

## A systematic approach to nuclear microscopy of water trees for a large number of field-aged HV cable samples

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### SUMMARY

In order to perform micro-PIXE measurements on water trees in underground HV cables when a large number of cable samples are involved, a sequence of tests has been devised to minimize time and effort for sample preparation, water tree detection and analysis. These tests include electrical diagnostic tests to predict the possible presence of water trees, optical microscopy on cable insulation to detect water trees in the samples screened by the electrical tests, scanning electron microscopy for detailed surface topography of water trees, and nuclear microscopy for elemental composition and distribution maps of water trees. Correlations among the results of the four types of measurements are discussed to evaluate the usefulness of the methodology. Copyright © 2005 John Wiley & Sons, Ltd.

**KEY WORDS:** water trees; HV cables; electrical diagnostic tests; optical microscopy; scanning electron microscopy; nuclear microscopy

### INTRODUCTION

Water treeing in the insulation of underground HV cables is a chronic problem that leads to premature degradation of the cables [1]. In spite of a great deal of attention given to solving the problem, there is as yet no satisfactory solution in sight [2]. Nuclear microscopy [3], a novel technique in this field, has the capability of analyzing the water trees on a microscopic scale in order to understand their behavior and characteristics. However, this sophisticated technique requires considerable time and effort for sample preparation and water tree detection, and involves the use of large-scale and expensive accelerator-based nuclear microprobe facilities where time is at a premium. Therefore, the cost of nuclear microscopy can be prohibitively high, both in terms of time and money, if one is to use it directly to look for and analyze water trees in the insulation sections of a large number of field-aged cables. Alternatively, one can scan the insulation sections with an optical microscope to detect water trees before nuclear microscopy is attempted. This too will be enormously labor intensive since the

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work involves removing all the layers of the cable samples, microsectioning the insulation and visually inspecting all the microsections at random to detect water trees. If, on the other hand, a way can be found to identify those collected cable samples that may possibly contain water trees, a lot of work can be minimized. Such a possibility exists through the use of some electrical diagnostic tests. If these electrical tests are able to isolate those cable samples that may contain water trees from all the failed cables collected from the field, then attention can be focused on these cable samples only.

Such an approach was adopted in this study. Selected electrical diagnostic tests were first carried out on the collected cable samples in order to predict the presence or absence of water trees. Only those samples indicated by the electrical tests were then followed up with optical microscopy to confirm the presence of water trees. Samples containing water trees as detected by optical microscopy were finally subjected to scanning electron microscopy (SEM) and micro-PIXE (microbeam Proton Induced X-ray Emission) measurements.

## EXPERIMENTAL METHODS

### *Sample collection*

Field-aged cable samples of three voltage ratings, namely 15 kV, 35 kV and 69 kV, in use in the Eastern Province of Saudi Arabia, were collected from three distinct locations: Dammam/Khobar residential districts, Jubail industrial city and a relatively rural area of Al-Hassa. Most of them were buried directly in the ground while some were inside concrete ducting in the ground. Many of the cable samples were extensively damaged due to electrical faults. The service life of the cable samples ranged from a few years to 20 years.

### *Sample preparation*

For the electrical tests, the cable jacket, lapped textile, copper screen (sheath), conductive crepe paper, and outer semi-conducting layers were removed up to a length of 20–25 cm from both ends. Some of the insulation (1–2 cm) was also removed from the cable ends to connect the cable conductor with the HV source. For the cable end that was to be connected to the measuring circuit the grounded sheath was not removed but folded back and connected to the measuring circuit. The two ends of the cables were cleaned appropriately to minimize leakage current and reduce the possibility of flashover on the surface. The conductor surfaces were also covered well with HV insulating tape so that a corona was prevented.

Preparation of samples for optical, scanning electron and nuclear microscopy measurements involved removal of all the outer layers (PVC jacket, metallic screen, textile layer, etc) as well as the inner conductor, leaving only the insulation together with its inner and outer semiconducting compound layers. Thin sections (5–10  $\mu\text{m}$ ) were microtomed from the cable insulations.

### *Electrical diagnostic tests*

Several electrical diagnostic tests have been suggested and tried by various authors [4] in order to predict the presence of water trees in field-aged HV cables. In this work, we limited our attention to three such tests: DC current, DC conductivity, and the partial discharge (PD) measurements.

