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Prospects for the processing of spent automotive catalysts with the extraction of precious, rare and rare-earth metals in Kazakhstan

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Abstract. Used automotive catalysts are a valuable source of rare and precious metals, which plays a key role in modern industry. These raw materials contain rare rare earths and precious metals, which makes their processing highly efficient and economically profitable. To develop a comprehensive processing technology, catalysts were studied using various analysis methods, such as chemical, X-ray, X-ray spectral, and electron microscopic analysis. The results of the phase analysis indicate that rare rare earths and noble metals are in various phase states, in the form of oxides embedded in the aluminosilicate matrix of the catalyst. These data are of critical importance for the creation of effective methods of metal extraction. The key approaches for processing these raw materials are pyrometallurgical and hydrometallurgical technologies, each of which has its own advantages and unique performance criteria. This article focuses on the physico-chemical studies of spent catalysts and the proposed method of their opening, which makes it possible to efficiently extract rare, rare-earth and precious metals. The research results can be used to optimize existing and develop new technological schemes for processing secondary raw materials.

Keywords: *spent automotive catalysts, rare metals, precious metals, rare earth elements, platinum metals, hydrometallurgical technologies, pyrometallurgical technologies, selective precipitation, secondary raw materials*.

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1. Introduction

Over the past three decades, a significant number of used cars have accumulated in Kazakhstan, and the country's vehicle fleet continues to grow at a rapid pace [1]. This has led to the formation of a vast volume of secondary technogenic raw materials, such as automotive catalysts. These catalysts, which contain valuable metals, are often dismantled and exported abroad for the extraction of precious, rare, and rare earth metals [2]. However, this approach not only deprives Kazakhstan of potential economic benefits but also creates environmental risks associated with the transportation and processing of waste abroad [3].

At the enterprise of JSC «Tau-Ken Altyn», located in the industrial park of Astana, work has begun in the field of recycling used catalysts to extract precious metals such as platinum, palladium, and rhodium [4]. However, the extraction of rare and rare earth metals (REM), whose global market demand is rapidly growing, has not been foreseen [5]. This limits the potential for comprehensive use of secondary raw materials and reduces the economic efficiency of recycling [6].

The relevance of comprehensive secondary raw material utilization is becoming increasingly evident for Kazakhstan, especially in light of the growing volumes of secondary waste [7]. Recycling used automotive catalysts is mainly aimed at extracting precious metals, while other important elements, such as rare and rare earth metals, remain undervalued [8]. This is because existing technologies focus on extracting precious metals, even though catalysts also contain significant amounts of rare and REMs, which can be economically profitable [9].

Existing recycling technologies, such as pyrometallurgy, ensure high yields of precious metal recovery but require substantial energy costs and specialized equipment [10]. This leads to increased capital and operational expenses, as well as the formation of waste, such as volatile substances and slag [11]. The main issue lies in the high cost of continuous furnace operation, which requires the use of secondary resources and increases the impurity content in the final product [12].

Modern pyrometallurgical methods, such as plasma smelting, allow for the minimization of emissions and improved recycling efficiency [13]. However, a key challenge remains addressing issues related to sulfur dioxide and chromium and other impurities [14]. Chlorination and fluorination methods are still relevant, although they are associated with the formation of toxic emissions, which requires efforts to reduce environmental impact [15].

Figure 1 presents the technological diagram of the pyrometallurgical method for recycling automotive catalysts. This process involves multiple sequential steps to recover platinum group metals (PGMs) from spent catalysts while minimizing waste generation.

