

# A study on influence of borax to polyester insulators

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## Abstract

*In this study the effect of borax on polyester insulators is investigated by evaluating the tracking and erosion resistance using the inclined plane test. The test procedure follows the ASTM D2303 standard. During the test, 50 Hz current was acquired from the ground electrode allowing a sampling rate of 48000 samples per second. The effect of borax concentration on the glass transition and the degradation temperature is studied by employing differential scanning calorimetry (DSC) analysis. The addition of borax to the unsaturated polyester resulted in an increase in the glass transition temperature, while the addition of 0.5% of borax by weight caused a decrease in the degradation temperature. The leakage current was processed using the Fast Fourier Transform (FFT) technique. The effect of borax on the degradation process of polymers was studied in a range of single harmonic components starting with the fundamental up to the 9th. The experimental data which was taken from borax added polyesters were shown to have higher harmonic components and lower fundamental leakage current than the pure polymer.*

**Key Words:** *Aging, boron compounds, flame retardants, polymers*

## 1. Introduction

Today polymers can be used at different voltage levels ranging from low to high with the help of additives and fillers. Polymers used in the electrical industry consist 50% of additives and fillers by weight. These additives can be flame retardants, UV stabilizers, curing agents, fillers, and such.

Flame retardants are additives that are added to the polymer system in order to prevent or delay ignition and fire. They can be halogens, non-halogens, metallic compounds or boron containing flame retardants. Brominated compounds and other halogen compound flame retardants have created an issue of concern due to their acute toxicity and long term side effects [1–8]. As a solution to this problem many researchers have attempted to find alternatives for these halogenated compounds. Aluminium trihydroxide (ATH) and silica are the most commonly used non-halogenic flame retardants in electrical industry. The formation of safe

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byproducts following a thermal decomposition of these additives makes them popular. However, the need for high concentrations of these additives is considered a major disadvantage [9–14].

Boric acid and borates have been used as flame retardants since early 1800s. However, until recent years phosphorous, halogens and several other materials were the compounds of interest that caught attention for investigation in detail rather than the borates. Boron compounds, such as zinc borates or calcium borates, are usually used in combination with other flame retardants and act as synergists for a variety of halogen flame retardants or of antimony oxide, as smoke suppressants [15–18].

Polyester is widely used in the electrical industry for its exceptional thermal, electrical or mechanical properties. Polyester resin mixed with several different fillers is becoming to be widely used throughout the electrical industry. It has also been reported that polyester resins are used with antimony trioxide and zinc borate in order to enhance their property as a flame retardant and the composites prepared with zinc borate were observed to yield successful results comparable to the performance of halogenated insulators [19]. Colemanite was reported to provide the most effective results among various borate compounds in the experiments that were conducted by the authors [20]. Also our previous studies based on the flame retardant properties of borax clearly indicate that below the flame onset temperature, borax forms a kind of glass sheet on the surface of the composite insulator, which eventually prevents the flame leading into the material [21].

This manuscript introduces an analysis of the different insulation behaviour of pure and borax added polyester specimens that were put through an artificial aging test method. The test samples were exposed to dry band arcing according to ASTM D2303 test standard [22]. The change in the thermal characteristics of polyester samples was studied using differential scanning calorimetry (DSC) analysis. The effect of borax concentration on the tracking and erosion resistance of polyester samples was studied by measuring the leakage current and then performing a Fourier analysis.

## 2. Materials and experimental setup

This study focuses on the electrical characteristics of borax added polyester through surface tracking tests. Surface tracking tests induce the contamination of the insulator via a contaminant applied on the surface of the insulator. Meanwhile, a conducting path is formed on the surface. This trace provides the surface characteristics of the organic insulator. Surface characteristics of polymers eventuate either through surface tracking or erosion. Tracking is the formation of a permanent conducting path, usually made of carbon, across a surface of insulation. Erosion is the deterioration of the insulation surface comparatively in a larger area than that of the tracking. In this case it roughens the surface and slowly penetrates the insulation material and at some stage will again give rise to channel propagation and tree-like growth through the insulation. The surface degradation phenomenon severely limits the use of polymer insulators in the outdoor environment. The rate of surface degradation depends upon the structure of the polymers and it can be drastically slowed down by adding appropriate fillers to the polymer which inhibits carbonization.

