



# Statistical Analysis of Mechanical Strength and Dielectric Breakdown of Polyester Nano-Alumina Composites

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## Abstract

The mechanical and electrical properties relation are very important, especially for brittle material. Nano Alumina-unsaturated polyester (PS/Al<sub>2</sub>O<sub>3</sub>) composites were prepared by casting PS with different percentages (1, 2, 3, and 4 wt%) of Nano alumina. Electrical breakdowns caused cracks to form around the breakdown point, and the length and quantity of cracks increased with dispersed powder added increases. The research involved determining Weibull modulus from an electrical strength test, and mechanical strength by piston-on-three-ball test. The results showed that Weibull modulus and dielectric breakdown increase during the rate of rising voltage (RRV) increases, especially 5 kV/s. the maximum Weibull modulus measured by the electrical breakdown was at a high RRV is 34.58 (PS/Al<sub>2</sub>O<sub>3</sub>). High RRV leads to electromechanical breakdown and electrothermal strength at low RRV. While the Weibull modulus by the piston-on-three-balls test is lower than the Weibull modulus calculated by electrical strength. Therefore, this mechanical test is more accurate in identifying defects that fail due to an increase in the affected area of the applied stress. Scanning electron microscope (SEM) images showing the homogeneous distribution of the powders within the polymeric matrix. Some pores were present in the structure of the composite despite the measures taken to increase the dispersion using ultrasound waves. Those pores caused fluctuating results in all electrical and mechanical tests.

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## 1. Introduction

Nanoscience is a large and comprehensive field of study that has grown substantially in recent years across the globe, and nanomaterials constitute the foundation of nanoscience and nanotechnology. [1] The matrix and reinforcement have a significant impact on the performance of nanocomposites. Polyester and alumina nanopowder, for example, is intended to increase flexibility, ductility, and processability, as well as stiffness and high thermal stability. Al<sub>2</sub>O<sub>3</sub> is a dielectric substance with a large band gap. It may occur in a variety of crystal forms, including gamma, delta, theta, and alpha [2][3]. Weibull Survival Probability Analysis has been created as an engineering design approach for composite and other materials components. A brittle component's failure

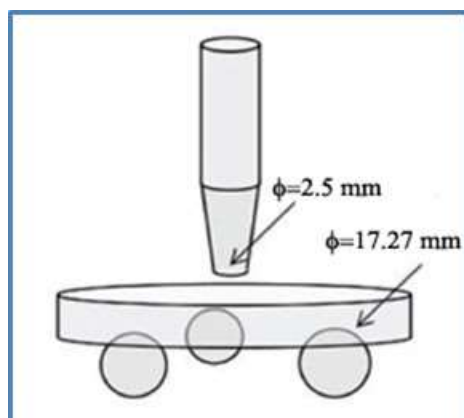
load is determined by the magnitude of the fault. As a result, it is a characteristic of the sample rather than a feature of the substance [4]. Because the faults have a size distribution, the failure load varies. Another way to put it is that under any given stress, a portion of the sample may survive. This score is defined by Weibull as the probability of survival ( $P_s$ ) under any tensile stress [5] as could be shown in Figure 1.

$$P_s = \exp \left\{ - \left( \frac{\sigma}{\sigma_0} \right)^m \right\} \quad (1)$$

Where,  $\sigma$ : maximum strength ( $\sigma = \sigma_E$  for Electrical Strength,  $\sigma = \sigma_M$  when the mechanical strength),  $\sigma_0$  = strength at survival probability is:  $(1/e) = 0.37$  (either  $\sigma_0 = \sigma_{0E}$  for Electrical Strength or  $\sigma_0 = \sigma_{0M}$  for Mechanical Strength). To determine the Weibull modulus, the natural logarithm must be taken for both sides of equation (1).

$$\ln \left( \ln \left| \frac{1}{P_s} \right| \right) = m \ln \sigma - m \ln \sigma_0 \quad (2)$$

The slope ( $m$ ) of the straight line might be calculated by drawing the connection between  $\ln(\ln|1/P_s|)$  and  $\ln M$ . The lesser the strength change, the greater the Weibull modulus value. [6] The relationship between electrical and mechanical characteristics is an important necessity in material assessment. [7] The dielectric breakdown is divided into two types: 1. Volume distribution 2. Surface deterioration [8] The inability of a dielectric to withstand an applied electric field is referred to as dielectric breakdown [9]. In the case of a solid, the breakdown mechanism is a complex phenomenon and changes with the time of the applied voltage. Different breakdown mechanisms can be divided into the intrinsic breakdown, electrothermal breakdown, electromechanical breakdown, streamer breakdown, and erosion breakdown [10]. The main purpose of this research is to determine the conditions under which the measured Weibull modulus can be used to characterize the dielectric structure and the homogeneity of the brittle polymer composites dispersed by alumina nanoparticles through electrical and mechanical methods.

