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New directions for the use of spent catalysts as sorbents for removing impurities from liquids

Nowe kierunki zastosowania zużytych katalizatorów jako sorbentów do usuwania zanieczyszczeń z cieczy

Anna Niemczyk-Wojdyla, Rafal Zawisz, Agnieszka Fornalczyk*), Joanna Willner, Agnieszka Sobianowska-Turek, Oksana Velgosova*)

Keywords: *recycling of spent catalysts; sorbents, removal of impurities from liquids, circular economy*

Abstract
Current approaches to recycling spent car catalysts are primarily focused on effectively recovering platinum group metals (PGMs), which are crucial raw materials for the economy. However, PGMs constitute only a small fraction of the total weight of the waste catalysts. The main component, both in terms of mass and volume, is the monolithic ceramic or metallic carrier. The residues left after the pyrometallurgical or hydrometallurgical recovery of platinum metals generate waste that burdens the natural environment when stored. Hence, there is a need for a balanced approach to the processing of used car catalysts, taking into consideration the effective recovery and management of other components besides the platinum group.
Cordierite, the predominant and widely used ceramic material in car catalytic converters, is notable for its exceptional chemical resistance and high-temperature durability during processing. It is also a low-porous material with a relatively small specific surface area, distinguishing it from conventional adsorbents, which usually possess highly developed specific surface areas and high porosity. This article presents preliminary studies that support the potential use of ceramic carriers derived from spent car catalysts as a novel material with sorption properties. Such an approach serves as an environmentally friendly alternative for the effective management of car catalyst waste and maximizes the added value derived from the recycling process, aligning with the principles of the Circular Economy.

Słowa kluczowe:*recykling zużytych katalizatorów, sorbenty, usuwanie zanieczyszczeń z cieczy, gospodarka obiegu zamkniętego*

Streszczenie
Obecne podejścia do recyklingu zużytych katalizatorów samochodowych skupiają się na efektywnym odzysku platynowców, które są surowcem o kluczowym znaczeniu dla gospodarki. Jednakże, platynowce stanowią jedynie niewielki procent całkowitej masy odpadowego katalizatora. Głównym składnikiem masy i objętości takich odpadów jest monolityczny nośnik ceramiczny lub metaliczny. Pozostałości masy katalitycznej po procesach pirometalurgicznego lub hydrometalurgicznego odzysku platynowców generują odpady, które obciążają środowisko naturalne w kontekście ich składowania. W związku z tym, istnieje potrzeba zrównoważonego podejścia do przetwarzania zużytych katalizatorów samochodowych, które uwzględnia efektywny odzysk i zagospodarowanie pozostałych komponentów poza platynowcami.
Kordieryt, jako główny i powszechnie stosowany materiał ceramiczny w katalizatorach samochodowych, wyróżnia się wyjątkową odpornością chemiczną i wysoką wytrzymałością temperaturową podczas procesów przetwarzania. Jest również nisko porowatym materiałem o mało rozwiniętej powierzchni właściwej, co odróżnia go od klasycznych adsorbentów, które zazwyczaj posiadają silnie rozwiniętą powierzchnię właściwą i są silnie porowate. W niniejszym artykule przedstawiono wstępne badania, które uzasadniają możliwość wykorzystania ceramicznych nośników pochodzących z zużytych katalizatorów samochodowych jako nowego materiału o właściwościach sorpcyjnych. Takie rozwiązanie stanowi przyjazną dla środowiska alternatywę w kontekście efektywnego zagospodarowania odpadowego katalizatora samochodowego oraz maksymalizacji wartości dodanej z procesu recyklingu, wpisując się w założenia Gospodarki Obiegu Zamkniętego.

1.Introduction

The contemporary industry involved in the management of catalyst waste, encompassing both the automotive and petrochemical sectors, primarily focuses on recovering the active phase of the catalyst. This active phase contains economically critical metals, including platinum group metals and other transition metals (such as rhodium, palladium, platinum, cobalt, nickel, vanadium, and molybdenum).

