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Evaluation of compatibility of neutron-shielding boron aggregates with Portland cement in mortar



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HIGHLIGHTS

• Setting time retardation of cement in mortar with boron minerals can be controlled.

• Isothermal calorimetry helps to develop optimal mix design for radiation shielding.

• The most beneficial boron-containing aggregate was boron carbide and colemanite.

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ABSTRACT

Enhanced neutron radiation shielding capacity of protective structures can be achieved using cementbased composites with boron-containing aggregates. Experimental tests were performed to evaluate the effect of boron aggregates (colemanite, ulexite, borax, boron carbide) and nanosilica on the setting time and hydration heat of cement in mortars using isothermal calorimetry. Boron leaching test from mineral aggregates were performed in water and saturated Ca(OH)₂ solution. Cement setting retardation effects were found qualitatively correlated with boron leaching from mineral aggregates. A linear dependence of compressive strength of borated mortars and heat released after 72 h of cement hydration was found. A maximum content of boron compounds in mortar, allowing for a systematical control of setting time, was evaluated.

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1. Introduction

Current developments in the use of nuclear reactors and other advanced sources of neutron radiation require further advancement of shielding solutions. Particle spallation centers or other particle accelerators for neutron bombardment are known to generate high density neutron fluxes, e.g. 10¹⁸n/s like in European Spallation Source (under construction) [1]. Highly penetrating neutron radiation eventually must be captured by the shielding system to provide the necessary radiological protection for personnel and the environment. There is therefore a potential for the application of concrete of enhanced radiation shielding capacity.

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Known solutions for relevant concrete mix design include special components of the mix of particular elemental composition [2,3]. Certain elements, like hydrogen, boron, cadmium, or rare earth elements, exhibit outstanding neutron shielding capacity. Considering the abundance, costs and shielding efficiency the boron containing minerals are selected as particularly desired aggregates for radiation shielding concrete. Several examples of concrete mix design containing boron-bearing compounds are given in [2] and in [4]. Because boron has a large cross-section for neutron capture, especially for the capture of thermal neutrons, its effective content in concrete can be as low as 1%. Boron compounds are known as cement setting retarders, but are only seldom used in retarding admixtures [5,6]. However, they have been recently proposed as retarding compounds both for ordinary Portland cement [7] and calcium sulfoaluminate (CSA) cement [8]. Portlandite solubility is increased on addition of boron compounds



but in [9] it was shown that a combined effect of K_2O and B_2O_3 was seen as an extension of the stability field of portlandite to much higher borate concentrations. The properties of cement required for application in radiation shielding concrete are explained in [10]. The negative impact of boron compounds on cement setting and concrete hardening should be taken into account in the radiation shielding concrete technology, [4,11–13]. This is because uncontrolled setting and hardening may induce inhomogeneity of concrete, cold joints or areas of increased porosity that would weaken the designed radiation shielding capacity. Cold joints occur when concreting procedure goes in layers and the upper layer is cast too late, i.e. after hardening of the lower layer. Instead of a full, monolithic connection between the layers a plane of weakness can be produced, therefore named cold joint. Such a weakness is not allowed in radiation shielding structures and therefore uncontrolled retardation or acceleration of concrete hardening is not acceptable. In known publications, such issue was only fragmentary dealt with.

Experimental and Monte Carlo N-Particle code (MCNP) studies on radiation shielding of boron enriched concrete demonstrated the effectiveness of some boron minerals, like colemanite, ulexite, borax, or boron products like boron-frits, boron carbide [14]. A slab attenuation experiment carried out to using ²⁵²Cf neutron source showed that the shielding effect of a colemanite concrete was about 1.7 times better than ordinary concrete [15]. According to Kharita [16] adding borax to concrete up to 0.5% by weight had no significant effect on strength of concrete, but it caused a significant increase of shielding efficiency in thick concrete shields, up to 80% better than unborated concrete. A neutron transport calculation of a shield containing colemanite was made by Yarar [17] using the removal-diffusion technique and the decrease in shield thickness, compared to ordinary Portland cement concrete, was determined. In spite of demonstrated benefits of boron minerals as radiation shielding compounds of concrete their effect on the durability of concrete is not well known.

The minerals are usually applied as fine grained aggregates, as recommended in [2] to achieve uniform neutron absorption characteristics of material. Boron additives of powder consistency are usually not used due to the greater potential of forming chemical solutions that act as a retarder in the concrete [18,19]. Because of contradictory effects on shielding efficiency and setting/strength properties of concrete an optimization study was performed by Yadollahi [20] that resulted in an optimal colemanite concrete mixture for thermal neutron shielding and compressive strength. The suitability of colemanite ore waste as cost-effective shrinkagereducing agent was shown in [21]. However, this and most of other relevant shielding-related studies refer to fully hardened concrete, including also strength properties, but not the early strength and related cement hardening characteristics. The paper [13] presents isothermal calorimetry results of two type of colemanite (pure powder and ore) and their effect on cement hydration process is varied.

