



## Original Article

# Gamma radiation attenuation, mechanical properties and microstructure of barite-modified cement and geopolymer mortars

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## ABSTRACT

The present study contributes to the development of alternative materials for radiation shielding, focusing on environmental sustainability and material cost efficiency. The primary aim was to evaluate the compressive and flexural strength, mineral composition, microstructure, and gamma-ray attenuation properties of cement mortars and geopolymer mortars containing barite powder. Mortars based on ordinary Portland cement (OPC) and fly ash geopolymers with varying amounts of barite powder were assessed for their shielding properties at energy levels associated with the decay of <sup>137</sup>Cs. From the results, key parameters such as the linear attenuation coefficient ( $\mu$ ), mass attenuation coefficient ( $\mu_m$ ), half-value layer (HVL), and tenth-value layer (TVL) were determined. The results showed that while cement-based composites exhibited superior gamma radiation attenuation compared to fly ash geopolymer mortars, the latter had higher mass attenuation efficiency, meaning less material density was required for the same level of shielding. Additionally, cement mortars had 23–25 % higher mechanical strength than geopolymer mortars. Importantly, the inclusion of barite powder improved the radiation shielding performance of both materials by 7–10 %, demonstrating its effectiveness in enhancing the protective properties of these mortars. This research highlights the potential of fly ash geopolymer mortars as viable, eco-friendly alternatives to traditional cement mortars in radiation shielding applications.

## 1. Introduction

Nuclear materials – including those used in nuclear reactors, radioactive waste, and medical imaging – pose unique challenges due to their ionizing radiation, [1–4]. Effective management and containment are crucial for safety and minimizing environmental and health risks. A key aspect of managing nuclear materials involves effective radiation shielding to protect humans and the environment from gamma radiation, [5,6].

Gamma rays are high-energy electromagnetic waves with significant penetrating power, making them difficult to shield against. They are commonly emitted by radioactive isotopes in nuclear power plants and used in medical imaging. As a result, materials used for shielding in these contexts must be carefully selected for their gamma-ray attenuation capabilities [7,8].

Cement based composites have traditionally been employed for

radiation shielding due to their density, strength, durability and versatility [9,10]. However, their structural integrity and long-term performance can be affected by exposure to gamma radiation [11]. This interaction, particularly in concrete structures exposed to gamma radiation in industrial and research settings, impacts both mechanical and chemical properties [12–15]. Understanding how gamma radiation affects concrete at the atomic and molecular levels is essential for applications such as nuclear power plants, radioactive waste storage, and certain medical facilities. In tandem with increasing environmental concerns, there is growing interest in alternative materials that offer similar or better performance with reduced environmental impact. In 2013, the International Atomic Energy Agency (IAEA) expressed interest in four alternative cementitious materials: geopolymers, magnesium phosphate cements, calcium aluminate cements, and calcium sulfoaluminate cements [3,4]. Despite progress, further work is needed on process optimization, precursor standardization, and durability

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assessments before widespread adoption of these alternative materials.

Geopolymers are a promising alternative to materials like glass [16, 17], modified plastics [18] or red mud [19,20] which currently undergoing intensive study. These inorganic materials are produced via low-temperature polycondensation of aluminosilicate precursors in an highly alkaline solution. They are classified as alkali-activated materials where the binding phase consists of aluminosilicate, forming an amorphous to semicrystalline 3D network of interconnected  $\text{SiO}_4$  and  $\text{AlO}_4$  tetrahedra. This network, resembling a pseudo-zeolite structure, differs from the chain-like calcium silicate hydrates in traditional cements. The aluminosilicate precursors can include calcined materials such as metakaolin, or various waste materials like fly ash from the combustion of coal, rice husk ash, straw ash, bottom ash [21–25], red mud [26,27], or waste red brick powder [28,29]. Using waste materials provides both economic and environmental benefits, making fly ash-based geopolymer an attractive alternative. Alkali activation of fly ash has been extensively studied [30–36], offering economic advantages and providing higher durability and resistance to aggressive acids [37,38], alkali-aggregate reaction [39,40], and high temperature exposure [41–43].

Few studies have examined the effect of high-energy radiation alkali-activated materials or geopolymers. Those that have compared shielding properties between alkali-activated slag and Portland cement found higher shielding capacities and better gamma irradiation resistance in slag-based materials [44–46]. Studies on fly ash-based glasses with barium–borate have reported superior gamma-ray shielding compared to standard concrete [47], while composites with barium sulfate have demonstrated potential to replace lead shielding in X-ray applications [48]. Further research has revealed that low doses of gamma radiation can enhance geopolymerization, though high doses may lead to structural degradation [49–51]. Fly ash-based geopolymers have shown comparable HVL and TVL to Portland cement concrete at low energy levels but were less effective at higher energies [52]. Metakaolin and fly ash geopolymer composites have been reported to exhibit a lower linear attenuation coefficient for gamma irradiation compared to cement composites, but they are more efficient at attenuating thermal neutrons [53]. Ahn et al. demonstrated that metakaolin-based geopolymers can be used to solidify sulfate-rich nuclear waste [54]. Research by Catenacci et al. [55] showed that although fly ash samples from different sources varied in chemical composition, these differences among Class C fly ashes resulted in only marginal changes in the linear attenuation coefficient. Instead, changes in aggregate quantity and composition had a more pronounced impact. Furthermore, gamma rays had negligible effects on the pore structure of the geopolymer, indicating that irradiation does not lead to significant structural changes. However, the dose rate does influence the radiolysis of water and the hydrogen production [56].

This study compares the shielding performance of cement-based and fly ash geopolymer mortars containing barite powder as a partial replacement of quartz sand. Barite, a high-density mineral, increases the mortar's density, which is beneficial for radiation shielding applications. Additionally, barite particles serve as fillers, enhancing the mechanical properties of the mortar. The prepared mixtures were comprehensively tested and analyzed for their microstructure and mechanical properties. The main objective was to evaluate the ability of these materials to attenuate low-energy gamma radiation from  $^{137}\text{Cs}$  gamma-ray source (662 keV). Fly ash geopolymer is a more sustainable, environmentally friendly, and cost-effective material compared to ordinary Portland cement, making it a potential alternative shielding material in non-load-bearing applications where high mechanical strength is not essential. The research provides new and original insights into the comparison of cement mortars and fly ash geopolymer mortars containing barite powder, with a focus on their mechanical properties, microstructure, and gamma-ray attenuation capabilities.

