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Reclamation of Aged Transformer Oil Using Thermally Activated Sludge Derived from Drinking Water Treatment

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Abstract

For electrical equipment (transformer) to last a long time, insulating oils are essential. The lifespan of a transformer is impacted by the ageing and degradation process of insulating oils under operating conditions. Since insulating oils are costly, regeneration is preferable to replacement. The most common materials used in this manner are aluminium oxides and silicates. In this work, aluminium oxides and silicates are obtained from the sludge produced from drinking water treatment. When water is treated with aluminium sulphate (alum), the produced sludge is enriched with silicon and aluminium oxides, as well as traces of Fe, Ca, Mg, Na, and K oxides. The produced sludges were thoroughly examined both quantitatively and qualitatively using different spectroscopic techniques. The performance of the extracted sludges is attributed to the large surface area. After the grinding method, the sludge was preheated at temperatures of 200°C, 400°C, 600°C, and 800°C for 2 hours to yield particles with high adsorption capacity, which increases with increasing activation temperature. Thus, a three-step lab-scale refining process was exactly applied. First, a physical oil refining process without a sludge refining cycle was performed to reduce water, gases, and sediments. Second, the sludge was used at varying doses with continuous stirring at different temperatures for 30 minutes to remove the aging products in the oil. Finally, three additional physical oils without sludge refining cycles were conducted. The optimum conditions were verified at 65°C with continuous stirring for 30 minutes using sludge dose 32% preheated at 800°C, under which the oil properties were improved significantly such as breakdown voltage, water content, colour, acidity, interfacial tension, dielectrically dissipation factor, furanic compounds content, total dissolved gases, and sulphur content were as follow 23 k.v./2.5 mm, 64 ppm, 6.5, 0.38 mg KOH/g oil, 19.3 mN/m, 0.44, 3818 ppb, 4247 ppm, and 0.39 % while after treatment process, the values were changed and became as follow 71 k.v./2.5 mm, 7 ppm, 1.5, 0.01 mg KOH/g oil, 40.2 mN/m, 0.0006, 109 ppb, 888 ppm, and 0.01 % respectively. It was found that the parameter values after the treatment process meet the IEC 60296-2020 standard for evaluating unused mineral insulating oils for transformers and switchgear.

Keywords: Insulating oils; Adsorbent material; Water treatment sludge; Transformer oil quality.

1. Introduction

Power transformers are among the most critical and expensive components in the electric power transmission and distribution system. Their failure can lead to significant outages and costly repairs, making proper maintenance essential to prolong their service life [1-5]. Although power transformers are designed to operate for over 30 years, many begin to fail after 20 years due to aging, internal faults, or deterioration of vital components [6]. One of the key elements affecting transformer reliability is the insulating oil, which plays a dual role: acting as a dielectric medium and as a coolant that helps dissipate heat generated during operation [7, 8]. However, under service conditions, transformer oil undergoes degradation due to exposure to oxygen, elevated temperature, and catalytic effects of metals or contaminants. This leads to oxidation, forming acidic by-products, sludge, and color changes, ultimately compromising the oil's dielectric and thermal performance [9-12].

To mitigate these effects and extend oil life, various purification and reclamation techniques have been developed. Among these, adsorption using natural or engineered clays has proven particularly effective. The most adsorbent material used in the reclamation of transformer oil is called fuller's earth, such as bentonite, montmorillonite, mesoporous bauxite, and kaolin clay have a high surface area and polar active sites capable of selectively removing polar degradation products from the oil while allowing the non-polar base oil to pass through unaffected. This process restores vital oil properties such as colour, total acidity, breakdown voltage, and dissolved gas levels [13, 14]. Recently, research work shows that the three eco-friendly inorganic fillers like activated bentonite, activated carbon, and silica fume, are used to reclaim aged transformer oil [15]. Most examined old oils had their sulphur level reduced by the use of kaolin clay. After being reclaimed by kaolin clay, the oil in each of the cases examined had no trace of corrosive sulphur on the copper strip surface [16].

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Interestingly, recent studies have highlighted the similarity in chemical composition between common adsorbents (e.g., bentonite and kaolin) and the alum generated as a waste product in water treatment facilities. Alum sludge, primarily composed of aluminum and silicon oxides, presents a disposal challenge due to its large volume and large storage requirements. However, its mineral content and textural properties suggest it could be repurposed as a low-cost, environmentally sustainable adsorbent for transformer oil reclamation [17-20].

This work explores the feasibility of using water treatment sludge, which is an activated thermally, as an alternative adsorbent for reclaiming aged transformer oil. The novelty of this study is centered not only to improve oil quality and transformer reliability but also to contribute sustainable waste management practices and obtain environmentally friendly materials that can be used as transformer oil treatment to improve their durability and operational efficiency. From an economic aspect, transformer oil is expensive. Hence, recycling aged transformer oil plays an important role in industrial applications. In addition, the adsorbent material (sludge) is readily available and freely accessible without additional cost in our Egyptian power plants. Moreover, the actual cost of the oil treatment process is negligible compared to the high price of virgin oil.

To the best of the author's knowledge, this is the first study on the reclamation of Egyptian transformer oil using a lab-scale refining unit and thermally activated sludge produced from drinking water treatment, which is available at Egyptian power plants as a waste. Moreover, this is the first study to employ both sensitive short-long orthogonal dual-pulse laser-induced plasma spectroscopy (SLODP-LIPS) and inductively coupled plasma-optical emission spectrometry (ICP-OES) as spectroscopic techniques for evaluating the elemental activated sludge from drinking water treatment. The identification of Fe, Ca, Mg, Na, and K compositions by SLODP-LIPS shows a great match with the ICP-OES standard performance.

2. Materials and methods

2.1. Aged Transformer Oil

The oil was collected from an extra-high voltage transformer with a rated voltage of 220/66 kV after more than thirty years of operation, and its different properties were estimated and listed in Table 1. Our results, shown in Table 1, indicate the presence of the greatest physical and chemical deterioration in the aged transformer oil sample, high dissolved gas concentration, and high content of furanic compounds.

Table 1: Test results of aged transformer oil sample.

Test		Unit	Standard Method	Results
Density at 20 ºC		Kg/m^3	ASTM D 1298	0.8811
Kinematic Viscosity at 40ºC		mm^2/s (cSt)	ASTM D 445	10.32
Flash Point closed		ºC	ASTM D 93	142
Flash Point open		ºC	ASTM D 92	150
Color		ASTM Color Scale	ASTM D 1500	6.5
Total Acidity		mg KOH/goil	ASTM D 974	0.38
Interfacial Tension		dyne/cm	ASTM D 971	19.3
Appearance		--------	--------	Dark with suspended matter
Water content		ppm	*IEC 60814*	64
Breakdown Voltage		KV/2.5mm	IEC 156	23
Dielectric dissipation factor at 90ºC		--------	IEC 60247	0.44
Cupper strip tarnish test		--------	ASTM D 130	Not Corrosive
Dissolved gas analysis	Hydrogen	ppm	ASTM D 3612	133
	Carbon dioxide			3054
	Ethylene			5
	Ethane			2
	Acetylene			0
	Methane			1

	Carbon monoxide			1052
Furanic Compounds	2-Furfural	ppb	ASTM D 5837	3609
	5-hyroxymethyl-2-furfural			89
	2-acetylfuran			90
	5-methyl-2-furfural			30
	Total Furanic compounds			3818

2.2. The adsorbent material (sludge)

The dried and grinded adsorbent material (sludge) resulting from water treatment is shown in Figure 1.

Fig. 1. The grinded dried sludge at 200°C.

The adsorbent material used in the treatment process was heated at 200°C, 400°C, 600°C, and 800°C for two hours, then kept in a desiccator.

