Politechnika Śląska Wydział Inżynierii Materiałowej

Rozprawa doktorska

## Opracowanie metody przetwarzania zużytych katalizatorów samochodowych w kierunku pozyskania z nich materiału o właściwościach sorpcyjnych

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## Abstract

The studies have been focused on processing and recycling of spent car catalysts, and more specifically the ceramic material, i.e. cordierite, which is the basic component of ceramic car catalysts. The main goal of the research is to develop an effective method of processing cordierite to obtain the material with attractive sorption properties. Cordierite, unlike classical materials with sorption properties, is characterized by a poorly developed specific surface area (below 1.0 m<sup>2</sup>/g). In addition, it is a material with an exceptional chemical and temperature resistance. Its surface activation in ordinary laboratory conditions requires at least several hours of treatment in an environment of hot and concentrated, strong inorganic or organic acids. The harsh chemical regime significantly reduces the potential benefits from the recycling process of spent cordierite according to the literature methods.

The studies have presented and developed an effective, environmentally friendly method of surface activation of cordierite based on the use of the electromagnetic mill to process cordierite. Different processing conditions in the electromagnetic mill were used. The variable parameters were the time of the processing of cordierite samples in the electromagnetic mill and the methods of activation: the dry method, i.e. without the use of a liquid phase, and the wet method with the use of water (first option) or 10% oxalic acid (second option) as the liquid phase. Cordierite after surface activation in the electromagnetic mill were tested as a adsorbent for the purification of used, contaminated transformer oils. The sorption's test results confirmed that the cordierite adsorbent obtained by the wet method with the use of 10% oxalic acid and 100-second processing time in the electromagnetic mill (sample M/II<sub>100</sub>) exhibited the highest sorption efficiency. For this adsorbent, a significant improvement in transformer oil performance parameters was achieved, comparable to the sorption efficiency of commercial absorbers - Fuller's earth and activated alumina. The research confirmed that the use of electromagnetic mill is a critical factor for improving the sorption properties of cordierite. A sample of cordierite which was devoid of processing in the electromagnetic mill did not exhibit sorption efficiency.

The cordierite samples were studied using the following physicochemical analyzes: the grain characteristics by IPS-U analyzer; the specific surface area by BET method and the chemical surface composition analysis by SEM-EDS method. The obtained physicochemical characteristics of the cordierite samples were affected by the method of activation in the electromagnetic mill. Sample M/II<sub>100</sub> exhibited the lowest degree of grain size dispersion, the smallest average grain size (about 0.062 mm), the highest increase in porosity (more than 110-fold) and the specific surface area (more than 245-fold). The SEM-EDS analysis confirmed that activation in the electromagnetic mill using wet method with 10% oxalic acid resulted in the changes of the elemental surface composition, i.e. increase in the silicon content, and decrease in the aluminum and magnesium content.

The studies have also determined the changes in the specific surface area of cordierite after processing in the electromagnetic mill over time, resulting from the changes in the selected parameters, i.e. the grain size and the grain shape of the cordierite samples. The developed models and relationships were an useful tool for better insight in the process of the surface activation of the cordierite samples over times while processed in the electromagnetic mill.

The studies have provided the basis of the knowledge about the environmentally friendly method of spent cordierite processing, the effective surface activation of cordierite and the changes in physiochemical properties of cordierite after the treatment in electromagnetic mill along with the examination of selected factors affecting the process of surface activation.