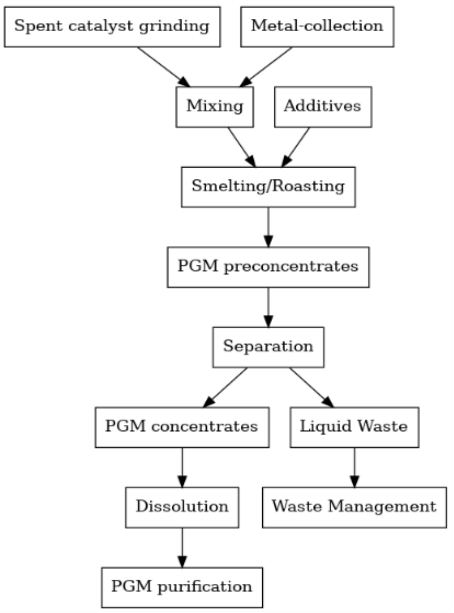

Figure 1. Pyrometallurgical and hydrometallurgical processes for PGM recovery

The process involves crushing, grinding, and smelting catalysts at temperatures above 2000°C with the addition of fluxes and collector metals [16]. After this, the precious metals are extracted from the resulting concentrate for further processing. However, components of the catalysts, such as rare metals and aluminum oxide, have significant economic value, and their joint extraction is complicated by the slag melting process [17].

Modern recycling methods aim to increase the efficiency of extracting all valuable components [18]. The development of technologies that minimize the losses of rare metals and their combinations is becoming increasingly relevant [19]. In addition, environmental and safety considerations during high-temperature processes must be addressed [20].

Improving catalyst recycling technologies could lead to better resource utilization and a reduction in the negative impact on the environment [21]. This, in turn, contributes to the sustainable development of the economy and the preservation of natural resources [22]. Investing in scientific research in this field promises new solutions and improvements in methods for extracting valuable metals [23].

In recent years, alternative hydrometallurgical procedures have been developed to improve extraction rates and the purity of the final product [24]. Despite these efforts, the efficiency of new methods has not yet reached the level of pyrometallurgical technologies, which continue to dominate the industry [25]. To improve the effectiveness of hydrometallurgical processes, new reagents and technologies need to be explored [26]. An important direction is the use of biodiversity and alternative energy sources, which helps reduce the negative environmental impact [27]. Therefore, further innovations in this area could significantly improve the sustainability and economic feasibility of metal recycling processes [28].

In light of the above, recycling used automotive catalysts in Kazakhstan represents an important direction for economic development and environmental improvement [29]. A comprehensive approach to the extraction of precious, rare, and rare earth metals could become a key factor in achieving sustainable development and reducing dependence on the import of valuable resources [30].

2. Materials and methods

2.1. Objects of research

The objects of the study were a representative sample of used automotive catalysts, selected to account for their morphological and chemical diversity. Before the experiments, the sample underwent mechanical treatment, including crushing, grinding to a fraction smaller than 100 µm, and homogenization to ensure uniform distribution of components. For the analysis of the elemental composition, X-ray fluorescence spectrometry (XRF) and scanning electron microscopy with energy-dispersive analysis (SEM-EDS) were used [1].

2.2 Analytical methods

During the physicochemical studies, chemical, X-ray phase, X-ray spectral, and electron probe methods of analysis were used. The sample preparation for ore was carried out according to the standard methodology [1].

The concentrations of precious, rare, and rare earth metals were determined using an inductively coupled plasma atomic emission spectrometer (ICP-AES), model 8300 DV (PerkinElmer Inc., USA). The instrument provided measurements in the spectral range of 165–782 nm with a resolution no lower than 0.006 nm (at a wavelength of ~200 nm) and a root mean square deviation of random error of 2.0%. Calibration curves, constructed from standard solutions with known concentrations of target elements, were used for quantitative analysis.

The efficiency of metal extraction (η, %) was calculated using the formula: $\eta = C \cdot V / m_0 \cdot w \times 100\%$, $\eta = m_0 \cdot w / C \cdot V \times 100\%$, where C is the metal concentration in the solution (mg/l), V is the solution volume (l), m_0 is the sample mass (g), and w is the metal mass fraction in the catalyst.

X-ray phase analysis was performed on a D8 Advance diffractometer (Bruker) using Cu Kα radiation (40 kV, 40 mA). The diffraction patterns were processed, including the calculation of interplanar distances, using the EVA software. Phase identification and matching were carried out using the Search/Match program and the PDF-2 powder diffraction database.

2.3 Statistical processing

Each experiment was repeated three times. The data were processed using OriginPro 2023 software, calculating the mean values and standard deviations.

