Journal of Energy Technology

JET Volume 17 (2024) p.p. 32-46
Issue 1, 2024
Type of article: 1.01
http://www.fe.um.si/si/jet.htm

ANALYSIS OF AGED CABLES` INSULATION WITH X-RAY FLUORESCENCE SPECTROMETRY

ANALIZA ELEKTRIČNE IZOLACIJE STARANIH KABLOV Z RENTGENSKO FLUOROSCENČNO SPEKTROMETRIJO

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Keywords: cable ageing, polymer electrical insulation, x-ray fluorescence spectrometry, preventive maintenance, acceptance testing

Abstract

The paper provides an analysis with X-ray fluorescence spectrometry of aged cable samples. The work encompasses a review of the electrical, mechanical and chemical properties, as well as measurement methods for determining those parameters. The analyses are based on physical principles that allow us to assess the condition of the cable insulation. Using the physical properties and measurement results, mathematical models can be formulated to describe the ageing of individual materials in the presence of specific influences. The goal of the paper is to establish the foundation for the development of a measurement method using X-ray spectral analysis. The methodology assumes that the presence of specific elements decreases or increases on the material's surface as it ages. By using a standard indenter modulus test, we can determine reference points for X-ray spectral analysis. Sampling these points on differently degraded cables enables the establishment of criteria for acceptable cable insulation.

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Povzetek

Delo zajema pregled električnih, mehanskih in kemijskih lastnosti ter merilnih metod za določanje njihovih parametrov. Analize temeljijo na fizikalnih osnovah, s pomočjo katerih lahko določamo stanje kabelske izolacije. S pomočjo fizikalnih lastnosti in merilnih rezultatov pa lahko oblikujemo tudi matematične modele, ki opisujejo staranje materialov ob prisotnosti posameznih vplivov. Cilj je zasnova podlage za oblikovanje merilne metode z rentgensko spektralno analizo. Metodologija temelji na predpostavki, da se s staranjem materiala na površju zmanjšuje oziroma povečuje prisotnost posameznih elementov. S pomočjo standardnega testa indenter modula lahko določimo referenčne točke, na podlagi katerih se izvede rentgenska spektralna analiza. Z vzorčenjem teh točk različno degradiranih kablov pa se vzpostavimo merila sprejemljivosti kabelske izolacije.

1 INTRODUCTION

The fundamental component of all systems utilising electrical energy is represented by electric cables. However, they are often overlooked and taken for granted. The primary purpose of conductors is to transmit electrical energy or information between components or devices. Conductors, typically cylindrical in shape, are surrounded by basic electrical insulation. Low and medium voltage cables, assuming polymer insulation, will be the focus of this paper. The conductors are categorised based on nominal voltage and current, determining their cross-section. Cables consist of one or more conductors, surrounded by a polymer sheath. Aging refers to the degradation of material and deterioration of the mechanical, electrical and chemical properties of electrical insulation, leading to cable destruction. External factors such as temperature, water presence, chemicals and UV light accelerate ageing. Thermal-oxidative degradation poses the greatest challenge, altering the material's structure and properties. Various non-destructive methods, including insulation resistance and dielectric loss measurements, are used to analyse insulation condition. Mechanical measurements, like tensile strength, and chemical analysis methods, such as thermogravimetric analysis, aid in assessing changes in the properties. Mathematical models or experimental data are relied upon to assess the remaining lifespan based on theoretical principles or data *[1 - 5].*

1.1 Polymer insulation

The electrical insulation separates conductive parts from each other electrically, meaning wires from each other and wires from the surroundings. The outer sheath, in addition to providing electrical protection, also offers mechanical protection to the conductors against environmental influences. An important role of electrical insulation is also to protect workers and users, as it shields them from direct contact and electric shock. Additionally, various colours of insulation facilitate easier orientation when connecting conductors to electrical components. Electrical insulation has standard electrical properties, which are categorised as follows *[6]*:

- volumetric resistance,
- surface resistance,
- insulation resistance,
- dielectric constant,
- dielectric losses, and
- dielectric strength.