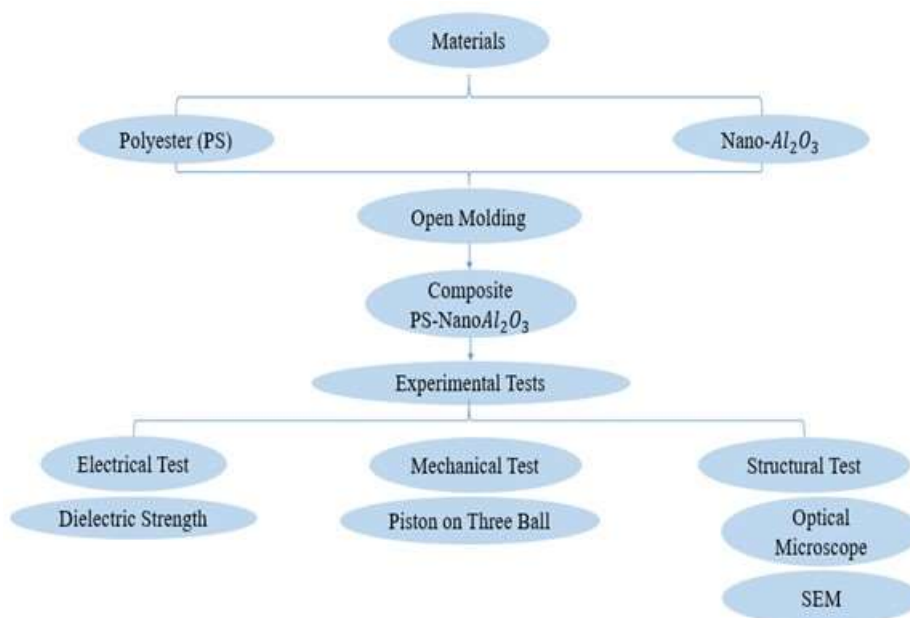


**Figure 1:** Piston-on-three ball biaxial flexural strength test. [11].

## 2. Experimental Procedure

### 2.1 Sample Preparation

The polymer-based nanocomposite employed in this study is composed of unsaturated polyester (manufactured by B-CHEM, MARMOLIT, Italy) with a hardener of 2 wt percent Di Benzoyl Peroxide Catalyst Paste. The fillers are nano Aluminum oxide (alumina) powder provided by Sinopharm Chemical Reagent Co., Ltd., with a purity of 99 percent and particle size of 20-30 nm. Various amounts of nano  $Al_2O_3$  were used (1, 2, 3 and 4 wt percent). Meanwhile, the mixture was stiffened in a silicone mold immersed in an ultrasonic water bath at 27 degrees Celsius. The experiment procedure could be summarized in Figure 2.



**Figure 2:** Methodology of Procedure.

## 2.2 Dielectric Strength test

Using a German high-voltage provider, the dielectric breakdown was measured in the range (0-60 kV) and frequency (50 Hz) (BAUR-PGO-S-3). The breakdown has been tested for various rising voltage rates (RRV) (0.5, 1, 2, 3, and 5 kV/s). Take 10 points for each RRV and then average them. The electric field may be used to determine dielectric strength, where  $E$  denotes the dielectric material's fail field:

$$\sigma_E = \frac{V_{br}}{t} \quad (3)$$

$V_{br}$  : the max. voltage applied to the dielectric, and  $t$  is the thickness of the insulating material. The dielectric strength of a solid dielectric is affected by several factors, like test Durand ation, impurities, or structural defects, humidity, ambient temperature, and whether it is direct current or alternating current. [12]

## 2.3. Mechanical Strength Test

Piston-on-three-ball test was used to calculate the mechanical strength (Fig 1), for 10 samples and the radius of the support ball circle was 17.27mm, the radius of the ball used the loading surface 2.5mm, and the sample's radius 20mm. The device provides max. force value, and the mechanical strength calculated by Equation 4. The piston-on-three-ball test is carried out by a piston-to-three-ball device, which approved the standard test (ASTM F394 in ISO-6872) in 1991 and calculates the strength ( $\sigma_M$ ) of all samples through this test [13].

$$\sigma_{biaxial} = \frac{3P(1-\nu)}{4\pi t^2} \left[ 1 + 2 \ln \frac{a}{r_0^*} + \frac{1-\nu}{1+\nu} \left( \frac{2a^2 - r_0^{*2}}{2c^2} \right) \right] \quad (4)$$

$$r_0^* = \sqrt{(1.6r_0^2 + t^2) - 0.675t} \quad (5)$$

Where,  $P$  is the total loading fracture (N),  $\nu$  the poisson's ratio (0.24 for all composites) [14],  $a$  the radius of the support ball circle,  $r_0$  radius of the ball used the loading surface,  $c$  sample's radius and  $t$  thickness of samples.