*DC leakage current measurement.* DC voltage was raised in steps of 10–20% of the test voltage and the voltmeter reading was recorded after the elapse of the discharging time (about 15 seconds) of the

capacitive current. The cable DC current is calculated as the voltmeter reading divided by the measuring resistance. It has been reported [5] that if the slope of the current–voltage characteristic changes from a linear to a non-linear relation, then this is a sign that the insulating material is deteriorating.

*DC conductivity measurement.* The circuit set-up for the DC conductivity measurement was the same as that for the DC current test. The main difference was in the procedure for applying the DC voltage. In this case, a short circuit is made after the application of the DC step voltage. The DC current vs. time characteristic was then measured [6,7].

*Partial discharge (PD) measurement.* The cable samples were excited from a 60 Hz AC voltage source. The voltage was increased to  $1.75 U_0$  where  $U_0$  is the rated power frequency voltage between conductor and earth or metallic screen for which the cable is designed. Any PD signal above 5 pC was taken as a measure of the cable deterioration as reported in the literature [8].

### *Optical microscopy*

Optical microscopy was carried out on those cable samples that were indicated by the electrical diagnostic tests to have the possibility of containing water trees. Insulation microsections, 5–10  $\mu\text{m}$  thin, were prepared from the insulation of these cable samples. The microslides were scanned under a microscope at a magnification of 100. A video camera attached to the microscope captured the images of the water trees observed which were stored on a PC. Hard copies of the water trees were printed out as a visual guide for later measurements using SEM and nuclear microscopy.

### *Scanning electron microscopy*

Scanning electron microscopy measurements were carried out on 5  $\mu\text{m}$  thin insulation sections using a JEOL SEM model JSM-5800. The probe resolution was typically 3.5 nm and an acceleration voltage of 20 kV was used. The energy dispersive spectroscopy (EDS) system with the SEM had an x-ray Si(Li) detector with an atmospheric thin window capable of detecting elements down to beryllium. Samples were coated with a thin layer of carbon.

### *Nuclear microscopy*

Micro-PIXE (microbeam Proton Induced X-ray Emission) measurements were carried out on 10  $\mu\text{m}$  thin insulation sections using the nuclear microprobe at the Research Center for Nuclear Microscopy of the National University of Singapore. A 2.1 MeV proton beam of 1  $\mu\text{m}$  spot size was used at a beam current of 30–50 pA. Spectra using scanning transmission ion microscopy (STIM), PIXE and Rutherford backscattering spectrometry (RBS) were acquired simultaneously.

## RESULTS AND DISCUSSIONS

### *DC current test results*

The DC current vs. voltage characteristics plot of an unused 35 kV cable sample is shown in Figure 1. The relationship was found to be linear implying that the insulations for these cables are in good condition, free of water trees. This is in agreement with the published data [5] that the current—

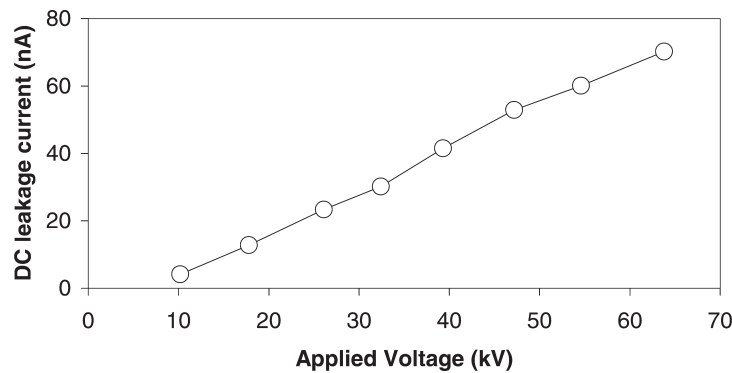


Figure 1. Current–voltage characteristic for an unused 35 kV cable sample.