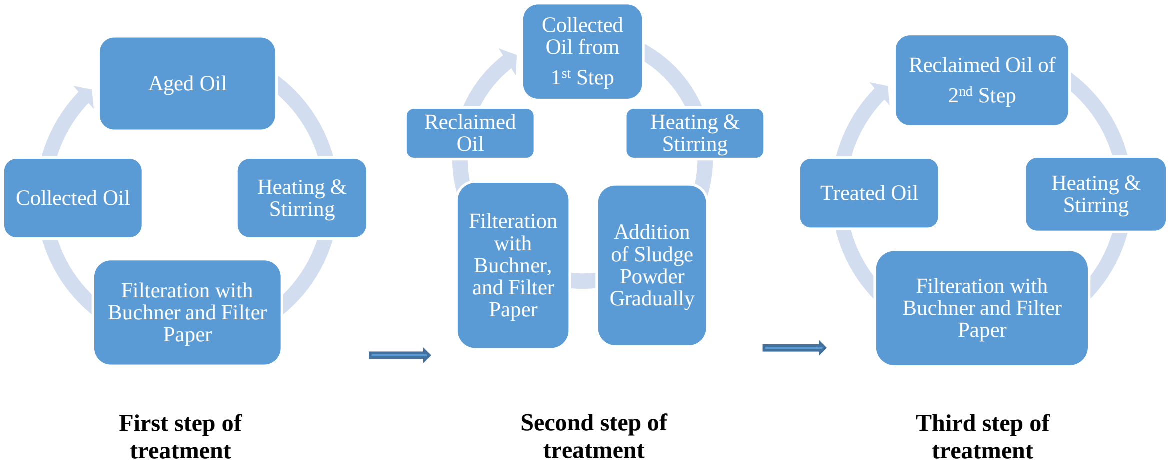
2.3. Treatment process of the aged transformer oil

The lab refining unit used for treating the aged transformer oil, as shown in Figure 2, consists of a vacuum pump, a Buchner funnel, filter papers grade 42, and a hot plate with a stirrer.

Fig. 2. Lab refining unit.

The treatment process was performed in 3 steps as shown in Fig. 3. The first step was a physical treatment (one cycle refining) without using the adsorbent material through passing the aged oil after heating it to 65°C (the optimum temperature) on the lab-scale refining unit, where a Buchner was installed with a vacuum pump and filter paper. The treated oil was finally collected in a conical Buchner flask. This step decreased the amount of undesirable water, gases, and sediments that will affect the performance of the adsorbent material during the chemical treatment. While, the second step was a chemical treatment using the heated adsorbent material at (200°C, 400°C, 600°C and 800°C) and lab-scale refining unit where the aged oil was heated with the adsorbent material of different doses (8%, 16%, 24% and 32%) with a continuous stirring at various temperatures (25°C, 40°C, 50°C and 65°C) for 30 minutes to remove the oxidation products, furanic compounds and any polar contaminants besides lightening the color of the oil.

The acidity and color of oil were measured under these conditions. Appearance, interfacial tension, dielectric dissipation factor, kinematic viscosity, elemental analysis of the oil, and furanic compounds content were measured under optimum conditions (the adsorbent material was activated at 800°C for two hours, the experiment temperature was 65°C for ½ hour). Finally, the third step involved using a physical treatment again without adsorbent (three cycles of refining) to remove the remaining water, gases, and sediments. Breakdown Voltage, Dissolved Gases, Water Content, Flash Point, Density, and Copper Strip Tarnish Test were measured after this step.



First step of treatment

Second step of treatment

Third step of treatment

Fig. 3. Schematic flow diagram of oil treatment process.

3. Results and discussion

3.1. Structure and chemical composition of the adsorbent material

Our study focused on the preparation of extracted sludge from drinking water, for clearing polluted transformer oil, and monitoring the elements in the extracted sludge. The objective was to investigate the impact of the adsorbent material sludge on enhancing the purification of oil adsorption efficiency. Quantitative and qualitative analysis of this extracted sludge using Optical Microscope, Energy-Dispersive X-ray spectroscopy (EDX), Inductively Coupled Plasma (ICP), and short-long orthogonal double pulse-Laser-Induced Plasma Spectroscopy (SLODP-LIPS).

3.1.1. Microscopic examination of adsorbent using optical microscopy

The adsorbent material that was heated at different temperatures (200°C, 400°C, 600°C, 800°C) was examined using an Optical Microscope LEICA DM750 with a camera LEICA ICC50 W. The images were performed at various magnifications (40x, 100x, and 200x) as shown in Figure 4. It was observed that the untreated adsorbent sample at all magnifications has a surface that appears relatively compact, smooth, with minimal porosity, and the particle distribution was dense and unstructured. Concerning the thermally activated adsorbent sample at 200°C, slight surface changes were observed at magnifications of 100x and 200x, respectively. In addition, small pores began to form, indicating the onset of structural modification due to mild thermal activation. When the adsorbent sample was thermally activated at 400°C, the porosity increased, and surface roughness became evident.

The pore formation was more pronounced, with visible inter-particle gaps suggesting initial decomposition of volatile components. Concerning the adsorbent sample thermally activated at 500°C, the surface morphology showed noticeable fragmentation and pore enlargement. A network of micro-channels was visible, particularly at 200x magnification. Regarding the adsorbent sample thermally activated at 600°C, a significant structural transformation occurred, and the particles appeared more separated, with increased surface area and well-developed pores, enhancing the adsorptive capacity. In addition, the adsorbent sample thermally activated at 800°C gives an adsorbent with a highly porous and fragile structure at this high temperature. Nevertheless, the agglomerates were reduced, and the surface was extensively modified, exhibiting interconnected pore networks and an increase in active sites.

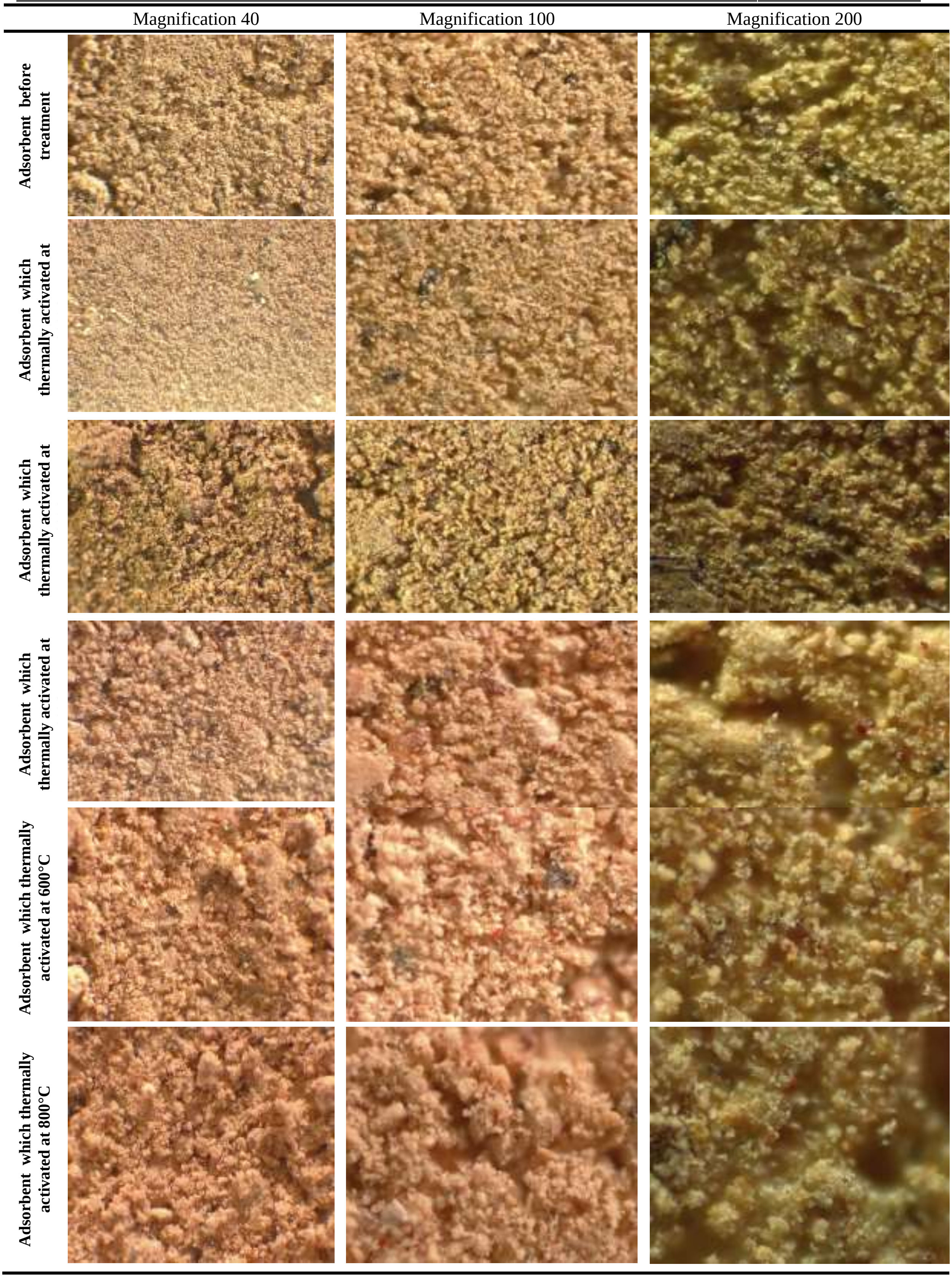
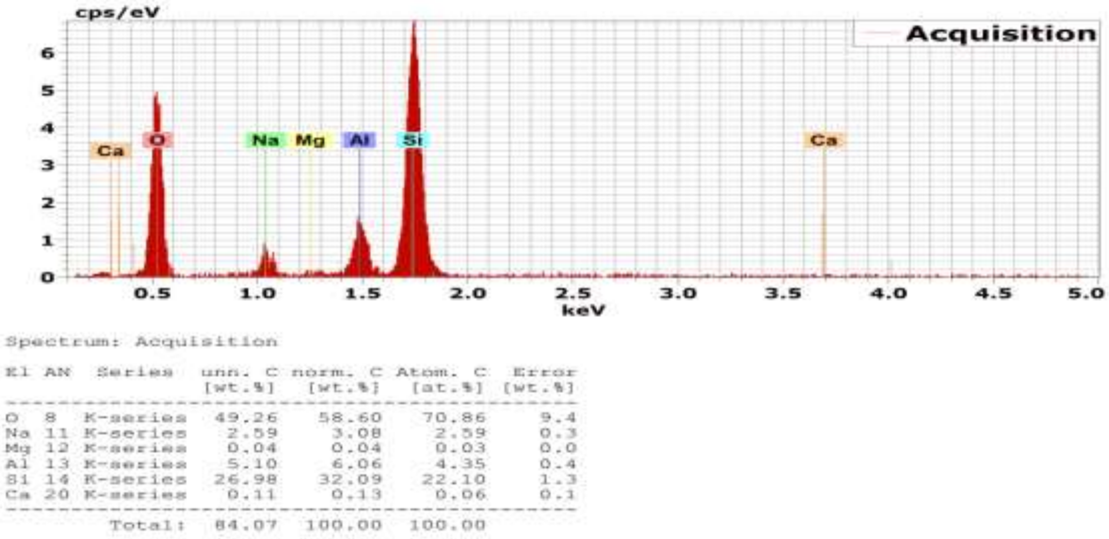


