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Complex physicochemical analysis of transformer oil parameters using the inductively coupled plasma mass spectrometry technique

Introduction. Transformers are crucial and expensive components of power systems, experiencing electrical, thermal, and chemical stresses. Transformer oil analysis is important for diagnosing transformer faults and assessing its remaining service life. The oil used in transformers degrades over time due to its interaction with electrical loads and heat from the core and windings. The oil degrades into low-molecular gases and carbon particles, which affect its dielectric properties and indicate potential problems. Analysis of dissolved gases in oil allows early detection of defects such as corona or arc discharges, as well as overheating. In addition, analysis of metal content in oil helps to clarify the type and location of the fault identified by gas analysis. **Novelty** of the proposed work lies in the study of the relationship between transformer oil parameters and its quality, as well as the effect of dissolved gases. The article proposes a method for determining how changes in these parameters affect each other. The obtained data are compared with the results of mass spectrometric analysis for a more accurate assessment of the transformer condition. **The purpose** of this paper is to explore the connection between the chemical properties of transformer oil and the elemental composition determined through inductively coupled plasma mass spectrometry (ICP-MS). **Methods.** The solution to the problem was carried out using the inductively coupled plasma mass spectrometry method from Agilent Technologies 7700e (USA) to measure the concentration of metals in transformer oil. **Results.** An inverse correlation has been identified between the acidity of transformer oil and its furfural content. Experimental evidence has shown that the water content has the most significant impact on decreasing the breakdown voltage of dielectric oil. It was found that CO gas has the greatest influence on the formation of furfural. It has been established that gaseous C₂H₂ plays an important role in the formation of acidic components. Correlations were found between the oil acidity and the concentrations of copper and iron and between the breakdown voltage and the amount of lead and aluminium in the transformer oil. A high concentration of copper in the oil indicates potential issues with the transformer windings, as well as in any bronze or brass components, and the concentration of iron in significant quantities indicates problems with the transformer core and tank. Moreover, as the breakdown voltage of the oil decreases, there is a marked increase in the concentrations of lead and aluminum. This suggests that significant amounts of lead are found in the transformer solder joints, while aluminum is present in the windings and ceramic bushings. **Practical value.** The advantage of the mass spectrometric method for detecting metals in transformer oils is the ability to accurately determine the type of fault and diagnose transformer problems. Research shows that this method allows early detection of potential problems and predicts the condition of the transformer. References 21, table 2, figures 8.

Key words: transformer oil, furfural component, breakdown voltage, mass spectrometer, dissolved gases analysis.