3. Results and discussion

3.1. Physico-chemical characteristics of man-made waste

Chemical Composition and Mineralogical Analysis

Table 1 presents the results of the chemical analysis of the initial sample, including the mass fractions of various elements. The data reveal a significant content of aluminum (22.57%) and zirconium (14.53%), along with the presence of rare-earth elements (La, Ce) and platinum group metals (Pt, Pd, Rh).

Table 1. Chemical composition of spent automotive catalysts

Name of the product	The content of the elements, %						
	Ca	Mg	Al	Si	Fe	Zn	Zr
The initial sample	0.76	2.83	22.57	8.31	1.28	0.44	14.53
	Ba	La	Ce	C	Pt	Pd	Rh
	1.48	2.67	6.21	0.036	0.0632	0.1510	0.0235

The X-ray diffraction (XRD) pattern presented in Figure 2 illustrates the results of the phase analysis of the initial sample, revealing a complex mineral composition. The primary identified phases include indialite ($Mg_2Al_4Si_5O_{18}$) – 62.4%, zirconium oxide (ZrO_2) – 14.3%, zirconium-ceria oxide ($ZrCeO_4$) – 9.6%, lanthanum-zirconium oxide ($La_2Zr_2O_7$) – 9.5%, and aluminum-lanthanum-magnesium compound ($Al_2La_2Mg_3$) – 5.4%. These findings indicate the presence of aluminum, zirconium, magnesium, and rare-earth element compounds, suggesting a possible crystalline structure with high thermal and chemical stability. The diffraction peaks correlate well with PDF database references, confirming the reliability of the phase analysis.

For accurate identification and confirmation of the presence of noble, rare, rare-earth, and associated metals, as well as for elemental mapping of mineral grains, electron probe scanning was performed using a scanning electron microscope (SEM) at high magnifications (Figure 3).