In addition to the electrical properties of the insulation itself, the thermal, mechanical and chemical properties are also extremely important, as they influence the material's effectiveness and its ability to provide electrical insulation. The composition of the material itself determines whether the material can withstand prolonged exposure to various external influences. Essentially, we want materials to be resistant to the following factors *[6]*:

- fire,
- high temperatures,
- aggressive liquids,
- UV light,
- moisture, and
- to ionising radiation.

Various polymeric or other insulating materials are used, due to the need for optimal electrical and mechanical properties of electrical insulation. However, the electrical and mechanical properties change or deteriorate throughout the material's lifespan due to the ageing process. Material ageing depends on the material's production, maintenance, and, above all, the conditions under which it operates.

1.2 Electrical and mechanical properties

The mentioned electrical properties of the insulating material constitute an essential part of the conductor, as the essence of insulation lies in separating conductive parts from each other and reducing electrical losses. The most fundamental property of electrical insulation is its resistance. Measurements of IR are considered complex, since it is necessary to consider multiple factors.

In practice, the determination of IR is carried out using the "Megger" IR test, which is based on a non-destructive method. IR measurements are influenced by various factors, including temperature and humidity, making measurement conditions crucial. Periodic measurements allow for tracking trends and assessing insulation and electrical equipment conditions over time *[7]*. According to the IEC 60364-6 Standard, an installation cable must have at least 1 MΩ IR when measured, with a voltage of 500 V [8]. The relative dielectric constant describes the material's ability to behave like a capacitor, with values ranging between 2 and 3.5 depending on temperature and humidity *[9]*. Increases in capacitance and decreases in IR are explained by the concept of tangent delta or dielectric losses. Moisture ingress or partial immersion of insulation in water can increase capacitance and decrease IR, leading to the appearance of a real component of the current. The measurement of dielectric losses, known as tanδ, enables the detection of unnoticed cable conditions, such as local cable soaking or high humidity. Dielectric strength expresses a material's resistance to high voltage, determining the voltage it can withstand before breakdown occurs *[10]*. PDs, which can degrade the insulation, occur due to exceeding the dielectric strength limit of the surrounding material. PDs can be categorised into internal discharge, surface discharge and corona discharge *[11]*. Changes in mechanical properties, such as cable cross-section, maximum tensile strength, elongation at break, and hardness, reflect insulation and cable sheath ageing. The indenter method is used commonly for measuring degradation and mechanical properties, involving a small ball pressing against the cable to determine the indenter modulus *[12]*.

1.3 Insulating materials and cable structure

The planning or selection of insulation depends primarily on the surroundings in which the cable will be located and the operating voltage. Depending on the complexity, the insulation material itself is also chosen accordingly. However, it should be noted that more resistant materials will fall into a higher price range. Cables using the sheath or insulation will be considered at a basic level:

- Poly vinyl chloride,
- Ethylene propylene rubber (EPR),
- Cross-linked polyethylene,
- Chloroprene or neoprene, and
- Chlorosulfonated polyethylene, or known commercially as Hypalon.

A typical structure of a basic cable is represented in Figure 1.

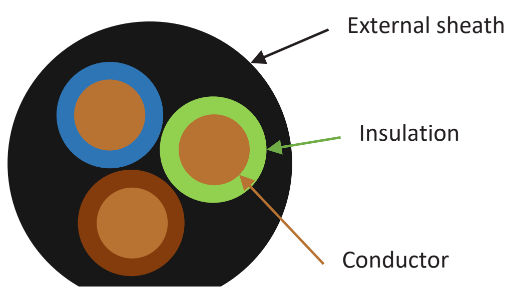


Figure 1: *Cross-section of a cable with three conductors in insulation and a sheath*

The cable used in our experiment consists of EPR used as the insulator and neoprene as the external sheath.

