

Technology and possibilities of recycling catalysts

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Abstract: The presented article focuses on the possibilities of recycling three types of catalysts. These catalyst types will undergo examination, measurement, and analysis with the aim of identifying which of these catalysts contains the necessary number of precious metals (PGM - Platinum Group Metals). PGM metals are among the rarest and most challenging-to-obtain elements on Earth, carrying a high risk of supply shortage. Nevertheless, they are crucial for the European Union (EU) and the automotive industry. Not every catalyst used in the market is suitable for recycling due to the absence of these precious metal particles.

1 Introduction

In the dynamic environment of modern industry and technological advancement, the concept of sustainability has emerged as a primary aspect. As societies strive to achieve a harmonious balance between economic growth and environmental care, the critical role of recycling processes becomes increasingly evident. Among these processes, catalytic recycling stands out as a key player on the path to a more sustainable future.

The necessity for catalysis recycling becomes even more apparent when considering the intricate composition of modern catalysts. Numerous industrial catalysts are designed with complex structures that incorporate rare and precious metals like platinum, palladium, and rhodium, collectively known as Platinum Group Metals (PGMs). These metals, acclaimed for their exceptional catalytic properties, confront a challenging paradox. While propelling the development of sustainable technologies, their scarcity and associated geopolitical intricacies pose significant supply chain risks. This dual nature of PGMs intensifies the urgency to implement effective recycling

procedures that ensure the responsible and efficient utilization of these valuable resources.

The need for catalytic recycling goes beyond the scope of resource conservation. It deeply resonates with the global effort to reduce the ecological footprint of various industrial sectors. Given that catalytic processes are an integral part of sectors such as energy production, transportation, and chemical manufacturing, the effective recycling of catalysts contributes to reducing energy consumption, lowering emissions, and minimizing waste generation. These outcomes align with international sustainability goals and regulatory frameworks aimed at mitigating climate change and promoting a circular economy [1].

2 Prepare of identification of sustainable catalyst

The identification of each used catalyst depended on the container code. The main characteristics of each catalyst included the car model and catalyst type. The type of catalysts was verified based on the metal content after chemical analysis [2].

Table 1 Identification of 3 catalysts on market [3]

Number	Canister code	Model Car
1	13106917	Opel Astra H
2	8200358551, C114	RENAULT ESPACE, 2200CC, DIESEL
3	3B0131701Q, 8D0178E, GLH	AUDI A6, 2400CC, DIESEL

Each catalyst was disassembled and de-canned to remove the metal container and obtain the ceramic catalyst, which was prepared for physico-chemical characterization.

The decanting process was conducted carefully to prevent the fracture of the ceramic monolith inside, allowing the evaluation of weight and dimensions [2].

Technology and possibilities of recycling catalysts

Martin Straka, Peter Kacmary, Jakub Kovalcik

Table 2 Dimension of catalyst [3]

Number	Weight, g	Height, cm	Diameter, cm
1	952.30	9.7	20.1
2	1026.75	14.4	10.7
3	573.50	11.9	11.4

The acquired dimensions of each catalyst were measured according to their shape. For cylinder-shaped catalysts, weight, height, and diameter were measured. Each ceramic catalyst was pre-processed for microscopic analysis and elemental analysis (XRF). The pre-processing involved steps of grinding, milling, and sieving to reduce the particle size below 250 μm . During the grinding phase, small pieces were taken from each catalyst, which underwent analysis by optical microscopy for cell observation and measurement of their dimensions [2].

3 Results - analysis through optical microscopy

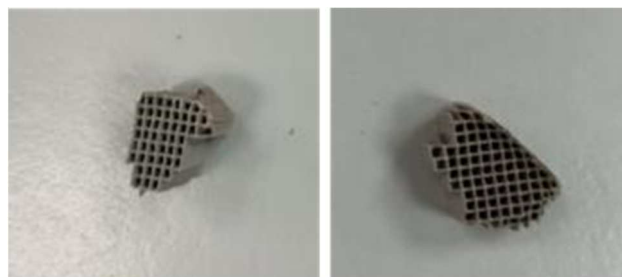
In the context of analyzing three different catalysts used in the study, an optical microscope from the AmScope ME520 (Figure 1) series was employed, along with the corresponding software. The data was acquired at a total magnification of 125x. A detailed description of the obtained information regarding each catalyst follows [2].