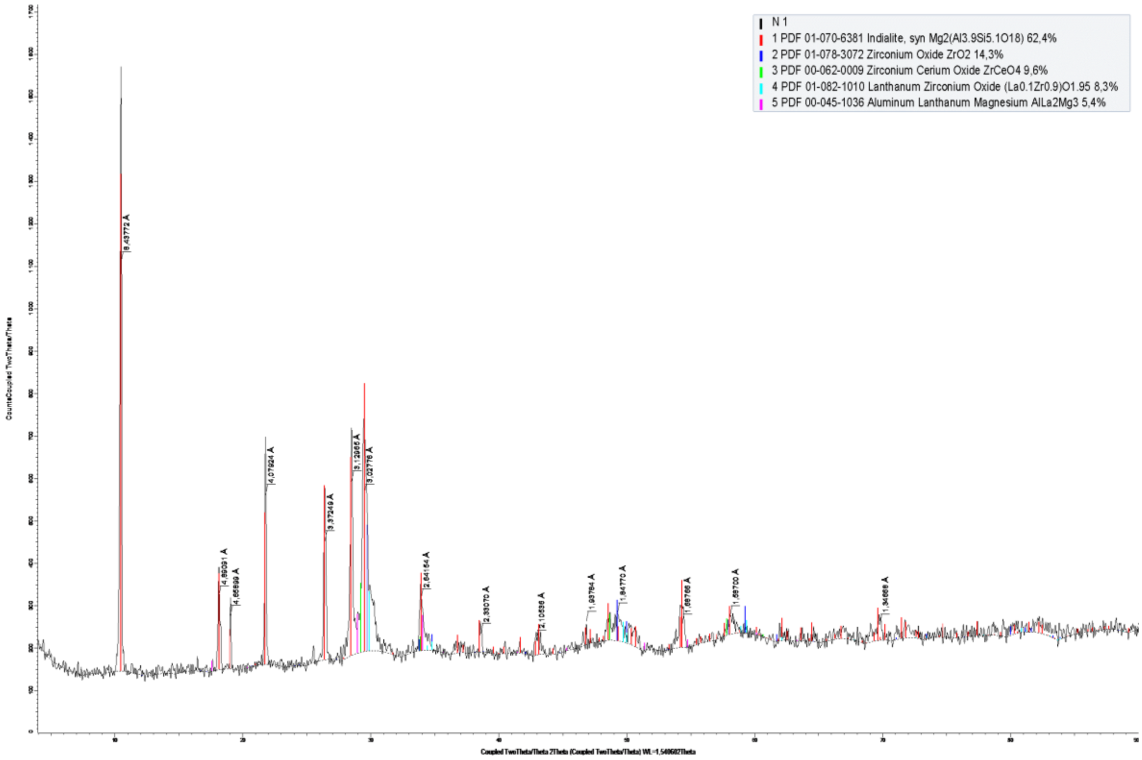

Figure 2. X-ray diffraction pattern of spent catalysts

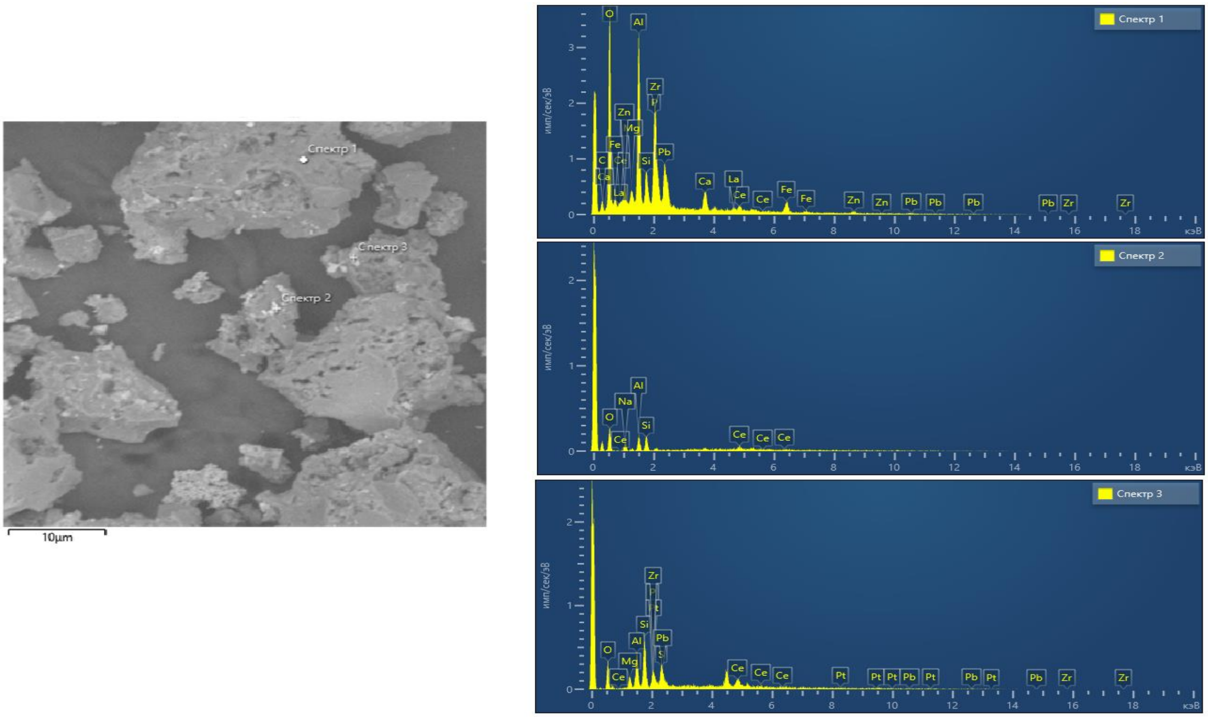

Figure 3. Micrograph and point spectral analysis of particles in the initial sample

The results of qualitative and quantitative elemental analysis, conducted in a randomly selected microscopic region, are presented in correlation with the surface topography obtained through scanning electron microscopy and a specialized electron probe microanalyzer. The acquired data are visualized in Figure 3, and the corresponding elemental composition spectrum is provided in Table 2.