The automotive catalytic converter market is the sector that utilizes the largest quantities of platinum group metals (PGMs), notably palladium, platinum, and rhodium. The automotive industry

*) **Anna Niemczyk-Wojdyla,** ID – 0000-0001-8568-7478, ania.i.niemczyk@gmail.com, Ad Moto Rafal Zawisz, Aleja Rozdzienskiego 188B, 40-203 Katowice, Poland, **Rafal Zawisz,** ID – 0000-0002-1123-5476, rafal@filtracjaoleju.pl, Ad Moto Rafal Zawisz, Aleja Rozdzienskiego 188B, 40-203 Katowice, Poland, **Agnieszka Fornalczyk** (corresponding author), ID – 0000-0002-2149-1422, agnieszka.fornalczyk@polsl.pl, Faculty of Material Engineering, Silesian University of Technology, 2A Akademicka st., 40-019 Gliwice, Poland, **Joanna Willner**, ID – 0000-0003-3369-3678, joanna.willner@polsl.pl, Faculty of Material Engineering, Silesian University of Technology, 2A Akademicka st., 40-019 Gliwice, Poland , **Agnieszka Sobianowska-Turek**, ID – 0000-0003-2154-8609, agnieszka.sobianowska-turek@pwr.edu.pl, Faculty of Environmental Engineering, Wroclaw University of Science and Technology, 27 Wybrzeże Wyspiańskiego str, 50-370 Wrocław, Poland, **Oksana Velgosova,** ID - 0000-0001-8903-2604, oksana.velgosova@tuke.sk, Faculty of Materials, Technical University of Košice: Košice, Slovakia

accounts for over 80% of the global demand for palladium, while rhodium finds its predominant use in the production of car catalysts, with nearly 90% of its global demand stemming from the automotive sector. Regarding platinum, the primary demand-driving sectors are the automotive and jewellery industries, which collectively contribute to around 60% of the worldwide demand [11].

A notable characteristic of the PGM market is that the economic demand for PGMs exceeds the level of extraction and production of these metals from natural resources. In simpler terms, global demand for PGMs surpasses the primary supply generated by the PGM mining industry. Between 2017 and 2021, the mining industry's production of palladium, platinum, and rhodium satisfied roughly 64% of the global demand for palladium, over 75% of the global demand for platinum, and approximately 68% of the demand for rhodium [11].

However, relying solely on PGM extraction does not suffice to meet the supply requirements. The disparity between the high global demand and the limited supply from PGM mining is supplemented by secondary supply, which involves producing PGMs from secondary raw materials, primarily used car catalysts. From 2017 to 2021, annual PGM production from secondary raw materials remained consistent, and for palladium and rhodium, it even exhibited an upward trend. In 2021, roughly 33% of the global demand for palladium, 24% of the global demand for platinum, and 36% of the world's rhodium demand were met through this secondary supply [11].

The catalytic carrier constitutes a substantial portion of the catalyst's mass, with the ceramic carrier accounting for about 99% of its mass, while the remaining 1% represents the active phase (including PGM metals). Although the ceramic carrier is not as economically valuable as the active phase of the catalyst, managing waste ceramic carriers poses a significant economic and ecological challenge for the industry.

Consequently, prioritizing the management of ceramic catalyst carriers after recovering critical metals from them contributes to a prudent approach to raw materials management. This, in turn, leads to a reduction in the consumption of raw materials and allows for the utilization of the industrial potential of waste.

In line with the EU's Circular Economy requirements, there is a need to comprehend and implement strategies that enhance the effective management of catalyst waste. The European Union, committed to achieving a sustainable future, advocates an approach that promotes the recovery, recycling, and reuse of raw materials.

By adopting a Circular Economy perspective in catalyst waste management, the industry stands to gain both economic and environmental benefits. Additionally, reducing primary raw material consumption and embracing secondary raw materials will help minimize the negative impact on the natural environment while increasing overall industry efficiency. Pursuing a more sustainable waste management approach is not only a necessity but also an opportunity to foster a resilient and sustainable economy for the future.

