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# Examination of microstructure of solidification product containing hazardous sludge

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## Abstract

The tests involved with the microstructure evaluation must be also added to the processes which significantly support the evaluation of long-term durability of solidification product (SP). Monitoring the microstructure by analyses such as scanning electron microscopy (SEM), X-ray powder diffraction (XRD) and differential thermal analysis (DTA) it can be assumed according to the new compounds being formed during the hydration whether the hydration products are modified. Moreover, it could also be determined how the pollutants contained in input hazardous waste (HW) are incorporated in solidification product (SP) matrix. Within this work four different types of sludges (S) containing dangerous substances, mainly heavy metals were selected as input hazardous waste (HW) for laboratory preparation of samples of the solidification products (SP) for the microstructure analyses. On the basis of the findings from microstructure analyses it can be assumed whether contaminants contained in input HW, sludges, is immobilized into the matrices of solidification products (SP) by incorporating into the hydration products of SP matrix.

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Keywords: Solidification product; contaminants; scanning electron microscop; differential thermal analysis; X-ray diffraction

## 1. Introduction

To still larger extent there is an effort to use the solidification/stabilization (S/S) process for transformation of hazardous waste (HW) in new secondary raw materials suitable for preparation of building materials. It has been

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In the Czech Republic the solidification product (SP) has been used in the form of granulate as technological material at landfills and also as reclamation material. In this way not only the disposal of hazardous waste (HW) or safe storage of produced solidification products (SP) at landfill for strongly lower fees are provided but also directly its application as useful material replacing natural resources of raw materials. Monitoring the microstructure of SP interior structure can be clarified together with physical and chemical mechanisms occurring during the S/S processes. The study of solidification product (SP) microstructure may also help to elucidate the way of contaminant incorporating in SP matrix structure. However, majority of minerals contained in SP can be defined only informatively, because the microstructure of SP has not been sufficiently examined, especially due to different chemical composition of various input hazardous waste (HW). Since the amount of contaminants are not reduced by S/S process and other physical and chemical barriers preventing these pollutants to proceed further to the environment are created it is necessary to pay attention also to SP long-term durability and verify stability of its properties. Applied solidification agents, homogenization grade of solidification mixture and ambient conditions mostly decide on the success of hazardous waste (HW) solidification.

Cement, fly ash and lime are the most frequently used solidification agents. Conner [4] pointed out that cement is widely used solidification agent of low-level radioactive, hazardous wastes, mixed wastes, and remediation of contaminated sites because it has many advantages. Shi et al. [5,6] stated that solidification/stabilization (S/S) process of contaminants by cements includes the following three aspects: chemical fixation of contaminants – chemical interactions between the main hydration products of the cement and the contaminants; physical adsorption of the contaminants on the surface of hydration products of the cements and finally physical encapsulation of HW (low permeability of the hardened solidification mixtures).

#### 2. Materials

#### 2.1. Input hazardous waste

Not only the suitable properties for solidification/stabilization (S/S) using inorganic binders were among the main parameters of the selection of input hazardous wastes (HW) but there was also the requirement that S/S was desirable also in economic point of view. The cost-effective criterion is represented by minimal annual production of HW set to 10 tons, because S/S of lower waste volume will not be effective. Therefore, four different sludge (S) types were selected as input HW for laboratory preparation of solidification products (SP). They were inorganic wastes containing dangerous substances. As the most suitable the following HW were selected: sludge from metal machining, neutralisation sludge (S1); sludge from wire drawing processes (S2) and two types of sludge from the chemical and physical processes containing hazardous substances (S3 and S4). Sludge types marked as S3 and S4 are formed by neutralising of galvanic baths intended for surface treatment of metal elements using Ca(OH)<sub>2</sub> and their subsequent drying. Sludges S3 and S4 can be classified according to Environmental Protection Agency (EPA's) F-code Resource Conservation and Recovery Act (RCRA) HW listings to the code F019 that is one of the wastes generated from common industrial and manufacturing processes. The wastewater treatment sludges F019 are generated from the treatment of the rinse and overflow wastewaters from the chemical conversion coating process. The highest production – 500 tons annually is registered with sludge S1 and S4 and on that ground it is necessary to resolve further processing of these HW types, because currently they are only stored at landfills for HW, which is not cost effective and it is unacceptable from the ecological point of view.

In order to examine the input HW, sludges, and to evaluate their negative impact on the environment the leachability test was carried out according to the European standard EN 12457-4. It has been demonstrated [7] that due to the complexity of the leaching process and the number of factors influencing release, no single leaching test or single set of leaching conditions (e.g. extraction medium) is appropriate for the entire range of leach testing objectives and applications. On the basis of water leachate pH it can be assumed that S1 sludge has neutral character, S2 is slightly acid and S3 and S4 are of alkali nature. With regard to the leachability test results, the evaluation with limits stated in the Regulation no. 294/2005 on conditions of waste storage at landfills and their

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