Fig. 4. Optical microscopy images of the adsorbent at different magnifications.

3.1.2. Energy Dispersive X-ray analysis

The adsorbent material resulting from water treatment (thermally activated sludge at 800°C) was analyzed using an Energy Dispersive X-ray spectrometer (EDX) ISIS Link Instrument P/C. Oxford Co., which is attached to SEM. Figure 5, shows spectra of the adsorbent material that was thermally treated at 800°C. This material was enriched with Al, Si, and Na oxides and traces of Mg, K, and Ca oxides.



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Spectrum: Acquisition

El AN  Series    unn. C norm. C Atom. C  Error
                 [wt.%]  [wt.%]  [at.%] [wt.%]
------------------------------------------------
O   8  K-series  49.26   58.60   70.86    9.4
Na 11  K-series   2.59    3.08    2.59    0.3
Mg 12  K-series   0.04    0.04    0.03    0.0
Al 13  K-series   5.10    6.06    4.35    0.4
Si 14  K-series  26.98   32.09   22.10    1.3
Ca 20  K-series   0.11    0.13    0.06    0.1
------------------------------------------------
          Total:  84.07  100.00  100.00
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Fig. 5. EDX spectra of thermally treated adsorbent material at 800°C.

3.1.3. Short long Orthogonal Double Pulse-Laser-Induced Plasma Spectroscopy (SLODP-LIPS) analysis on the sludge

The experimental setup for SLODP-LIPS is detailed in earlier studies [21-28]. The process involved grinding and mixing acceptable sludge powders into a uniform consistency using a Moulinex blender. For the SLODP-LIPS analysis, the sludge powders were compacted into pellets, which were then irradiated with laser flux to record their emissions. Specifically, the powder was compressed into disks measuring 50 mm in diameter and 7 mm in thickness using a cylindrical die and compacted under hydraulic pressure (approximately 9.5×10^6 Pa) for 30 minutes. This step ensured the formation of well-compact pellets, minimizing potential signal inaccuracies during SLODP-LIPS analysis. The prepared samples were stored in labeled petri dishes without any additives and kept in a moisture-free, evacuated desiccator to prevent contamination. These pellets demonstrated sufficient durability to resist the high temperatures and shock waves generated by laser pulses. The results showed that the extracted sludge materials exhibited enhanced adsorption efficiency compared to the control samples. SLODP-LIPS proved to be a powerful and sensitive tool for monitoring the presence of heavy metals such as Si, Al, Fe, Ca, Mg, K, Na, and O. Notably, the findings from SLODP-LIPS aligned closely with those obtained through ICP-OES, providing further validation of its reliability. This innovative approach has significant potential for contributing to advances in transformer oil technologies. The SLDP-LIPS analytical system, developed relatively recently, offers several advantages in elemental identification by exciting atoms in a sample without selectivity [29-35]. The technique involves directing a pulsed laser beam onto the sample surface, leading to ablation, atomization, ionization, and subsequently the formation of a plasma plume from a small amount of material (just a few nanograms). The physical properties of the sample, along with experimental parameters such as photon absorption, shock wave generation, vaporization, stress wave formation, melting, and laser shielding, significantly affect plasma formation. The plasma generated emits discrete radiation as well as a continuum spectrum, which cools within microseconds. Its characteristic spectral pattern results from discrete transitions of excited atoms or ions within the plasma, allowing for elemental identification in the sample. This emitted light is then collected by a spectrometer for detailed analysis of the target's composition. SLODP-LIPS is especially versatile, as it works with liquids, solids, and gases regardless of their conductivity and requires minimal or no sample preparation. Additionally, it can analyze extremely hard materials such as semiconductors, superconductors, and ceramics for simultaneous multi-elemental analysis. However, SLODP-LIPS has certain limitations, including the need for suitable standards for reliable quantitative analysis, its relatively high cost, and matrix effects that can interfere with multi-elemental assessments. Typically, SLODP-LIPS achieves precision ranging from 5% to 10%, depending on variables such as sample matrix properties, laser excitation characteristics, and sample uniformity. Spectra derived from multiple positions on the pellet or sample surface are averaged to produce a single representative spectrum for both blank and annealed samples. Laser-induced plasma spectroscopy of the sludge material produced spectra in the wavelength range of 250–975 nm, as illustrated in Figure 6. Most of the observed atomic and ionic spectral lines are tabulated in Table 2 via the NIST database [NIST 2024 Atomic spectra database].

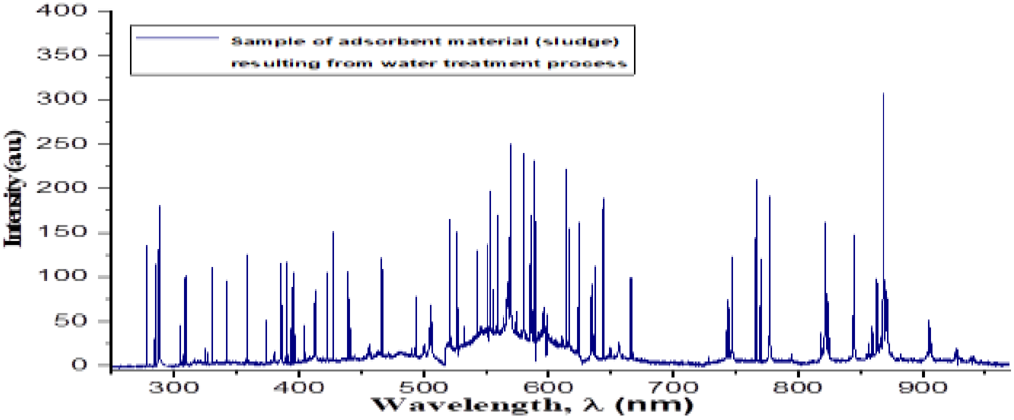


Fig. 6. SLODP-LIPS emission spectrum of the adsorbent powder burned at 800 ºC.

Table 2. List of major spectral lines observed.

Element	Spectral Lines
Si (I), Si (II)	288.16, 390.55, 494.76, 615.51, 742.35, 794.40, 855.68, 385.60, 413.09, 504.10, 505.59, 637.14, 666.05,
Al (I), Al (II)	309.30, 394.40, 396.20, 569.60, 586.18, 422.68, 466.40, 559.33
Fe (I)	330.64, 342.42, 358.21, 373.48, 404.58, 440.77, 520.85, 526.95, 542.92
Ca (I)	551.28, 616.22, 643.91
Mg (I)	277.90, 284.80, 765.90, 821.30
K (I)	580.18, 766.49, 769.89
Na (I)	427.36, 439.00, 588.99, 589.59
O (I)	777.19, 926.601
N (I)	939.28

3.1.4. Inductively coupled plasma (ICP) spectroscopy

The activated adsorbent material was thermally treated at 800°C (optimum temperature) and was well analyzed using an inductively coupled plasma spectroscopy (ICP-OES spectrometer Model optima 8300 optical emission spectrometer Perkin Elmer). This technique was used to measure and identify the elements of the adsorbent material. Table 3 shows the percentages of the elements Al, Fe, Ca, Mg, K, and Na in the adsorbent material. the results indicate a high concentration of Al%, a low concentration of Fe% and Ca%, and traces of Mg%, K%, and Na%.