1.3.1 Ethylene propylene rubber

The ethylene-propylene rubber is a polymer chain manufactured through the reaction of ethylene and propylene. Both basic molecules are in a gaseous state at room temperature and atmospheric pressure. We obtain both molecules by splitting natural gas or petroleum *[13]*. The structures of the individual molecules are illustrated in Figure 2.

Figure 2: *Chemical structure of ethylene and propylene [13]*

The carbon (C) double bonds of both molecules can react with other molecules in various ways. Ethylene can bind with another ethylene or with propylene. Similarly, propylene can bind with another propylene or with ethylene. This forms a long chain of EPR. It is important to note that this chain is not two-dimensional, but three-dimensional, with the connection between molecules and their arrangement being random *[13]*.

EPR is one of the most commonly used polymers for low and medium voltage cables. As mentioned, the basic components include ethylene and propylene, and additional components such as hexane, diene and a catalyst solution are also classified as basic ingredients. All these components must be extremely pure and dry for the manufacturing process. The amorphous EPR itself has very poor mechanical properties, so special additives need to be introduced to improve both its mechanical and electrical characteristics *[14]*.

Materials such as clays, talc, fillers, silica, aluminium oxide, and other minerals, are added to the base EPR mixture to produce electrical insulation. In addition to these primary additives, other additives, such as metal oxides, plasticisers, antioxidants, zinc oxide, lead oxide, and other substances, are introduced into the mixture to optimise the manufacturing process.

Since EPR is fully saturated and nonpolar, this polymer exhibits excellent resistance to ozone, oxidation, high temperatures, water and polar solvents. EPR with a lower percentage of ethylene is amorphous and easier to process, while EPR with a higher percentage of ethylene demonstrates better mechanical properties. The resistance of EPR to all the beforementioned factors, as well as its resistance to corona, formation of water trees and other mechanical stresses, presents a significant advantage, and is a reason for the frequent use of this polymer as electrical insulation. As mentioned earlier, more than 10 different additives are introduced to EPR. Additionally, paraffinic, and naphthenic oils are added to the mixture as plasticisers and for optimising the extrusion process *[15]*.

1.3.2 Neoprene

Chloroprene, also known as neoprene, is an emulsion polymer composed of 2-chlorobut-1,3-diene. Due to the presence of chlorine, neoprene is polar, which is reflected in its exceptional mechanical properties, such as tensile strength, elasticity, resistance to weathering, chemicals, oils and fire. In addition to improved mechanical properties, neoprene also exhibits good electrical properties, making it a popular material for cable insulation. Neoprene is also used frequently for cable sheaths *[16]*.

As the temperature increases, the IR of neoprene decreases, a result of ion movement and the transition of neoprene from the crystalline phase to the amorphous phase. During this transition, the ions in the neoprene create dipoles, leading to increased conductivity. This phenomenon stems from the breaking of C-Cl bonds, causing the release of chlorine ions. These ions then react with ZnO, resulting in an increased concentration of impurities such as $ZnCl_2$ in the neoprene. This, in turn, causes increased electrical conductivity *[17]*. In addition to this reaction, chlorine also reacts with lead, forming $PbCl_2$, although these molecules are less problematic. $ZnCl_2$ is particularly challenging, as it is soluble in water, potentially causing corrosion of the surrounding materials *[18]*. Table 1 provides typical values for the electrical and mechanical properties of neoprene and EPR used in electrical insulation or sheathing.

Table 1: *Characteristics of insulation material based on neoprene [19, 20]*

Characteristics		Neoprene	EPR
Maximum operating temperature [°C]	Normal	90	40
	Maximum	130	120
	Short-term	250	180
Tensile strength [kg/mm^2]		2,8	0,95
Maximum elongation [%]		600	250-550
Specific resistance [Ωcm]		10^{11}	10^{15}-10^{17}
Dielectric constant [1kHz]		9	3,17-3,34
Dielectric strength [kV/mm]		19,7-27,6	35,4