Вступ. Трансформатори є важливими та коштовними компонентами енергосистем, які зазнають електричних, теплових та хімічних навантажень. Аналіз трансформаторної оливи важливий для діагностики несправностей трансформатора та оцінки його терміну служби, що залишився. Олива, що використовується в трансформаторах, з часом руйнується через його взаємодію з електричними навантаженнями та теплом сердечника та обмоток. Олива розкладається на низькомолекулярні гази та частинки вуглецю, що впливає на її діелектричні властивості та вказує на потенційні проблеми. Аналіз розчинених газів у оливі дозволяє завчасно виявити дефекти, такі як коронний або дуговий розряд, а також перегрів. Крім того, аналіз вмісту металу в оливі допомагає уточнити тип і місце дефекту, виявленого газовим аналізом. **Новизна** запропонованої роботи полягає у вивченні зв'язку між параметрами трансформаторної оливи та її якістю, а також впливом розчинених газів. У статті запропоновано метод визначення того, як зміни цих параметрів впливають одна на одну. Отримані дані порівнюють з результатами мас-спектрометричного аналізу для більш точної оцінки стану трансформатора. **Метою** статті є дослідження зв'язку між хімічними властивостями трансформаторної оливи та елементним складом, визначеним за допомогою мас-спектрометрії з індуктивно зв'язаною плазмою (ICP-MS). **Методи.** Рішення проблеми здійснювалося за допомогою методу мас-спектрометрії з індуктивно зв'язаною плазмою Agilent Technologies 7700e (США) для вимірювання концентрації металів у трансформаторній оливі. **Результати.** Між кислотністю трансформаторної оливи та вмістом у ній фурфуролу виявлено зворотну залежність. Експериментальні дані показали, що вміст води має найбільш значний вплив на зниження напруги пробою діелектричної оливи. Встановлено, що найбільший вплив на утворення фурфуролу має газ СО. Встановлено, що в утворенні кислотних компонентів важливу роль відіграє газоподібний C₂H₂. Були виявлені кореляції між кислотністю оливи та концентрацією міді та заліза, а також між напругою пробою та кількістю свинцю та алюмінію в трансформаторній оливі. Високий вміст міді в оливі вказує на потенційні проблеми з обмотками трансформатора, а також у будь-яких бронзових або латунних компонентах, а концентрація заліза в значних кількостях вказує на проблеми з сердечником трансформатора та баком. Крім того, у міру зниження напруги пробою оливи спостерігається помітне збільшення концентрації свинцю та алюмінію. Це свідчить про те, що значна кількість свинцю міститься в паяних з'єднаннях трансформатора, тоді як алюміній присутній в обмотках і керамічних втулках. **Практична цінність.** Перевагою мас-спектрометричного методу виявлення металів у трансформаторній оливі є можливість точного визначення типу несправності та діагностики проблем трансформатора. Дослідження показують, що цей метод дозволяє завчасно виявляти потенційні проблеми та прогнозувати стан трансформатора. Бібл. 21, табл. 2, рис. 8.

Ключові слова: трансформаторна олива, фурфуроловий компонент, пробивна напруга, мас-спектрометр, аналіз розчинених газів.

Introduction. By sampling transformer oil and conducting various tests, it is possible to diagnose numerous faults in the transformer and evaluate its remaining service life and overall condition. Transformer oils, like most insulating and dielectric materials, decompose and deteriorate during prolonged use. This is attributed to the oil's role in resisting electrical loads and facilitating heat transfer from the core and windings. The

condition of dielectric oil is influenced by contamination, its type, and the presence of acidic compounds like metal sulfide particles. Besides chemical degradation, dielectric oil also deteriorates due to physical contamination. When exposed to partial discharges, electrical arcs, and rising temperatures, dielectric oil breaks down into low molecular weight gases that dissolve in the oil, along with

carbon particles. The behavior of each type of insulation oil is influenced by the way carbon particles are transformed. Hence, analyzing dielectric oil is crucial for assessing the condition of a transformer and identifying its potential issues [1, 2].

Dissolved gas analysis (DGA) in insulation oil is a reliable method for detecting transformer faults early. These are typically used in transformers hydrocarbon (mineral) oils or silicones as insulating fluids because of their superior dielectric properties, heat transfer efficiency, and stability. These insulating fluids typically undergo minimal decomposition under normal conditions. However, damage can lead to the degradation of both the liquid and solid insulation materials [3]. Gases like H₂ (hydrogen), CH₄ (methane), C₂H₄ (ethane), C₂H₂ (acetylene), CO (carbon monoxide), and CO₂ (carbon dioxide) can dissolve in transformer oil, indicating decomposition caused by thermal or electrical stresses. Quantitative examination of these gases can help in detecting issues like corona, arc, spark discharge, and oil overheating, all of which can impact the transformer's operational lifespan. Furthermore, analysing metals in transformer oil supports dissolved gas analysis by specifying the nature and source of potential problems identified through gas analysis [4, 5].

High-energy faults not only damage transformer insulation materials like oil, paper, and wood but also generate metal particles that disperse into the oil. These particles can subsequently circulate throughout the transformer, mainly through the oil flow. Different components of a transformer produce distinct types of metal particles, which can manifest individually or in various compounds, and may appear in different concentrations. Identifying these particles can help narrow down the potential components contributing to failures [6].