Table 3. List of elemental percentage in the adsorbent observed.

Element	Percentage (%)
Al	11.6
Fe	2.21
Ca	1.75
Mg	0.63
K	0.28
Na	0.194

3.1.5. Comparison between SLODP-LIPS results and the ICP results of the adsorbent

To validate the results obtained from SLDP-LIPS, a comparison was made with ICP analysis for elements Si, Al, Fe, Ca, Mg, Na, and K as detailed in Table 4. The results demonstrate a strong correlation between the SLDP-LIPS and ICP-OES results, underscoring the reliability of SLDP-LIPS for elemental analysis. The results showed that the adsorbent was enriched with Si, Al oxides, and small amounts of Fe, Ca oxides, with traces of Mg, K, and Na oxides.

Table 4. Percentage results of SLODP-LIPS and ICP results of the heavy elements.

Element	SLDP-LIPS results (%)	ICP results (%)
Al	11.9	11.6
Fe	2.42	2.21
Ca	1.84	1.75
Mg	0.68	0.63
K	0.31	0.28
Na	0.21	0.194
Si	24.1	-----

3.1.6. Thermo gravimetric analysis (TGA) of the activated sludge

Thermo gravimetric analysis of the adsorbent material, which was thermally activated at 800°C, was conducted using Thermo gravimetric DE Terminator Lecco - Mac-500.ST.Joseph, which continuously measures sample weight loss across temperatures ranging from ambient to 900 °C. Samples were placed in an alumina crucible and heated to 900 °C at a rate of 10 °C / min with nitrogen as the circulating gas. The resulting thermo gram, depicted in Figure 7, reveals four distinct decomposition stages, the first stage (from 55°C to 100°C), where a weight loss of 25.2 % was observed, primarily attributed to the evaporation of moisture content. Second stage (from 100°C to 220°C), and this phase showed a 30.9% weight loss, corresponding to the decomposition of hydrated hydroxides such as [Al (OH) 3.xH2O] and others. The third stage (from 220°C to 600°C), where a weight loss of 34.6 % was observed, is associated with the carbonization processes. The final fourth stage (from 600°C to 900°C) involved a 39.2% weight loss related to the calcination process.

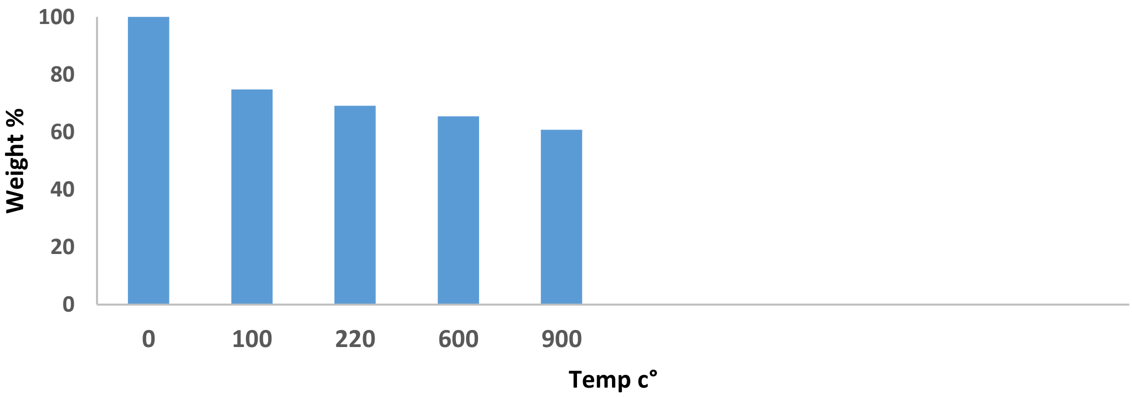


Fig. 7. Thermo gravimetric analysis of the adsorbent material observed.

3.2. Characterization of the transformer oil

3.2.1. Comparison between SLDP-LIPS results and the ICP results of the aged oil before and after treatment

Short-long Orthogonal Double Pulse-Laser-Induced Plasma Spectroscopy (SLODP-LIPS) analysis and Inductively Coupled Plasma Spectroscopy (ICP spectroscopy) were conducted on the oil before and after treatment with the adsorbent to measure and identify the elements inside the oil and study the effect of treatment on their concentrations. The comparison between results is shown in Table 5. The results of both techniques were consistent with each other. It was observed that the concentration of Si, Al, and Fe increased in the oil after the treatment process with sludge, while the concentration of Ca, Cu, Zn, Mg, Na, and K decreased. This means that it was an ion exchange between the sludge and oil during the adsorption process. whereas the metal cation in the adsorbent was replaced by another low-valence metal cation (i.e., Si^{4+} was replaced by Al^{3+}, Fe^{3+}, and Al^{3++}, Fe^{3+} were replaced by Mg^{2+}, Ca^{2+}, Zn^{2+}, Cu^{2+}). These substitutions lead to a charge deficit, which is compensated by cations such as H^+, Na^+, and K+ from the oil.

Table 5. List values of both SLODP-LIPS and ICP results of the aged oil before and after treatment.

Elements	SLDP-LIPS results (ppm)		ICP results (ppm)	
	Before treatment	after treatment	Before treatment	after treatment
Al	0.158	0.413	0.152	0.394
Ca	1.26	0.786	1.23	0.723
Cu	1.38	1.201	1.34	1
Fe	0.428	1.18	0.423	1.13
Na	1.61	1.48	1.59	1.44
Zn	0.326	0.071	0.321	0.09
K	0.327	0.216	0.321	0.208
Mg	0.225	0.142	0.224	Nil
Si	0.116	0.516	-----	-----

So, carboxylic acids, oxidation products, and other polar contaminants presented in the aged oil will bind to its positively charged site with the negatively charged site of the adsorbent material by means of hydrogen bonds and Van der Waals forces, as sketched in Figure 8.

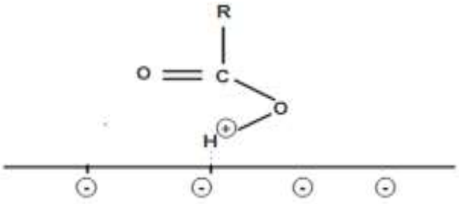


Fig. 8. Hydrogen bond between carboxylic acid and adsorbent material showed.

3.2.2. Elemental analysis of oil before and after treatment

The elemental analysis (C, H, N, and S) was determined by [Leco-Truspect (CHN) Analyser, Lecco Corporation, 3000 LAKE View AVE. ST. Joseph, MI-USA]. The results before and after final treatment are listed in Table 6.

Table 6: The elemental analysis of oil.

Elements	Percentage before treatment (%)	Percentage after treatment (%)
C	85.43	84.65
H	12.57	12.34
N	0.25	0.19
S	0.39	0.01

In Table 6, the data reveal that the concentration of carbon content in oil was reduced by the adsorbent. The reduction in carbon element by the treatment may be attributed to the elimination of carbon impurities present in the aged oil due to ageing conditions. The results also indicate a significant reduction in sulphur content by the adsorbent material. This reduction is beneficial since the presence of sulphur in transformer oils is undesirable.

3.2.3. Effect of the chemical treatment on the acidity of oil

The acidity (neutralization value) of the oil is a measure of its acidic constituents or contaminants. The acidity of used oil is caused by acidic oxidation products. The oil's dielectric and other properties will change as a result of reactions between solid, water contaminant, acids, and other oxidation by products. Acids also alter the breakdown of cellulosic materials and can lead to the corrosion of transformer metal components [35, 36]. (Figures 9a, 9b, 9c and 9d) show total acidity test results with variation of the adsorbent material's activated temperature (200°C, 400°C, 600°C, 800°C) and its amount (8%, 12%, 24%, 32%) at various experiment temperatures (25°C, 40°C, 50°C, 65°C) and experiment duration (½ hour). These figures illustrate how the adsorbent material eliminates acidity and oxidation products from the oil. The acidity was improved and reduced from 0.38 to 0.01 mg KOH/g oil. Besides, as the activation temperature increases, the acidity of the oil decreases.