2. METHODOLOGY

The term "ageing" denotes the inevitable degradation of electrical and mechanical properties, accompanied by chemical structural changes in materials, polymers, or cable insulation, leading ultimately to their dysfunction or failure. Ageing in polymeric materials is an inherent process affecting all plastic materials. As cables age, both the electrical and mechanical properties of the constituent materials deteriorate, resulting in increased material brittleness and vulnerability to external factors. Chemical changes in polymers, such as alterations in the glass transition temperature, crystallisation, and melting, intensify with ageing, necessitating laboratory analyses for detection. X-ray Fluorescence Analysis (XRF) is a rapid, precise and non-destructive analytical method used for determining the chemical composition of materials, applicable across various industries, including metallurgy, cement, petroleum, polymers, food, geology and mining. XRF, which can detect elements ranging from beryllium to uranium, operates in energy-dispersive and wavelength-dispersive modes, with a focus on energy dispersion here. This method's accuracy and reliability make it suitable for analysing both standard and non-standard samples, enabling the identification of ageing-related changes such as the reduction of elements like chlorine and barium in aged cables, facilitating the establishment of acceptance criteria *[21-24]*.

2.1 Theoretical background of XRF

X-ray beam or X-ray can be perceived as electromagnetic radiation, or as a photon with a specific energy. For easier understanding, we will describe it as a high-energy form of electromagnetic radiation with a wavelength between 10 picometers and 10 nanometers.

The spectrum of X-rays and other electromagnetic radiation with specified wavelength and corresponding energy is shown in Figure 3.

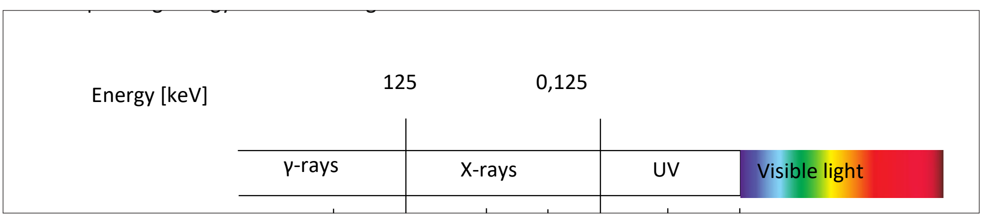


Figure 3: *Spectrum of electromagnetic radiation*

X-rays are generated through the conversion of kinetic energy acquired by an electron during acceleration with high voltage into electromagnetic radiation due to collisions and interactions *[23]*. To produce X-rays, a generator and electrodes are utilised, where the X-ray tube furnishes the requisite environment and electrons, while the generator provides the electrical voltage to electrodes. Matter interacts with X-rays through fluorescence, Compton scattering and Rayleigh scattering, with X-ray energy absorption contingent on the material's thickness, density, composition and the energy of the X-ray itself. The atom's classical model comprises a nucleus containing positively charged protons and neutrally charged neutrons, encircled by an electronic shell housing negatively charged electrons arranged in orbitals *[25]*. Electron ejection occurs when an atom is impacted by an X-ray beam with greater energy than the binding energy of the electron, leading to radiation absorption and fluorescence intensity. X-ray spectrometry determines element concentration quantitatively, employing empirical calculations or sophisticated theoretical methods, along with standard reference samples for accuracy *[26]*.

2.2 Analysis of an aged sheath and insulation of a cable sample

The portable X-ray spectrometer NITON XL3T 980 GOLD++ was utilised for the sample analysis. The analyser was equipped with a large geometrically optimised silicon detector and an X-ray tube of 50 keV. The adjustment of sample thickness can be made using the analyser software, which is always crucial in polymer analysis. Polymers are composed of lightweight materials, allowing deep penetration of X-rays into them. Easy sample analysis is facilitated by the software, as not only the energy at which responses occur are displayed, but also the corresponding elements. Figure 4 shows the analyser performing sample measurements.