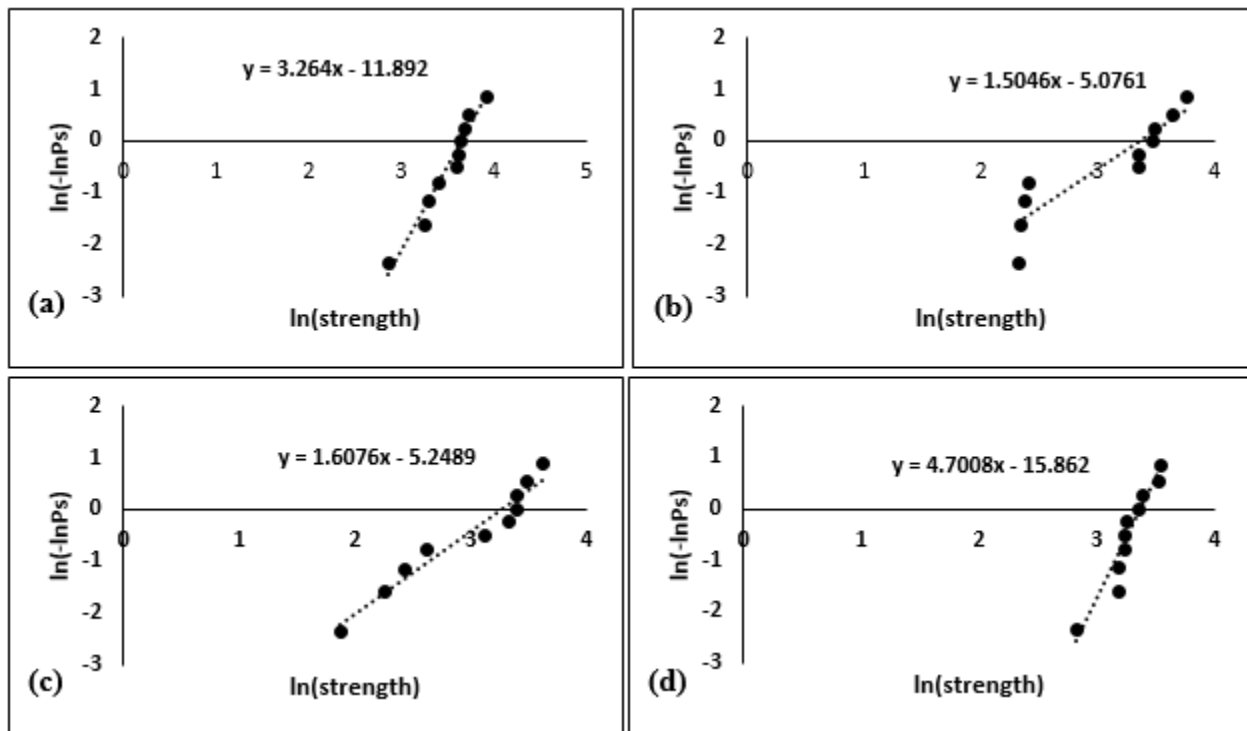
## 2.4. Optical Microscope Test

Using an optical microscope of a type ((MEIJI) Japan), which is equipped with a digital camera connected to a