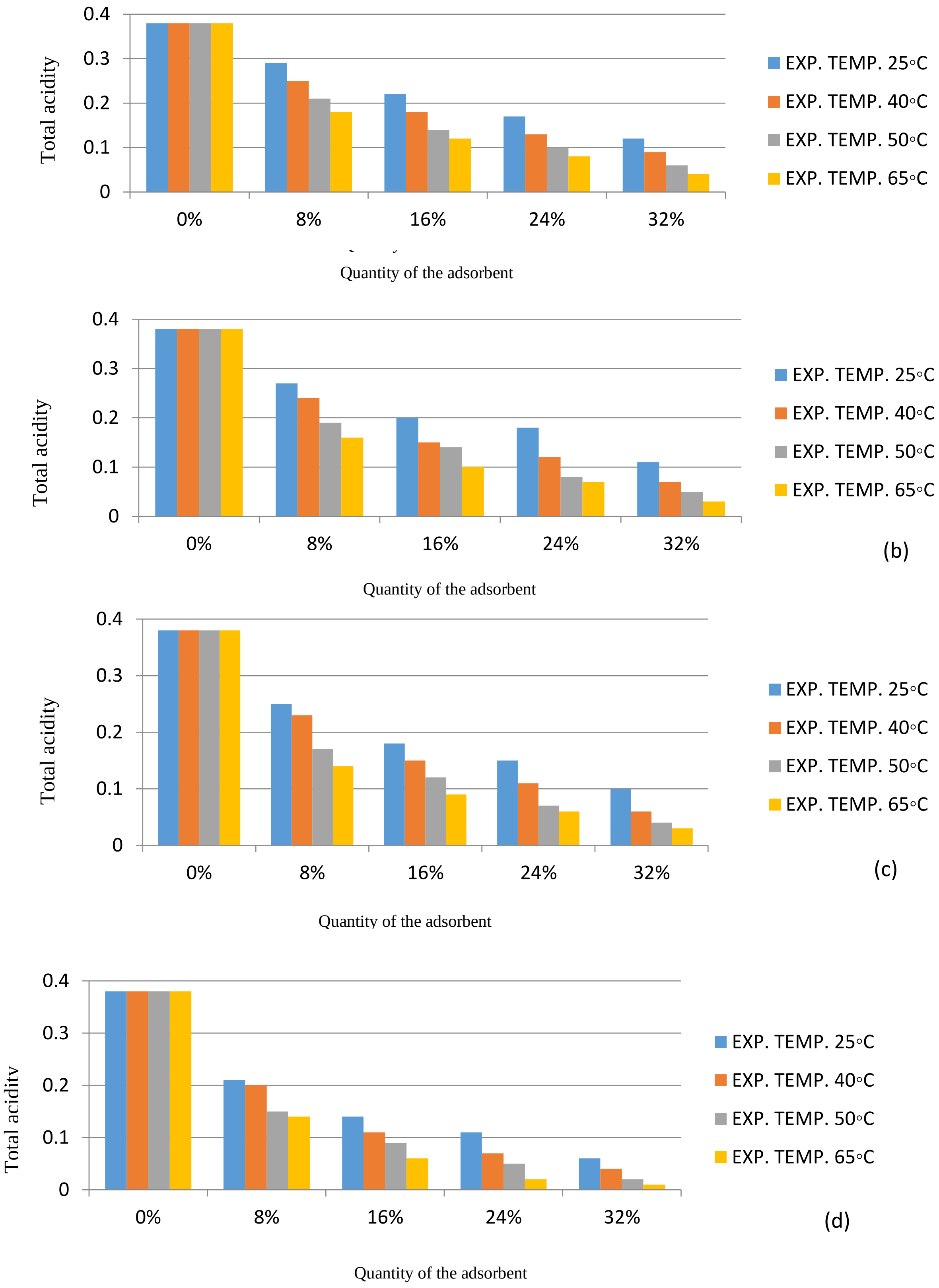
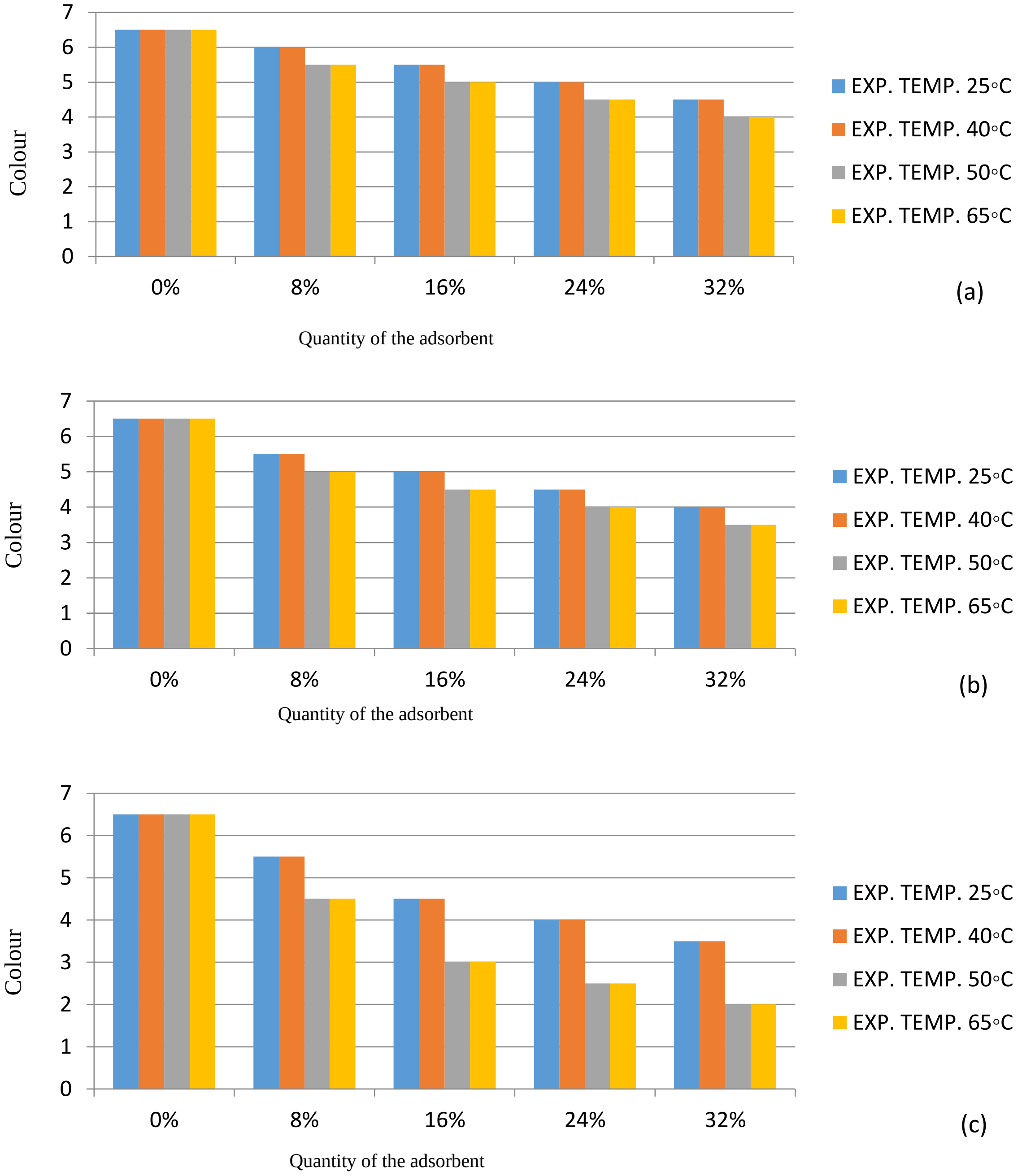


Fig. 9. Change of acid number with adsorbent material dose, which was activated at different temperatures, (a; 200°C, b; 400°C, c; 600°C, d; 800°C).

3.2.4. Colour of oil

By comparing it to a set of colour standards, the colour of insulating oil is measured in transmitted light and converted into a numerical value. A high or rapidly increasing colour number may indicate pollution or oil degradation. [Colorimeter, serial number: 10an-10, Precision Scientific (USA)] was used to measure the colour of oil. Increasing the temperature of the activated adsorbent (200°C, 400°C, 600°C, 800°C) and increasing its dose (8%, 16%, 24%, 32%) at various experiment temperatures (25°C, 40°C, 50°C, 65°C) results in a colour drop, as shown in (Figures 10a, 10b, 10c and 10d). The experiment lasts for ½ hour. The colour of oil before and after treatment was shown in Figures 11a and 11b. It was observed that as the activation temperature increases, the color of the oil decreases. The optimum conditions indicate that the adsorbent material is more effective at 65°C with continuous stirring for 30 minutes using a sludge dose of 32% preheated at 800°C. The maximum decrease in colour was 1.5.