Figure 1 AmScope ME520 [5]

3.1 Process of identification of sustainable catalyst via optical microscopy

Catalyst 1

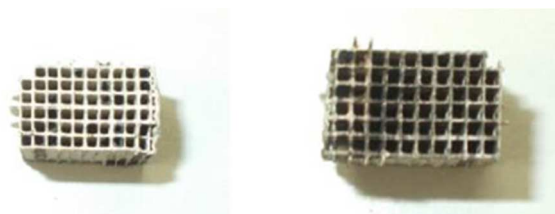
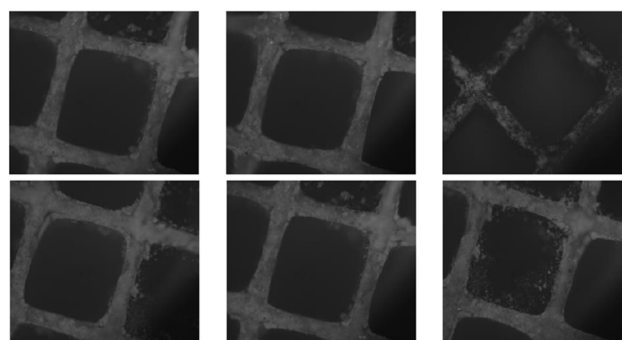
Two minor fragments were gathered (Figure 2) from the exhausted catalyst for the purpose of capturing images from various regions under the optical microscope, wherein the density of cells and the thickness of the ceramic monolith walls were assessed [2].


Figure 2 Pieces from catalyst number 1 [3]

Utilizing the optical microscope images, the density of cells and the thickness of the monolith's washcoat were evaluated. The findings derived from the optical microscope revealed an estimated cell density of around 527 cells per square inch (cps). Furthermore, measurements and calculations yielded a cell wall thickness of 0.193 mm and a washcoat thickness of 0.049 mm.

Catalyst 2

To gain visual insights from diverse regions under the optical microscope, two minor fragments were gathered (Figure 3) from the exhausted catalyst number 2. This facilitated the assessment of cell density and ceramic monolith wall thickness. The images obtained through the optical microscope were also utilized to gauge the cell density and washcoat thickness of the monolith. Specific regions (Figure 4) were chosen to quantify the catalyst's cell density [3].


Figure 3 Pieces from catalyst number 2 [3]

Figure 4 Optical microscope images from catalyst number 2 [3]

Catalyst 3

To capture a variety of perspectives under the optical microscope, two minor fragments were gathered (Figure 5) from depleted catalyst number 3. This procedure aimed to

Technology and possibilities of recycling catalysts

Martin Straka, Peter Kacmary, Jakub Kovalcik

measure the cell density and the thickness of the ceramic monolith's walls. The cell density and the washcoat thickness of the monolith were determined based on the images obtained through the optical microscope. Specifically designated regions, (Figure 6), were chosen to quantify the catalyst's cell density [3].

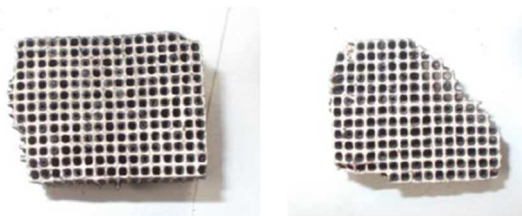


Figure 5 Pieces from catalyst number 3 [3]

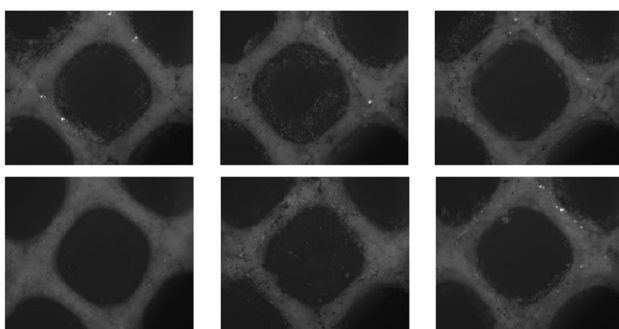


Figure 6 Optical microscope images from catalyst number 3 [3]

3.2 Chemical analysis

In the realm of elemental analysis, the importance of preliminary processing and accurate sample selection cannot be overstated. For every ceramic catalyst, an individual procedure involved milling, ensuring that 80% of the sample achieved a particle size below $250\mu\text{m}$, accomplished via a knife mill (Figure 7). The particle size of each sample was subsequently verified with sieves tailored for sizes below $250\mu\text{m}$ [5].