Common metals found in transformer oil include aluminum, copper, iron, lead, silver, tin, and zinc. Two techniques used to analyze these metals in oil are mass spectrometry with inductively coupled plasma (ICP-MS) and mass spectrometry with atomic absorption. The amounts of metals in the oil must be measured using these techniques. Typically, the metal atoms in a sample are liberated by high temperature burning of the metal particles, making them amenable to accurate examination via these methods. The presence of these atoms in an atomic absorption flame or inductively coupled plasma can be determined by measuring discrete frequency absorption or positive ion emission (ICP) from the atoms, as well as the metal's ion concentration and composition. The Agilent Technologies 7700e inductively coupled plasma mass spectrometer was used in this study [7, 8].

Recently, ICP-MS has been gradually replacing flame atomic absorption spectrometry due to the significantly greater availability and performance of modern equipment while maintaining undoubted advantages, including the ability to record low detection limits for chemical elements and isotopes, down to ng/l and occasionally even pg/l levels, low consumption of the analyze, the ability to conduct multielement analysis, and high sensitivity and resolution of the analyser [9–11].

In previous studies [3–5, 12], the quality of transformer oil and the DGA have been individually

investigated and quantified as parameters for assessing transformer performance or diagnosing malfunctions. However, these works did not explore the interdependencies between the electrical and physicochemical parameters of transformer oils.

The goal of the paper to approach the study of how changes in transformer oil parameters affect each other, as well as the effect of dissolved gases on oil quality. In addition, the study compares these parameters with data obtained by mass spectrometry, which serves as a criterion for assessing the condition of transformers

Materials and methods. The research used a mass spectrometer with inductively coupled plasma (Agilent Technologies 7700e, USA) to measure and assess the concentrations of metals in the transformer oil (Fig. 1) [7].

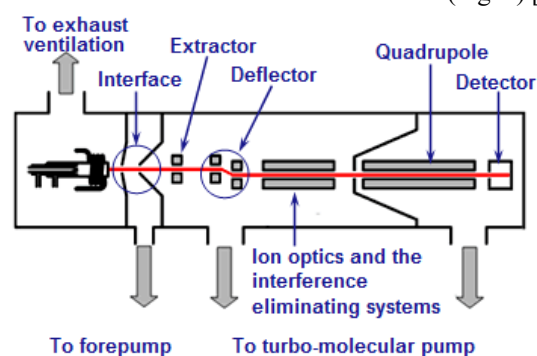


Fig. 1. Diagram of the ICP-MS Agilent 7700e [7]

To mineralize the samples, a «Speedwave Xpert» microwave system (Germany) equipped with small-volume vessels for working with microsamples was used to control the temperature (Table 1). Dispensers with volumes ranging from 100 to 1000 l and 1 to 10 ml, made by Pipet4u and Eppendorf (Germany), along with disposable tips and polypropylene tubes with capacities of 15 and 50 ml, were utilized.

Table 1
Procedure for microwave decomposition of transformer oil

Reagents	Acid	Volume			
	HNO ₃ (65 %)	8 ml			
Procedure	A 100 mg (0.1 ml) sample was added to the vessel, followed by the addition of 8 ml of HNO ₃ for mineralization. The mixture was then thoroughly shaken or stirred with a clean Teflon or glass rod. Before sealing the vessel, it should be allowed to sit for at least 10 min before being heated in a microwave oven according to the specified program				
Program	Step	T, °C	P, bar	t, min	P, %
	1	145	80	10	80
	2	170	80	10	80
	3	190	80	20	90
	4	50	60	10	0

To mineralize the materials and generate calibration solutions, 65 % nitric acid (HNO₃) was utilized. Furthermore, a 30 % solution of hydrogen peroxide (H₂O₂) from Suprapur (Merck, Germany) was used to quickly dissolve the samples during mineralization. The

solutions were diluted using deionized water with a resistivity of 18.2 MΩ·cm. The instrument's calibration accuracy was verified by analyzing a standard sample of drinking water.

Chromatography was used to detect the amounts of gases dissolved in the oil (H₂, CO, CO₂, CH₄, C₂H₆, C₂H₄, C₂H₂, N₂, and O₂). This was accomplished using an automated KRISTALLUX-4000 M gas chromatograph, which featured both a flame ionization detector and a thermal conductivity detector.