2.Material characteristics and possibilities of recycling of spent catalysts

Spent Auto Catalysts (SAC) on ceramic carrier are comprised of three essential components: active metals – Precious Group Metals (PGMs – mainly Pt, Pd, Rh), a cordierite substrate, and a washcoat (γ-alumina, γ-Al_2O_3) infused with diverse additives [20] (Fig.1). Within the auto-catalyst structure, the washcoat layer firmly adheres to the surface of the cordierite substrate, effectively dispersing PGM particles throughout. This design enhances the catalytic efficiency of the converter. The catalytic carrier is further enclosed within a fibrous material and housed within a stainless steel shell [7], ensuring its structural integrity and longevity.

Fig.1. View of spent auto catalysts on ceramic carrier
Ryc.1. Widok zużytych katalizatorów samochodowych na nośniku ceramicznym

Numerous scientific publications have extensively explored the methodologies for recovering precious metals from spent catalysts, and the process itself is well-established and widely implemented in industrial settings. Pyrometallurgical techniques are commonly employed, involving the remelting of ceramic catalyst carriers along with a metal collector and fluxes [22]. This procedure results in the collection of precious group metals (PGMs) predominantly in a metal collector. Alternatively, hydrometallurgical methods, such as the use of aqua regia, chlorination, and mixed processes, are also utilized to extract PGMs from the spent catalysts. These techniques have proven effective in recovering valuable metals for potential reuse or recycling purposes.

The main solid waste generated during the processing of used car catalysts is the monolith, which constitutes about 84.5% of the weight of the ceramic catalyst [6,9 15]. The major phases of an auto-catalyst are found to be cordierite ($2MgO{\cdot}2Al_2O_3{\cdot}5SiO_2$), alumina (ϒ-$Al_2O_3$), and some additives, such as CeO_2, La_2O_3, and ZrO_2 [20]. Most commonly, the composition of primary oxides in cordierite lies within the range of: 48-52% silicon oxide (SiO_2), 32-36% aluminum oxide (Al_2O_3), and 8-14% magnesium oxide (MgO) [8-9]. Table 1 presents samples of materials applied (containing Al_2O_3, particularly intriguing due to its exceptional sorption properties) to industry used to make monolithic carriers.

Table 1. Materials applied in industry as monolithic carriers [7]
Tabela1. Materiały stosowane w przemyśle jako nośniki monolityczne

Name	Chemical Components
Alumina (α, γ)	Al_2O_3
Cordierite	$2MgO{\cdot}2Al_2O_3{\cdot}5SiO_2$
Mullite	$3Al_2O_3{\cdot}2SiO_2$
Magnesium spinel	$MgO{\cdot}Al_2O_3$
Cordierite-mullite	$2MgO{\cdot}2Al_2O_3{\cdot}5SiO_2$-$3Al_2O_3{\cdot}2SiO_2$
Mullite-aluminium titanate	$3Al_2O_3{\cdot}5SiO_2$-$3Al_2O_3{\cdot}TiO_2$
Zeolite	$Me_2O{\cdot}xAl_2O_3{\cdot}ySiO_2{\cdot}zH_2O$
Zirconium dioxide	ZrO_2
Zirconium dioxide-magnesium spinel	ZrO_2-$MgO{\cdot}Al_2O_3$

The ceramic material applied in spent auto catalysts exhibits several advantageous functional properties, making it suitable for use as a catalyst core [4]. Notably, it is known for its high chemical resistance and thermal durability, as well as its excellent adhesive properties when in contact with the γ-Al_2O_3 washcoat oxide phase [4]. However, owing to its exceptional thermal and chemical resistance, this material presents a considerable challenge in the processing of used car catalysts [14,17,19]. The difficulty lies in effectively processing and extracting valuable metals from the ceramic substrate, adding complexity to the recycling and recovery of precious group metals (PGMs) from spent automotive catalysts.

Table 2 provides an overview of the key performance characteristics of cordierite [2].

Ceramic monoliths typically take the form of cylindrical bodies. In terms of cross-section, the prevailing configuration for a ceramic monolith is oval; nevertheless, monoliths with a circular cross-section also exist (Fig. 1) [2]. The monolith's shape and spatial arrangement result from the extrusion process carried out in specialized moulds under high pressure [2]. These monoliths are manufactured with wall thicknesses on the order of thousandths of an inch, and the count of channels per one square inch of cross-sectional area ranges from 350 to 1600 [8]. A commonly employed structure, for instance, is 400/6.5, denoting 400 channels per square inch and a wall thickness of 0.0065 inch. However, thinner wall structures like 400/4 or 600/4 are gaining popularity [8].