Figure 4: *Analyser NITON XL3T 980 GOLD++*

The sample under analysis is a cable previously used in a lighting installation exposed to high temperatures exceeding 45 °C, locally even higher. The healthy section of the cable experienced temperatures ranging from 40 to 50 °C, while the partially degraded section endured temperatures between 55 and 65 °C. The completely degraded portion was subjected to temperatures ranging from 75 to 85 °C. Moreover, the environment also exhibited high humidity, persisting for 40 years. IR measurements were conducted on all sections, to establish a reference for the electrical insulation and sheath condition. The cable comprises a neoprene sheath and EPR insulation, with an unknown material filler between them. Given the cable's exposure to a dusty environment, potential impurities need identification and removal. The initial analysis involves IR measurements on all sections, including the sheath and conductors. The indentation modulus will gauge the electrical insulation condition and aid in interpreting the X-ray spectral analysis results.

Figure 5 shows the most degraded section of the cable sample.

Figure 5: *Part of the degraded cable sample*

3. RESULTS

3.1 IM measurements

The IM measurements were taken as a reference on individual sections of the cable. The condition of the cable was divided into three parts: healthy (soft), partially damaged (medium), and damaged (hard) cable. The IM-sheath measurements were conducted on all three sections of the cable, and XRF measurements were also performed on all three parts. Table 2 shows the sheath measurements with the indentation modules.

Table 2: *The sheath measurements with the indentation modulus*

	Healthy (soft) sheath of cable IM [N/mm]	Partially damaged (medium) sheath of cable IM [N/mm]	Damaged (hard) sheath of cable IM [N/mm]
Average	13,5	66,2	193,7
Standard Deviation	0,9	50,1	38,6

From the average value and Standard Deviation of the measurements shown in Table 2, significant deviations were observed in a very small section of the cable, indicating varying degrees of sheath degradation relative to the extent and length of the cable.

IM measurements were conducted on the degraded sections of insulation, specifically on all three wires: yellow-green, blue, and brown. Table 3 displays the IM values on the individual sections of the brown insulation.

***Table 3:** The insulation measurements with the indentation modulus*

	Healthy (soft) sheath of cable IM [N/mm]	Partially damaged (medium) sheath of cable IM [N/mm]	Damaged (hard) sheath of cable IM [N/mm]
Average	11,5	17,8	24,9
Standard Deviation	1,2	4,2	9,9

The measurements revealed the greatest degradation on the brown wire, while no significant signs of degradation were observed on the other wires. Consequently, the most detailed analysis was performed on the brown insulation.

In this case, the Standard Deviation was much smaller than that of the sheath measurement. However, for more precise results, the highest and lowest points were disregarded. It should be emphasised that, due to the non-critical state of IM in the insulation, it was smaller than in the sheath.

3.2 XRF measurements

The results of the jacket measurements are shown in Figure 6.