## 2. Materials and methods

### 2.1. Materials

#### 2.1.1. Geopolymer based composites

Geopolymer mortars were prepared using fly ash from the high-temperature combustion of coal at the Dětmarovice Power Plant (CZ). A commercial water glass solution (Vodní sklo, CZ) combined with a 40 % sodium hydroxide solution served as the alkaline activator. The fine aggregate used was standard natural siliceous sand, conforming to EN 196–1 [57], with a specific gravity of  $2.65 \text{ g/cm}^3$  and a maximum grain size of 2 mm. Tap water was also used in the mixture. Barite powder ( $<0.5 \text{ mm}$ , specific gravity  $4.20 \text{ g/cm}^3$ ) was used as a partial replacement for the sand by mass. The specimens were labeled as follows: the reference mix without barite powder was denoted GEO, while those with 10 % and 20 % barite powder were labeled GEO-B-10 and GEO-B-20, respectively. The chemical compositions of the fly ash and water glass are presented in Table 1, while Table 2 shows the chemical composition of barite powder. The compositions of the tested geopolymer mortars are summarized in Table 3. The water-to-fly ash ratio was 0.53, including water from both the alkaline activator mixture and additional mixing water. Four prisms ( $40 \times 40 \times 160 \text{ mm}$ ) were cast for each composition. Three prisms were used to determine the mechanical properties and for microstructural analysis, while one prism was reserved for gamma radiation tests. After casting, the molds were sealed with PE foil to prevent water evaporation and cured for 24 h in an electric furnace at  $60 \text{ }^\circ\text{C}$  to accelerate the setting. Following this, the specimens were placed in a plastic bags and stored under laboratory conditions ( $22 \pm 2 \text{ }^\circ\text{C}$ ) according to standard procedure until testing.

#### 2.1.2. Cement based composites

In all the cement mortar mixes, ordinary Portland cement (CEM I 42.5R) was used along with a fine aggregate - standard natural siliceous sand in accordance with EN 196–1 [57]. The sand had a specific gravity of  $2.65 \text{ g/cm}^3$  and a maximum grain size of 2 mm. Tap water was utilized for mixing. Barite powder served as a substitute for sand, and the same amount of barite was incorporated in both the geopolymer and cement mortar. The specimens were labeled as follows: reference without barite powder (CEM), and those containing 10 % and 20 % barite powder as CEM-B-10, and CEM-B-20, respectively. The chemical composition of cement is provided in Table 4, while Table 5 outlines the composition of cement mortars. A constant water to binder ratio of 0.5 was maintained for all mixes. Four prisms ( $40 \times 40 \times 160 \text{ mm}$ ) were cast for each composition for mechanical and microstructural analysis. All specimens were cured in water at the temperature of  $20 \pm 1 \text{ }^\circ\text{C}$  for 28 days, following the standard procedure before testing.

### 2.2. Methods

#### 2.2.1. Mineral and chemical composition

The mineral composition of fly ash geopolymers and cement mortars was analyzed using X-ray diffraction (XRD). After 28 days of curing, the mortar specimens were dried at  $50 \text{ }^\circ\text{C}$  for three days, crushed into small pieces (1 mm diameter), and ground using a ceramic mortar. The ground material was sieved through a  $0.032 \text{ mm}$  sieve. A Bruker D8 DISCOVER diffractometer with a 40 kV voltage and 40 mA lamp current, equipped with a copper lamp as an X-ray source, was used. Diffraction patterns were collected over a  $2\theta$  range from  $5^\circ$  to  $65^\circ$  with a step size of  $0.02^\circ$  and a 1-s counting time using coupled theta-2theta geometry. The Diffrac.Eva V5.2 software was utilized to analyze the XRD patterns and determine the crystallite phases.

Fourier-Transform Infrared Spectroscopy (FTIR) was employed to analyze the chemical composition of the specimens, prepared similarly to those for XRD analysis. FTIR measurements were conducted with a the Bruker Vertex 70 equipment in ATR mode (Bruker Platinum Diamond ATR stage), with 8 scans averaged at a  $2 \text{ cm}^{-1}$  resolution.

**Table 1**

Chemical composition and LOI of fly ash and water glass in wt.% by XRF.

	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MgO	CaO	SO <sub>3</sub>	Na <sub>2</sub> O	K <sub>2</sub> O	TiO <sub>2</sub>	MnO	P <sub>2</sub> O <sub>5</sub>	LOI
Fly ash	51.67	23.31	7.08	0.36	4.45	0.01	0.77	2.95	1.00	1.14	0.04	5.83
WG <sup>a</sup>	29.53	–	–	–	–	–	16.96	–	–	–	–	–

<sup>a</sup> Content of H<sub>2</sub>O in WG was equal to 53.51 %.**Table 2**

Chemical composition of barite rock and LOI in wt.% by XRF.

SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MgO	CaO	SO <sub>3</sub>	Na <sub>2</sub> O	BaO	TiO <sub>2</sub>	Mn <sub>2</sub> O <sub>3</sub>	SrO	LOI
4.88	0.15	0.26	0.14	2.39	30.23	0.04	59.26	0.10	0.06	1.43	1.10

**Table 3**

The mixture proportions of geopolymer mortars.

Mix ID	Fly ash (g)	WG (g)	40 % NaOH	Quartz sand (g)	Baryte (g)	Water (ml)
GEO	350	253	27	1050	–	30
GEO-B-10	350	253	27	940	175	30
GEO-B-20	350	253	27	830	350	30

Background scans were performed before sample scan to account for humidity and carbon dioxide fluctuations in the air. All scans were conducted under ambient conditions (~25 °C, 50 % relative humidity).

### 2.2.2. Density and mechanical properties

After 28 days of curing, density was determined by measuring the dimensions and weight of the specimens. Flexural and compressive strength tests followed the procedures outlined in EN 196-1 [57], three-point loading with standardized 40 × 40 × 160 mm beams. The EZ testing machine (50 kN load capacity, loading rate of 50 N/s) was used to determine flexural strength, while a 2000 kN Controls testing machine, applying a constant loading rate of 500 N/s, was employed to determine compressive strength. Each test was completed when the specimens failed and split into two parts, resulting in three flexural strength values and four compressive strength values per mortar type. The remaining halves of the mortar beams were retained for further testing.

### 2.2.3. Microstructure

The microstructure of both fly ash geopolymer and cement mortar specimens was examined using a JEOL JSM-6460 LV high-vacuum scanning electron microscope (SEM). Fresh split surfaces were analyzed using secondary electrons (SE), while polished surfaces were studied with back-scattered electrons (BSE). Specimens were carbon-coated, and conductive tape was applied to ensure conductivity. A voltage of 15 kV and an aperture of 120 μm were applied, maintaining a working distance of 9–11 mm. Observations were conducted at magnification ranging from 30 × to 300 ×.

Nitrogen adsorption/desorption experiments (BET analysis) were conducted after 28 days of curing to analyze the pore structure. Specimens were prepared as per the XRD procedure, and measurements followed the Barret-Joyner-Halenda (BJH) method using a Quantachrome Autosorb IQ analyzer. The samples were degassed at 105 °C for 2 h to remove moisture and gas contaminants, in accordance with the standard

**Table 4**

Chemical composition and LOI of cement CEM I 42.5R in wt.% by XRF.

SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MgO	CaO	SO <sub>3</sub>	Na <sub>2</sub> O	K <sub>2</sub> O	TiO <sub>2</sub>	MnO	P <sub>2</sub> O <sub>5</sub>	LOI
19.26	4.72	3.21	2.88	61.86	3.12	0.22	0.93	0.24	0.09	0.13	3.10

for this characterization. Compared to the long-term vacuum drying process at low temperature, the short-term degassing process is more suitable for the pretreatment of the samples to be analyzed in this technique [58,59]. The desorption isotherms as well as the pore size distribution and the cumulative volume of pores registered from 1 nm to 100 nm were obtained as test results.