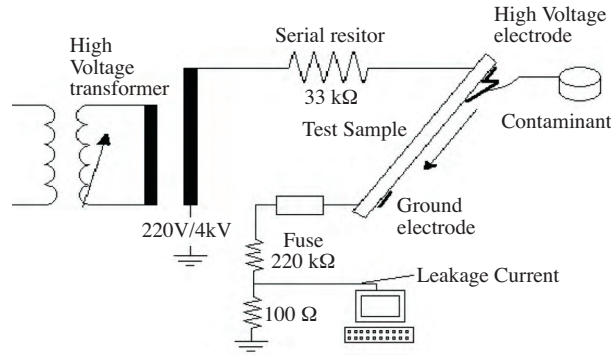
In this study, polyester was used as the base polymer and borax was selected as the fire retardant. The chemical formulation of borax is  $\text{Na}_2\text{O} \cdot 2\text{B}_2\text{O}_3 \cdot 10\text{H}_2\text{O}$  and consists of 36.5%  $\text{B}_2\text{O}_3$  and 47.2%  $\text{H}_2\text{O}$  by weight. Its high structural water content was another parameter making it desirable for selection.

Test specimens consisting 0.1%, 0.2%, 0.3%, 0.4%, 0.5%, 0.6%, 0.7%, 0.8%, 1.0%, 1.2% and 1.5% of borax by weight were prepared. The polyester samples have been prepared by using 0.25% MEKP (Methyl Ethyl Ketone Peroxide) and 0.25% cobalt as an accelerator. The borax mineral particles had a mean diameter of

less than 35  $\mu\text{m}$  and they were added to the unsaturated polyester resin. The final products with the dimensions of 100 mm by 55 mm by 9 mm were cured in an oven at a temperature of 45 °C for the duration of 4 hours.

In order to investigate the surface characteristics of borax added samples, the inclined plane test method (IPT) was used. The IPT setup was based on the ASTM D2303 test standard. There are three instances during the IPT test that requires the termination of the test:

- When the tracking length reaches 25 mm close to the ground electrode
- When the leakage current value exceeds 60 mA for two seconds or longer
- When the sample is eroded [23].



**Figure 1.** Schematic view of the inclined plane test setup.

The schematic diagram of the experimental setup is shown in Figure 1. According to the ASTM D2303 standard, a 33 k $\Omega$ , 400 W resistor was used in order to limit the short-circuit current. The resistor was connected in series with the high voltage source. The contaminant had a composition of 0.1% ammonium chloride and 0.2% TritonX100.

Preliminary tests have been conducted prior to the initiation of the experiments. Based on the results of these preliminary analyses, the experiments were designed so that they would be completed once the deterioration on the surface of the sample reached the ground and high voltage electrode in order to be able to better observe the characteristics of this deterioration. Again based on the preliminary tests and on other studies conducted using polyester, the applied voltage was selected as 4.0 kV. The liquid flow rate was reported as 36 ml/hr for this voltage level in the ASTM D2303 test standards and therefore, this value was adopted in the tests. Ten samples were tested for each borax concentration level.

When modelling the deterioration behaviour of the samples, the leakage value when the current reached its critical value was taken as the initial value as described by the ASTM D2303 standard. In this study leakage current was monitored using a sound card. It was connected to a resistor divider which is in series with the ground electrode as shown in Figure 1. The effect of the sound card on the measured value created a deviation of 0.025% from its true value which could be neglected during the calculations. The leakage current was recorded at a sampling rate of 48 kS/s.

In order to understand the degradation process clearly, Fourier analyses were used to decompose the recorded leakage current. Because the leakage current was non-sinusoidal, it can be represented in the form of a Fourier series as,

$$i(t) = \sum_{n=1}^{\infty} \sqrt{2}I_n \sin(n\omega_1 t + \delta_n), \tag{1}$$

Where  $I_n$  and  $\delta_n$  are the rms value of the current and the phase angle of the  $n^{th}$  harmonic, respectively;  $\omega_1$  is the angular frequency of the fundamental. In the equation, DC terms were ignored for simplicity.

The total harmonic distortion (THD) of the leakage current was calculated using 3<sup>rd</sup> to 9<sup>th</sup> odd harmonics as

$$THD = \frac{I_3^2 + I_5^2 + I_7^2 + I_9^2}{I_1}, \tag{2}$$

since the leakage current was symmetric about the time axis even harmonics do not consider when calculating total harmonic distortion.

