	26.59	26.06	25.5	24.00	23.47	22.97	21.09	20.55	20.01
N1	26.89	26.69	26.12	25.81	24.38	24.00	23.11	21.83	21.11	20.39	19.08	18.27
N3	29.09	27.84	27.06	26.55	25.28	25.00	24.25	23.54	22.86	22.35	20.89	20.03
N5	35.61	35.18	34.68	34.31	33.72	33.04	32.04	31.32	30.85	29.67	28.86	28.3
N7	39.85	39.34	38.79	36.96	35.12	34.56	34.37	32.89	32.31	31.85	30.34	29.74

Table 2 also shows that the electrical properties of LDPE composites filled with Al_2O_3 are reliable at 7 % nano particle size and have a dielectric strength greater than that of micro Al_2O_3.

As shown in Table 2, the dielectric strength of nano Al_2O_3 composites is greater than that of micro Al_2O_3 composites at all concentrations. By increasing the filler concentration, the LDPE composite achieves maximum dielectric strength. The dielectric strength decreases when the filler concentration exceeds critical values.

The physical properties of the samples, such as shrinkage and deformation, are affected by continuously raising the temperature (Fig. 4). High temperatures reduce dielectric strength. Lower filler contents, such as in nano composite, can result in greater flexibility, ease of processing during product manufacturing, and improved electrical performance of polymers.

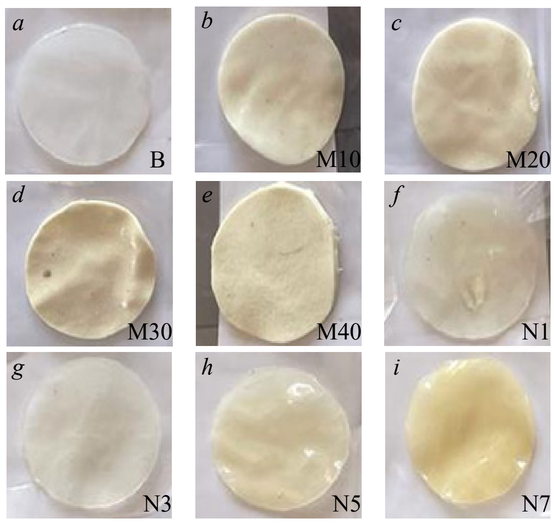

Fig. 4. A photograph of the samples after 30 min of exposure to a temperature of 120 °C

Thermogravimetric analysis. TGA provides the variation in the weight of sample loss with respect to temperature in a controlled environment. The release of moisture or gases from the material's breakdown causes weight loss as the temperature rises. TGA provides ageing stability information within short test times.

TGA analyses of the samples were done to comprehend the thermal performance. TGA of samples with various micro Al_2O_3 loadings is displayed in Fig. 5.

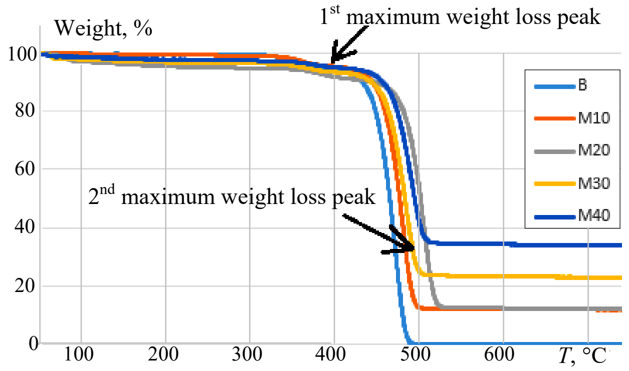

Fig. 5. TGA for LDPE with different micro Al_2O_3 filler concentration

Temperature has no discernible impact on weight for all samples in the temperature range of 35 °C to 450 °C, as demonstrated in Fig. 4. The 1st maximum weight loss peak on the TGA curve above 450 °C is caused by Al_2O_3 filler water loss. The burning of the LDPE side chains may be the cause of the 2nd maximum weight loss peak. When the temperature was raised from 35 °C to 700 °C, the weight loss of pure LDPE was the least. When compared to all composite samples and a blank one, the weight loss of composites filled with 40 wt.% micro Al_2O_3 provides the highest thermal stability.

Figure 6 studies the effect of thermal stability on LDPE samples filled with various concentrations of nano Al_2O_3 filler to determine changes in the weight of a sample in relation to changes in temperature. Thermal stability is the ability of a polymeric material to withstand the effects of heat while preserving its properties, such as toughness, strength, or elasticity, at a certain temperature.

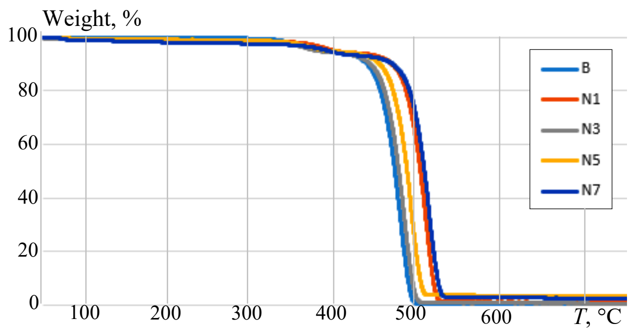

Fig. 6. TGA for LDPE with different nano Al_2O_3 filler concentration

Mechanical test results. Studying the tensile strength of LDPE composite samples. The maximum stress that a material can sustain when being stretched or pulled before necking is known as tensile strength. The load at break is divided by the initial minimum cross-sectional area to determine tensile strength.