### 2.2.4. Gamma-ray attenuation parameters

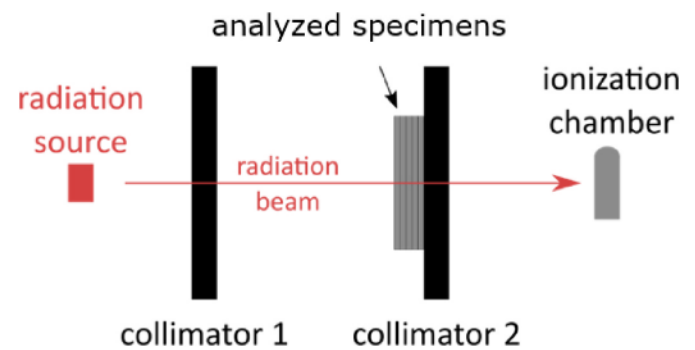
To estimate the gamma-ray attenuation parameters, a test stand was prepared according to the EN 61331-1 standard [60,61] (Fig. 1). The tests were conducted in an accredited calibration laboratory (accreditation No. AP 070 issued by the Polish Center for Accreditation). Based on the previous investigation on radiation shielding of concrete slabs [61], measurements were performed using a Tema Sinergie IM6M automatic irradiator with <sup>137</sup>Cs gamma-ray reference source and a G5 type graphite ionization chamber [62]. The active center of the detector was set at a distance of 85 cm from the source, at a point with an air kerma rate of  $\dot{K}_{air} = 30.4 \pm 1.5$  mGy h<sup>-1</sup>.

Based on dosimetric measurements, the linear attenuation coefficient ( $\mu$ ), the half-value layer (HVL), and the tenth-value layer (TVL) were determined. The linear attenuation coefficient was obtained as a

**Table 5**

The mixture proportions of cement mortars.

Mix ID	Cement [g]	Water [ml]	Quartz sand [g]	Barite [g]
CEM	450	225	1350	–
CEM-B-10	450	225	1245	175
CEM-B-20	450	225	1140	350

**Fig. 1.** Schematic view of the gamma-ray attenuation test stand.

curve-fitting parameter of the attenuation rate ( $k$ ) dependent on the thickness of the shielding layer ( $d$ ). The attenuation rate ( $k$ ) is defined as the ratio of the ionization current of the ionization chamber without a shield  $i_0$  to the value of the ionization current behind a shield of thickness  $d$ ,  $i(d)$ :

$$k(d) = \frac{i_0}{i(d)} \quad (1)$$

$$k(d) = e^{\mu d} \quad (2)$$

The HVL and TVL were calculated from  $\mu$ :

$$HVL = \frac{\ln(2)}{\mu} \quad (3)$$

$$TVL = \frac{\ln(10)}{\mu} \quad (4)$$

The mass attenuation coefficient, which characterizes how easily a material can be penetrated by gamma radiation, was calculated as the attenuation coefficient normalized to density:

$$\mu_m = \frac{\mu}{\rho} \quad (5)$$

The study of radiation attenuation by concrete specimens was carried out for five shielding layer thicknesses. The relative uncertainty in determining the attenuation rate  $k$  for all settings did not exceed 5%. The uncertainty  $\mu$  ( $u_\mu$ ) was determined as the uncertainty of curve fitting by the least squares method. The uncertainties of HVL and TVL were determined from the uncertainty of  $u_\mu$  as follows:

$$u_{HVL} = HVL \cdot \frac{u_\mu}{\mu} \quad (6)$$

$$u_{TVL} = TVL \cdot \frac{u_\mu}{\mu} \quad (7)$$

After 28 days, previously cast specimens of  $40 \times 40 \times 160$  mm were cross-cut into 9 mm thick slices. Five consecutive slices were placed between collimators 1 and 2 (Fig. 1) and irradiated.

### 3. Results and discussion

#### 3.1. Mineral and chemical composition

The X-ray diffraction (XRD) mineral analysis, shown in Figs. 2 and 3, indicates the presence of crystalline phases in the analyzed specimens, derived from the used starting materials used. In geopolymer specimens, quartz, mullite, and hematite were identified, while in cement mortars, quartz and portlandite were detected. As seen in Fig. 2, quartz and mullite were the major crystalline phases, while hematite was a minor in the reference geopolymer specimen. These phases originated from the fly ash, which contained crystalline material without pozzolanic reactivity [63]. However, much of the matter in both geopolymer and cement binder is amorphous and not observable via XRD. New characteristic peaks were observed in the XRD patterns of modified mortars, corresponding to the partial replacement of quartz sand with barite powder, consistent with previously published data [64,65]. The intensity of these peaks increased with barite content, Figs. 2 and 3.

The chemical composition of the specimens, determined by Fourier-Transform Infrared Spectroscopy (FTIR), is shown in Figs. 4 and 5. To facilitate comparisons, data for both barite and the analyzed mortars were included. Upon analyzing the FTIR spectral profile of the reference cement mortar (CEM), the following features were identified:

- A strong band at  $452 \text{ cm}^{-1}$  with a shoulder at  $513 \text{ cm}^{-1}$ , associated with in-plane and out-of-plane Si-O vibrations.
- A broadband in the  $800\text{--}1250 \text{ cm}^{-1}$  range, centered at  $1035 \text{ cm}^{-1}$  with a shoulder at  $1163 \text{ cm}^{-1}$ , attributed to the stretching mode of Si-O bond  $\text{SiO}_4$  tetrahedra.
- Bands at  $796$ ,  $777$ , and  $694 \text{ cm}^{-1}$  attributed to the symmetrical stretching and bending Si-O vibrations {Saikia, 2014 #117}.
- Bands at  $1472$ ,  $1419$ , and  $876 \text{ cm}^{-1}$  corresponding to carbonate species.
- Broadband in the  $3100\text{--}3600 \text{ cm}^{-1}$  range, along with a weak band at  $1647 \text{ cm}^{-1}$ , attributed to the OH stretching and bending modes in water associated with the cement matrix [66].
- The presence of a peak related to the OH stretching vibrations of portlandite at  $3643 \text{ cm}^{-1}$  [66].