Moreover, the DSC was performed on the polyester samples. Differential scanning calorimetry is a technique in which the difference in energy input into a material and a reference material is measured as a function of temperature. In this study the glass transition  $T_g$  and degradation  $T_d$  temperatures of pure and borax added samples providing data on the melting behaviour were observed using Mettler Toledo, Model 822. The experiments were performed in nitrogen atmosphere, at a heating rate of 10 °C/min. The scanning temperature range was 25–400 °C.

### 3. Results and discussion

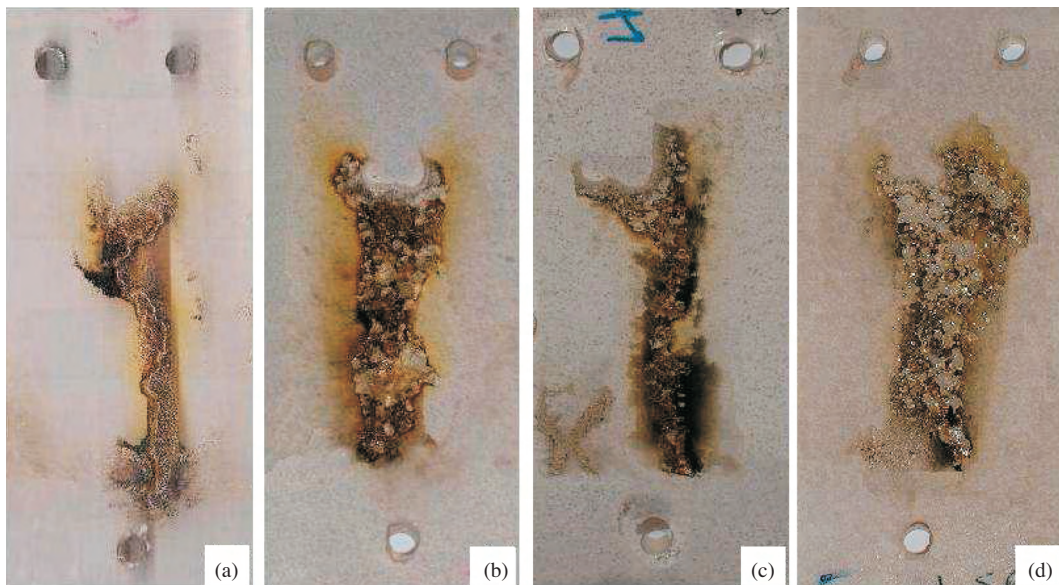
#### 3.1. Inclined plane test results

All samples were tested at an applied voltage of 4 kV with 36 ml/h contaminant flow rate. The effect of borax concentration on tracking initiation time is shown in Table 1. The average tracking time for pure polyester samples was found as 18 minutes. The average tracking times for borax added samples were larger than the one measured for unsaturated polyester. Borax addition to polyester approximately increased the tracking initiation time of the samples approximately more than two times. The tracking initiation times as well as their corresponding coefficients of variation given as percentages were given in Table 1. While this variation was 1.65% for the pure polyester samples, for the samples with borax added polyesters, this variation, which is acceptable for an experimental study, ranged between 4.32% and 7.89%.

**Table 1.** The effect of borax concentration on the tracking initiation time.

Borax Concentration [% by weight]	Tracking initiation time [min]	Coefficient of variation [%]
0	18:24	1.65
0.1	42:06	6.37
0.2	54:34	4.32
0.3	59:41	4.99
0.4	59:29	6.62
0.5	55:13	7.14
0.6	45:12	7.82
0.7	48:01	6.33
0.8	46:51	6.19
1.0	62:17	6.82
1.2	52:11	7.89
1.5	52:43	7.40