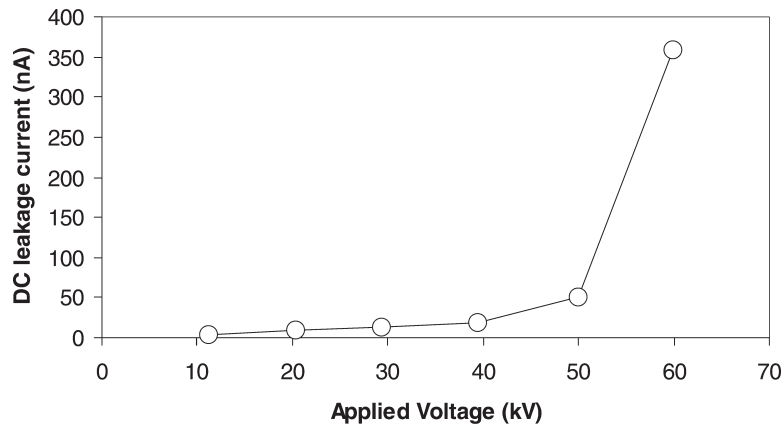


Figure 2. Current–voltage characteristic for a used 69 kV cable sample.

voltage characteristics should be linear for undamaged cable insulation. Similar results were obtained for cable samples of other voltage ratings.

A typical current–voltage characteristic measured for a used 69 kV cable sample is shown in Figure 2. The leakage current started to become non-linear generally when the applied DC voltage was raised to around 40 kV. This non-linear relationship became very sharp when the voltage was above 50 kV. This result indicates deterioration of the cable insulation with the possibility of the presence of water trees in these cable samples.

#### *DC conductivity test results*

DC conductivity measurements on an unused cable sample showed a very fast decay of DC current which indicated that the cable insulation is not electrically damaged. This is in conformity with the results obtained from the DC current measurements for the same cable sample. The DC current–time characteristic for the same used 15 kV cable sample for which the leakage current measurements were performed earlier, is shown in Figure 3. A long decay time for the conductivity current was

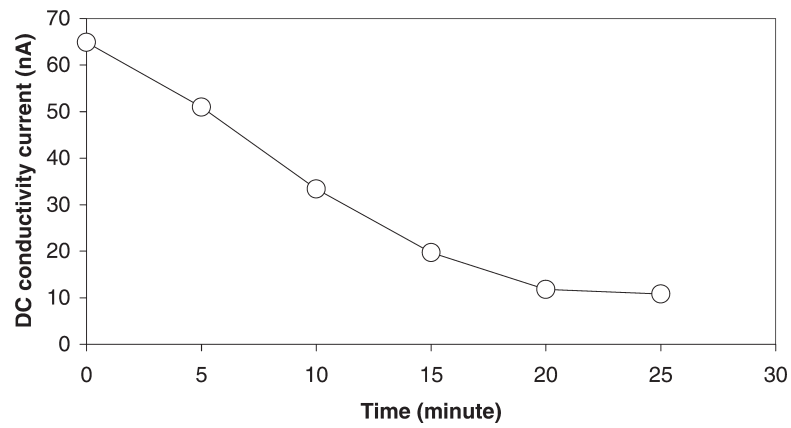


Figure 3. DC conductivity current–time characteristic for the used 15 kV cable sample indicating insulation deterioration.