Table 2. General characteristics of operational properties of synthetic cordierite [2]

Tabela 2. Ogólna charakterystyka właściwości użytkowych syntetycznego kordierytu [2]

Parametr	Evaluation of operational properties
Chemical resistance	• highly resistant to the effects of acids and bases, as well as high temperatures • used in the production of durable ceramic materials
Hardness	• at a level of 9.0 on the Mohs scale – one of the hardest ceramic materials • can be difficult to machine, requiring the use of specialized tools
Thermal stability	• very high thermal stability, indicating resistance to high temperatures • an ideal for manufacturing high-temperature components
Low thermal expansion	• very low thermal expansion, implying its volume remains unchanged with temperature variations • often used in the production of insulating elements
Electrical insulation	• very good electrical insulation • used in the production of electrical insulators and other electronic components
Wear resistance	• high wear resistance – an ideal material for cutting tools, machine components
Brittleness	• somewhat brittle and can easily fracture or be damaged upon impact or bending
Thermal conductivity	• low thermal conductivity compared to other ceramic materials, • problematic in certain applications where rapid heat conduction is required
Mechanical resistance	• good compressive and frictional mechanical properties • low bending strength, meaning it can easily crack under bending stress

The literature suggests various approaches for the management of waste cordierite from car catalysts [18]. In the pyrometallurgical recovery process of the precious group metal (PGM) fraction, the differences in melting temperatures between PGM and cordierite are exploited, leading to the collection of insoluble cordierite in the slag, which is then separated from the collector metal alloy and PGM [14]. Another proposed method involves transforming the cordierite phase in the slag, initially forming amorphous glass, followed by crystallization resulting in the formation of ceramic glass of the $CaAl_2$-SiO_8 and $CaMgSi_2O_6$ types, which finds industrial applications [5, 24].

In hydrometallurgical processes used for PGM recovery, cordierite remains unreacted and can be separated from the post-process extraction solution through filtration [4]. Moreover, if the cordierite ceramic monolith undergoes successful chemical decomposition, it can yield aluminum, magnesium, and silicon compounds, providing secondary products that can be employed as catalytic carriers, ceramic coatings, additives to polymers, or adsorbents for water purification [4,7].

The literature also describes potential hydrometallurgical processing methods for catalytic waste materials containing aluminium compounds similar to cordierite [3, 7, 12]. However, these processing pathways often require the use of high temperatures and chemically aggressive reagents, such as calcination at temperatures ≥600 °C in the presence of NaOH, which can limit the overall benefits of recycling [1, 2, 21]. Nonetheless, the analysis of potential processing routes indicates the possibility of obtaining not only α-Al_2O_3 but also more valuable raw materials, such as boehmite [γ-AlO(OH)] or γ-Al_2O_3, from waste catalytic materials containing aluminium compounds [1, 7, 12, 13, 21, 25]. Boehmite is widely used in various industries, including catalyst production, ceramic coatings, polymer additives, and adsorbents [25]. Similarly, γ-$A2l_2O_3$ can be recycled into the material cycle as a catalytic carrier and a dispersing medium for the catalytically active phase [21]. Another promising avenue is further processing of γ-$A2l_2O_3$ through electrolysis to obtain pure aluminium, avoiding the production of environmentally harmful red mud typically generated in the industrial Bayer method [7].

Details of the concept for recovering aluminium in the form of useful compounds from solutions containing $NaAlO_2$ or other aluminium salts were elaborated in publication [21]. According to this method, selective precipitation of aluminium ions using urea as a precipitating agent under hydrothermal conditions results in the formation of boehmite. Subsequent calcination of the obtained boehmite (at 500 °C for 4 hours) leads to the production of γ-Al_2O_3.

The literature presents concepts for processing waste cordierite material into more economically valuable products, such as boehmite or γ-Al_2O_3. However, these concepts involve energy-intensive processes and the use of toxic and corrosively aggressive chemicals, which is a significant drawback, making them impractical for industrial implementation.