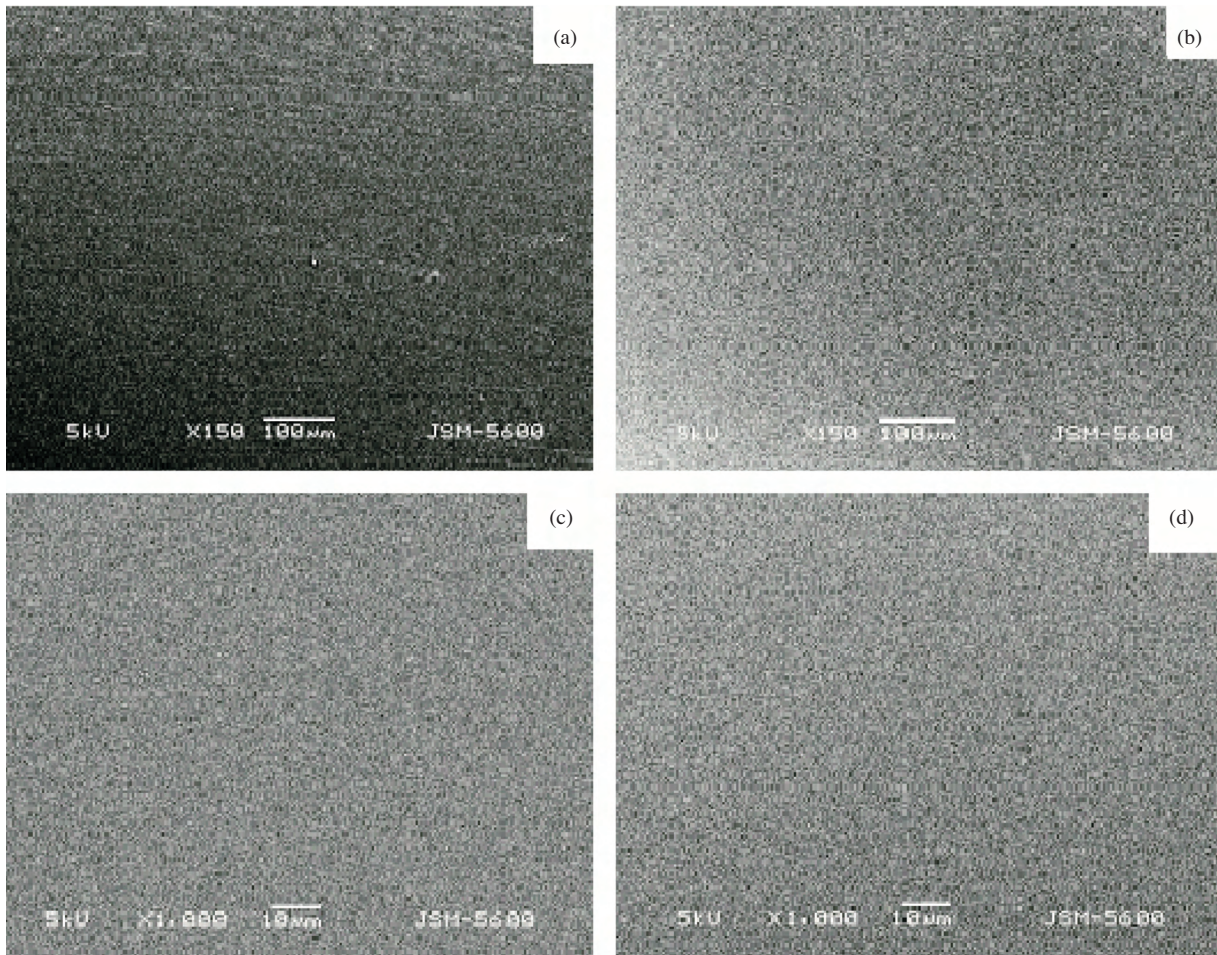
As mentioned before, in order to understand clearly the effect of borax on the surface behaviour of the polyester samples, we kept the experiment running even after the length of the erosion exceeded the 25 mm limit from ground electrode. In Figure 2, the scanned photographs of the representative samples of pure polyester as well as 0.5% (wt), 1.0% (wt) and 1.5% (wt) borax added samples were given. As it can be seen from the figures, adding borax to polyester changes the surface deterioration behaviour from tracking to erosion. In Figure 2(a), the progression of the degradation in pure polyester samples is given and the behaviour can be described as of the tracking type. In Figures 2(b), 2(c) and 2(d) the characteristic degradation process for 0.5%, 1.0% and 1.5% borax added polyesters were given respectively. Here the surface behaviour of the borax added samples changed in response to erosion rather than the tracking. The eroded areas of the 0.5%, 1.0% and 1.5% borax added polyesters were all greater than that of the tracked pure polyester sample.



**Figure 2.** Typical change of surface in the polyester samples with the following borax concentrations (a) Pure polyester, 0.0% borax, (b) 0.5% (wt) borax added into the polyester, (c) 1.0% (wt) borax added into the polyester, (d) 1.5% (wt) borax added into the polyester.