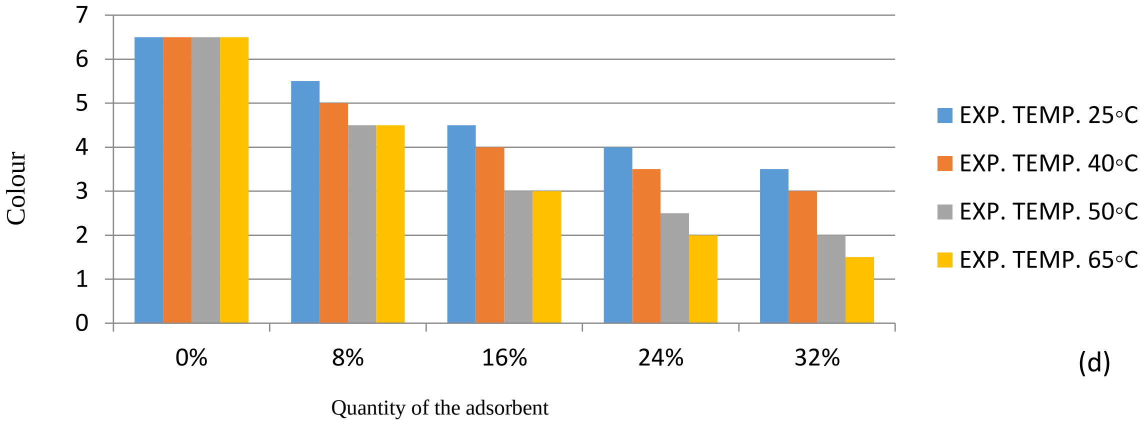


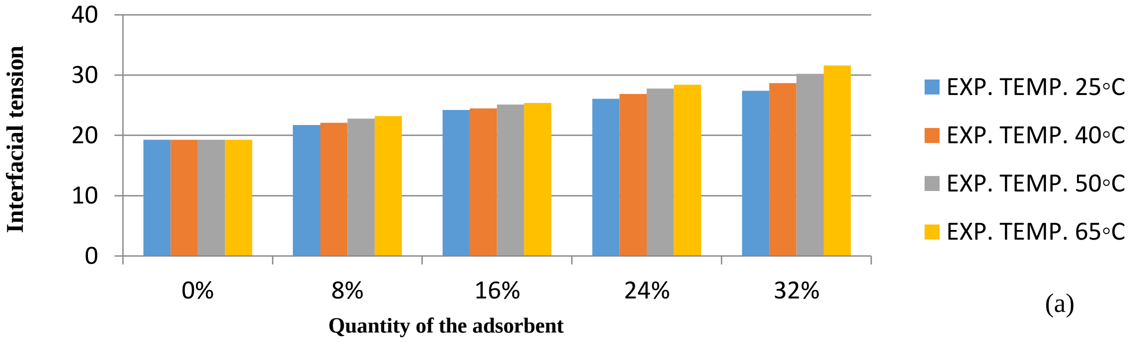
Fig. 10. Change of colour with activated adsorbent material dose at different temperatures, (a; 200°C, b; 400°C, c; 600°C, d; 800°C).

a: Untreated oil **b: Treated oil**

Fig. 11. Colour of oil before and after treatment showed.

3.2.5. The interfacial tension

The interfacial tension between oil and water of transformer oil was measured by [Tensiometer - Sigma702ET – serial number: 72065] according to ASTM D 971. (Figures 12a, 12b, 12c and 12d) showed an improvement and an increase in the interfacial tension after the chemical treatment. It was observed that as the activation temperature increases, the colour of the oil decreases. The maximum increase of the interfacial tension was (40.2 dyne/cm), like virgin oil.



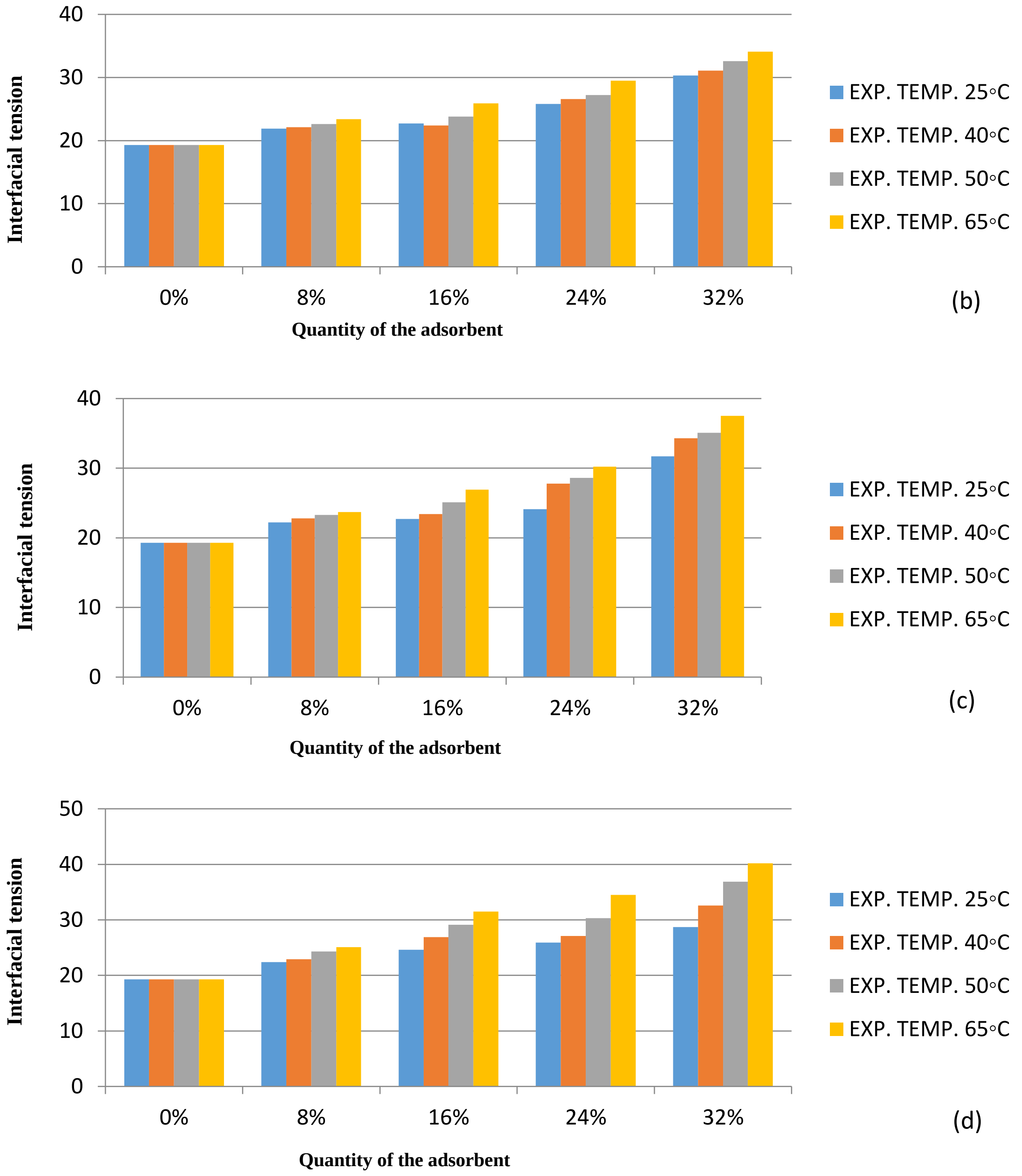


Fig. 12. Change of the interfacial tension with adsorbent material dose, which was activated at different temperatures, (a; 200◦C, b; 400◦C, c; 600◦C, d; 800◦C).

3.2.6. Dielectric Dissipation Factor (DDF) of the aged oil

The dielectric dissipation factor of transformer oil was measured by [Fully automatic dissipation factor and resistivity measuring system DTL-Austria-serial number: 073178074] according to IEC 60247. (Figures 13a, 13b, 13c and 13d) showed an improvement and a decrease in the dielectric dissipation factor after the chemical treatment. It was observed that as the activation temperature increases, the DDF of the oil decreases. The maximum decrease of the dielectric dissipation factor was (0.0006).