Table 2. Results of electron probe spectral analysis of catalyst particles

Element	Spectrum 1	Spectrum 2	Spectrum 3
	%	%	%
C	7.98	-	-
O	36.71	-	24.67
Mg	0.95	-	3.90
Al	11.14	41.70	10.57
Si	2.25	58.30	15.71
P	4.69	-	3.67
Ca	2.65	-	-
Fe	4.76	-	-
Zn	3.25	-	-
Zr	8.67	-	8.4
La	1.74	-	-
Ce	2.97	-	14.53
Pb	12.24	-	9.05
S	-	-	7.94
Pt	-	-	1.57
Итого	100	100	100

The presented micrograph, obtained using scanning electron microscopy (SEM), visualizes the particles of the initial sample with characteristic morphology. The image reveals a heterogeneous material structure containing both large aggregates and fine-dispersed inclusions. The scale bar of 10 µm indicates the micron-sized nature of the particles, while the marked regions (Spectrum 1, Spectrum 2, Spectrum 3) represent zones of localized spectral analysis.

The results of energy-dispersive X-ray spectroscopy (EDS) for these points demonstrate a complex elemental composition, including aluminum (Al), silicon (Si), magnesium (Mg), zirconium (Zr), iron (Fe), lead (Pb), rare-earth elements (La, Ce), and platinum (Pt). Variations in elemental concentrations across different spectra indicate the heterogeneous composition of the samples.

The spectral analysis data confirm the presence of noble and rare-earth elements, highlighting the potential value of material recycling. The obtained results are visualized in the spectrograms on the right, displaying the intensities of characteristic X-ray lines corresponding to the marked areas in the micrograph.

Table 2 presents the results of electron probe spectral analysis of catalyst particles in three different regions of investigation (Spectrum 1, Spectrum 2, Spectrum 3). The data reflect the mass fractions (in %) of the chemical elements detected in localized areas of the sample.

The analysis reveals significant variations in elemental composition across different spectral zones. In Spectrum 1, oxygen (36.71%), aluminum (11.14%), zirconium (8.67%), and lead (12.24%) are predominant, indicating the presence of oxide compounds and metallic elements. Spectrum 2 is dominated by aluminum (41.70%) and silicon (58.30%), suggesting the presence of aluminosilicate phases. Spectrum 3 exhibits high concentrations of silicon (15.71%), oxygen (24.67%), cerium (14.53%), and sulfur (7.94%), along with platinum (1.57%), confirming the presence of rare-earth and noble metals.

The overall element distribution highlights the complex and heterogeneous composition of catalyst particles, including oxides, aluminosilicates, rare-earth elements, and noble metals, which is crucial for further research into their catalytic properties and recycling potential.

Elemental mapping of catalyst particles from spent automotive catalysts, performed using energy-dispersive spectroscopy (EDS) at 750× magnification, is presented in the image. The multilayer element distribution map (left) visualizes the heterogeneous structure of the material with varying concentrations of key elements.

The mapping results (Figure 4) confirm the complex composition and inhomogeneous element distribution within the material, emphasizing the need to consider these factors when developing efficient extraction methods for valuable components.