Experiments. Figure 1 shows a diagram of the ICP-MS Agilent 7700 instrument. The measurements were carried out on an ICP-MS system from Agilent Technologies 7700e (USA) under stable operating conditions [7–9]. The ICP-MS method is based on the use of an argon ICP as an ion source and a quadrupole mass spectrometer.

Figure 1 shows a diagram of the main parts of the instrument using an Agilent 7700e ICP-MS instrument as an example. The sample introduction system included a peristaltic pump, an atomizer, and a spray chamber. The solution of the studied substance was removed by a peristaltic pump at a speed of 0.1 ml/min. An aerosol was obtained from the sample solution and passed through a two-pass spray chamber. The fine aerosol obtained from the sample (leaving the spray chamber) directly enters a tube that directs the aerosol into a horizontally mounted plasma burner. The gas entering the three-cylinder plasma burner is called plasma, auxiliary gas, or carrier gas (supplied to the atomizer). A four-turn coil (inductor) is attached to the end of the burner, and a high-frequency signal (27.12 MHz) is supplied to it. After the plasma is enriched with electrons in a strong high-frequency field, collisions of argon atoms are ensured (i.e., plasma «combustion» is supported). At the plasma centre, the temperature reaches the range of 8000 to 10000 K. The aerosolized sample is instantly freed from the solvent and ionized. Furthermore, a beam of ions from the analysed sample is formed and introduced into the mass spectrometer through a system of cones and lenses. The ions then entered the quadrupole analyser. Only ions with a specific mass-to-charge ratio (m/z) can pass through the centre of the quadrupole under a specific combination of applied voltages.

The quadrupole provides a very fast (sawtooth) change in voltage because it can scan the entire mass range (from 2 to 260 Da) in 100 ms. As a result, mass spectra displaying the intensity vs. mass can be recorded for all elements virtually simultaneously. After passing through the quadrupole, the ions are detected by an electron multiplier. Table 2 shows some ICP-MS data from the experiments.

Table 2

Experimental mode	
Plasma, generator power, W	1450
Argon flow rate, l/min	1.2
Sample supply rate, l/min	1
Mass-spectrometer resolution, Da	0.2
Vacuum without plasma, Torr	4·10 ⁻⁴
Dynamic cell, gas	Helium
Time of measurement, s	0.1–0.5

Results and discuss. The analytical findings from 50 transformers, which included dissolved gases, oil quality parameters, and metals in the oil, were utilized to

determine the change or departure of oil quality parameters from one another to evaluate the transformer's performance and early diagnosis. The top results were chosen from 30 different analyses.

Carbon monoxide (CO) and carbon dioxide (CO₂) emissions found in transformer oil are indicative of a malfunction that may cause the deterioration and breakdown of paper insulation. The gases that indicate transformer overload include ethane (C₂H₆), ethylene (C₂H₄), and methane (CH₄). Acetylene gas (C₂H₂) indicates that there may have been an arc inside the transformer, which could have been brought on by a tap changer contact failure that resulted in internal shorts. The concurrent presence of methane, ethane, ethylene, carbon monoxide, and carbon dioxide gases (CH₄, C₂H₆, C₂H₄, CO and CO₂) in the dielectric oil signals the combustion of the transformer's paper insulation. The presence of hydrogen indicates the formation of partial discharges, and this gas is produced in most types of faults [13–15].

Transformer oil always contains oxygen, which leads to the formation of the gases CO and CO₂ and acidity. As the temperature rises in the transformer, oxide and acid components trigger a hydrolysis reaction, leading to the decomposition of the paper insulation molecules, resulting in pyrolysis. The products of hydrolysis and pyrolysis react to form furfural, which is generated from oxygen, acid, moisture, as well as CO and CO₂ gases. The cause of transformer oil and paper insulation degradation is the acid, moisture and oxygen contained in furfural [16–19].