computer that contains different magnification powers (40X, 100X, 400X),

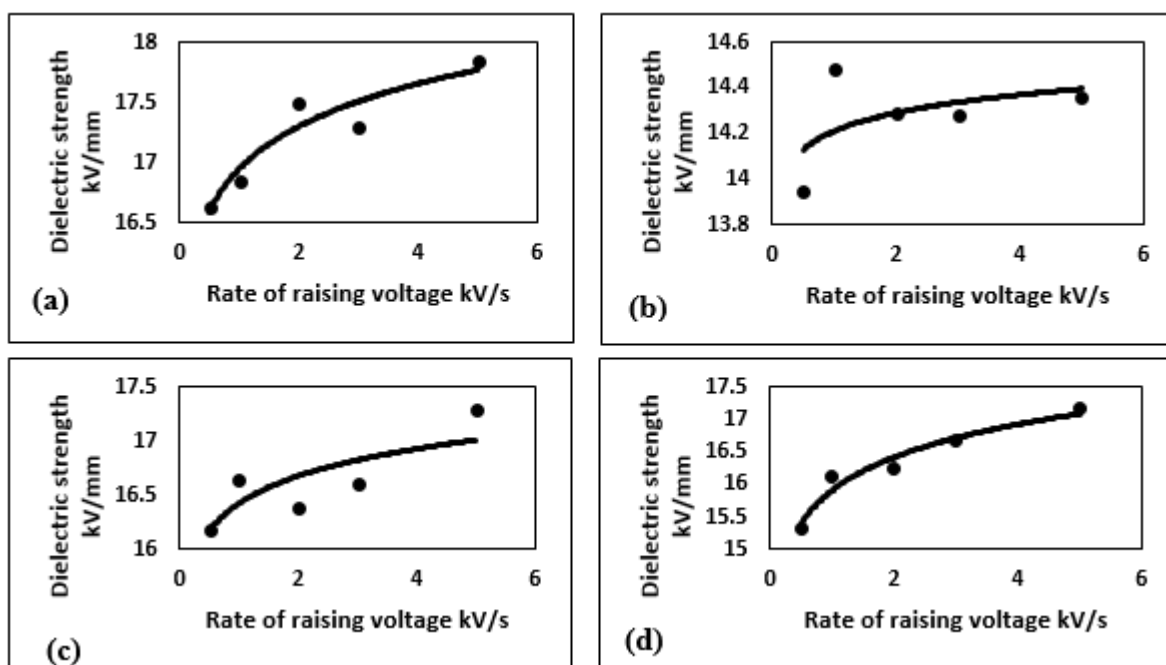
### 3. Results and Discussion

The strength of a piston-on-three-ball is a kind of mechanical strength; it is also a feature of resistance evaluation, as it may replicate the material's tensile strength weakness. [15] The Weibull modulus is one of the greatest statistical approaches for describing randomness, and "changes in observed values may be detected." The results of the tests on the polyester-nano- $\text{Al}_2\text{O}_3$  composite samples are shown in Figure 3. They also illustrate the change in the piston-on-three-ball strength value on the x-axis, which is a logarithm of the maximum mechanical strength ( $\ln |\sigma E|$ ) at which mechanical failure occurs. The y-axis depicts the samples' survival probability without violating the log-log inversion of  $\ln(-\ln P_s)$ . The relationship ( $P_f = 1 - P_s$ ) may be used to determine the likelihood of failure. The slope of the line indicates the Weibull modulus values ( $m=3.264, 1.5046, 1.6076, \text{ and } 4.7008$ ). The pistons on three balls obtained this strength value (Figure 1). The RRV is the most critical metric in the dielectric breakdown test. The magnitude and nature of the electrical breakdown may help identify and explain why the value changed. As seen in Figure 4, the dielectric strength rises as the RRV increases. The low RRV indicates that the heat generated between the electrodes (the sample contact area) rises as the leakage current increases. Additionally, the cumulative impact of electrochemical transformation and corrosion caused by the heating process will degrade the material's structure and increase dielectric breakdown. When utilized as a high voltage insulator, the electrical breakdown value at a low enhancement rate indicates the sample's working value. Additionally, when the increasing voltage is greater, the breakdown value reflects the instant the circuit is opened or closed. [16]



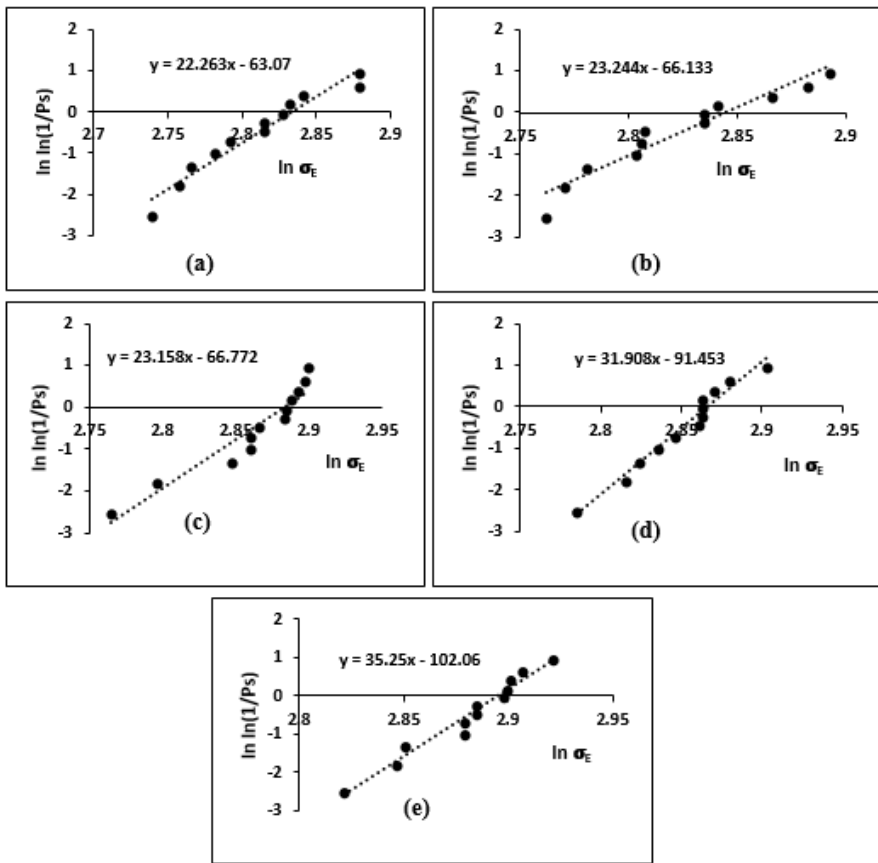
**Figure 1:** Weibull Distribution by piston-on-three-ball test (a) 1 wt%  $\text{Al}_2\text{O}_3$ , (b) 2 wt%  $\text{Al}_2\text{O}_3$ , (c) 3 wt%  $\text{Al}_2\text{O}_3$ , (d) 4 wt%  $\text{Al}_2\text{O}_3$ .