The aim of present study is to estimate the limits for the content of boron compounds used in cement mortars considering setting time and hydration heat of cement, as well as early mortar strength. Expecting the presence of boron to produce negative effects, an attempt was made to experimentally solve the optimization problem: finding a mix design with a maximum boron content at which setting time can be systematically controlled. The scope of the study includes four types of additives containing boron compounds.

The criterion of maximum boron content is assumed here as a technically justified simplification for mortar mix design for enhanced shielding capacity. Exact and final evaluation of the radiation shielding is normally carried out on whole structure when both the elemental composition of the material and the shape and dimensions of the structure influence the shielding efficiency

2. Experimental

2.1. Materials and specimens

The measurements were made on cement mortar samples with constant water to cement ratio w/c = 0.5, modified by addition of boron containing aggregate. The following materials were used:

- ordinary Portland cement CEM I 42.5 R (EN 197-1:2011), Table 1,
- deionized water,
- sand for testing of cement strength complying with EN 196-1:2016 [22],
- fine grained boron-containing aggregates:
 - a) colemanite with grain size 0.5–2 mm (Ca₂B₆O₁₁·5H₂O, content of B = 15.78 wt%),
 - b) ulexite with grain size 0.5–2 mm (NaCaB₅O₉·5H₂O, content of B = 13.34 wt %),
 - c) sodium tetraborate pentahydrate (borax) with grain size 0–1.6 mm (Na₂- B_4O_7 - $5H_2O$, content of B = 14.85 wt%),
 - boron carbide with grain size 90–125 μm and 94% of purity (B₄C, content of B = 73.56 wt%, take into account the purity of the raw material),
- amorphous nanosilica with grain size \leq 30 nm (nSiO₂) as water dispersion.

For heat of hydration measurements, mortar samples were prepared using the following mass ratios: cement:water:sand 1:0.5:0.75. At these proportions, the binder's volume share is 44% – it was intentionally high to extract a strong signal of the hydration heat generated. The additive, in the form of mineral or synthetic boron aggregate, was used as a partial replacement for quartz sand. The target content of boron in aggregates was from 2 to 10% per cement mass. This could not be achieved for borax and ulexite because of significant setting delay, therefore the range of boron content was diminished down to 0.33% and 1% of boron per cement mass, respectively. For B₄C this range was increased up to 40% B per cement mass because of weak influence on cement setting and hardening characteristics. As an additive for selected mortars, an amorphous nanosilica in form of an aqueous dispersion was added in an amount of 1 and 2% per cement mass; water present in the dispersion was taken into account by correction the amount of water added.

2.2. Test methods

An isothermal calorimetry method was used for the determination of progress of the cement hydration in Calmetrix I-Cal 2000 HPC isothermal calorimeter at the temperature of 23 °C for 72 h. The ASTM C 1679 [23] standard was used to interpret the results. The compressive strength of mortar specimens was determined according to the standard EN 196-1 on $40 \times 40 \times 160$ mm prisms. The strength was determined after 72 h and after 28 days of hardening under standard conditions of constant temperature 20 ± 2 °C and high humidity RH > 95%. Boron leaching test from mineral aggregates was performed to determine the concentration of boron in solutions obtained by shaking mixtures of aggregates (colemanite, ulexite, borax) with solvent (water or saturated Ca(OH)₂ solution) at solid/liquid ratio 1:10. Shaking was carried out at 23 °C for 3, 6 and 24 h. Boron concentration was determined by ICP-MS method using Agilent 7700x from Agilent Technologies, following [24]. Each measurement was repeated six times.

3. Test results

The rate of heat release during cement hydration in mortars with additives is shown in Fig. 1a–f. The content of the additive is indicated as the ratio of boron mass to cement weight. Ulexite

Table 1

Properties of cement CEM I 42.5R (EN 197-1:2011).

Compressive strength	Unit	Value		Requirement
After 28 days	MPa	59.1		≥42.5 ≤62.5
Physical properties		Unit	Value	Requirement
Initial setting time Water to the standard consis Soundness Specific surface area (Bleine Chemical composition	5	min % mm cm²/g Va	172 28.4 0.3 4000 lue	≥60 No requirements ≤10 No requirements Requirement
Loss on ignition Insoluble residue Sulphates as SO ₃ Cl ⁻ Na ₂ O _{eq} (Na ₂ O + 0658 K ₂ O)	%	3.11 0.69 2.81 0.084 0.70		≤5.0 ≤5.0 ≤4.0 ≤0.10 No requirements

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