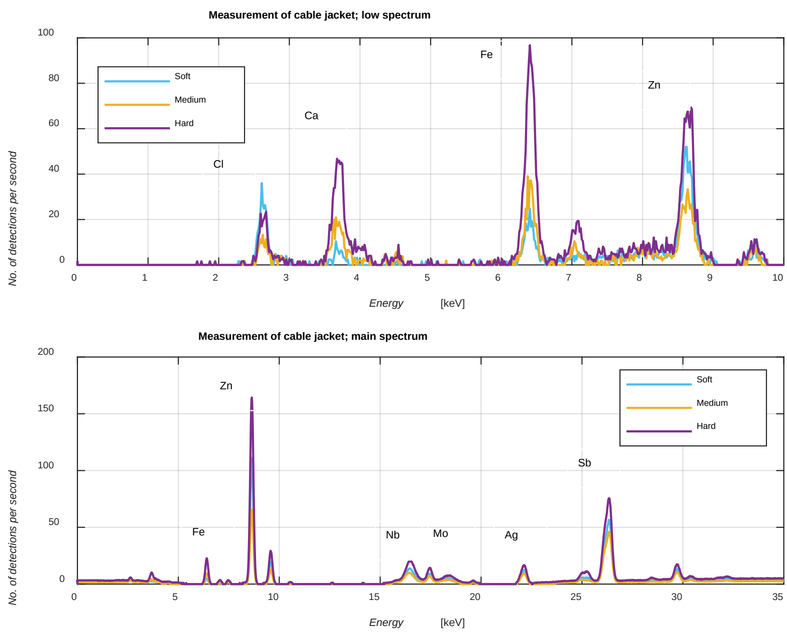
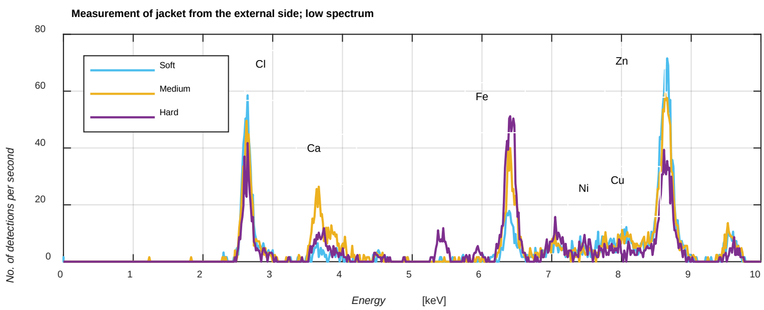


***Figure 6**: The results of the sheath measurements with XRF*

The measurements in Figure 6 indicate that the presence of chlorine generally decreases with ageing, which could be attributed to the degradation of the crystal structure, especially the covalent bonds of chlorine in the neoprene. An increase in calcium value with ageing was observed, suggesting potential leaching from the interior, as a higher presence of calcium was detected in the healthy section of the cable during the filler measurement. Higher iron values were recorded in the more degraded sample, possibly indicating contamination or leaching from the interior, a scenario applicable to zinc as well. The presence of zinc in cable insulation is expected, as it is added to the polymer to improve the mechanical properties in the form of zinc ions. It should be noted that the sheath remains on the cable, allowing the possibility of X-ray penetration depth measuring filler and insulation. Based on this assumption, measurements were conducted where individual parts were removed and measured separately. The results of the jacket measurements from the outside of the cable are presented in Figure 7.



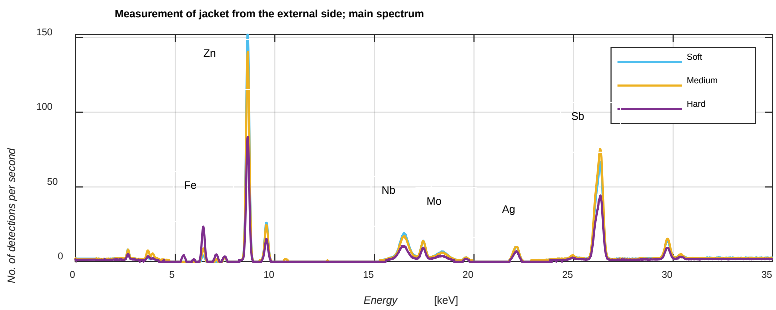


Figure 7: *The results of the jacket measurements from the removed cable's exterior with XRF*

Similar trends as those observed in the cable measurements emerged in these measurements (Figure 7). With the ageing of the cable, the presence of chlorine decreases, while the presence of iron increases. However, zinc presence decreased in this case, contrary to when the insulation was still on the cable. This difference in results could be attributed to different measurement points, contamination, or indicates that zinc may be present in the filler or insulation.

Additionally, the measurements detected the presence of nickel and copper, with the concentration of copper decreasing with ageing, suggesting contamination. The presence of these two materials generally indicates contamination. In the main spectrum, niobium and molybdenum showed a decreasing trend with ageing. Similarly, antimony exhibited such a trend, with slight deviations observed in the moderately aged sample.

The results of sheath measurements from the inside are depicted in Figure 8.

