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Physical and chemical characterization of technogenic pozzolans for the application in blended cements



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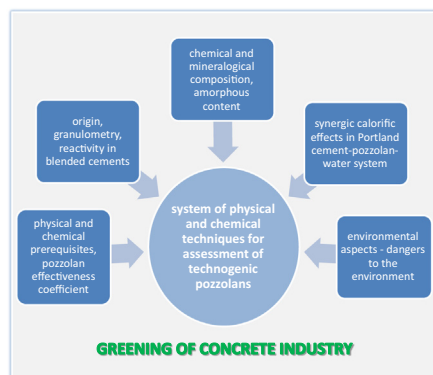
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HIGHLIGHTS

- System of physical and chemical techniques for assessment of technogenic pozzolans.
- Physical and chemical prerequisites, reactivity, performance in blended cements.
- Evaluation of synergistic calorific effects.
- Pozzolan effectiveness coefficient as a new criterion.
- Practical application demonstrated for fly ash from a coal power plant.

GRAPHICAL ABSTRACT



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ABSTRACT

Technogenic pozzolans present a diverse group of materials. They differ in their origin, chemical and mineralogical composition, granulometry, or amorphous content. However, the analysis of their properties before utilization as supplementary cementitious materials is often insufficient which may lead to unexpected results in some cases. In this paper, a system of physical and chemical techniques for the characterization of technogenic pozzolans is proposed, which takes into account both functional and environmental aspects. The system is hierarchical and consists of three sets of methods which assess consecutively the fundamental physical and chemical prerequisites, reactivity, and performance in a blend with Portland cement. The practical application of the methodology is demonstrated for the example of fly ash from a coal power plant as the most common technogenic pozzolan. Experimental results show that the proposed system has good prerequisites to serve as a good guidance for both research and building practice.

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1. Introduction

The application of technogenic pozzolans as common components of cements used in concrete industry gains on importance at the present time. The motivation is mostly environmental and economical, with the lack of availability of natural pozzolans in

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some territories as an additional supporting argument. However, contrary to natural pozzolanic materials, which mostly need only conditioning of particle characteristics by sieving and grinding [1], technogenic pozzolans may require a more sophisticated processing.

Calcination of various types of clays or shales can be considered as one of the simplest ways how to obtain a man-made pozzolan [2–4]. Some industrial byproducts, such as silica fume [5], fly ash [6], waste glass [7], or residues of ceramic industry [8] can even be used either in their original form or just after milling. Incineration of agricultural waste [9,10] or municipal solid waste [11,12] presents another source of possible pozzolanic materials. Among other waste materials having a potential to be used as pozzolans belong, e.g., fluid cracking catalyst residues [13], or thermally treated sewage sludge [14].

The current trend of utilizing a wide range of waste materials as pozzolanic additions to cement in highest possible extent brings though, despite the apparent environmental and economical benefits, potential dangers which were not experienced, at least not in such magnitude, with the previous generations of pozzolans. For example, some waste materials can contain heavy metals or harmful organic substances in increased amounts which are supposed to be incorporated into the cement matrix [15]; they may require specific measures at the manipulation in raw state. Some potential pozzolans can contain soluble salts or alkalis in too high concentrations [16] which can have negative effects on the durability of cement composites. Certain waste processing techniques can affect negatively pozzolanic properties, some others can have enormous economical demands [17].

The current diversity of technogenic pozzolans, which differ in their origin, chemical and mineralogical composition, granulometry, or amorphous content and the continuous supply of new materials to this group from various industrial and agricultural sources brings increasing requirements to their physical and chemical characterization. However, the growing complexity of an appropriate incorporation of potential technogenic pozzolans into the cement matrix is often underestimated.