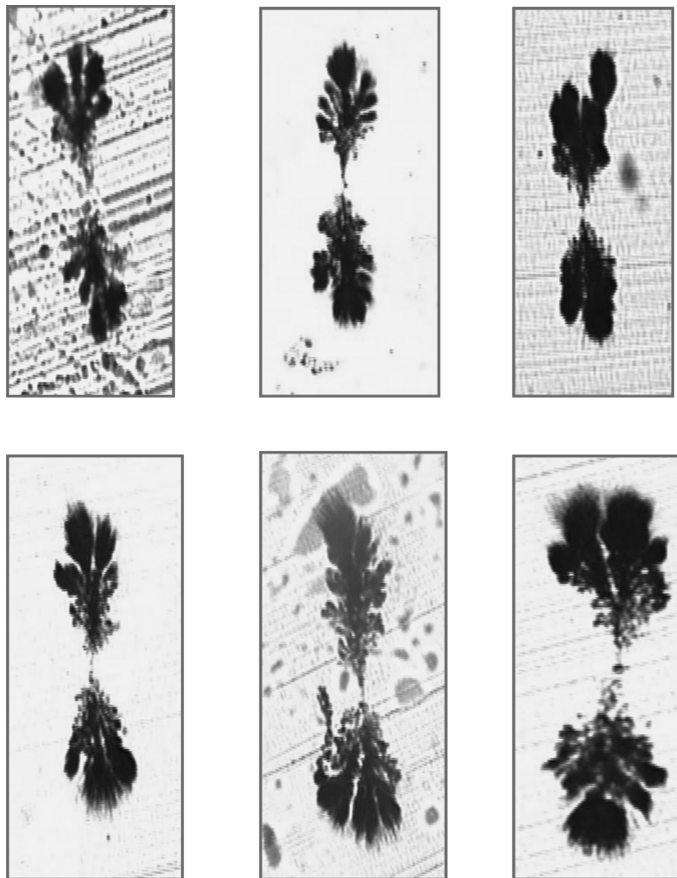


Figure 4. A view of some typical bow-tie trees in 10  $\mu\text{m}$  thin sections of the insulation of 35 kV cable samples.

observed which indicates that these cable samples have insulation deterioration, possibly due to water trees.

From the above results, it is clear that the DC leakage current and conductivity tests data are in agreement with each other. The same cables that were tested by the two techniques gave similar indications of the dielectric deterioration of the cable insulation, possibly due to the growth of water trees. Therefore, either of these two tests can be used as a possible diagnostic tool for screening a large number of field-aged cables for the presence of water trees.

#### *Partial discharge test results*

Partial discharge (PD) measurement on a used 35 kV cable sample at 25 kV applied voltage gave a value of 85 pC, which is far above the accepted PD levels of 5 pC. This high level of partial discharge

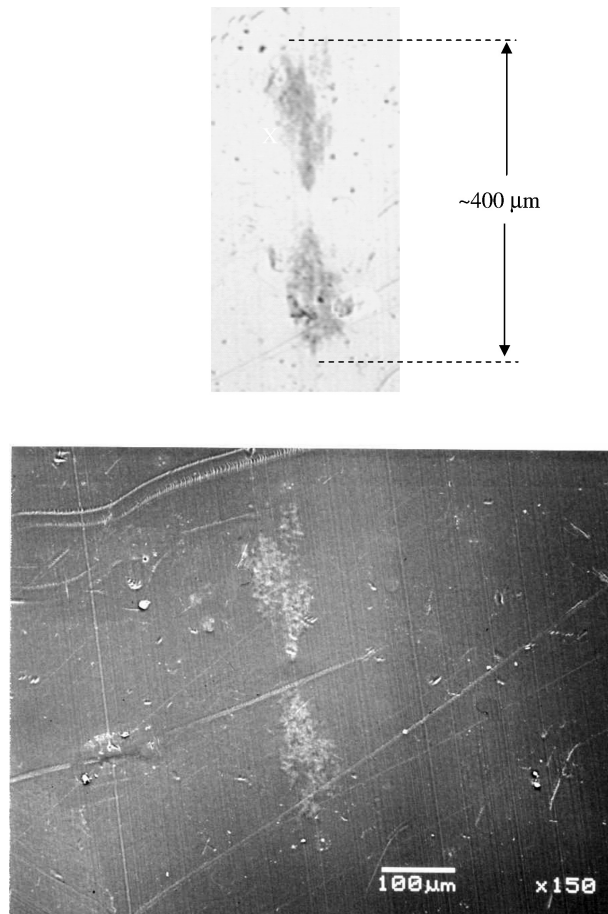


Figure 5. Optical microscope view (top) and SEM backscattered-electron image (bottom) of a water tree in a 5  $\mu\text{m}$  thin insulation section of a 69 kV cable sample.

indicates that the cable sample has insulation deterioration due to either the existence of microvoids, or the presence of water trees.