Figure 2 illustrates the inverse relationship between the furfural component, which forms from the degradation of the transformer's paper insulation, and the acid component. Figure 2 shows that a 1 ppm increase in the acid component results in a 0.6 ppm decrease in the furfural component. The primary causes of acid formation in transformer oil are oxygen and oil oxidation. The degradation of transformer paper insulation and the formation of furfural are attributed to processes involving oxygen, hydrolysis, and pyrolysis [20]. Figure 2 clearly demonstrates that the concentration of furfural decreases as the acid content in the transformer oil increases.

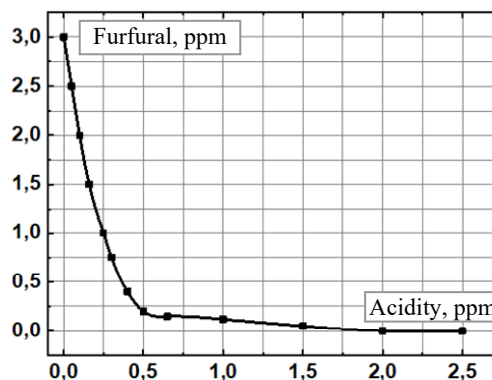


Fig. 2. Relationship between acid and furfural components of transformer oil

The dependence of furfural on the water content of the oil is depicted in Fig. 3. Apart from ambient moisture, the hydrolysis of paper insulation results in the creation of moisture within the insulating oil. As the temperature

increases, these bubbles evolve into partial discharge and hydrogen production [21].

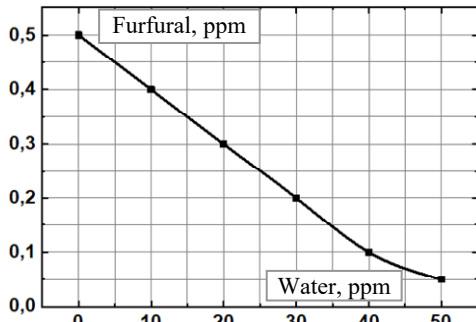


Fig. 3. Variation in furfural content with water content

Carbon monoxide is a gas produced during the decomposition of transformer paper insulation and has the most significant impact on the furfural component. Figure 4 shows that at low CO concentrations, the furfural content in the transformer oil does not change. However, as the heat increases and the paper insulation degrades, the carbon dioxide content increases, which increases the furfural content. The furfural component is a key parameter for assessing the degree of polymerization and estimating the remaining service life of transformer paper insulation.

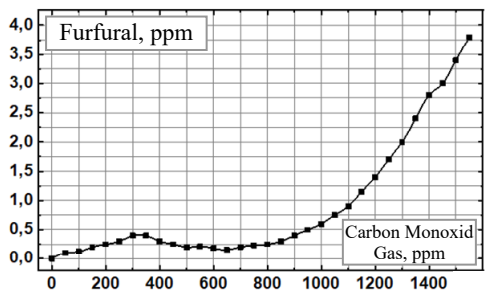


Fig. 4. Dependence of furfural on carbon monoxide

The breakdown voltage of transformer oil is a crucial indicator of its quality, reflecting its dielectric strength against factors such as arcing. The parameter exerting the most significant influence on this breakdown voltage is the water content. As the water content increases, the electrical conductivity of the oil also increases, thereby reducing its dielectric strength against electrical stress. Specifically, the breakdown voltage of the oil decreases by 1 kV for every increase of 1 ppm in water content.

Figure 5 illustrates the relationship between the concentration of copper and iron and the acidity of transformer oil. The data from the figure show that as the acidity of the oil increases, there is an exponential increase in the concentration of these metals. This confirms the correlation between oil acidity levels and the accumulation of metals such as copper and iron, which can indicate transformer issues.

Oil acidity is a critical parameter that directly affects its quality and operational safety. When the oil acidity exceeds 1 ppm, active corrosion of transformer components such as the core, windings, and tank occurs. This corrosion leads to the formation of iron and copper particles in the oil.