In the FTIR spectra of the reference geopolymer mortar, the following features were identified:

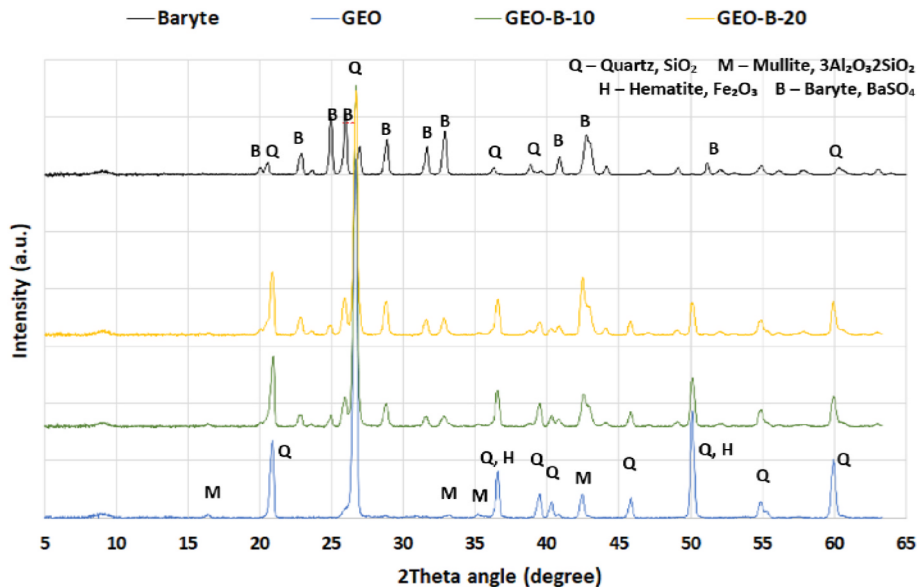


Fig. 2. XRD diffractogram for the fly ash geopolymer mortars.

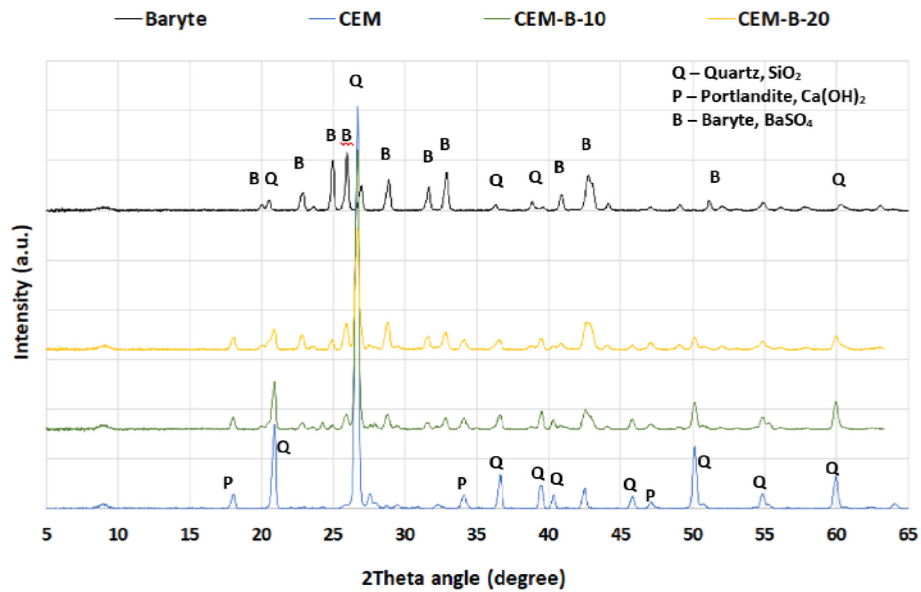


Fig. 3. XRD diffractogram for the cement mortars.

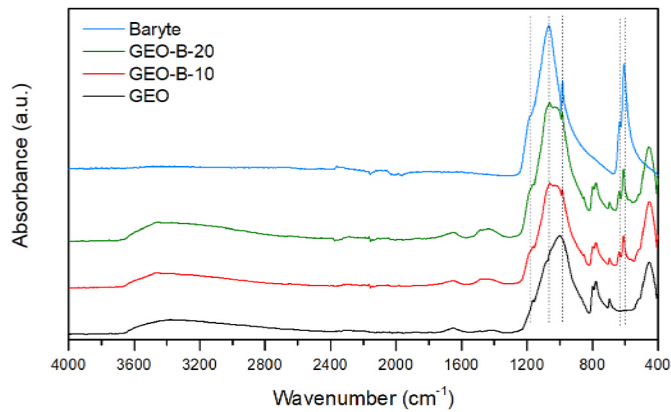


Fig. 4. FTIR spectra for the fly ash geopolymer based mortars.

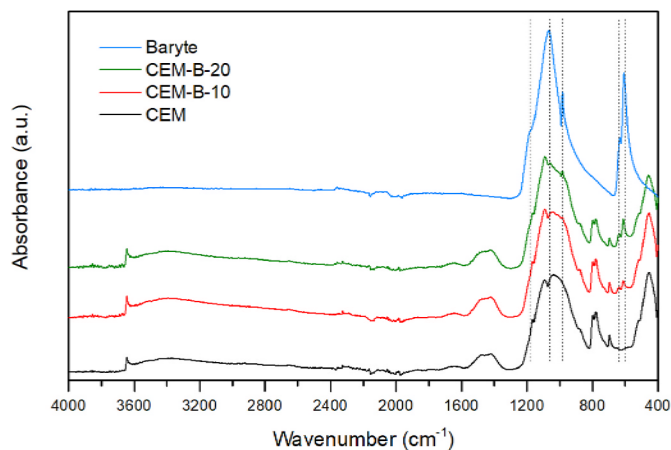


Fig. 5. FTIR spectra for the cement based mortars.

- A band at  $452\text{ cm}^{-1}$  with a shoulder at  $513\text{ cm}^{-1}$ , associated with in-plane and out-of-plane Si-O vibrations.
- Bands at  $796$ ,  $777$ , and  $694\text{ cm}^{-1}$  assigned to the symmetrical stretching and bending Si-O vibrations.

- Broadband in the  $800\text{--}1250\text{ cm}^{-1}$  range, centered at  $1000\text{ cm}^{-1}$  with shoulders at  $1163$  and  $1080\text{ cm}^{-1}$ , attributed to the asymmetrical stretching mode of Si-O and Al-O bonds.
- Broad band at  $1412\text{ cm}^{-1}$  that corresponds to the carbonates.
- Weak broad bands centered at  $3360$  and  $1648\text{ cm}^{-1}$  attributed to the OH stretching and bending modes, respectively, in water associated with the geopolymer matrix.

The bands at  $1163$ ,  $1090$ ,  $796$ ,  $777$ ,  $694$ ,  $513$ , and  $452\text{ cm}^{-1}$  are associated with quartz, present as aggregate in both types of mortars [67,68]. The presence of barite in the mortars (CEM-B-10, CEM-B-20, GEO-B-10, and GEO-B-20) is confirmed by bands at  $1058$ ,  $982$ ,  $635$ , and  $608\text{ cm}^{-1}$ . The former two bands are linked to asymmetrical and symmetrical stretching vibrations of  $\text{SO}_4^{2-}$  [69], while the latter two are attributed to the out-of-plane bending vibration of  $\text{SO}_4^{2-}$  group [69]. In fly ash geopolymer, the broad peak centered around  $1000\text{ cm}^{-1}$  can be assigned to Si-O-T (T = Si or Al) asymmetric stretching vibrations in the geopolymer's 3D aluminosilicate structure [70–72]. The shift of the peak from  $1000\text{ cm}^{-1}$  to approximately  $1061\text{ cm}^{-1}$  is attributed to the addition of barite. In the case of cement-based mortars, the strong band at  $1035\text{ cm}^{-1}$  with a shoulder at  $1046\text{ cm}^{-1}$ , is associated with asymmetric stretching vibrations in C-S-H gels. However, the slight shift towards a higher wavenumber compared to literature data [66,73,74] may result from a higher condensation degree of  $\text{SiO}_4$  tetrahedra in the C-S-H phase. It can be concluded that no influence the molecular structure of fly ash geopolymer or cement matrix, as there are no significant differences in the FTIR spectra between reference mortars and mortars containing 10 % and 20 % barite powder, aside from the increased intensity of signals associated with barite.