An alternative concept aiming to minimize solid waste in the processing of spent car catalysts was proposed in publication [17]. This concept involves utilizing a hydrometallurgical method based on chloride leaching of the precious group metal (PGM) fraction in $HCl+H_2O_2$ solution. The recycling process is designed to generate four secondary products from the used car catalyst, substantially reducing waste streams. The primary product is the PGM fraction separated through cementation with aluminium (Al) powder. Unreacted cordierite, separated by filtration, can be repurposed as a refractory material, while soluble salts, known as alum, can find application in bleaching agents and the cosmetics industry. Cerium, on the other hand, is isolated after the PGM recovery step through precipitation using Na_2SO_4 and K_2SO_4 as precipitating agents. The cerium sulphate salts obtained in this manner are converted to cerium oxide, a versatile material extensively used in glass, ceramics, catalysts, and high-hardness materials. However, it is essential to note that this process is currently only developed conceptually without practical implementation [10, 17].

Considering the aforementioned literature concepts and the growing trend towards a circular economy, it is evident that developing an effective and ecologically beneficial method for managing waste cordierite is a crucial challenge in the realm of recycling used car catalysts.

The purpose of this article is to demonstrate the viability of utilizing waste materials, specifically used car catalysts, as a novel sorption material for industrial oil filtration. The article should be regarded as a preliminary research report conducted as part of "Implementation doctorate" [23].

3. Materials and methods

3.1. Preparation of material for research

For the sorption process, the material of ceramic cordierite of SAC with a three-functional catalytic layer, coming from the domestic market for the purchase of this type of waste, was used. Initial preparation of the ceramic monolith material, aimed at removing solid impurities from the surface layer, included: dry cleaning by blowing with compressed air, washing in an ultrasonic bath with the addition of a degreasing agent and initial crushing, further grinding and homogenization in a ball mill (fraction <0.500 mm).

The platinum group metals (Pt, Pd, Rh) were removed from the surface of the ground ceramic support in the leaching process carried out according to the methodology described in the paper by Trinh et al. [20] using a formic acid solution in the first stage, and then 2.0 M HCl with the addition of 1.5 M $NaClO_3$ (90°C, 2h). The efficiency of metals extraction was obtained at: Pd=93.5%, Pt=93.8%, Rh=94.2%.

The leaching residue – the material of the ceramic cordierite – was filtered, washed several times alternately with an aqueous $NaHCO_3$ solution and pure water, and finally dried at 50°C for 12 hours.

Fig. 2 and Fig. 3 show the diagram of the subsequent stages of the preparation of the SAC material together with the results of the qualitative analysis of the powder of the ceramic material (XRD method) with the visible main structure in the form of crystalline cordierite $2MgO{\cdot}2Al_2O_3{\cdot}5SiO_2$. The phase composition was analyzed by X-ray diffraction (XRD) using a PANALYTICAL Empyrean diffractometer equipped with detector PIXCel and Johansson monochromator. Measurements were carried out on powder samples (after grinding) in the angle range 2θ 10÷80° with a measurement step of 0.02° with the X-ray tube generator parameters: 45kV and 40mA.

The cordierite material prepared in this way was then subjected to sorption tests. To determine the sorption potential of SAC cordierite material, preliminary purification tests for used industrial oil were carried out. The tests were conducted in a flow system, in a sorption column filled with an adsorbent prepared from ground SAC waste material, which was placed on a layer of inert material (Fig. 3). The bed was then flooded with used industrial oil until the pores were completely saturated, then left to drain completely, where the oil sample flowed by gravity through the fixed SAC bed in the sorption column.

Fig.2. Scheme of the SAC cordierite material preparation process together with the sorption stage on the SAC bed

Rys.2. Schemat procesu przygotowania materiału kordierytowego SAC wraz z etapem sorpcji na złożu SAC

