



The effect of alternations in mineral additives (zeolite, bentonite, fly ash) on physico-chemical behavior of Portland cement based binders

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HIGHLIGHTS

- Binders with 30% of mineral additives gave physico-chemical properties matching to standard cement.
- Aluminosilicate additives behave as pozzolana increasing initial hydration rate and early strength gain.
- Heulandite and montmorillonite mineral phases did not interfere with the cement hydration.
- Additional water (DTA&FTIR detected) present in sorptive clays enables ion-exchange chemistry.
- PCA of XRD/DTA/FTIR data sets enabled neat differentiation of composite binders and reference cement.

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ABSTRACT

Instrumental analyses accompanied by analytical modeling tools were employed to assess physico-chemical changes induced by variations in chemical composition of cementitious composites, i.e. mortar binders. Coal combustion ash was utilized as pozzolanic mineral additive. The binders' mix-design was supplemented with sorptive clays to prevent migration of toxic elements from fly ash. The experiment was established on the premise of clay's ion-exchanging ability. Ten binders comprising cement CEM I 42.5 and fly ash, zeolite and/or bentonite additions were prepared in accordance with chemometric experimental design rules. Chemical composition was determined via XRF method. The changes in mineral phases and crystallinity were traced by X-ray diffraction. Hydration mechanisms and thermal behavior were investigated via DTA/TGA. The chemical bonds were identified by FTIR. Morphology of hardened samples was detected by SEM. Mathematical tools employed data sets of instrumental analyses to form a clear differentiation between binders and to assess changes caused by adoption of mineral additives in the mix designs. Sorptive clays showed pozzolanic behavior, thereby causing no incapacitation to the cement hydration mechanism, and classifying as a possible economical resources which can be used in production technology of construction materials to redeem environmental pollution issues of building industry.

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1. Introduction

Contemporaneous building composites, such as concrete and mortar, are still the main material basis used in the construction technology. Civil-engineering projects require significant amount of non-shaped materials; thereby the new solutions for their advanced performances have to be economically and environmentally sustainable. The fabrication of building materials implies high

consumption of natural resources, often accompanied by pollution caused by cement manufacturing and industrial wastes production [1]. Research studies practice a continuous tendency for upgrading of the performances of classic construction materials by incorporation of raw resources with special features [2–8]. The main step towards establishing sustainability in the construction composites processing is achieved through the reduction of quantity of materials characterized as expensive due to the high-maintenance manufacturing [9]. One of the groundbreaking solutions in building materials design was the application of industrial byproducts and economical primary resources as binders [9–12]. The main

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criterion imposed on these composites is to exhibit the properties at least equal to those of standard building materials [13,14].

The use of coal combustion byproducts in the building industry is in the accordance with ecological requirements; thereby it became a common practice [13,15,16]. Fly ash is recognized as a standard bonding agent replacement because its characteristics are matching to those of Portland cement [15–18]. The coal ash based products are still actual theme for the research since this secondary raw material contains a small yet detectable amount of toxic elements. If released into the environment, the non-degradable heavy metals are starting to accumulate. Therefore, finding a solution for isolating of the pollutants and preventing their emission is obligatory [13,19–21]. Immobilization of heavy metals within the microstructure of a composite can be achieved by application of natural sorptive clay as an addition to a cementitious binder [22–24]. Clay characterized with hydrophilicity and ion exchange ability is classified as sorbent. Thereby, bentonite and zeolite are economical primary resources ascribed with high sorptivity, but also with a certain extent of pozzolanic properties [24,25]. Bentonite minerals belong to the smectite group, whereas the most abundant mineral montmorillonite is composed of two SiO_4 tetrahedra enclosing an octahedral sheet of AlO_4 ions. The 2:1 layer structure exhibits excellent sorptive properties, as the weak Van Der Waals forces tie the piled layers of basic units simplifying the cationic exchange in the interlayer space [26–28]. Owing to favorable cation exchange capacity, bentonite is mainly used as a sorbent of heavy metals and radionuclides [29,30]. Zeolite is a crystalline framework of SiO_4 and AlO_4 tetrahedra that are linked by oxygen atoms situated in the structural intersections [31]. This clay is a typical molecular sieve with frameworks interconnected by micro-channels [32]. Ion-exchange chemistry of zeolite is enabled by the presence of extra-framework cations that can be relocated through the formed maze of cavities and pores [33,34]. The structural empty spaces in the range of molecular dimensions (3–10 Å) provide a space for reception of Na^+ , K^+ , Mg^{2+} , Ba^{2+} , Ca^{2+} cations, transition metal ions, molecules or ion groups (H_2O , NH_3 , CO_3^{2-} , NO_3^-) [35–37].