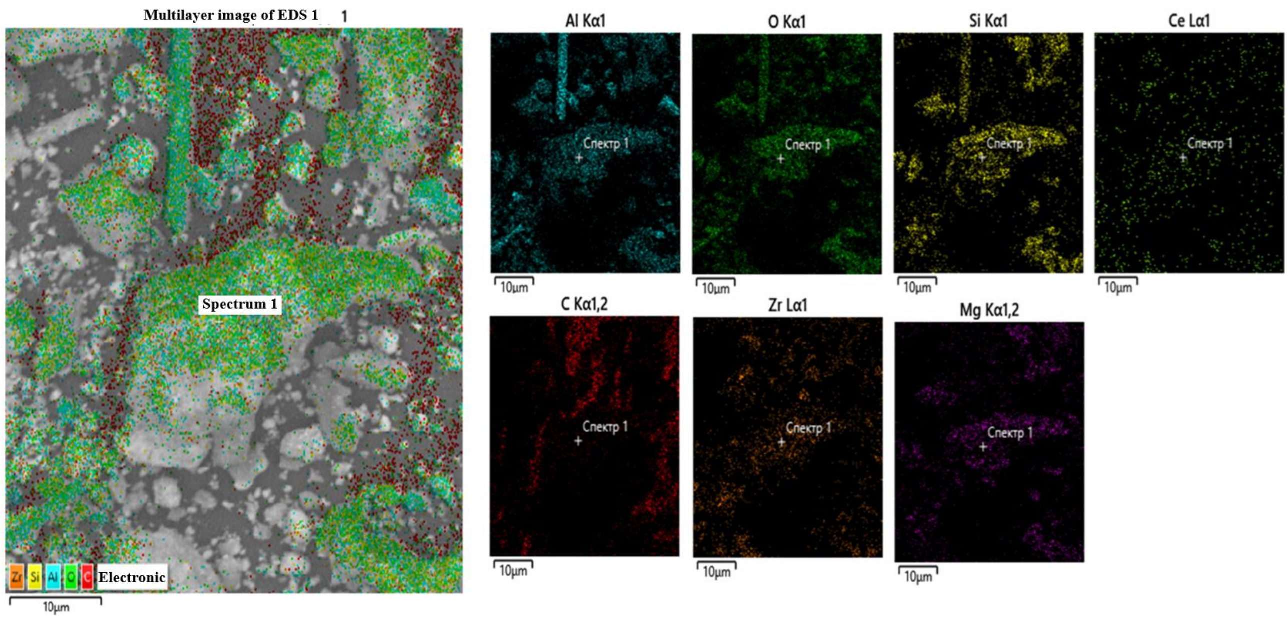

Figure 4. Elemental mapping of grains and particles of automotive catalysts

Elemental mapping of catalyst particle grains revealed a non-uniform element distribution in spent catalysts. The primary components localized in specific regions are aluminum (Al) and silicon (Si), indicating an aluminosilicate matrix of the catalyst. Oxygen (O) is relatively evenly distributed, suggesting the presence of oxide phases. Carbon (C) is detected in limited areas, likely associated with carbon-containing residues or decomposition products of organic compounds. Cerium (Ce) and zirconium (Zr) identified in localized regions point to the presence of rare-earth oxide phases, characteristic of automotive catalysts. Magnesium (Mg) is also present in the catalyst structure, though its distribution is highly uneven. However, noble metals were not detected in the analyzed areas, confirming their heterogeneous distribution within the material.

The obtained results indicate that spent catalysts contain not only noble metals but also rare and rare-earth elements, making them a promising target for recycling. Consequently, studies on the extraction of valuable components, particularly rare and rare-earth metals, were conducted using thermal treatment with concentrated sulfuric acid at the initial stage of the research.

4. Conclusions

Recycling of spent automotive catalysts is a crucial task for both the economy and the environment of Kazakhstan. The conducted studies have confirmed the presence of significant amounts of noble metals (Pt, Pd, Rh), rare and rare-earth elements (La, Ce), as well as aluminum and zirconium oxides, making these catalysts a valuable secondary raw material.

An analysis of existing recycling technologies has shown that traditional pyrometallurgical methods demonstrate high efficiency in extracting noble metals but are associated with high energy consumption and environmental risks. The results of X-ray phase analysis and energy-dispersive spectral analysis have confirmed the complex composition of catalysts, highlighting the need for further improvement in comprehensive recycling technologies.

Thus, the development and implementation of new technological solutions aimed at maximizing the recovery of all valuable components will significantly enhance the economic efficiency of recycling, reduce dependence on imported rare metals, and minimize environmental impact. Investing in scientific research and modernizing recycling facilities is a key step toward the sustainable development of Kazakhstan's metallurgical industry.

Author contributions

Conceptualization: S.S.K.; Data curation: A.T.K.; Formal analysis: S.S.K.; Funding acquisition: S.B.Y.; Investigation: S.B.Y.; Methodology: K.T.Y.; Project administration: S.B.Y.; Resources: S.B.Y.; Software: S.S.K., K.T.Y.; Supervision: S.S.K., K.T.Y.; Validation: S.B.Y.; Visualization: A.T.K., S.B.Y.; Writing – original draft: A.T.K., S.B.Y.; Writing – review & editing: S.B.Y. All authors have read and agreed to the published version of the manuscript.