Copper can be found in the windings or in components made of bronze or brass, while iron is present in the core and tank of the transformer. These particles can result in significant transformer malfunctions, such as decreased electrical strength and increased risk of short circuits.

To determine the breakdown voltage of the oil, which can serve as an indicator of its contamination and degradation, a standard test cell is used. The breakdown voltage is measured according to the international standard IEC 60156, which describes the testing methodology and result interpretation. This standard allows for an objective assessment of transformer oil quality and its suitability for continued use.

Thus, the data in Figure 5 highlight the importance of monitoring oil acidity and regularly analyzing its composition to ensure the reliability and safety of transformer operation.

Threshold levels for metals in transformer oil are not universally established, but accumulating data and documented cases are making metal analysis in oil an increasingly valuable tool for the early detection of transformer faults before they escalate into serious issues. Relying on a single report for metal analysis is insufficient to fully assess the transformer's condition; establishing correlations between transformer oil parameters and elemental analysis obtained through techniques such as ICP-MS is crucial for a comprehensive understanding.

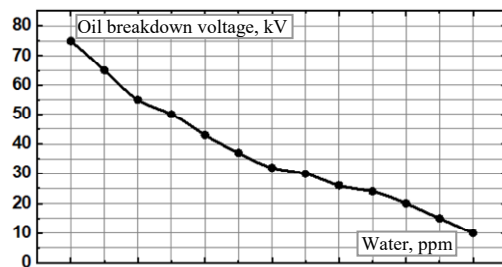


Fig. 5. Change in the breakdown voltage of transformer oil relative to the water content

Significant levels of iron and copper were detected in oils with acidity levels higher than 1 ppm. In fact, corrosion of several parts, including the transformer's core, windings, and tank, occurs as the acidity of the oil increases. As a result, iron and copper particles accumulate in the oil, causing transformer failure. Figure 6 illustrates how the amounts of iron and copper increase exponentially with the acidity of the oil. Iron is present in a transformer's core and tank, while copper is present in the windings and other bronze or brass components.

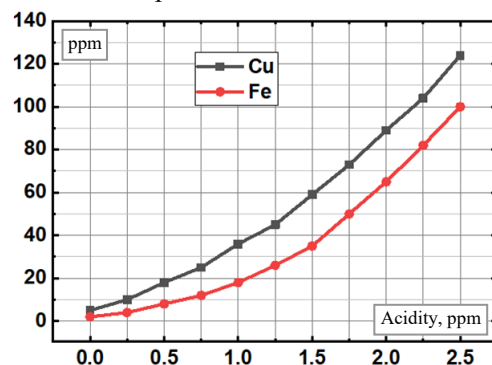


Fig. 6. Dependence of the acidity of transformer oil on the concentration of copper and iron

The primary factor affecting the transformer's performance the most is the variation in oil breakdown voltage. This is mainly influenced by the presence of contaminants and foreign particles in the dielectric oil,

which decreases both the breakdown voltage and the insulation effectiveness. Among these factors, the presence of water in the oil has the most significant impact on the breakdown voltage. According to findings in the literature, higher water content correlates directly with a reduced service life of the transformer. The value of furfural decreases with increasing temperature since the concentration of furfural is inversely related to the level of the acid component. CO₂ and CO gases have the greatest impact on transformer performance. These gases are formed when the paper insulation of the transformer decomposes into transformer oil. C₂H₆ has the most significant impact on the water content of transformer oil. Additionally, the most influential component of oil acidity is C₂H₂ gas. The advantage of using a mass spectrometric method to detect metals in transformer oils is to determine the type of fault and accurately diagnose transformer problems.

Additionally, as the breakdown voltage of the oil decreases, the concentrations of lead and aluminium increase sharply due to the decomposition of the dielectric oil under the influence of electrical voltage. The presence of particles like iron filings and impurities in dielectric oil leads to a reduction in both its breakdown voltage and dielectric strength. Figure 7 shows that at lower breakdown voltages, the concentrations of aluminium and lead increase significantly. Lead is commonly found in solder joints, connectors, and other ancillary components of transformers, while aluminium is present in the windings and ceramic bushings.