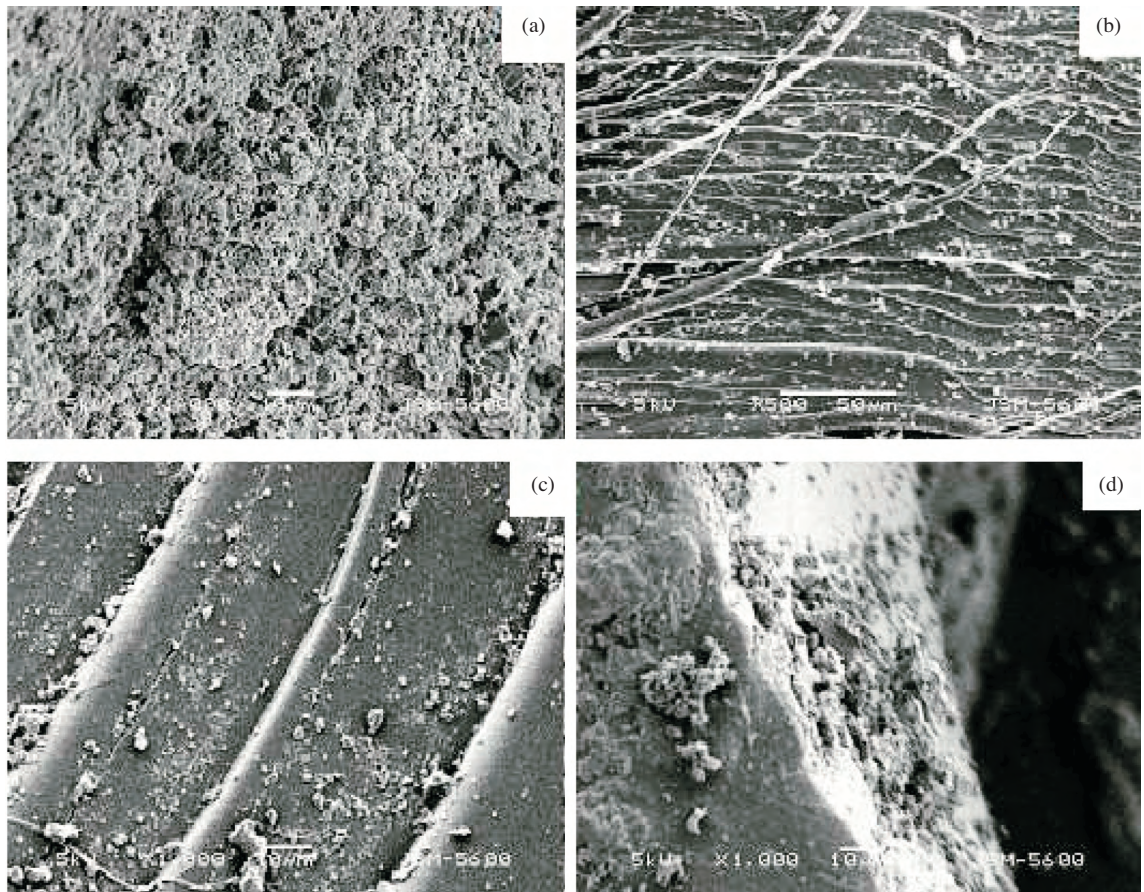
Because the borax used in this study was of a size measurable in microns and that the amount we use was very small, the scanning electron microscope (SEM) analysis was used to make sure that the borates in the polyester were dispersed evenly in the sample prior to its use in the tests. In Figure 3, SEM analysis of the surface pictures of the representative samples taken prior to the IPT test was given. Prior to the IPT test all the samples were shown to possess similar smooth surfaces. The SEM pictures recorded following the IPT test were used to investigate and understand the physical changes on the surface of the representative pure polyester and borax added polyester samples (Figure 4). Enlarging the surface pictures provide easier visualization of the change in the surface degradation characteristics. The pure polyester sample was observed to attain a sponge-like surface after going through the IPT test (Figure 4(a)). However the borax added samples following the IPT test were observed to be eroded rather than deteriorated by tracking. The change in the surface degradation characteristics lead to the conclusion that during the IPT test continuous discharges on the sample surface increased the temperature of the surface of the insulator which eventually accelerated the

breakdown process. When subject to high temperatures, borax minerals produce a thin glass like sheet on the surface of the insulator, which prevented thermal degradation (Figure 4(b), 4(c) and 4(d))