*Optical microscopy results*

Bow-tie water trees observed in a 35 kV used cable for which electrical tests provided a positive result are shown in Figure 4. The density of the water trees observed was estimated to range from  $1000/\text{cm}^3$  to  $12\,000/\text{cm}^3$ . Lengths of the trees observed varied from about  $100\text{ }\mu\text{m}$  to about  $500\text{ }\mu\text{m}$ . No vented trees could be found in the microslides investigated. Similar results were obtained for the 69 kV cable

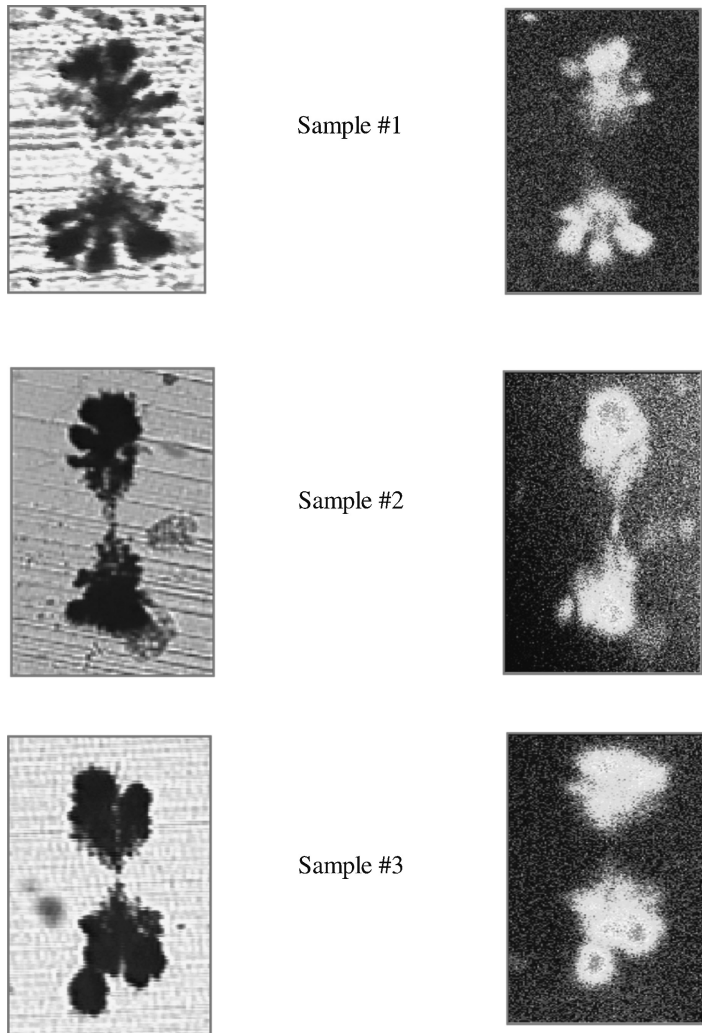


Figure 6. Optical images (left) and STIM images of water trees in 35 kV cable samples.



samples. However, no water tree could be detected in the 15 kV cable samples although they were indicated to have water trees by the electrical tests. This negative correlation for the 15 kV cable sample indicates that the cable deterioration may have been due to causes other than water trees.

### *Scanning electron microscopy results*

SEM measurements on 5  $\mu\text{m}$  thin insulation sections found to contain water trees by optical microscopy showed the effectiveness of this technique to generate high-resolution images of water tree microstructure in insulation specimens. A backscattered electron image of a water tree in a cable sample along with its optical microscope image is shown in Figure 5. Composition analysis using this technique could detect only one major element (pb) in the water trees investigated.

### *Nuclear microscopy results*

The STIM images of some water trees in a 35 kV cable sample are shown in Figure 6 along with the corresponding optical microscope images. A typical x-ray elemental spectrum of a water tree is shown in Figure 7. Elements generally seen in this cable sample are Si ( $\sim 100$  ppm), S (10 000–30 000 ppm), Ca (100–150 ppm), Cu (50–80 ppm) and Pb (70 000–200 000 ppm). The relatively large amount of Pb in the water trees points to the existence of Pb contaminated soil in the area where the cables were laid. The insulation matrix spectrum (Figure 8) does not show any contaminant except some S (491 ppm). The likely source of S may be the antioxidants used to treat cable insulation to inhibit oxidation.