### 3.2. Density and mechanical properties

The density of the tested specimens is presented in Table 6 and mechanical properties determined after 28 days of curing are presented in Table 7. Geopolymer mortars exhibited lower density compared to cement mortars. The influence of barite powder on mortar density is clearly visible: as the barite content increases, the mortar density also increases, which was expected for both types of mortars [75,76]. The density of the geopolymer mortar with 10 % barite content was approximately equal to the density of the reference cement mortar. However, the density of the geopolymer mortar with 20 % barite content was comparable to the cement mortar with 10 % barite.



**Table 6**

Density of the fly ash geopolymers and cement mortars, after 28 days of curing.

Specimen description	Density (g cm <sup>-3</sup> )
GEO	2.02 ± 0.03
GEO-B-10	2.16 ± 0.01
GEO-B-20	2.30 ± 0.03
CEM	2.23 ± 0.01
CEM-B-10	2.32 ± 0.01
CEM-B-20	2.44 ± 0.02

**Table 7**

Mechanical properties of tested fly ash geopolymers and cement mortars.

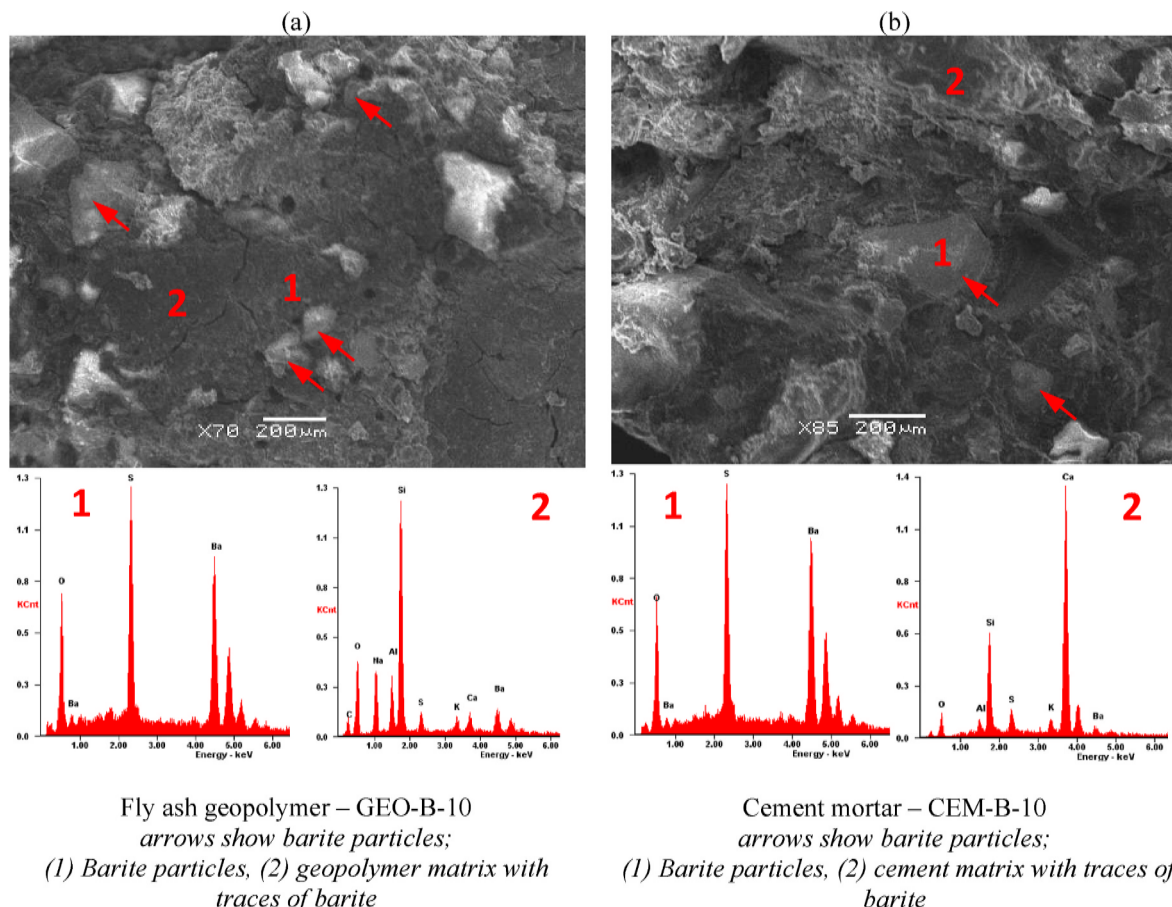
Specimen description	Compressive strength	Flexural strength
	MPa	
GEO	35.3 ± 0.2	5.9 ± 0.1
GEO-B-10	35.7 ± 0.5	6.5 ± 0.1
GEO-B-20	36.5 ± 0.3	6.4 ± 0.1
CEM	47.3 ± 0.5	7.9 ± 0.2
CEM-B-10	48.0 ± 0.3	8.1 ± 0.1
CEM-B-20	48.5 ± 0.4	8.3 ± 0.1

Geopolymer mortar specimens exhibited approximately 25 % lower compressive and flexural strength compared to cement mortar specimens. The compressive strength of the reference cement mortar was 47.3 MPa, while the reference geopolymer mortar achieved 35.2 MPa. Similarly, the flexural strength values were 7.9 MPa and 5.9 MPa, respectively. Replacing part of the fine quartz sand with barite powder resulted in a slight increase in strength, which can be attributed to

improved compaction of aggregate particles and densification of the mortar structure. Similar results were reported by Saidani et al. [75], who demonstrated that a minor substitution of sand (5 % by volume) with barite powder (<0.100 mm) did not adversely affect compressive strength. Conversely, Gökçe et al. [77] reported that a higher substitution of fine aggregate with barite (≥50 %) led to a decline in mechanical properties, with a more pronounced negative effect on flexural strength than on compressive strength. However, in this study, no such detrimental effects were observed.