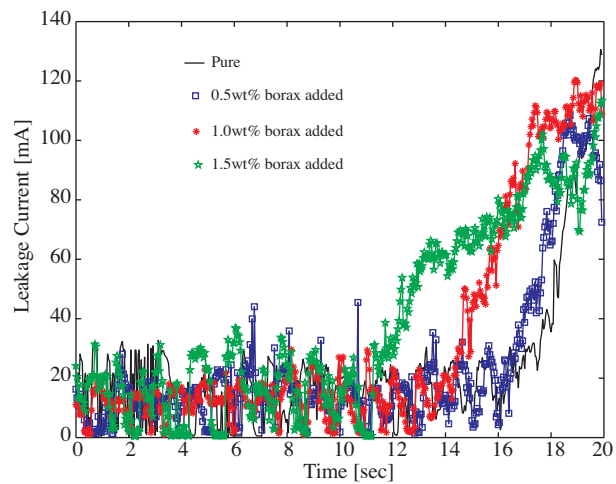


**Figure 3.** SEM pictures before inclined plane test: (a) pure polyester sample enlarged 150 times, (b) 0.5% (wt) borax added polyester sample enlarged 150 times, (c) 1.0 % (wt) borax added polyester sample enlarged 1000 times, (d) 1.5% (wt) borax added polyester sample enlarged 1000 times.

In order to compare the differences during the progression of the degradation and investigate the effect of borax in polyester samples, the leakage current during the whole degradation process was monitored. As mentioned before in Table 1, the degradation time of pure and borax added samples were different. To see the difference clearly, the leakage current recordings during the last 20 seconds prior to the degradation of the sample were taken and given in Figure 5. Before the degradation process began, no differences were noted for the pure and borax added samples. Irrespective of the added borax concentration, the calculated values of harmonic distortion were higher than that of pure borax. For instance the complete degradation of pure samples approximately took 2 seconds but in 1.5wt% borax added samples, the complete degradation process between the ground electrode and high voltage electrode took 7 seconds. That was 3.5 times the pure specimen as shown. Similarly, the degradation time was longer in other samples with borax additives in comparison to that of the pure polyester.

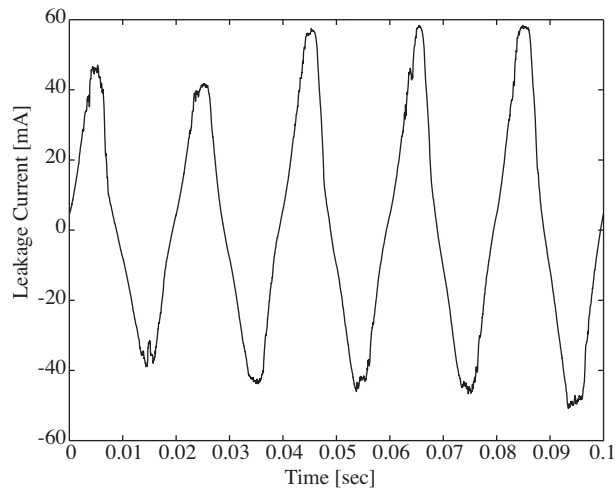


**Figure 4.** SEM pictures after inclined plane test: (a) pure polyester sample enlarged 1000 times, (b) 0.5% (wt) borax added polyester sample enlarged 500 times, (c) 1.0 % (wt) borax added polyester sample enlarged 1000 times, (d) 1.5% (wt) borax added polyester sample enlarged 1000 times.



**Figure 5.** The leakage current for the pure, 0.5 wt%, 1.0 wt% and 1.5 wt% borax added polyester samples during the IPT test.

According to ASTM D2303 the tracking process is over when the leakage current exceeds 60 mA for 2 seconds. Therefore, in this study when evaluating the leakage current and its harmonics, the last five cycles in the leakage current before its value reached 60 mA were taken into consideration. Using these last five cycles the harmonic distortion factors (DF) and the total harmonic distortion (THD) of pure and borax added polyester samples from the 3<sup>rd</sup> up to the 9<sup>th</sup> harmonic was computed. The leakage current signal for the pure polyester samples before its value reaches 60 mA was given in Figure 6. By evaluating the odd harmonics from this last 5 cycles before its value reaches 60 mA is concluded in Table 2



**Figure 6.** The leakage current signal for the pure polyester samples before its value reaches 60 mA.

**Table 2.** Mean Odd harmonic distortion values against borax concentration.

Borax concentration [wt %]	HD <sub>I3</sub> , [%]	HD <sub>I5</sub> , [%]	HD <sub>I7</sub> , [%]	HD <sub>I9</sub> , [%]	THD, [%]
0	14.864	4.663	2.318	1.351	15.808
0.1	18.881	6.328	2.396	1.505	20.113
0.2	20.171	6.921	1.677	0.827	21.407
0.3	24.072	5.569	2.981	0.968	24.906
0.4	18.137	4.688	2.487	1.101	18.930
0.6	15.459	5.227	1.915	0.859	16.454
0.7	18.658	6.862	2.295	0.944	20.035
0.8	17.147	5.515	2.386	0.993	18.197
1	19.461	6.863	2.893	0.898	20.857
1.2	19.659	8.021	2.406	1.045	21.394
1.5	21.606	5.792	3.063	1.004	22.600