Electrical breakdown occurs with a low RRV and is referred to as "electro thermal breakdown," while electrical breakdown occurs at a high RRV and is referred to as "pure electrical breakdown." [17] In both circumstances, damage will occur between the sample's "contact point" and the electrode, which may be observed with the naked eye or with the use of a basic optical microscope. Microcracks arise as a result of damage, as seen in Figure 4.

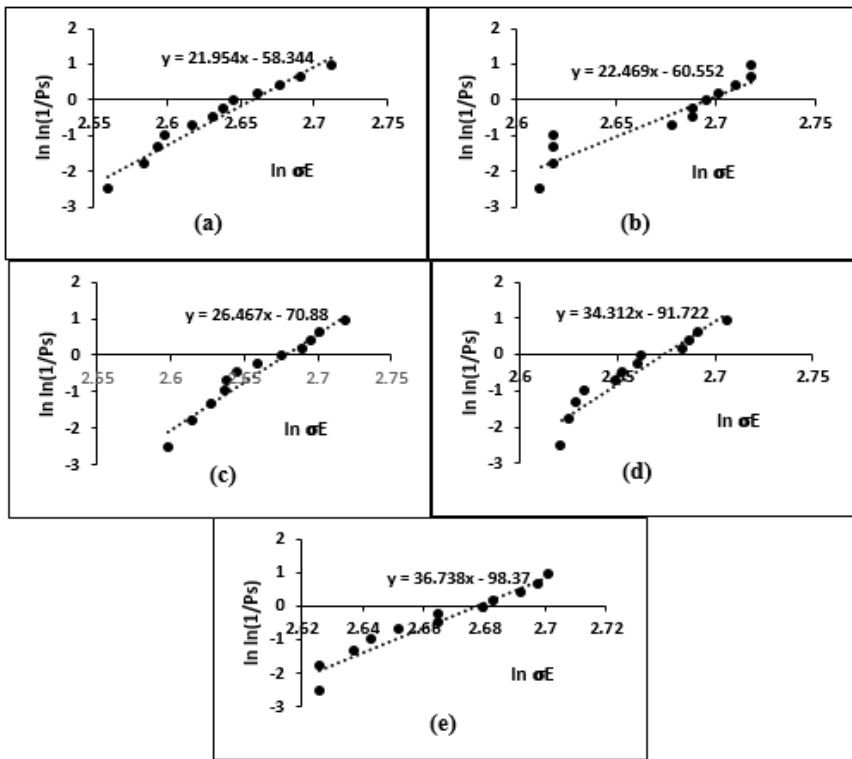


**Figure 2:** Variation of dielectric strength with RRV for (a) 1 wt% Al<sub>2</sub>O<sub>3</sub> (b) 2 wt% Al<sub>2</sub>O<sub>3</sub> (c) 3 wt% Al<sub>2</sub>O<sub>3</sub> and (d) 4 wt% Al<sub>2</sub>O<sub>3</sub>.