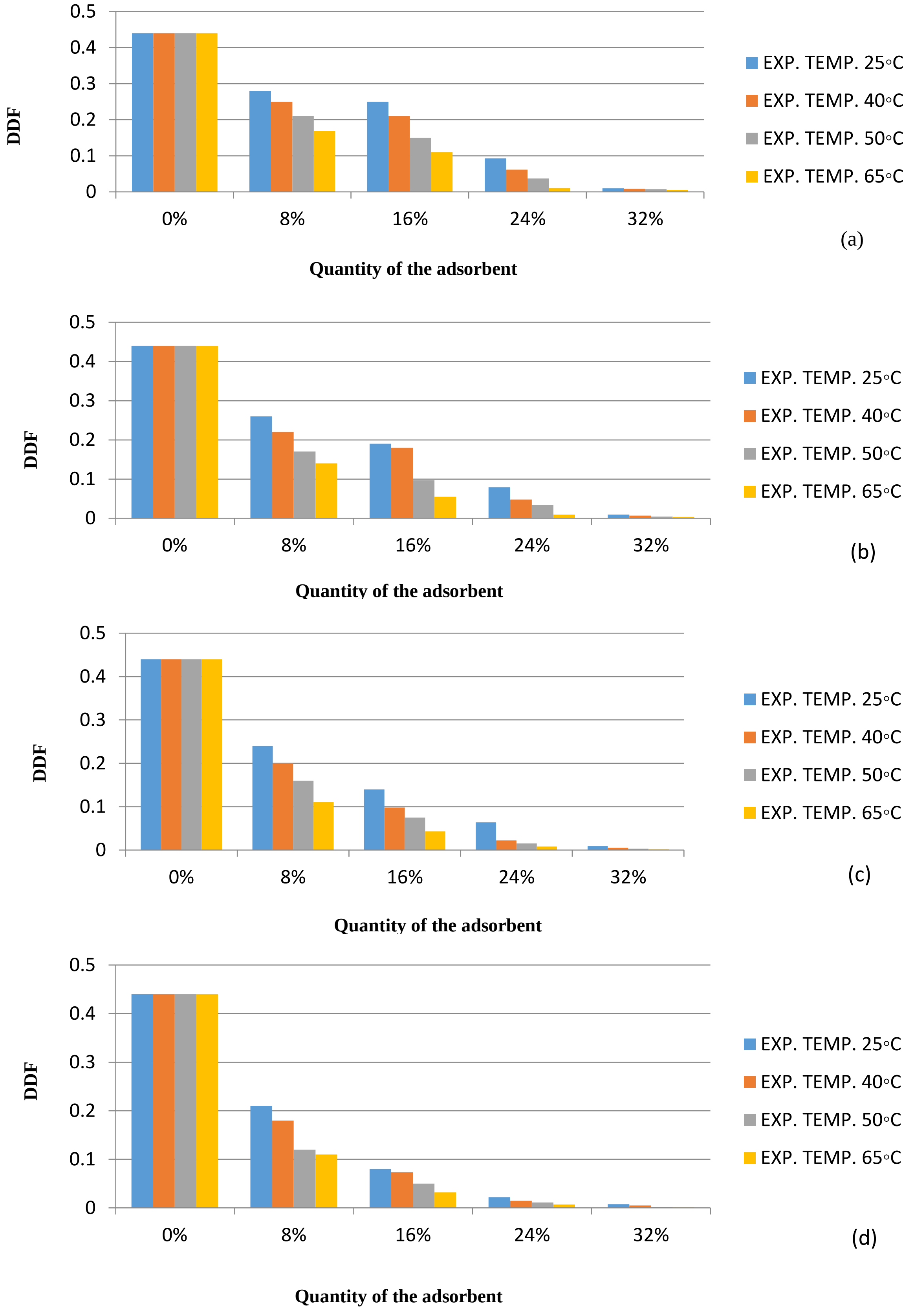


Fig. 13. Change of DDF with adsorbent material dose which was activated at different temperatures, (a; 200°C, b; 400°C, c; 600°C, d; 800°C).

3.2.7. Furanic compounds content of the aged oil

Furanic compounds are by-products of insulating paper aging in oil-filled transformers. Monitoring the furanic content of oil samples taken from in-service transformers would be used as an indication of paper aging. Therefore, it is important to have insulating oil free from 2-furfural content [37-38].

The furanic compounds content of transformer oil was measured by [DIONEX ULTIMATE 3000 HPLC device] according to ASTM D 5837. Table 7 showed a decrease in furanic compounds content after the chemical treatment at the optimum conditions (the adsorbent material was activated at 800°C for two hours, the experiment temperature is 65°C for ½ hour). The maximum decrease of the total furanic compounds content was (109 ppb).

Table 7: The gradual decrease of the furanic compounds content (ppb) with the doses of the adsorbent.

Furanic Compounds Results (ppm)	Aged oil	Adsorbent dose			
		8%	16%	24%	32%
5-Hydroxy methyl-2-furaldehyde	89	53	38	16	1
2-Furaldehyde	3609	2225	1850	560	49
5-Methyl-2-furaldehyde	30	25	18	15	13
2-Acetylfuran	90	79	64	58	46
Total Furanic compounds	3818	2382	1970	649	109

3.2.8. Copper strip tarnish test

The copper strip tarnish test was measured by using [Kohler, model: K25339 and serial number: R61890106, USA] according to ASTM D 130. Figure 14 shows copper strips with the virgin oil and the aged oil before and after treatment; the results obtained revealed a non-corrosive condition for three strips.

Fig. 14. Copper Strips with different oils (virgin oil, oil before and after treatment).

Table 8 shows the copper strip tarnish test results before and after chemical treatment with optimum conditions (the adsorbent material was activated at 800°C for 2 hours, the experiment temperature was 65°C for ½ hour), indicating that the adsorbent has no corrosive effect on the oil.

Table 8: The copper strip tarnish test after the physical treatment.

Test	Aged Oil	Adsorbent dose			
		8%	16%	24%	32%
Copper strip tarnish test	Not corrosive	Not corrosive	Not corrosive	Not corrosive	Not corrosive

3.2.9. Appearance

The transformer oil appeared dark with suspended matter, which could indicate the presence of contaminants, including fibers, dust, carbon particles, free water, or insoluble sludge. Table 9 showed improvement of the appearance after the chemical treatment at the optimum conditions (the adsorbent material was activated at 800°C for two hours, the experiment temperature is 65°C for ½ hour). The oil became clear, free from sediment and suspended matter.

Table 9: Appearance of oil before and after treatment with the adsorbent.

Test	Aged Oil	Adsorbent dose 8%	 16%	 24%	 32%
Appearance	dark with suspended matter	Clear, free from sediment and suspended matter			

3.2.10. Kinematic viscosity of the oil

The kinematic viscosity of the oil was measured by [viscometer Koehler" K2337] according to ASTM D 445. Table 10 showed the variation of kinematic viscosity of oil after the chemical treatment at the optimum conditions (the adsorbent material was activated at 800°C for two hours, the experiment temperature is 65°C for ½ hour), where the kinematic viscosity decreased as a result of removing the aging products.

Table 10: The gradual decrease of oil viscosity with the doses of the adsorbent.

Test	**Aged Oil**	**Adsorbent dose** 8%	 16%	 24%	 32%
Kinematic Viscosity at 40°C (mm^2/s)	10.32	10.21	10.11	10.05	10.04

3.2.11. Flash point of the oil

Flash point of oil were measured by [Pensky-marten - APM-7-serial no. 20311-Tanaka (Japan)] according to ASTM D 93. Table 11 showed the flash point of oil after every cycle of the physical treatment. There is no a significant change in flash point of oil after treatment.

Table 11: Variation of oil flash point (closed cup).

Test	**Aged Oil**	Adsorbent dose 8%	 16%	 24%	 32%
Flash Point closed (°C)	142	142	142	140	142

3.2.12. Density of the oil

The density of oil before and after treatment was measured using the hydrometer according to ASTM D 1298. Table 12 showed the variation of the density of oil after every cycle of the physical treatment. There is no significant change in the density of oil after treatment.

Table 12: Variation of oil density after the physical treatment.

Test	Aged Oil	Adsorbent dose 8%	 16%	 24%	 32%
Density at 20°C (g/ml)	0.8811	0.8816	0.8815	0.8817	0.8815

3.3. The effect of the physical treatment (final step of treatment) on:

3.3.1. The breakdown voltage of oil

The breakdown voltage of transformer oil was measured by [Megger OTS100AF device] according to IEC 156 after every cycle of the refining process in the third step of treatment. Table 13 showed improvement and an increase in breakdown voltage of oil after every cycle of the refining process. The value changed from 23 kV/2.5 mm for aged oil to 71 kV/2.5 mm for the oil after treatment at the optimum conditions (temperature 65 °C for ½ hour per cycle).

Table 13: Variation of breakdown voltage during the three cycles of physical treatment.

Test	Aged Oil	After one cycle	After two cycles	After three cycles
Breakdown voltage (k.v./2.5mm)	23	39	57	71

3.3.2. Water content in the oil

The water content of aged and treated oil was measured by [Karl Fisher - MKE500-serial no. Lcb32a22- KEM (Japan)]. Table 14 showed improvement and a decrease in water content after every cycle of the refining process. The value changed from 64 ppm for aged oil to 7 ppm for the oil after treatment at the optimum conditions (temperature 65 °C for ½ hour per cycle).