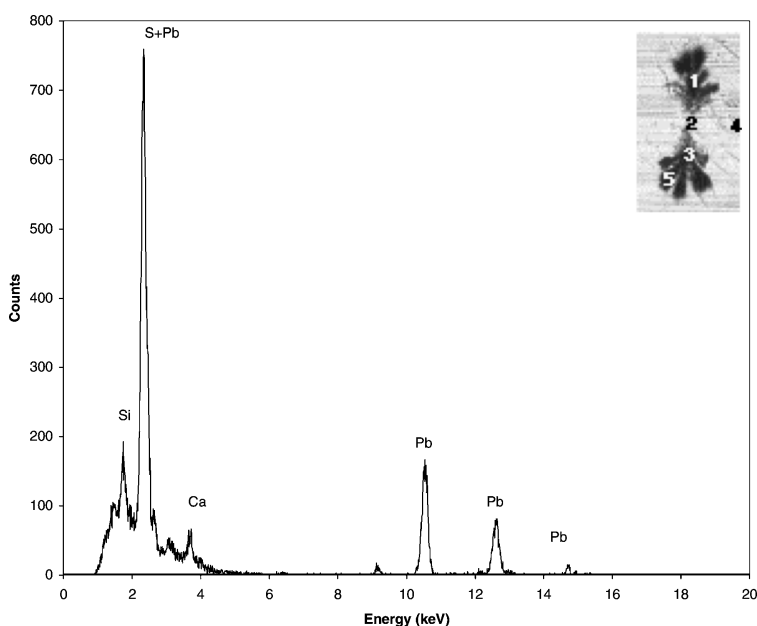


Figure 7. A typical micro-PIXE spectrum from a spot inside a water tree in a 35 kV cable sample. The water tree with the spots analyzed is shown in the inset.



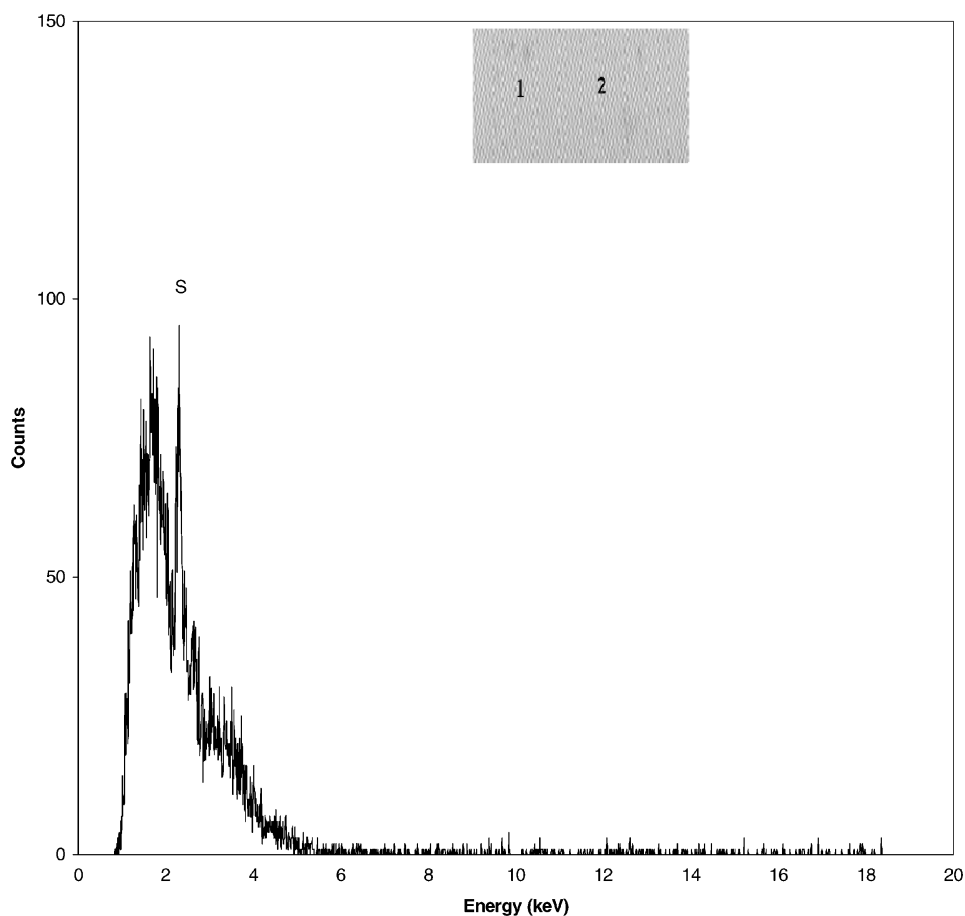


Figure 8. A typical micro-PIXE spectrum from a spot on the insulation matrix of a 35 kV cable sample. A section of the semicon layer with the spots analyzed is shown in the inset.