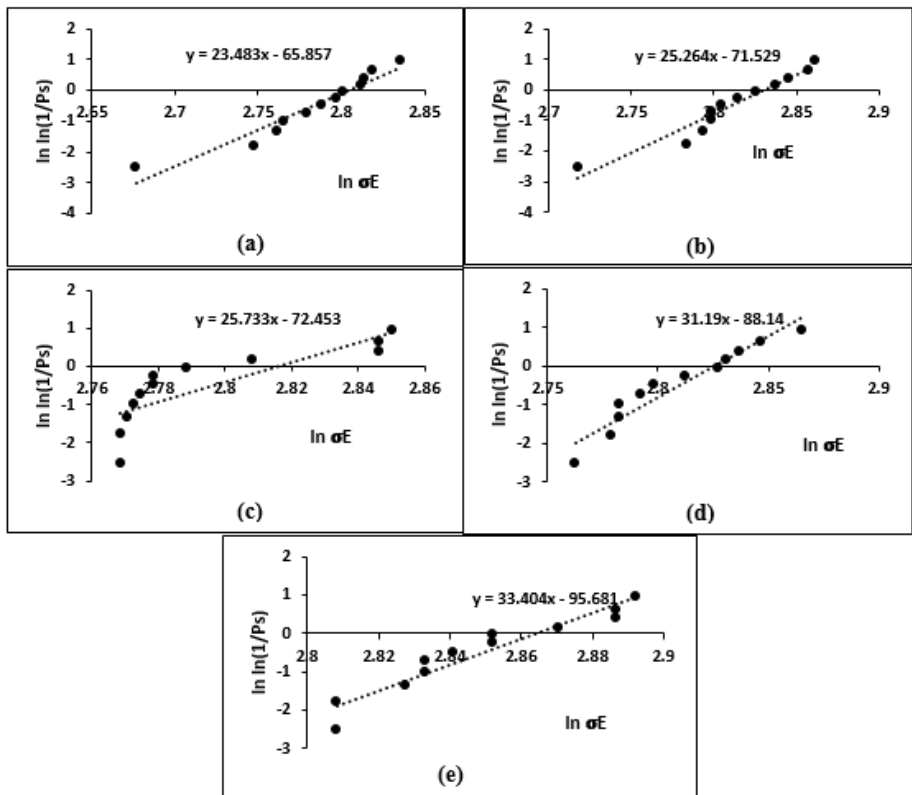
Figure 5 shows the change of survival probability with dielectric breakdown under different RRV. Electric strength is mainly related to changes in electrical energy and thermal energy. The high temperature associated with the voltage can cause a breakdown. The Weibull modulus can be calculated from the slope of the straight line. In the comparison between Figures 5, 6, 7, and 8, we noticed that the Weibull modulus of the nano-Al<sub>2</sub>O<sub>3</sub> additive is different for the same RRV. The difference in Weibull modulus values means that the repetitive value of the result is small, in other words, the measure of the maximum value of the electrical field in the composite sample has a huge change. It demonstrates that two approaches enhance the degree of breakdown point damage: the first method involves electrochemical changes, which are visible at (0.5, 1 and 2kV/s), and the microcracks are small and will not shatter the sample. The second one indicates that when the RRV is increased to 3 kV and 5 kV/s, it was noted that no melting zone appeared, and only microscopic cracks appeared due to electromechanical breakdown, which is similar to cracks arising from mechanical impact. Increasing the percentage of alumina added to 4%, the samples showed areas of damage on the surface with a lesser percentage of low additives. The reason for this is that the alumina nanoparticles hinder the growth of micro cracks resulting from the breakdown in addition to the increase in the melting temperature of the composite. The Weibull modulus increases as the RRV increases. The reason is the thermal effect of the slow RRV, which lead to increased leakage currents due to the reduction of the energy gap of the dielectric material [18]. The surface morphology of the polyester-alumina samples is presented in Figure 9. Significant damage areas are observed at the boundary of the electrical breakdown area, especially in the case of adding 1% at low voltage rise rates (0.5kV/s). Damage may cause perforation of the sample due to the melting of the contact point between the electrodes and the sample surface as shown in Figure 9. In the case of an electromechanical breakdown, the scattering is evident around the point of the electrical breakdown. Also, increasing the percentage of nano-alumina reduces the damage size. Figure 10 shows the shape of the specimen after fracture by the piston-to-three-ball method. The geometric regularity of the parts means the homogeneity of the structure of the compound material. It can be seen that the damage is greater in the case of electrothermal breakdown. Table 1 lists all Weibull modulus values of samples with different RRV.



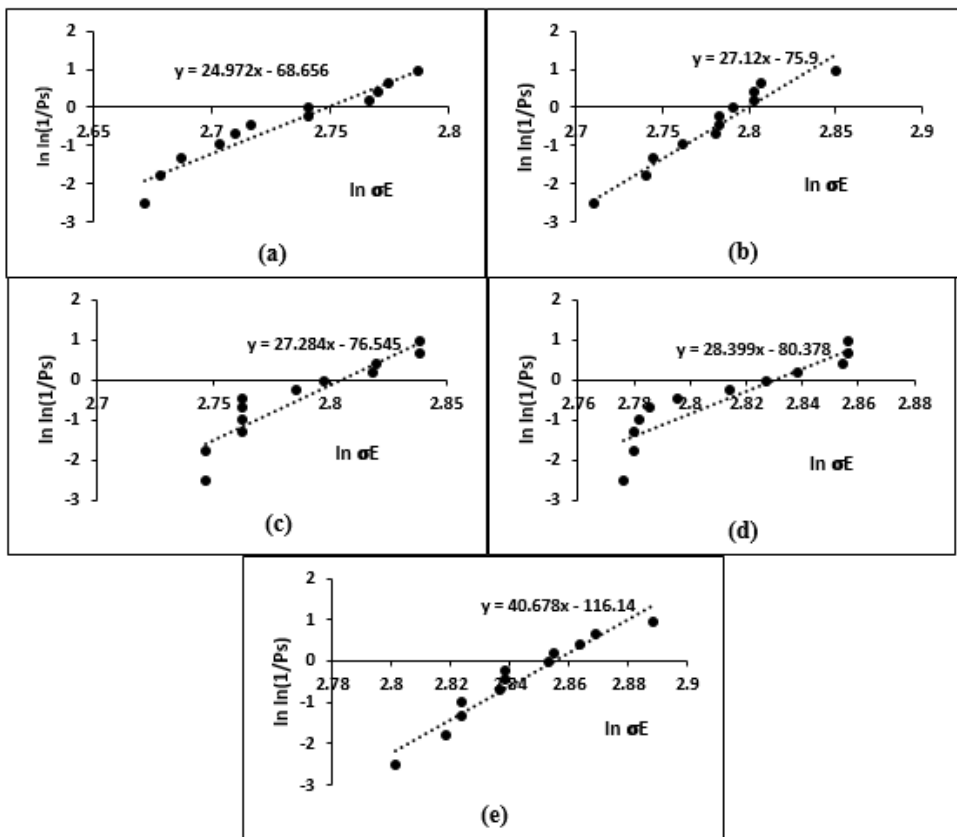
**Figure 3:** Weibull distribution of dielectric strength at various RRV: (a) 0.5, (b) 1, (c) 2, (d) 3 and (e) 5 kV/s, for 1 wt% Al<sub>2</sub>O<sub>3</sub>.