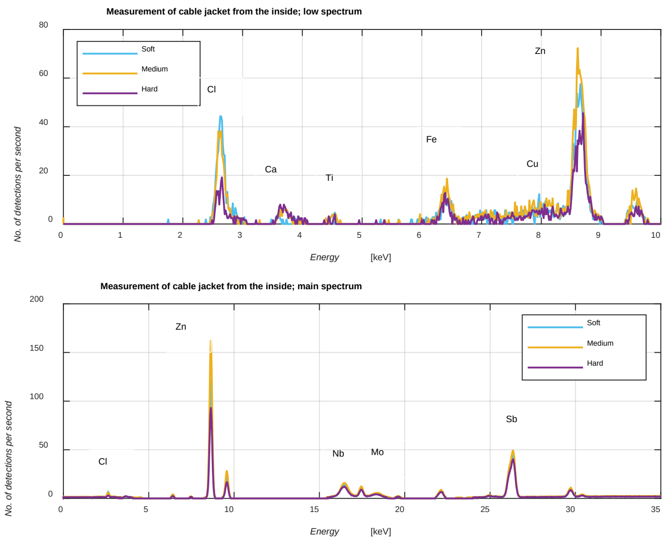


Figure 8: *The results of sheath measurements from the inside of the cable*

Figure 8 presents a pronounced decrease in chlorine concentration.

Iron and zinc maintained a decreasing concentration trend with ageing. No clear trend was observed for other elements, as measurements from the moderately healthy sheath deviated. This deviation may be attributed to contamination of the inner sheath due to the filler.

Figure 9 shows the results of filler measurements from all three parts of the cable.

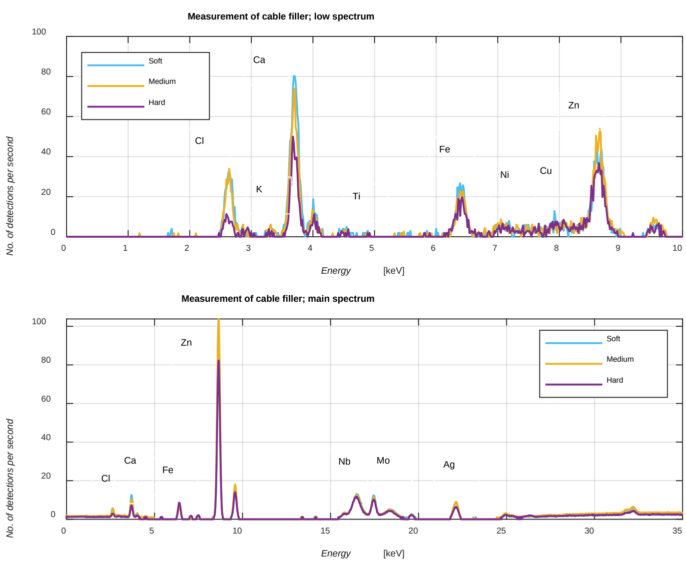


Figure 9: *The results of the filler measurements*

From the results presented in Figure 9, a trend of decreasing chlorine concentration with ageing is evident, which also applies to the filler. The most pronounced decrease was observed in the calcium. The presence of calcium may indicate the use of clay as an additive in the polymer, hence, monitoring this element is acceptable. Similar trends to those observed in other measurements were also noticeable for iron and zinc. The measurements also revealed concentrations of potassium, titanium, nickel and copper. The main spectrum measurements indicated decreasing trends for niobium, molybdenum and antimony.

The measurements of the cable insulation, specifically the brown-coloured insulation, which exhibited the greatest deviation in IM, are shown in Figure 10.