The x-ray spectrum from the inner semiconducting compound surrounding the cable insulation showed (Figure 9) the presence of a large amount of Cu (3000 ppm) in addition to some S (519 ppm). However, the outer semiconducting compound (Figure 10) was found to contain a large number of elements such as Si (4000 ppm), S (2700 ppm), Cl (1400 ppm), Ca (639 ppm), V (153 ppm), Fe (294 ppm), Ni (186 ppm), Cu (9000 ppm) and Zn (666 ppm). The presence of a large number of impurity elements in the outer semiconducting compound used in these cable samples could be due to diffusion from the surrounding soil through the outer layers. It is also possible that these contaminants were present in the original raw materials.

The distribution maps of several elements, from major to trace quantities, detected in some of the water trees are shown in Figure 11. These maps demonstrate the unique capability of nuclear microscopy for non-destructive water tree analysis that can be useful to understand the problem of premature degradation of HV cables. An interesting feature of these distributions is that the elements are more or less uniformly spread over the water trees. This lends supports to the theory that water

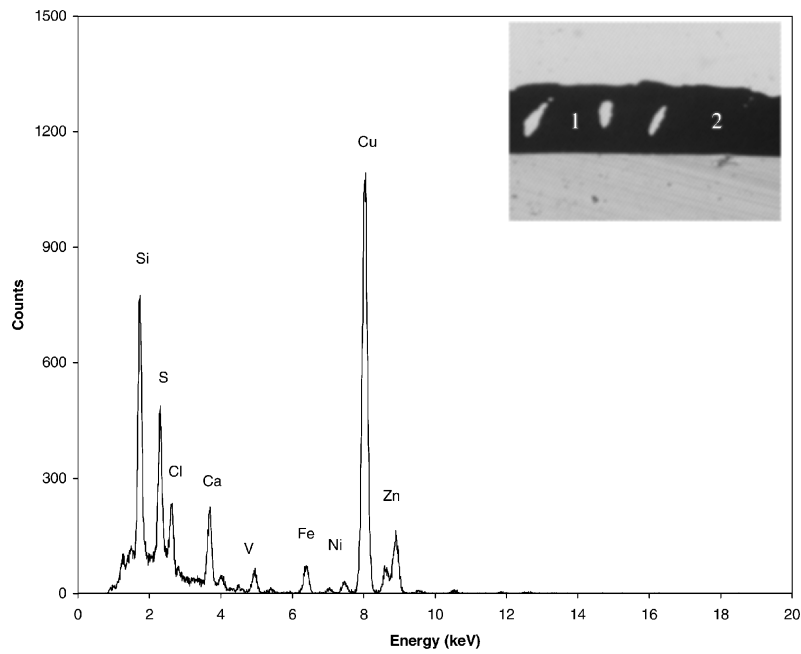


Figure 9. A typical micro-PIXE spectrum from a spot on the inner semicon layer of a 35 kV cable sample. A section of the semicon layer with the spots analyzed is shown in the inset.

