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Mössbauer Analysis of Iron Oxides in Topsoil

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Abstract

Analysis of the reports of the Regional Inspectorate for Environmental Protection in Katowice, allows the exact location of critical points associated with increased emissions of PM10 and PM2.5, consequently also technogenic magnetic particles (TMPs) in Upper Silesia. TMPs are ferrimagnetic iron minerals which arose during high temperature technological processes. Their presence in soils can indicate the soil contamination by potentially toxic elements (PTE) because TMPs due to their structure and well developed surface area have the ability to bind metals and metalloids. In iron analysis very effective tool is Mössbauer spectroscopy because of the high sensitivity of this method and precise phase analysis capabilities. It enables the determination of iron speciation in environmental samples and provides very precise information about the chemical, structural and magnetic properties of material.

The main objective of this study was identification of iron (hydro)oxides occurring in soils being under deposition of industrial dusts for long time period. Topsoil samples collected in the vicinity of power station, metallurgical and coke plants were subjected to this investigation. After standard initial preparation, samples were divided into granulometric fractions ($\emptyset = 0.05-0.1, 0.1-0.25$, and 0.25-0.5 mm) which then underwent the magnetic separation using the isodynamic magnetic separator operated on different currents. In such a way each granulometric fraction of an original sample was separated into 6 subsamples (one nonmagnetic and five with increasing magnetic susceptibility).

Afterwards, subsamples were subject of magnetic, morphological and mineralogical analyses (e.g., bulk magnetic susceptibility and temperature dependence of magnetic susceptibility and X-ray powder diffraction and scanning electron microscopy with energy dispersive spectroscopy – SEM/EDS).

The main research method was Mössbauer spectroscopy. The ⁵⁷Fe Mössbauer spectra were recorded at room temperature with a constant acceleration spectrometer with ⁵⁷Co:Rh source (activity ~50 mCi). The metallic iron powder (α -Fe) absorber was used for velocity

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Fig. 1. Room temperature ⁵⁷Fe transmission Mössbauer spectra with schematic component fitting: for sample A – present both the magnetic and non-magnetic fraction; and B – without magnetic fraction, and present non-magnetic.

and isomer shift calibration of the Mössbauer spectrometer. The mineralogical analysis of the spectra was based on the Mössbauer Mineral Handbook (Stevens *et al.* 2005).

The Mössbauer spectra measured at room temperature were fitted with two quadrupole doublets and one or two magnetic sextets. The paramagnetic doublets may originate from the following sources: (I) Fe³⁺ and Fe²⁺ cations in silicate minerals, (II) Fe3⁺ in oxyhydroxides (β -FeOOH and γ -FeOOH), and/or (III) from iron-containing compounds in the form of ultra-fine particles in the superparamagnetic state (Kopcewicz and Kopcewicz 2001). First Zeeman sextet (H = 29 T) are connected with pyrrhotite, another one (H = 50 T) was identified as magnetite or maghemite.

Keywords: magnetic susceptibility, Mössbauer spectroscopy, TMPs, topsoil.

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