Table 14: Change of water content during the three cycles of physical treatment

Test	Aged Oil	After one cycle	After two cycles	After three cycles
Water content (ppm)	64	40	23	7

3.3.3. Dissolved gas analysis (DGA) in the oil

Dissolved gas analysis is used to discriminate between normal and abnormal conditions of the transformer [39, 40]. Gases in the oil were analysed by [Gas Chromatograph "DANI Auto sampler GC1000"] according to ASTM D 3612. Table 15 showed a decrease of dissolved gases in the oil after every cycle of the refining process. The total dissolved combustible gases decreased from 961 ppm for aged oil to 128 ppm for the oil after treatment at the optimum conditions (temperatujanre 65 °C for ½ hour per cycle).

Table 15: Dissolved gas analysis of the aged and treated oil.

Dissolved Gases (ppm)	Aged Oil	After one cycle	After two cycles	After three cycles
Hydrogen	133	63	35	17
Carbon monoxide	1052	410	303	104
Carbon dioxide	3054	2128	1360	760
Methane	1	1	1	1
Ethane	2	3	3	4
Ethylene	5	4	2	2
Acetylene	0	0	0	0
Total dissolved gases	4247	2609	1704	888

3.4. The effect of overall treatment (three steps of treatment) on improving of aged oil properties

A comparative study of the required specifications for virgin oil relative to IEC 60296-2020 and the test results of virgin oil, aged oil before and after final treatment, are listed in Tables 16, 17.

Table 16: Comparison of results according to IEC 60296-2020.

Test	Unit	Standard Method	Aged oil before treatment	Aged oil after treatment	Results of virgin oil	Limits of IEC 60296-2020
Density at 20 °C	Kg/m^3	ASTM D 1298	0.8811	0.8815	0.8157	Max. 0.895
Kinematics Viscosity at 40°C	mm^2/s (cSt)	ASTM D 445	10.32	10.04	11.43	Max. 12
Flash Point (closed cup)	°C	ASTM D 93	142	142	174	Min. 135
Color	ASTM Color Scale	ASTM D 1500	6.5	1.5	0.5	Max. 1.5
Total Acidity	mg KOH/g oil	ASTM D 974	0.38	0.01	0.01	Max. 0.01
Interfacial Tension	dyne/cm	ASTM D 971	19.3	40.2	41	Min. 40

Appearance	--------	--------	Dark with suspended matter	Clear, free from sediment and suspended matter	Clear, free from sediment and suspended matter	Clear, free from sediment and suspended matter
Water Content	PPm	IEC 60814	64	7	17	Max. 30/40[1]
Break down Voltage	k.v./2.5mm	IEC 156	23	71	44	Min. 30/70[2]
Dielectric dissipation factor at 90ºC	--------	IEC 60247	0.44	0.0006	0.00159	Max. 0.005
Cupper strip tarnish test	--------	ASTM D 130	Not Corrosive	Not Corrosive	Not Corrosive	Not Corrosive

1-for bulk supply/for delivery in drums.
2-before/after laboratory treatment.

Table 17: Comparison of results of furanic compounds analysis according to IEC 60296-2020.

Furanic Compounds (ppm)	Standard Method	Aged oil before treatment	Aged oil after treatment	Results of a virgin oil	Limits of IEC 60296
2-Furaldehyde	ASTM D 5837	3609	49	47	<50
5-Hydroxy-methyl-2-furaldehyde		89	1	30	<50
2-Acetylfuran		90	46	10	<50
5-Methyl-2-furaldehyde		30	13	15	<50
Total Furanic compounds		3818	109	102	<200

From the results obtained, it was clear that the chemical treatment with the adsorbent material, which adsorbed the aging and oxidation products, improved colour, appearance, acidity, dielectric dissipation factor, kinematic viscosity, and furanic compounds. Additionally, as a result of physical treatment using filtration, vacuum dehydration, water content, sediment, and suspended matter decreased, which raises the oil's breakdown voltage. The oil density, flash point, and copper strip tarnish test results before and after treatment showed no significant changes, and the obtained values were within the acceptable limits of virgin oil. Table 18 showed a comparison between the test results of dissolved gas analysis of virgin oil and the aged oil before and after treatment. As a result of the physical treatment using the degassing process, dissolved gases were reduced, like the behaviour of virgin oil.

Table 18: Comparison of results of dissolved gases analysis of the studied oils.

Dissolved Gases (ppm)	Standard Method	Aged oil before treatment	Aged oil after treatment	Results of virgin oil
Hydrogen	ASTM D 3612	133	17	5
Carbon monoxide		1052	104	120
Carbon dioxide		3054	760	650
Methane		1	1	3
Ethane		2	4	2
Ethylene		5	2	2
Acetylene		0	0	0
Total dissolved gases		4247	888	782

Therefore, all test results of the oil after the treatment processes were within the recommended limits of IEC 60296-2020, and the oil became suitable for reusing several times.

4. Conclusion

In this paper, a lab-scale refining unit and thermally activated sludge were used to remove water, gases, sediments, oxidation products, and furan compounds resulting from the degradation of transformer paper insulation and other aging products in the aged transformer oil sample. The extracted sludge was thoroughly examined by using quantitative and qualitative spectroscopic systems. The performance of the extracted sludge's is attributed to the large surface area. The results obtained from SLODP-LIPS, ICP-OES, and EDX spectroscopic elemental analysis techniques showed that the sludge has approximately the same constituents of fuller's earth, such as bentonite, bauxite, and kaolin clay, as those used before in oil treatment.

The properties of the treated transformer oil were improved significantly. Whereas, the appearance was improved from dark with suspended matter to clear, free from sediments and suspended matter. The breakdown voltage was increased from 23 kV/2.5 mm to 71 kV/ 2.5 mm. The water content decreased from 64 ppm to 7 ppm, and the color decreased from 6.5 to 1.5. While the total acidity decreased from 0.38 mg KOH/g oil to 0.01 mg KOH/g oil, the interfacial tension was increased from 19.3 mN/m to 40.2 mN/m, respectively. The dielectric dissipation factor of oil was decreased from 0.44 to 0.0006, and total furan compounds decreased from 3818 ppb to 109 ppb. In addition, the total dissolved gases decreased from 4247 ppm to 888 ppm, and the sulphur content decreased from 0.39% to 0.01%, consequently meeting IEC60296-2020 for evaluation of unused mineral insulating oils for transformers and switchgear.

It is worth mentioning that an advanced technique, namely, SLODP-LIPS, was employed in our study to characterize the elements in sludge. The results obtained using this technique were consistent with those of well-established methods such as ICP-OES and EDX, which enhances the reliability and accuracy of the findings.

To the best of the author's knowledge, this is the first study on the treatment of Egyptian transformer oil using a lab-scale refining unit and thermally activated sludge produced from drinking water treatment, which is available at Egyptian power plants as waste.

Economically, adsorbent material (sludge) is available in our Egyptian power plants. It is freely accessible without any cost, and the actual cost of oil treatment is negligible compared to the costly virgin oil.

5. Conflicts of interest:

There are no conflicts to declare.

6. Formatting of funding sources:

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7. Acknowledgment:

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8. Authors contributions:

Ahmed M. El Deeb, and Ashraf I. Hafez conceived the presented idea, concept, design the experimental plan and its execution. Ahmed M. El Deeb and Ahmed A.I. Khalil carried out the experiments and different tests, designed the experimental plan, participated in data acquisition and conducted the analytic calculations and calibrations. Ahmed I. Hashem, Ibrahim El Tantawy El Sayed and Abdel Aleem H. Abdel Aleem assisted in reviewing the manuscript, overcoming obstacles, achieving rapid process, providing technical and specialized guidance based on their experience in the field. Ahmed M. El Deeb, Ahmed A. I. Khalil and Ashraf I. Hafez participated in data analysis, interpretation of the test results, helped in shaping the research and contributed to the final manuscript.

9. List of abbreviations

Abbreviations	Full meaning
Alum	Aluminium sulphate
IEC	International Electrotechnical Commission
ASTM	American Standard Test Method
ppm	Part per million
ppb	Part per billion
k.v.	kilovolt
cSt	centistokes
Exp.	Experiment
Temp.	Temperature
EDX	Energy-Dispersive X-Ray Spectroscopy
ICP-OES	Inductively Coupled Plasma – Optical Emission Spectroscopy
SLODP-LIPS	Short long Orthogonal Double Pulse-Laser-Induced Plasma Spectroscopy

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