Many research studies use for pozzolan characterization only a granulometry test, an oxide composition test, and a strength-based test, which may not be sufficient in some cases. If the results of these three basic tests are in accordance, the pozzolan can be assessed as basically suitable for application in blended cements. Nevertheless, sometimes the strength values are worse than it could be expected from the granulometric and composition tests and there is no apparent reason for this discrepancy. To give only couple of examples of recent investigations where such problem was observed, Garcia-Lodeiro et al. [18] found the 28-days compressive strength, f_c , of mortar with 60% clinker and 40% of incinerator waste $\sim 60\%$ in a comparison with the reference mortar where CEM IV (65% clinker and 35% fly ash) was used. Lim et al. [19] measured f_c of blended cement paste with 20% of palm oil fuel ash and 80% CEM I and obtained 84% of the reference paste f_c . Chiou and Chen [20] used thermally treated mix of pulp sludge and textile sludge as a partial replacement of CEM I and found for 10% pozzolan content 88% of f_c measured for the reference mix, for 20% replacement it was 68%, and for the blend with 30% of pozzolan only 33%. Afshinnia and Rangaraju [21] observed for the mix with 20% glass powder with appropriate fineness and composition as CEM I substitute 83–86% f_c , as compared with the reference concrete.

In this paper, we introduce a system of physical and chemical techniques which, in a comparison with most studies published before, makes possible a more complex characterization of technogenic pozzolans. In addition to the common tests mentioned before, also a thorough analysis of reactivity in ideal conditions is included. At the performance assessment of the potential pozzolan

in a blend with Portland cement, two new criteria are proposed. The first is based on the identification of possible synergistic calorific effects in the Portland cement-pozzolan-water system, the second one expresses, using the compressive strength measurement, the effectiveness of a specific amount of pozzolan replacing Portland cement in the blend on a 0 to 1 scale. The practical application of the proposed system of characterization techniques is demonstrated for fly ash from a coal power plant as a typical example of technogenic pozzolans.

2. System of physical and chemical methods for the characterization of technogenic pozzolans

The proposed system consists of three sets of methods which are applied consecutively. The first is focused on fundamental physical and chemical prerequisites of a material to be considered a pozzolan suitable for blended cements. It includes a granulometric test, an oxide/elemental analysis, and molecular spectroscopy. The second one is aimed at the reactivity of the potential pozzolan, which is examined using a quantitative X-ray diffraction (XRD) analysis, a test of pozzolanic activity, and a scanning electron microscopy (SEM) study involving energy dispersive spectroscopy (EDS). The third set of methods targets the performance of the supposed pozzolan in a blend with Portland cement. The basic tests involve the determination of early age hydration heat development and paste compressive strength, the additional tests deal with the environmental aspects.

2.1. Physical and chemical prerequisites

A supposed pozzolan should include very small particles. Therefore, the common sieve analysis is not a suitable granulometric method and laser diffraction should be preferred as a more appropriate particle sizing technique. There is not a lower limit for the particle size but the material is supposed to have a suitable fineness to work properly in a system with cement from both physical and chemical points of view. The material itself can potentially have pozzolanic properties (as for its chemical composition) but if the particles are too big, they cannot be utilized effectively and the pozzolanic reaction is too slow to contribute to the functional properties of the hydrated system. Therefore, an upper limit for particle size should be considered. The recommendations of European and American standards this upper limit to be 45 μm (in [22] in the form of 40% residue on the 45 μm sieve, in [23] 34% on the same sieve) can serve as a basic guidance in that respect. Nevertheless, this criterion should be used with the supposed application of a particular construction element in mind. In other words, it is necessary to decide if a slower strength increase caused by the slower course of pozzolanic reaction is acceptable or not and if a possible achievement of later higher strengths can be utilized effectively.

The oxide/elemental analysis examines the supposed pozzolan from a chemical point of view. X-ray fluorescence (XRF) spectroscopy as the most suitable method for that purpose should be used in the oxide mode at first. In the obtained oxide composition SiO_2 and Al_2O_3 must prevail. Otherwise, the material would not be able to react with portlandite in cement to form (in the presence of water) CSH and CAH phases. Therefore, a lower limit for the SiO_2 and Al_2O_3 content should be taken into account. The simple criterion of $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3 > 70\%$ by mass defined in ASTM C618 [23] for the Class F fly ash can serve for a basic assessment in that respect. It should be noted that the oxide composition can also indicate an excessive presence of some salts potentially harmful for cement composites (e.g., sulfates, chlorides) or alkalis. However, at this stage such findings are not preclusive as these components may not affect negatively the pozzolanic properties of a

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