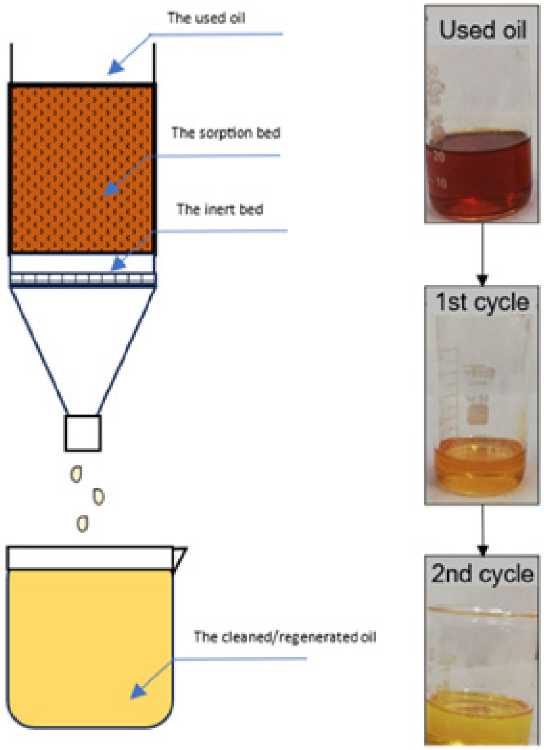

Fig.3 The results of the visual assessment test of the colour of used industrial oil after the use of a new sorption material

Rys.3 Wyniki wizualnego badania oceny barwy zużytego oleju przemysłowego po zastosowaniu nowego materiału sorpcyjnego

3.2. Purification tests of used industrial oil on a sorption bed made of SAC material

As an indicator of the effectiveness of the SAC sorption bed in determining the possibilities of regeneration of industrial oils, one of the basic and the simplest assessment methods, which is commonly used in laboratory tests and industrial practice was used – visual observation. Therefore, the essence of this stage of research was based on the visual assessment of the change in the colour of the oil sample and its turbidity after passing through the sorption bed in relation to the contaminated oil sample.

Fig. 3 presents the results of the sorption test carried out in two cycles, with a visible change in the colour of the samples. The sample of used industrial oil was dark brown in colour and had high turbidity, which indicated high contamination of the oil. After the oil passed through the SAC bed once, an intermediate colour was obtained, and finally, after the second sorption cycle, a light straw colour and high transparency of the sample were obtained – such colour parameters characterize pure and regenerated oil.

For the purposes of precise and objective assessment of the oil samples' colour, reference was made to ISO 2049 standard: Petroleum Products – Colour Determination (ASTM scale). The colour of the oil was determined using the ASTM D1500 colour chart with a scale of 0.5 (lightest) to 8.0 (darkest) (Fig. 4). Colour detection and measurements were performed using a PFXi 195 series colorimeter from Lovibond Tintometer (operating range: 420-710 nm, spectral gap: 10 nm, reading results on the ASTM D1500 colour scale) using a 33 mm cuvette.

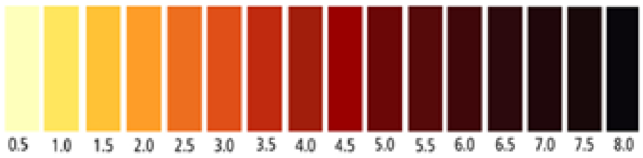

Fig.4. Colour scale according to ASTM D1500 standard
Ryc.4. Skala kolorów zgodna z normą ASTM D1500

Based on the colour scale according to the ASTM D1500 standard, the colour of the initial sample was determined as: 5.0, samples after the 1st sorption cycle: 2.0, and oil samples after the 2nd cycle: 1.0. An analysis of selected physicochemical parameters was also carried out for oil samples before and after passing through the SAC sorption bed by the rules adopted for industrial applications.

The filtered oil is evaluated using the following standards: IEC 62021 (for acid number), IEC 60814 (for water content), IEC 60156 (for breakdown voltage), IEC 60247 (for resistivity and dielectric loss factor), and ISO 2719 (for flash point).

In determining the following parameters, we relied on the assumption that the acid number is a measure of the oil's acidity, expressing the amount of KOH needed to neutralize all existing free acidic compounds present in 1 gram of oil. An increase in the acid number is usually caused by the presence of acidic compounds, such as carboxylic acids, that result from oxidation reactions in oil. Organic acids and other oxidation products, when combined with water and solid impurities, worsen the dielectric properties of the oil. The rate at which the acid number of used oil increases serves as an indicator of the oil's aging rate. It is generally accepted that the acceleration of sludge and sediment formation, as well as cellulose degradation, occurs when the oil's acid number exceeds 0.1 mg KOH/g. The level of the acid number is used as a general guideline to determine when the oil should be replaced or regenerated.