### 3.3. Microstructure

The microstructure of the reference specimens, including the hardened geopolymer produced through the alkaline activation of fly ash and the hardened cement mortar (both with and without barite powder), was examined using scanning electron microscopy (SEM). Figs. 6 and 7 present SEM images that illustrate the microstructure of the fractured and polished surfaces of the specimens after a 28-day storage duration under air-dry conditions. Evenly distributed angular grains of barite are visible in the micrographs. The SEM image (Fig. 7) reveals incompletely reacted fly ash particles, alongside various products of geopolymerization. The geopolymer matrix results from the reaction between the amorphous components of fly ash and an alkaline activator, with some unreacted fly ash particles still visible. The binding material formed through the alkaline activation of fly ash is observed between the barite particles and sand grains, forming a compact structure bonded to the grain surface (Fig. 7). This material mainly consists of silicon, aluminum, and sodium (Fig. 6). The SEM micrographs also show the presence of microcracks in geopolymer mortar, which likely contribute to the lower flexural strengths by acting as stress concentrators. In



**Fig. 6.** The microstructure of a: a) geopolymers mortars and b) cement mortars containing barite powder observed on split surfaces using SEM.

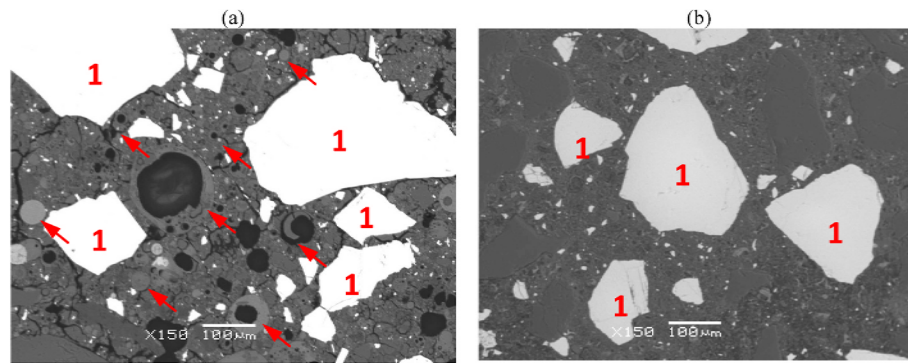


Fig. 7. The microstructure of a) geopolymer mortar GEO-B-20, b) cement mortar CEM-B-20 observed on polished surfaces using SEM, 1- barite particles, arrows show fly ash grains.

contrast, the cement specimens exhibit a compact and dense structure, with C-S-H gels filling the spaces between aggregate particles.

SEM analysis further demonstrated the even distribution of barite particles in both the cement and geopolymer matrices. This uniform distribution of barite is significant, as aggregate distribution in concrete affects its gamma-ray attenuation properties [78]. A homogeneous spread of barite particles reduces the paths for gamma radiation, thereby enhancing the material's ability to attenuate and resist the harmful effects of gamma radiation. In the SEM micrographs of geopolymer specimens, circular and smooth fly ash grains are visible, indicating that the alkaline reaction primarily occurs on the surface layer of the fly ash. Similar observations were reported by Rajczyk and Janus [63] on the fracture surfaces of geopolymer specimens with fly ash.

The comprehensive results of the nitrogen adsorption and desorption measurements are provided in Table 8 and visually presented in Figs. 8–10. A monotonic increase in nitrogen adsorption was observed in both reference mortars across relative pressures of 0.1–0.9, followed by a rapid increase at relative pressures between 0.9 and 1.0 (Fig. 8). However, the amount of nitrogen adsorbed by the geopolymer mortar was only half of that observed for the cement mortar. The addition of barite increased nitrogen adsorption in both mortars, although this increase was more pronounced in geopolymer mortars at low relative pressures and in cement mortars at high pressures.

The surface area and pore volume of the reference geopolymer were slightly lower than those of the cement mortar. However, when part of the sand was substituted with barite powder, the surface area, cumulative pore volume, and pore size increased in geopolymer mixes. The greater the barite content, the larger the surface area and the cumulative pore volume. In contrast, the surface area and pore volume of cement-based mortars modified with barite powder remained nearly equal to those of the reference mortar, suggesting that substituting quartz with barite does not significantly affect the microstructure of cement mortar.

Pores smaller than 10 nm are generally classified as gel pores, while

those those larger than 10 nm are described as capillary pores [59]. The pore size distribution curves for the geopolymer mortars showed unimodal profiles ranging from 1 to 100 nm, with a primary peak centered around 8 nm, indicating the dominance of gel pores. In cement mortars, however, the primary peak was around 40 nm, suggesting that capillary pores were predominant [71].

Variations in the distribution of gel pores in geopolymer mortars may serve as an indicator of the reaction degree [79]. Geopolymers containing barite powder exhibited the highest content of gel pores, which could imply a slightly lower reaction degree compared to the reference mortar. However, upon examining the SEM microphotographs, it appears that a high porosity observed in geopolymers is largely due to microcracks and large voids left by fly ash cenospheres, which are not detectable via BET analysis. This explains the lower flexural and compressive strengths of fly ash geopolymer mortars. Nonetheless, the increasing presence of gel pores resulting from the addition of barite does not appear to negatively impact the mechanical properties of geopolymer mortar.

#### 3.4. Gamma-ray attenuation parameters

The gamma-ray attenuation parameters for the five-layer specimens, including the linear attenuation coefficient ( $\mu$ ), tenth-value layer (TVL), and the half-value layer (HVL), are presented in Table 9. The results indicate that the cement reference mortar, containing 100 % normal-density sand, has a higher linear attenuation coefficient and lower TVL and HVL compared to the geopolymer reference mortar with the same type of sand. For both geopolymer and cement-based mortars, the influence of barite is noticeable. As the barite content increases, there is a clear rise in  $\mu$  and a corresponding decrease in HVL and TVL. This effect is slightly more pronounced in the cement mortars. The increase in the linear absorption coefficient from the reference CEM to the CEM-B-20 cement mortars was 10 %, while the corresponding increase between GEO and GEO-B-20 geopolymer mortars was 7 %. Similarly, the decrease in the tenth-value layer was 9 % for cement mortars and 6 % for geopolymer mortars.

The mass attenuation coefficient, which normalized the attenuation coefficient by the material's density, exhibits the opposite trend. While the values for cement-based mortars remained relatively unchanged as barite content increased, they decreased by 6 % between the GEO and GEO-B-20 geopolymer mixtures. Nonetheless, the mass attenuation coefficient of the geopolymer was higher than of reference cement mortar, implying that geopolymer exhibits better shielding properties for materials of the same density compared to cement-based mortars.

The results of gamma radiation attenuation from a  $^{137}\text{Cs}$  source clearly demonstrate differences between geopolymer and cement-based mortars, as well as the influence of barite filler. Geopolymer mortars exhibited a lower linear attenuation coefficient and higher TVL and HVL compared to cement-based mortars, yet a higher mass attenuation

Table 8

The BET surface area and BJH pore volume of geopolymer and cement based mortars modified by barite powder.