In Table 2 the mean values of harmonic distortion factors and total harmonic distortion values are given. The 3<sup>rd</sup> and the 5<sup>th</sup> harmonic distortion factors and the total harmonic distortion value increased remarkably with the addition of borax. The increase in the 7<sup>th</sup> harmonic distortion factor was not sharp but subtle. The change in the 9<sup>th</sup> harmonic distortion factor value was independent of the amount of borax addition. The total harmonic distortion for pure polyester sample was calculated as 15.808. For 0.1%, 0.2%, 0.3%, 0.4%, 0.5%, 0.6%,



0.7%, 0.8%, 1.0%, 1.2% and 1.5% borax added polyester samples, the total harmonic distortion values were computed to be larger than that of the pure polyester sample.

The basic electrical model of an insulating material is given as a resistor in series with a capacitor or a resistor in parallel with a capacitor. The dielectric constant of polyester at 60 Hz is 5 [24] and for the sodium borates the dielectric constant at 1 kHz is 31 [25]. When these two different materials were blended, impurities with higher capacitance than polyester were introduced into the composite sample. In this experiment the voltage was set to 4.0 kV and no difference was observed in the leakage current harmonics between the pure and the borax added polyesters prior to the initiation of degradation. Therefore the sole source of the difference in harmonics that was observed during the degradation process was the result of the borax minerals rather than because of the arcs that occurred during the evaporation of the contaminant as the polyester specimen was getting eroded.

During the IPT test, the continuous arcs as well as the scintillations observed on the surface of the pure polyester samples caused temporary hot spots to occur. These spots accelerated the formation of free carbon which finally lead to the carbonized black tracking images as seen in Figure 3(a). Adding borax to polyester changed the degradation behavior and the specimens were mostly damaged by the erosion of the surface as seen in Figure 3(b), 3(c) and 3(d). The tracking process and the degradation time of these samples was delayed for about three times longer than that of the pure polyester samples as seen in Table 1. When the tracking process started following the first few discharges initiating the carbon path near the ground electrode, the discharges also took place in drips near the flowing contaminant through the surface between the high voltage source and the ground electrode. This behaviour might have been the result of the extended test duration (since the drips near the ground electrode may also be heated ) or the borax particles in the polyester bulk surface might have changed the behaviour. Following the test the degraded samples were re-investigated under the scanning electron microscope and the glass like sheet rather than the carbon paths on the surface of the borax added samples were observed. It was presumed that this glass like sheet was formed when borax mineral was degraded under the continuous arcs. During the test, this surface helped the polyester to be more resistive to arcs and therefore the degradation was not complete only in 2 seconds as seen in pure polyester. For the degradation between the ground and the high voltage electrode to be completed for the borax added samples, it took much longer times as seen in Figure 4.

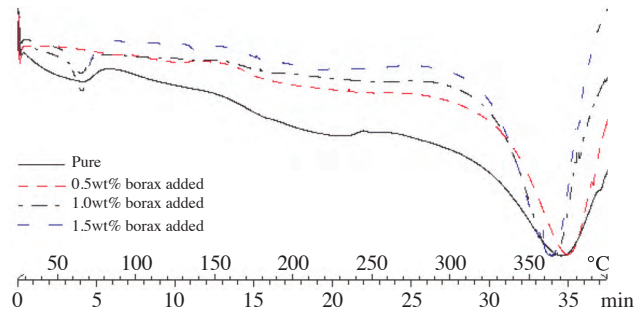
### 3.2. DSC analysis

In order to understand clearly the effect of borax on the degradation behaviour of the polyester samples, the DSC analysis was performed. The change in the glass transition and degradation temperatures of pure, and borax added polyester samples is given in Table 3. For representative purposes, 0.5% (wt), 1.0% (wt) and 1.5 % (wt) borax added samples were selected. For pure polyester samples the glass transition temperature was 166.03 °C and degradation temperature was 370.0 °C. In the samples with 0.5% (wt) borax added polyester the glass transition temperature was measured as 167.99 °C which was 1.99 °C higher than that of the pure sample. Also its degradation temperature was measured as 374 °C which was 4 °C higher than that of the pure sample. For the 1.0% (wt) borax added samples the glass transition temperature was measured as 168.97 °C which was 2.94 °C higher than that of the pure sample. However the degradation temperature was measured as 364.76 °C which was 1.24 °C lower than that of the pure polyester sample. From the DSC thermogram spectra of the 1.5% (wt) borax added sample, the glass transition temperature was measured as 170.25 °C which was 4.25 °C higher than that of the pure sample. On the other hand, the degradation temperature was measured