Figure 7 Knife mill [3]

Each sample underwent a process of homogenization and was subsequently divided into four distinct sections. To yield more comprehensive and representative outcomes, two minor samples were obtained. These were subsequently subjected to drying in a BINDER oven (120°C , 2 hours) (Figure 8), in preparation for XRF

analysis aimed at quantifying the content of Platinum Group Metals (PGMs) [5].



Figure 8 Dryer [3]

3.3 X-ray Fluorescence (XRF) analysis

The loading of PGMs was ascertained using X-Ray Fluorescence spectroscopy. XRF analysis, a method characterized by accuracy, speed, non-destructiveness, and repeatability, eliminates the need for chemical preparation. Consequently, chemical reagents are unnecessary, thereby minimizing costs. The XRF spectrometer (Vanta Olympus 2017, Waltham, MA, USA) (depicted in Figure 20) comes with an inherent calibration from the manufacturer, allowing precise measurement of Pt, Pd, and Rh in used catalysts with average PGM concentrations of 1000ppm, 1700ppm, and 300ppm, respectively. Despite the existing calibration of the X-ray Fluorescence (XRF) analyzer, conducted an additional calibration to enhance the precision of XRF measurements. Through this supplementary calibration, Pd was calibrated within a loading range of 1270-2730ppm, Pt within a range of 614-2760ppm, and Rh within a range of 237-322ppm. The PGM content of the two minor samples from each catalyst was gauged, and their average was computed (Tab.3). The homogeneity of the catalyst samples was verified through these measurements [7].

3.4 Calcination process

As previously noted, the microscopic examination revealed the presence of organic residues within the structure of the catalytic converter. Consequently, a calcination procedure was undertaken on small-scale samples to quantify the mass of these organic deposits within each catalyst. Additionally, the calcined samples were subjected to XRF analysis to evaluate how these organic compounds influenced the detection of PGM concentrations. The quantification of organic deposits was based on the comparison of sample mass before and after the calcination process, conducted at a temperature of 750°C for a duration of 5 hours. The impact of calcination was evident through visual observation, particularly by noting the color alteration in the various samples [7].

Technology and possibilities of recycling catalysts

Martin Straka, Peter Kacmary, Jakub Kovalcik

4 Conclusion

In summary (Table 3), a total of 3 used catalysts were examined for the purpose of their comprehensive physicochemical characterization. This characterization encompassed catalyst identification, preprocessing, and XRF analysis. These steps aimed to prepare the samples for chemical analysis and determine the content of PGMs (platinum, palladium, rhodium). Calcination was performed to identify potential organic residues in each sample. The samples underwent XRF analysis before and after calcination to assess the impact of organic compounds on PGM detection.

Regarding the ultimate identification of the provided used catalysts, those containing rhodium and platinum or/and palladium were classified as Three-Way Catalysts (TWC). Conversely, catalysts primarily containing platinum or/and palladium were mostly identified as Diesel Oxidation Catalysts (DOC). Nevertheless, spent catalytic converters with low concentrations of platinum or/and palladium could potentially be labeled as Dual-Function Catalysts, considering additional information such as the vehicle model and manufacturing year [7].

Table 3 Final summary of the results of the three catalysts [3]

Number	Pt, ppm	Pd, ppm	Rh, ppm
1	-	1945	348
2	620	-	-
3	2434	-	-

As a result of this detailed analysis, the following research findings were obtained:

- Only catalyst number 1 is identified as a suitable for recycling thanks to the values of Pd and Rh.
- Only Pt was detected in the other catalysts [7].

Acknowledgement

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