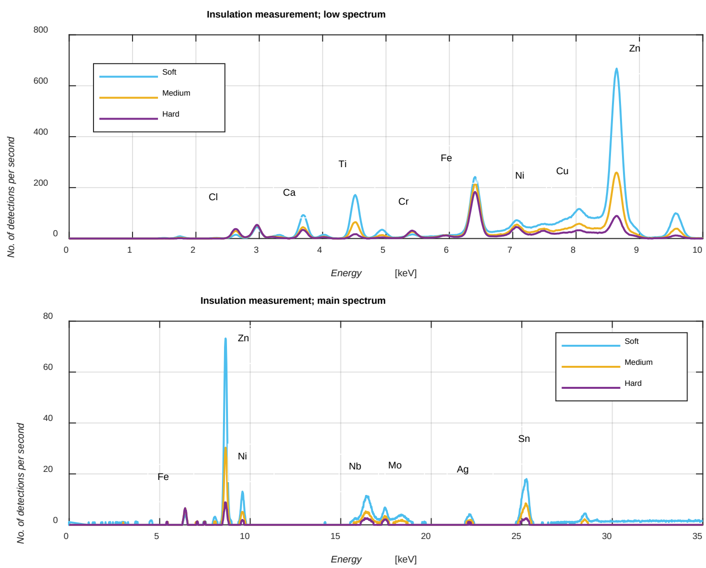


Figure 10: *The results of the measurements on the brown-coloured insulation*

In the low-spectrum measurements, the presence of chlorine, calcium, titanium, chromium, iron, nickel and copper reappeared, as shown in Figure 10. Calcium and nickel are elements present in the EPR itself. However, the measurements showed a decreasing concentration trend with ageing. Additionally, a decrease in zinc was observed, which is added to the EPR as a zinc oxide binder. The main spectrum measurements exhibited pronounced trends in niobium, molybdenum and antimony, where the concentrations decreased significantly with ageing.

Table 4 displays the proportion of individual elements in the healthy, partially healthy, and degraded samples.

Table 4: *The proportion of elements in cable samples of the insulation*

Element	Proportion of elements in the healthy sample [ppm]	Proportion of elements in the partially healthy sample [ppm]	Proportion of elements in the degraded sample [ppm]
Chlorine	2954,79	7973,02	23321,85
Chromium	58,76	326,63	941,11
Nickel	23,12	164,97	638,58
Tungsten	66,07	59,69	44,53

The results in Table 4 indicate a trend of decline or increase. The increase in concentration sometimes occurs due to the precipitation of this element on the surface.

4 DISCUSSION

Cables are crucial for integrating renewable sources into the power grid and ensuring smooth operation across all energy system stages. They play a vital role in critical infrastructures like nuclear power plants, ensuring uninterrupted safety system operation. Preventive maintenance is essential for early fault detection. Various methods, including mechanical and chemical property assessments, are used for cable maintenance and condition assessment. Efforts to simplify and standardise assessment methods involve establishing acceptability criteria. X-ray spectral analysis monitors the concentration trends of elements in cable sheaths and insulation during ageing, aiding in issue identification.

In addition to understanding chemical composition, considering Standard measurements with known acceptability criteria that determine cable condition is crucial. Trends of decreasing or increasing element concentrations are determined by performing IR measurements and XRF measurements on individual cable sections. Decreasing chlorine concentration in the sheath indicates the breakdown of covalent bonds in the crystal structure of the neoprene, indicating the ageing of this material. The presence of calcium in the filler suggests the use of clay in the polymer itself, while the decreasing trend in its concentration indicates material precipitation with ageing. In the cable insulation, a significant decreasing trend in antimony concentration was observed, which is used in polymers to add flexibility and fire resistance.

It is important to note that there is no universal method for assessing cable condition, as cable insulation consists of various materials with different properties. However, employing multiple methods can provide a comprehensive picture. This thesis serves as a foundation for further research and the potential application of X-ray spectral analysis, which is a useful method for monitoring the condition of cable electrical insulation.

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Nomenclature

(Symbols)	(Symbol meaning)
UV	Ultraviolet
IM	Indenter module
tanδ	Dielectric losses
VLF	Very low frequency
Un	Nominal voltage
PD	Partial discharges
IAEA	International Atomic Energy Agency
EPR	Ethylene propylene rubber
IEC	International Electrotechnical Commission
XRF	X-ray fluorescence spectrometry
IR	Insulation resistance