as 363.60 °C, that is, 6.40 °C lower than that of the pure polyester sample.

**Table 3.** The effect of borax concentration on glass transition and degradation temperatures.

Borax concentration (weight %)	$T_g$ (°C)	$T_d$ (°C)
0	166.00	370.00
0.5	167.99	374.10
1.0	168.97	364.76
1.5	170.25	363.60



**Figure 7.** DSC thermogram of pure, 0.5% (wt), 1.0% (wt) and 1.5% (wt) borax added polyester samples.

The DSC spectra of pure 0.5% (wt), 1.0% (wt) and 1.5% (wt) borax added polyester samples are given in Figure 5. The thermograms were different for the pure polyester sample than the DSC thermograms of the 1.0% (wt) and 1.5% (wt) borax added samples. It can be seen from the curve that there was a clear endothermic peak at about 30–90 °C which was similar to what has been observed in the spectra for the 1.0% (wt) and 1.5% (wt) borax added samples corresponding to the temperature of dehydrating process of the borax mineral. The DSC studies indicated that the thermal characteristics of the polyester altered with borax containing water molecules in its chemical composition, causing an endothermic reaction before the polyester degraded.

The addition of borax prevented the formation of free carbon on the surface of the composite samples as it was confirmed by the SEM analysis (Figure 4). When subject to high temperatures, the borax mineral produced a thin glass like sheet on the surface of the insulator which prevented thermal degradation and the local heating of the water molecules appearing in the chemical composition of borax, caused an endothermic reaction which also decelerates the degradation process. However it is not yet well understood, which of these mechanisms is more efficient in improving the electrical performance of the borax added composite insulators.

## 4. Conclusion

Based on the experimental studies carried out on the polyester resin with different borax concentrations, the following conclusions are drawn:

- According to the test results the tracking initiation time increased with borax concentration.
- Increasing the borax concentration in polyester samples, changes the tendency of the resulting composites to direct them to damage through erosion rather than surface tracking. Also the eroded area of polyester samples extends with borax concentration.

- In comparison to the pure samples, the leakage current values of the borax added samples increased slightly throughout the tracking profile.
- Harmonic distortion factors of samples for the 3<sup>rd</sup> and 5<sup>th</sup> show that increasing the borax concentration increases the DF values. The change in 7<sup>th</sup> and 9<sup>th</sup> harmonic distortion factor value is observed to be independent of the borax concentration.
- The DSC analyses reveal clearly, that the specimens produced by the samples with different borax concentrations differ amongst themselves considerably in glass transition temperatures and also in the degradation temperatures. The glass transition temperature of polyester samples slightly increased with increasing borax concentration. Except for the 0.5% (wt) borax added sample, the degradation temperature of the test samples decreased with increasing borax concentration.

In summary, the present work shows that the addition of borax to polyester increases the lifetime by shifting the glass transition temperature (Figure 7) and by physically forming a thin glass like sheet on the surface of the insulator (Figure 4). The entire degradation process was monitored by leakage current analysis, and no differences were noted prior to degradation for pure and the borax added polyester samples. The only difference was observed as the degradation started when the harmonic components of the borax added polyester samples were larger than that of the pure polyester samples. However it is not clear whether it is because of the borax addition or because of the erosion process itself.

After a thorough literature survey, it was found that, to our best knowledge, there were no data in relation to the surface degradation characteristics of polyester filled with borax. The effect of borax additive in polyester on the surface tracking has been investigated for the first time in this study. The change in the insulating surface containing this compound which is abundant in Turkish reserves has been reported. From the measurements and observations in this study it can be concluded that the surface degradation phenomena is rather complex in polyester-borax composites. Therefore, in order to better understand the electrical behaviour of the composite further studies are necessary. Further application studies regarding the flame retardant behaviour of the borax added polyesters and their application areas on high voltage materials are currently in progress.

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