**Figure 4:** Weibull distribution of dielectric strength at various RRV: (a) 0.5, (b) 1, (c) 2, (d) 3 and (e) 5 kV/s, for 2 wt% Al<sub>2</sub>O<sub>3</sub>.



**Figure 5:** Weibull distribution of dielectric strength at various RRV: (a) 0.5, (b) 1, (c) 2, (d) 3 and (e) 5 kV/s, for 3 wt% Al<sub>2</sub>O<sub>3</sub>.



**Figure 6:** Weibull distribution of dielectric strength at various RRV: (a) 0.5, (b) 1, (c) 2, (d) 3 and (e) 5 kV/s, for 4 wt% Al<sub>2</sub>O<sub>3</sub>.

Scanning electron microscope images (SEM), showing the surface morphology of the prepared samples. In addition to homogeneity clarification of the distribution of the components in the composite body (especially, the distribution of dispersed powders in the polyester polymer matrix). The dispersing effect of nano-alumina powder on the polyester matrix was better than that of other oxides. Homogeneous distribution of the powders was observed, especially in the percentage of 1%, as shown in Figure 11 a, with the presence of some pores. A clear change occurred in the surface morphology when the percentage of addition was increased up to 4%, and smaller granular aggregations were formed than when dispersing with other oxides [19][20], as shown in Figure 11 b. This means changing the course of the microscopic cracks as a result of applied mechanical or electrical stresses.

Table 1: Values of Weibull modulus for samples with different RRV.

Sample	0.5 kV/s	1 kV/s	2 kV/s	3 kV/s	5 kV/s
1 wt% Nano Al <sub>2</sub> O <sub>3</sub>	22.263	23.244	23.158	31.908	35.25
2 wt% Nano Al <sub>2</sub> O <sub>3</sub>	21.954	22.469	26.467	34.312	36.738
3 wt% Nano Al <sub>2</sub> O <sub>3</sub>	23.483	25.264	25.733	31.19	33.404
4 wt% Nano Al <sub>2</sub> O <sub>3</sub>	24.972	27.12	27.284	28.399	40.678