Specimen	Surface area ( $\text{m}^2 \text{g}^{-1}$ )	Pore volume ( $\text{cm}^3 \text{g}^{-1}$ )	Gel pores (<10 nm) (%)	Other mesopores and macropores (%)
GEO	4.99	0.067	63.48	36.52
GEO-B-10	6.53	0.085	58.86	41.14
GEO-B-20	8.59	0.102	60.16	39.84
CEM	6.77	0.097	52.72	47.28
CEM-B-10	8.03	0.104	46.91	53.09
CEM-B-20	7.68	0.102	44.22	55.78

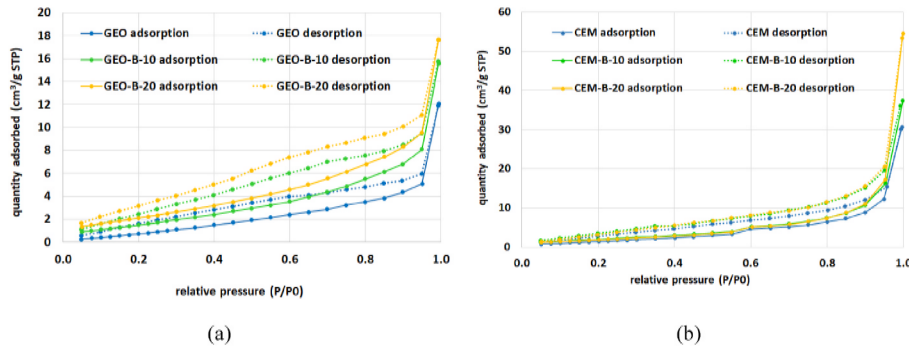


Fig. 8. Nitrogen adsorption and desorption isotherm characteristics of hardened: (a) fly ash geopolymer and (b) cement mortar.

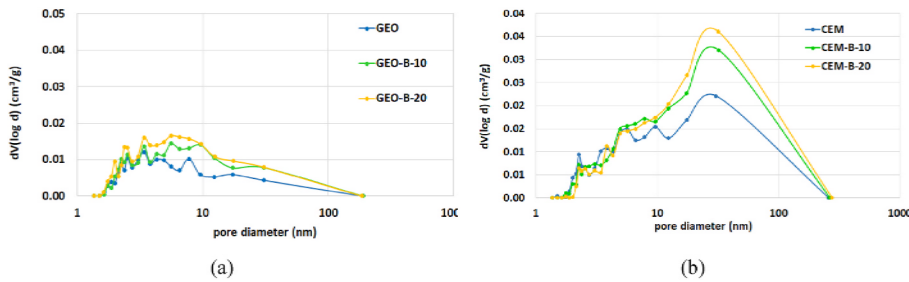


Fig. 9. BJH results of hardened: (a) fly ash geopolymer and (b) cement mortar; pore characteristics –  $dV(\log d)$ .

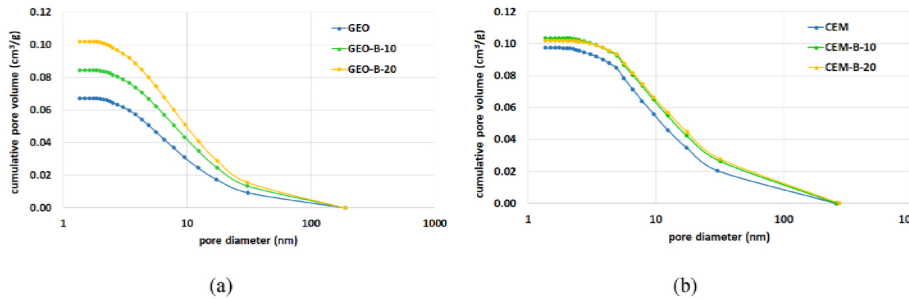


Fig. 10. BJH results of hardened: (a) fly ash geopolymer and (b) cement mortar; pore characteristics – cumulative pore volume.

**Table 9**  
The results of the attenuation of  $^{137}\text{Cs}$  gamma radiation for analyzed specimens.

Specimen description	Linear attenuation coefficient	Uncertainty of $\mu$	Mass attenuation coefficient	Half-value layer	Tenth-value layer
	$\mu$ ( $\text{cm}^{-1}$ )	$\mu_\mu$ ( $\text{cm}^{-1}$ )	$\mu_m$ ( $\text{cm}^2 \text{g}^{-1}$ )	HVL (cm)	TVL (cm)
CEM	0.1746	0.0008	0.0783	3.97	13.19
CEM-B-10	0.1840	0.0005	0.0793	3.77	12.51
CEM-B-20	0.1920	0.0013	0.0787	3.61	11.99
GEO	0.1695	0.0003	0.0839	4.09	13.58
GEO-B-10	0.1752	0.0013	0.0811	3.96	13.14
GEO-B-20	0.1812	0.0015	0.0789	3.83	12.71

coefficient. Hassan et al. [52] analyzed concrete and geopolymer made with blast furnace slag or fly ash and found that slag-based geopolymer achieved the highest linear and mass attenuation coefficient (0.1642  $\text{cm}^{-1}$ ; 0.782  $\text{cm}^2 \text{g}^{-1}$ , respectively) compared to fly ash-based geopolymer (0.1387  $\text{cm}^{-1}$ ; 0.761  $\text{cm}^2 \text{g}^{-1}$ ) and ordinary concrete (0.1380  $\text{cm}^{-1}$ ; 0.730  $\text{cm}^2 \text{g}^{-1}$ ) when subjected to  $^{137}\text{Cs}$  gamma irradiation. Similarly, Catenacci et al. [55] reported that the linear attenuation coefficient of mortars exposed to  $^{60}\text{Co}$  gamma irradiation decreased in the

following order: slag-based geopolymer, fly ash-based geopolymer, and cement-based mortar. In the conducted research, higher values for both the linear and mass attenuation coefficients were obtained for the reference cement mortar (0.1746  $\text{cm}^{-1}$ ; 0.783  $\text{cm}^2 \text{g}^{-1}$ ) and the reference fly ash geopolymer (0.1695  $\text{cm}^{-1}$ ; 0.839  $\text{cm}^2 \text{g}^{-1}$ ).

The relationship between  $\mu$  and density in the analyzed mortars is shown in Fig. 11, alongside literature data for cement and fly ash geopolymer mortars [55], fly ash geopolymer and cementitious concretes containing barite [80], and ordinary concretes [81]. Fig. 11 illustrates that for both geopolymer-based and cement-based mortars, there is a direct relationship between density and  $\mu$ . Specifically, as the density of the mortar increases, the linear absorption coefficient also increases. This is consistent with theoretical expectations because denser materials contain more mass per unit volume, which enhances their ability to absorb radiation. The data aligns well the trend reported by NIST for the ordinary concrete, which reinforces the reliability of the observed relationship between density and the linear absorption coefficient in the tested materials.