Determining the water content in the oil is particularly important, as water in the oil primarily appears due to moisture ingress from the atmosphere, resulting from enclosure leaks in the equipment, or during improper maintenance of the internal components of the unit. The presence of water is unfavourable for two reasons:

a) It reduces the dielectric strength of the oil.
b) It acts as a source of oxygen, which participates in oxidation reactions responsible for the aging process of the oil.

On the other hand, measuring the surface tension is significant, as based on the value of this parameter, the presence of even very low concentrations of dissolved polar substances can be determined. A decrease in the surface tension value below a critical threshold indicates the formation and precipitation of sludges in the oil.

Another significant parameter is the dielectric loss factor and resistivity, which are highly sensitive to the presence of solid particles, dissolved polar and ionic particles, as well as colloids in the oil. Changes in contamination levels can be monitored by measuring these parameters, even when the concentration of contaminants is so low that it approaches the chemical detection limit. Generally, there is a relationship between the dielectric loss factor and resistivity, with resistivity decreasing as the dielectric loss factor increases. It's usually not necessary to conduct both tests on the same oil. Analysing the dielectric loss factor is a more common test.

The operational parameters of the oil after the regeneration test on a specific bed were compared with a reference sample, which consisted of crude oil prior to the sorption test. All sorption tests were conducted in line with the standard test conditions for a stationary sorption system.

The results are presented in Table 3. It can therefore be concluded that the results of the preliminary visual tests together with the analysis of basic parameters confirm the beneficial effect of the SAC sorption bed on the quality parameters of industrial waste oil.

Table 3. Changing the basic physicochemical parameters of the oil.
Tabela 3. Zmiana podstawowych parametrów fizykochemicznych oleju

Analysed parameter	Oil before passing through the sorption bed	Oil after passing through the sorption bed
Acid number [mg KOH/g]	0.20	0.05
Water content [mg/kg]	22.3	12.5
Surface tension [mN/m]	21	33
Breakdown voltage [kV]	45	62
Loss factor tgδ at 90°C	0.056	0.004
Resistivity at 90°C [Gωm]	22	154

4. Summary

Presented the preliminary research results, evaluating the sorption potential of SAC cordierite for the regeneration of industrial oils, which were based on the visual assessment of a simple colour change test of oil samples together with the analysis of basic physicochemical parameters [16].

The results of the tests clearly indicate the sorption capacity of the SAC cordierite material and are the basis for further more advanced research. Currently, research is being carried out to develop a method of processing SAC monoliths to obtain a material with sorption properties from them [3] and to develop an innovative concept of using high-energy grinding with an electromagnetic mill for surface activation of cordierite, as an economical and environment-friendly alternative to classical methods with a harsh chemical regime.

In particular, two research directions are being considered:

1) The use of SAC cordierite activated in the mill as a sorption bed for the purification/regeneration of used industrial oils, along with comparative tests using commercial adsorbents as a sorption bed. The research includes a detailed analysis of changes in the functional and physicochemical parameters of industrial oils and the SAC cordierite material.
2) Determination of the sorption potential of cordierite SAC as a material capable of removing metal ions from polluted waters. The search for new and cheap sorbents used to remove heavy metal compounds, organic compounds, dyes, or pharmaceuticals that are harmful to health and the environment is a current issue widely discussed in the scientific literature.

The essence of the problem is the replacement of expensive traditional sorbents with cheap ones, which are often produced from waste materials.

Proposed in the article the innovative approach to the problem of managing the SAC cordierite material is an example of a solution that maximizes the potential benefits of its recycling. In the case of positive results of further research on the sorption material, it will be possible to effectively and environmentally friendly use of SAC waste cordierite, which will become a useful adsorption material with the possibility of its use for the purposes of another secondary recovery process, which is the regeneration of used industrial oils and/or metal ions removal from aqueous solutions.

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Data Availability

The specific sources of the data shown in Table 1 have been web archived from the footnoted websites and the archival links will be made available upon request.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper. ■

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