The necessity of finding an adequate solution for immobilization of toxic elements within fly ash and cement binding composites has prompted the research on possibilities of sorptive clay applications. In this study, the changes in physico-chemical behavior of cementitious binders with mineral additives (zeolite, bentonite, fly ash) were assessed in dependence of their starting chemical composition. Differential thermal analysis, thermogravimetry, Fourier transform infrared spectroscopy and scanning electron microscopy were employed to characterize ten experimental binders. Principal component analysis was used to discriminate and classify the obtained results of instrumental analyses. The combination of multiple experimental tests and chemometric analyses is a crucial step in acquiring the best product, as the chemometric deciding significantly assists in the selection of optimal binder for further engineering application [38–42]. The main goal is utilization of industrial byproduct and sorptive clays as economical resources, which are not incapacitating cement hydration, in the production of novel environmentally-friendly cementitious binders.

2. Experimental

2.1. Raw materials

Experimental binders were prepared from cement, fly ash, bentonite and zeolite used in different proportions in the mix designs. The base raw material for all composites was standard Portland cement, CEM I 42.5R. 97.5% of cement particles were in the 0.0–

Table 1

Chemical composition of raw materials used in the mix design of experimental binders.

Oxide, %	Cement	Fly ash	Zeolite	Bentonite
SiO_2	21.82	62.96	64.88	59.26
Al_2O_3	6.59	17.85	11.95	13.32
Fe_2O_3	4.75	6.99	1.76	3.36
CaO	62.36	5.93	6.18	7.01
MgO	2.17	2.33	0.57	0.05
K_2O	0.78	0.96	0.99	0.03
Na_2O	0.19	0.34	0.78	0.1
TiO_2	0	0.05	0.01	0.23
LoI	1.34	2.59	12.88	16.64

0.63 mm range, with 292 m^2/kg specific surface area (SSA). Fly ash, as a product of lignite combustion, was collected directly from the power-plant filter. The samples were randomly taken and preserved in hermetically sealed boxes. Fly ash showed 283 m^2/kg SSA according to BET method. The bentonite clay was of volcanic tuff origin with the montmorillonite content surpassing 70%. The crude bentonite samples from the deposit were acquired by standard sampling campaign. Prior to the laboratory analyses and preparation of the binders, bentonite was pulverized in an agate stone mill. Thereby, the sample obtained 79.1 m^2/kg SSA. Used natural zeolitic tuff appeared as pale yellow pelitic rock composed of zeolitized volcanic ash, with small amounts of quartz, plagioclase and biotite and covered by limonitic film. The pulverized zeolite sample had 75.5 m^2/kg SSA. The chemical composition of the raw materials was analyzed via X-ray Fluorescence method on a XRF spectrometer. The representative samples (100 g) were pulverized in a laboratory vibratory mill prior to the testing (Standard: SRPS EN196-2:2015). Averaged values are given in Table 1. The loss on ignition (LoI) was determined as a weight difference between 20° and 1000 °C.

2.2. Preparation of the experimental binders

Eleven binders were prepared. The sample marked with S_0 contained only Portland cement, as it was used for comparison with the composite samples (S_{1-10}). The ratios of raw materials were chosen in accordance with the rules of chemometric experimental design. Experimental data for the analysis were derived using the Simplex-lattice design, with 3 levels and 3 parameters (10 mixtures and 1 control). The mixing ratios are shown in Table 2.

The dry mixtures were homogenized using a laboratory mixer. The water was added during mixing. The water content needed for self-flow consistency was determined via trial mixes. The green mixes were cast in steel molds (20 × 20 × 20 mm) and kept in a climate chamber at 20 ± 2 °C for 24 h. Afterwards, the samples were removed from the molds and stored under the same conditions for next 24 h. During the next 5 days, the samples were cured at 20 ± 2 °C and 65 ± 5% humidity. The hardened samples were crushed and/or pulverized for further analyses.

2.3. Instrumental analyses

The chemical composition of the binders was analyzed via XRF spectrometer (Table 3).

Simultaneous differential thermal and thermo-gravimetric analyses were conducted on a DT/TGA analyzer in the 25°–1000 °C temperature range in an alumina pan at a constant heating rate of 10 °C/min and static air flow.

The mineral phase composition of the samples was determined using the X-ray diffraction (XRD) analysis. The XRD patterns were obtained on an automated diffractometer using a Cu tube operated at 40 kV and 30 mA. The instrument was equipped with

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