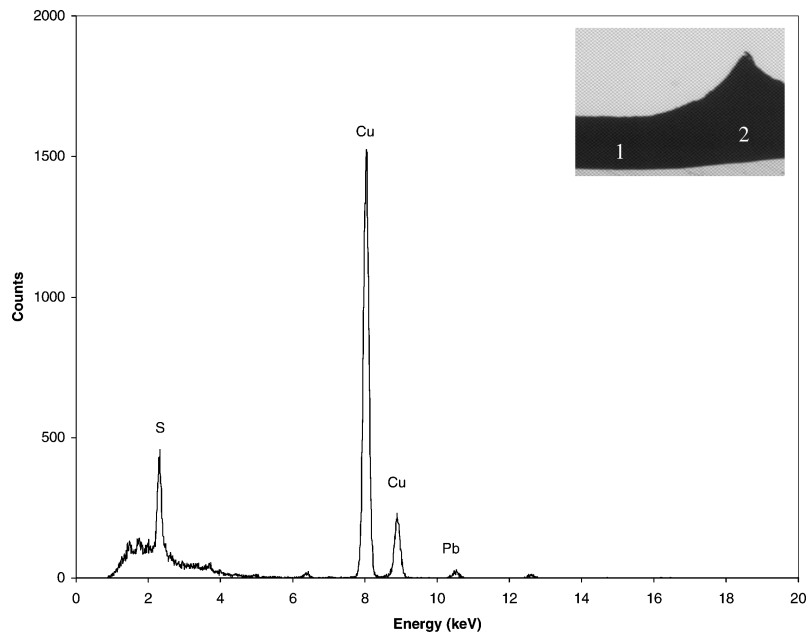


Figure 10. A typical micro-PIXE spectrum from a spot on the outer semicon layer of a 35 kV cable sample. A section of the semicon layer with the spots analyzed is shown in the inset.

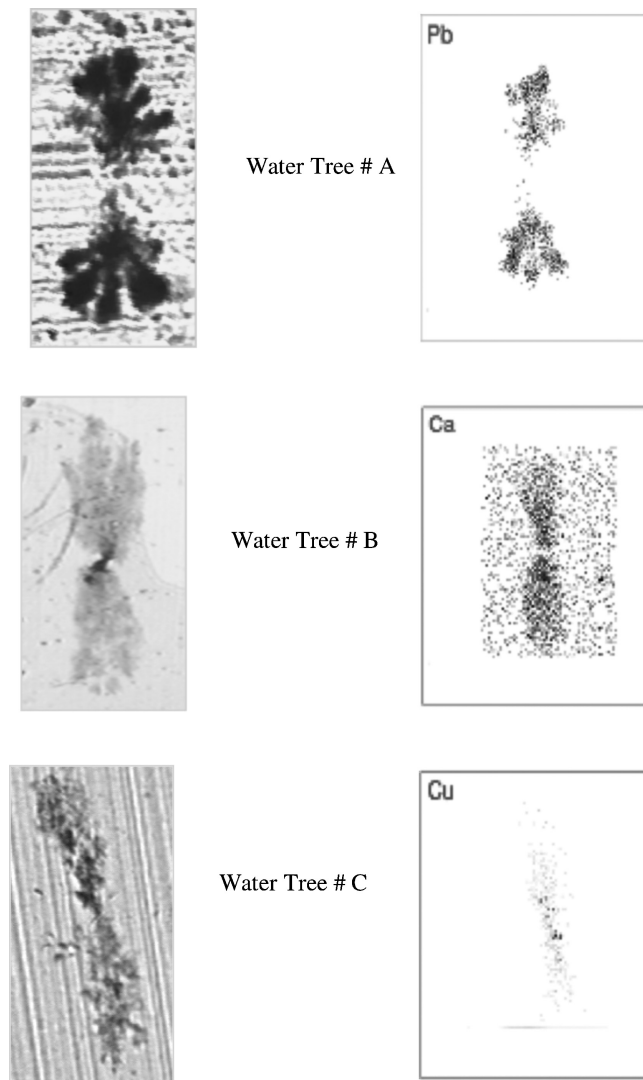


Figure 11. Optical image (left) and elemental distribution maps of water trees in 35 kV cable samples.

trees are composed of microvoids interconnected by micro-channels or tracks, which are filled by water and ions that penetrate the polymer under the action of an electric field [9].

## CONCLUSIONS

The analytical approach consisting of a series of tests in sequence was found to be effective in analyzing water trees in a large number of cable samples. The electrical diagnostic tests used proved to

be useful in screening the samples for the presence of water trees. Optical microscopy provided a visual confirmation of water trees in the screened samples. Scanning electron microscopy could produce high-resolution images of the surface topography of water trees. Nuclear microscopy provided useful data on major and trace element composition and distribution maps of the water trees. Such data can be useful in understanding premature degradation of underground HV cables.

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#### AUTHORS' BIOGRAPHIES



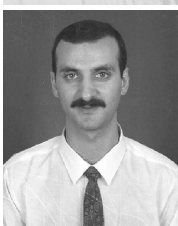
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