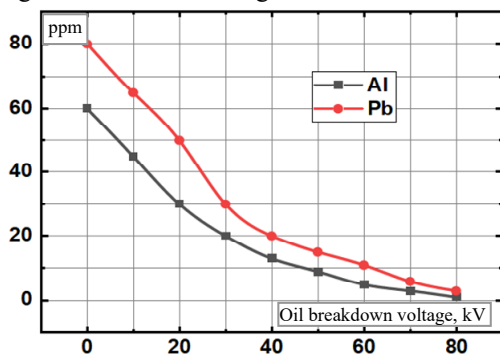


Fig. 7. Dependence of the oil breakdown voltage on the aluminium and lead concentrations

Elemental analysis of several oils revealed the presence of tin, silver, and zinc. It was experimentally revealed that a high concentration of water in the oil leads to partial discharge, which in turn increases the electrical conductivity of the oil, and sparking occurs, leading to the failure of several components of the transformer. Figure 8 shows that with a high concentration of water in the transformer oil, the concentrations of tin, silver, and zinc increase.

Tin, silver, and zinc may be present in the terminal, bolts, connectors and some peripheral components of the transformer, and their presence in the oil indicates failure of these components.

According to the findings, increasing acidity in transformer oil causes exponential increases in copper and iron concentrations. Copper levels above normally indicate problems with bronze or brass windings and components. Significant iron concentrations suggest difficulties with the transformer's core and tank, whereas large levels of aluminium indicate problems with the

ceramic bushing. Tin, silver, and zinc concentrations in the oil usually indicate wear on the tips and bolts. A significant amount of lead suggests that there may be difficulties with the transformer's solder joints, connectors, and peripheral components.

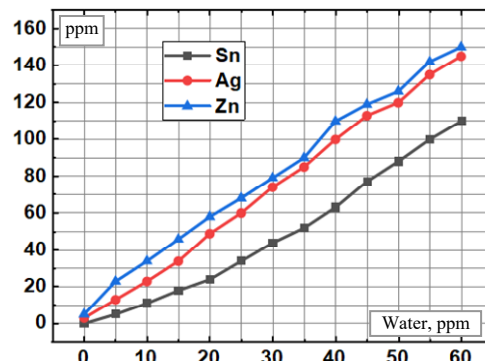


Fig. 8. Dependence of the amount of water in oil on the concentration of tin, silver, and zinc

Conclusions. The analysis revealed a significant correlation between moisture concentration, aluminum, lead, and the breakdown voltage of transformer oil. At equal concentrations, aluminum has a more pronounced effect on the breakdown voltage of the oil compared to lead.

Experimental studies have established a positive correlation between moisture content and the concentrations of tin, silver, and zinc in high-voltage transformer oil. The most critical factor affecting transformer performance is the change in the oil's breakdown voltage. The presence of particles and impurities in the oil reduces the breakdown voltage and, consequently, the insulation strength of the oil.

The most significant factor influencing the breakdown voltage is the water content in the oil. Therefore, water content is the key parameter that greatly reduces the transformer's lifespan, which aligns with previous research findings. Furfuryl alcohol is inversely proportional to oil acidity: as acidity increases, furfuryl alcohol decreases. Gases like CO₂ and CO have a major impact on transformer performance since they are formed during the decomposition of paper insulation. The gas C₂H₆ has the greatest influence on the oil's water content, while the gas C₂H₂ most significantly affects oil acidity.

The use of mass spectrometry for detecting metals in transformer oil allows for precise fault diagnosis and identification. Research indicates that with increasing oil acidity, the concentration of copper and iron rises exponentially. High levels of copper suggest issues with windings or components made of bronze or brass, while significant iron concentrations point to problems with the transformer core and tank. Aluminum is typically associated with ceramic bushings, while the presence of tin, silver, and zinc indicates wear on terminals and bolts. Elevated lead levels can indicate problems with soldered connections, connectors, and other peripheral components.

Conflict of interest. The authors declare that there is no conflict of interest.

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