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Conflicts of interest

The authors declare no conflict of interest.

Data availability statement

The original contributions presented in this study are included in the article. Further inquiries can be directed to the corresponding author.

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Қазақстанда асыл, сирек және сирек жер металдарын шығарумен пайдаланылған автомобиль катализаторларын қайта өңдеу перспективалары

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Аңдатпа. Пайдаланылған автомобиль катализаторлары қазіргі заманғы өнеркәсіпте шешуші рөл атқаратын сирек кездесетін және асыл металдардың құнды көзі болып табылады. Бұл шикізатта сирек кездесетін сирек кездесетін және асыл металдар бар, бұл оларды өңдеуді жоғары тиімді және үнемді етеді. Кешенді қайта өңдеу технологиясын әзірлеу үшін химиялық, рентгендік фазалық, рентгендік спектрлік және электронды микроскопиялық талдау сияқты әртүрлі талдау әдістерін қолданатын катализаторлар зерттелді. Фазалық талдау нәтижелері сирек кездесетін сирек кездесетін және асыл металдардың әртүрлі фазалық күйлерде, оксидтер түрінде, катализатордың алюминий Силикат матрицасындағы кіріктірілген қоспалар түрінде болатындығын көрсетеді. Бұл деректер металдарды алудың тиімді әдістерін жасау үшін маңызды мән береді. Бұл шикізатты өңдеудің негізгі тәсілдері пирометаллургиялық және гидрометаллургиялық технологиялар болып табылады, олардың әрқайсысының өзіндік артықшылықтары мен бірегей тиімділік критерийлері бар. Бұл мақалада пайдаланылған катализаторларды физика-химиялық зерттеулерге және оларды ашудың ұсынылған әдісіне назар аударылады, бұл сирек кездесетін, сирек кездесетін және асыл металдарды тиімді алуға мүмкіндік береді. Зерттеу нәтижелерін қолданыстағы шикізатты қайта өңдеудің жаңа технологиялық схемаларын оңтайландыру және әзірлеу үшін пайдалануға болады.

Негізгі сөздер: *пайдаланылған автомобиль катализаторлары, сирек металдар, асыл металдар, сирек жер элементтері, платина металдары, гидрометаллургиялық технологиялар, пирометаллургиялық технологиялар, селективті тұндыру, қайталама шикізат.*

Перспективы переработки отработанных автомобильных катализаторов с извлечением благородных, редких и редкоземельных металлов в Казахстане

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Аннотация. Отработанные автомобильные катализаторы представляют собой ценный источник редких и благородных металлов, который играет ключевую роль в современной промышленности. Данное сырье содержит редкие редкоземельные и благородные металлы, что делает их переработку высокоэффективной и экономически выгодной. Для разработки комплексной технологии переработки исследовались катализаторы с использованием различных методов анализа, такие как химический, рентгенофазовый, рентгеноспектральный и электронно-микроскопический анализ. Результаты фазового анализа указывают на то, что редкие редкоземельные и благородные металлы находятся в различных фазовых состояниях, в виде оксидов, встроенных включений в алюмосиликатной матрице катализатора. Эти данные дают критическое значение для создания эффективных методов извлечения металлов. Ключевыми подходами для переработки данного сырья являются пирометаллургические и гидрометаллургические технологии, каждая из которых обладает своими преимуществами и уникальными критериями эффективности. В этой статье акцентируется внимание на физико-химических исследованиях отработанных катализаторов и предложенном способе их вскрытия, что позволяет эффективно извлекать редкие, редкоземельные и благородные металлы. Результаты исследований могут быть использованы для оптимизации существующих и разработки новых технологических схем переработки вторичного сырья.

Ключевые слова: *отработанные автомобильные катализаторы, редкие металлы, благородные металлы, редкоземельные элементы, платиновые металлы, гидрометаллургические технологии, пирометаллургические технологии, селективное осаждение, вторичное сырье.*

Publisher's note