Fig. 12 illustrates the relationship between the half-value layer and the density of geopolymer and cement-based mortars. This figure is key for understanding how effective these materials are at attenuating radiation, particularly in terms of the HVL, which is a crucial parameter



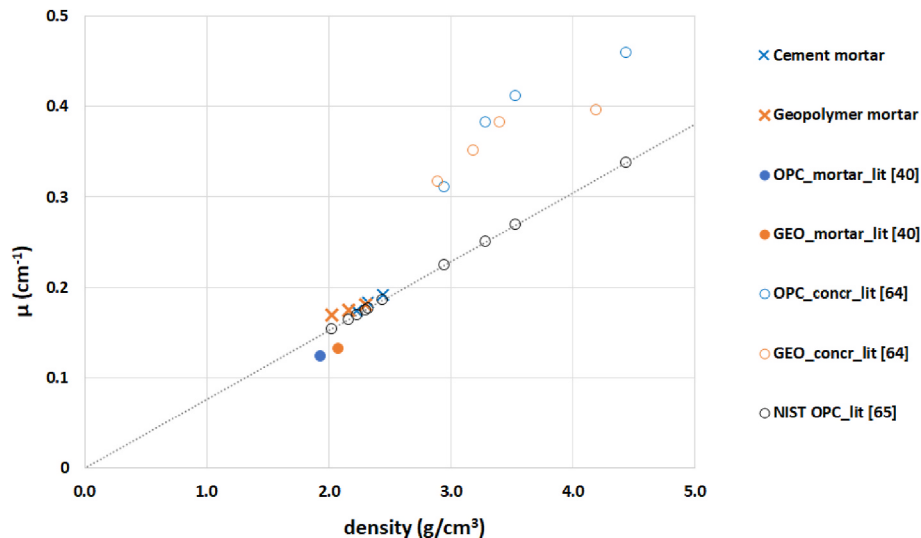


Fig. 11. The relationship between linear absorption coefficient  $\mu$  and density of the geopolymer and cement based mortars compared to literature data; OPC – ordinary Portland concrete.

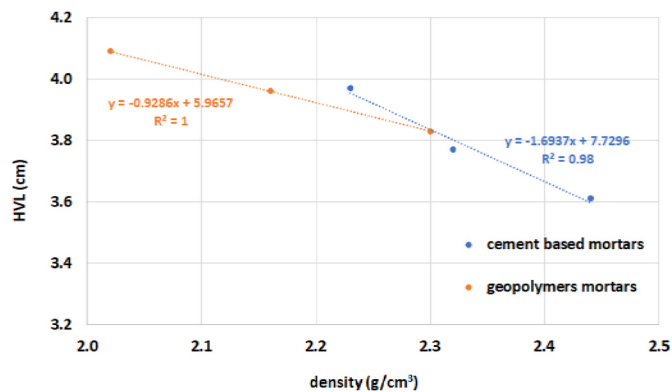


Fig. 12. The relationship between the half-value layer and density of the geopolymer and cement based mortars.

for radiation shielding applications. As shown in Fig. 12, there is a clear relationship between HVL and density for both geopolymer and cement-based mortars. This relationship aligns with the general principle that denser materials tend to have lower HVL values because they provide more mass for radiation to interact with, resulting in more effective attenuation. As known, with increasing concrete density, HVL and TVL values decrease [72]. The results in Fig. 12 demonstrate that the HVL of both geopolymer and cement-based composites is comparable, falling within a range of 3.6–4.1 cm. This indicates that both types of materials – geopolymers and traditional cement-based mortars – perform similarly when it comes to radiation shielding. However, the critical point is that fly ash-based geopolymers achieve these HVL values with lower material densities compared to traditional cement-based mortars. This suggests that geopolymer mortars, particularly those made with fly ash, are more efficient in terms of radiation attenuation relative to their density. In other words, less material mass is required to achieve the same level of radiation shielding as cement-based mortars.

#### 4. Conclusions

The present study reports a comprehensive dataset on the mineralogical (XRD), structural (FTIR), microstructural (SEM), and mechanical properties of cement-based and fly ash geopolymer mortars with varying amounts of barite powder as a partial replacement for quartz sand. All

mixtures were exposed to gamma irradiation from a  $^{137}\text{Cs}$  source.

Cement mortars exhibited 23–25 % higher compressive and flexural strengths compared to fly ash geopolymer mortars. This difference is likely due to the well-established hydration reactions in cement, which form stronger C-S-H gel, a known contributor to mechanical strength. However, both mortar types saw an improvement in mechanical properties when part of the sand was replaced with barite powder, indicating that barite's high density positively influences the structural integrity, particularly in geopolymer mortars.

The pore structure varied significantly between the two mortar types. Cement-based mortars had a 30 % higher surface area and cumulative pore volume compared to geopolymer mortars, with capillary pores dominating the structure. In contrast, the fly ash geopolymer mortars had a greater proportion of gel pores, which contributed to their lower overall porosity. The addition of barite powder had little effect on the cement mortars but significantly increased the volume of gel pores in the geopolymer mortars. These findings align with the scientific understanding that geopolymer gels tend to form fewer, smaller pores compared to the capillary pore structure typical of cementitious binders.

Microstructural analysis revealed that the addition of barite powder had minimal impact on the chemical reaction products in both types of binders, but it slightly enhanced pore refinement in geopolymers. Despite this, geopolymers were more prone to crack formation, particularly under flexural stress, which reduced their flexural strength. This behavior can be explained by the brittleness of geopolymer networks compared to cementitious matrices, which are more resilient under tensile or bending loads.

Cement mortars with 100 % normal-density sand had a higher linear attenuation coefficient ( $\mu$ ) and correspondingly lower HVL and TVL values compared to geopolymer mortars. This suggests that the higher density of cement-based materials provides better shielding efficiency against gamma rays. However, geopolymers exhibited a higher mass attenuation coefficient, meaning they could achieve comparable gamma-ray attenuation at lower densities, which underscores their potential as lightweight radiation shielding materials. This observation is supported by the scientific theory that geopolymer materials, with their unique chemical composition, can offer effective attenuation despite their lower density compared to cement.

Regardless of the mortar composition, adding barite powder improved the gamma-ray shielding properties by 7–10 %. This enhancement can be attributed to barite's high atomic number and density, which increase the material's overall ability to absorb radiation. Barite acts as an effective filler for increasing the density of the material,

thus reducing both the HVL and TVL values, which is consistent with theoretical expectations in radiation physics.

These findings demonstrate that while traditional cement mortars offer higher mechanical strength, fly ash geopolymers, particularly when modified with barite powder, present a viable alternative for applications requiring sustainable materials with efficient radiation shielding capabilities. This combination of properties makes geopolymer mortars especially promising for specialized construction applications, such as in radiation-sensitive environments.

Future research could explore how different special aggregates can enhance gamma and neutron shielding properties in geopolymer-based materials without compromising mechanical performance, while also ensuring long-term durability, particularly in relation to long-lived radioisotopes.

#### CRediT authorship contribution statement

**Daria Józwiak-Niedźwiedzka:** Writing – review & editing, Writing – original draft, Validation, Supervision, Methodology, Investigation, Formal analysis, Conceptualization. **Pavel Rovnaník:** Writing – review & editing, Writing – original draft, Validation, Resources, Investigation, Formal analysis, Conceptualization. **Mariusz Dąbrowski:** Writing – review & editing, Investigation. **Jakub Oško:** Writing – review & editing, Writing – original draft, Investigation, Formal analysis. **Michał Kuć:** Writing – review & editing, Investigation. **Maciej Maciak:** Writing – review & editing, Investigation.

#### Data availability

Original data are available on the request.

#### Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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