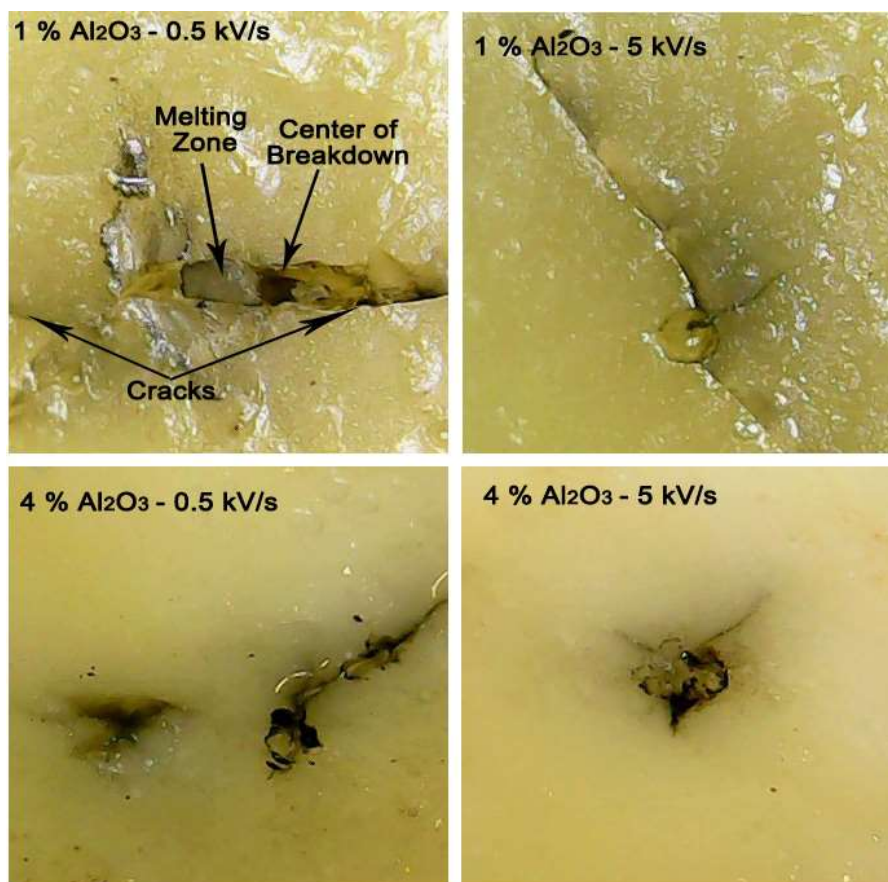
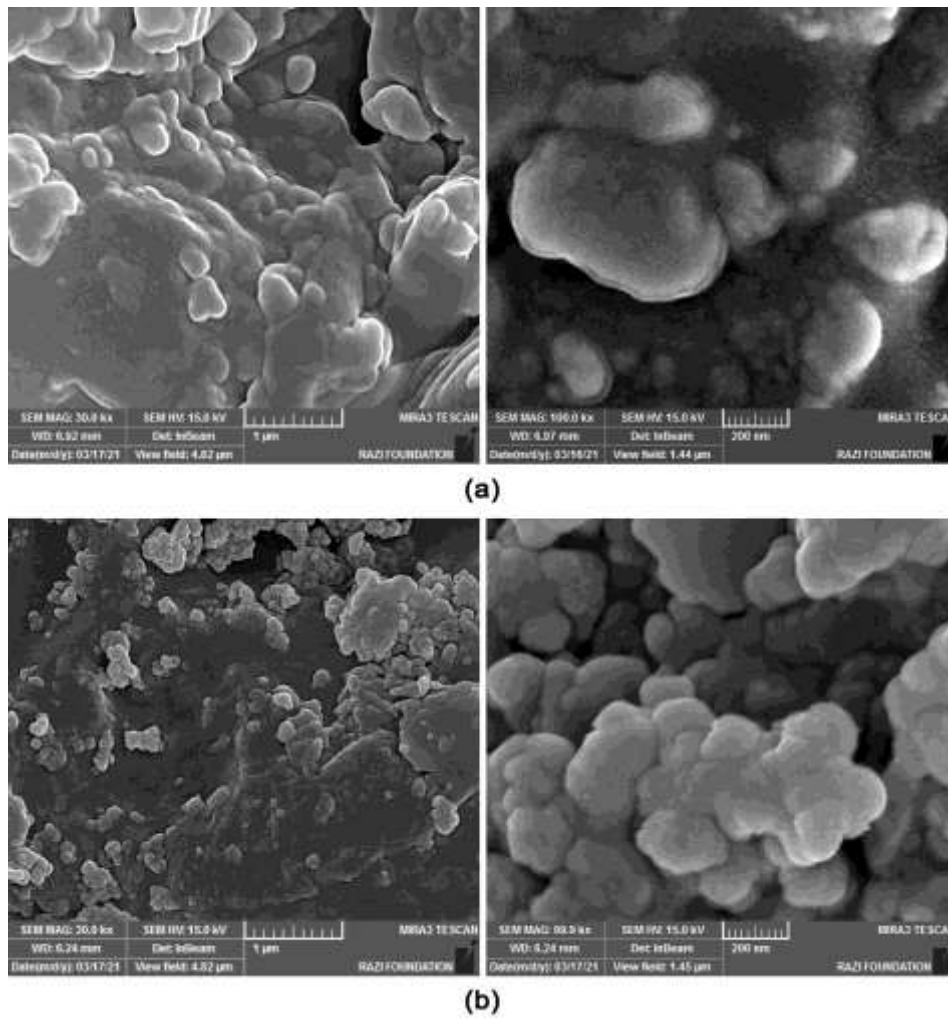


Figure 7: Trace of electrical breakdown damage for 1% Al<sub>2</sub>O<sub>3</sub> and 4% Al<sub>2</sub>O<sub>3</sub> with 0.5, 5 kV/s.





**Figure 8:** Broken specimen after pistons on three balls test.



**Figure 9:** SEM image of PS with (a) 1%  $\text{Al}_2\text{O}_3$  and (b) 4%  $\text{Al}_2\text{O}_3$  composites.

#### 4. Conclusions

The values of Weibull modulus measured for the composites using electric field were much higher than those measured by applying mechanical stress fracture. The increase in the Weibull modulus means an increase in the homogeneity of the distribution of the powder dispersed within the composites. The calculated Weibull coefficients in the case of mechanical strength correspond to the Weibull coefficients measured in the case of electrical breakdown measured at high RRV. High RRV means that an electromechanical breakdown has occurred in the dielectric, and therefore some interrelationships can be found between the mechanical and electrical properties. Heat is generated as a result of decreasing the RRV, which leads to the appearance of dangerous damage to the samples that can be considered destructive. The granular agglomerations that occurred as a result of adding nano-powders, greatly affected the surface morphology as well as the insulator properties.

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#### Conflict of Interest

The authors declare that they have no conflict of interest.

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