The results from 3 samples of each test have been averaged to reduce error because the sheet's structure is not homogeneous and because the sheet (20 cm × 20 cm) obtained through mixing is not. Table 3 displays the tensile strength of micro and nano Al_2O_3 filled LDPE composites as a function of filler loading.

Table 3

Tensile strength (MPa) for nano and micro Al_2O_3/LDPE composites

Sample	Tensile strength, MPa			Average values of tensile strength
	1st	2nd	3rd	
B	10.27	6.73	7.4	8.13
N1	9.83	8.61	12.24	10.23
N3	10.83	12.75	9.23	10.93
N5	11.88	11.27	10.96	11.37
N7	11.36	14.32	11.91	12.53
M10	11.25	10.13	10.19	10.53
M20	10.32	12.31	12.75	11.8
M30	10.19	12.10	12.3	11.75
M40	12.21	10.92	10.41	11.18

As shown in Table 3, LDPE composites with Al_2O_3 loading improve tensile strength at a 7 wt.% loading level. As the amount of micro Al_2O_3 increases, the tensile strength of LDPE composites decreases in all values.

Table 3 indicates that increasing micro Al_2O_3 concentrations result in a substantial improvement in tensile strength. In comparison to the other concentrations for the same filler, LDPE loaded with 7 wt.% nano Al_2O_3 records the highest tensile strength.

Studying the elongation at break of LDPE composite samples. Table 4 displays the elongation at break characteristics off LDPE composites with micro- and nano sized Al_2O_3 loadings.

Table 4

Elongation at break for nano and micro LPDE/Al_2O_3 composites

Sample	Elongation at break (Ɛ-F max %)			Average values of elongation at break
	1st	2nd	3rd	
B	110.32	101.8	103.52	105.21
N1	116.54	114.36	109.65	113.2
N3	129.23	117.65	121.39	122.7
N5	142.5	135.62	136.85	138.32
N7	144.92	136.42	140.85	140.73
M10	105.52	106.58	113.52	108.54
M20	120.65	125.02	119.54	121.7
M30	112.65	118.2	111.95	114.3
M40	102.35	100.65	98.9	100.63

For LDPE composite samples, the values of elongation at break decrease as the amount of micro Al_2O_3 increases.

Table 4 demonstrates how the addition of nano Al_2O_3 can enhance elongation at break. When nano Al_2O_3 is added at a 7 wt.% concentration, the composite exhibits greater break elongation than pure LDPE.

Discussion. By interfering with the polymer crystal and filling spaces and gaps, an inorganic filler – whether micro or nanosized – works to improve the fundamental polymer's electrical, mechanical, and thermal properties.

Heat exposure of the insulator throughout various operating situations reduces the cable's lifespan. It is critical to conduct a thermal ageing test to determine the effect of temperature and exposure duration on the value of the polymer's dielectric strength. According to the results of the thermal ageing test, the value of the insulating strength declined as the time of temperature exposure increased.

Compared to the percentage of micro filler, a small amount of nano filler gave better results in dielectric strength and tensile strength. Dielectric properties of LDPE composite loaded with Al_2O_3 are reliable at 7 wt.% nano scale and have maximum dielectric strength.

Conclusions. This paper demonstrated the effect of adding micro- and nano- Al_2O_3 to Low Density Polyethylene (LDPE) composites. The experimental results lead to the following conclusions:

- The physical properties of the samples are affected when they are exposed to high temperatures for extended periods of time. This effect causes deformations in the samples, which cause them to become more solid.
- The high temperature and the length of time that the samples are exposed to high temperatures have a negative impact on their dielectric strength.
- The thermal ageing has been decreased by adding Al_2O_3 filler to LDPE.
- With the addition of nano filler, the electrical performance has been greatly enhanced. Lower filler contents, as in nano composite, can contribute to greater flexibility, ease of processing during product manufacturing, and improved thermal ageing performance of samples.
- The optimal Al_2O_3 filler concentration for reducing thermal ageing in LDPE composites is 7 % nano Al_2O_3.
- Thermogravimetric analysis (TGA) of nano composite outperforms that of micro composite. A7 wt.% nano Al_2O_3 filler composite provided the best dielectric strength and TGA.
- The addition of micro-Al_2O_3 filler reduced the mechanical properties of LDPE. By increasing the amount of nano-Al_2O_3 filler in the sample to 7 wt.%, the tensile strength (MPa) and elongation at break (%) characteristics are enhanced.

In the future, it is proposed to blend two or three fillers with LDPE and investigate their electrical, mechanical, physical, and thermal characteristics. It is also suggested that composite samples be immersed in water to investigate the effect of water leakage on the electrical characteristics of the insulator.

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Conflict of interest. The authors declare no conflict of interest.

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