ЗБОРНИК РАДОВА

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Друштва за заштиту од зрачења
Србије и Црне Горе

04-06. октобар 2023. године

Будва, Црна Гора

ДРУШТВО ЗА ЗАШТИТУ ОД ЗРАЧЕЊА
СРБИЈЕ И ЦРНЕ ГОРЕ

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XXXII СИМПОЗИЈУМ ДЗЗСЦГ

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Овај Зборник је збирка радова саопштених на XXXII Симпозијуму Друштва за заштиту од зрачења Србије и Црне Горе који је одржан у Будви, Црна Гора, 04-06.10.2023. године. Радови су према обрађеној проблематици груписани у једанаест секција. Сви радови у Зборнику су рецензирани од стране Научног одбора, а за све приказане резултате и тврдње одговорни су сами аутори.

Југословенско друштво за заштиту од зрачења основано је 1963. године у Порторожу, а од 2005. носи име "Друштво за заштиту од зрачења Србије и Црне Горе". На XXXII Симпозијуму, ове године обележавамо веома значајан јубилеј - ***60 година организоване заштите од зрачења на нашим просторима****.*

Од оснивања, Симпозијуми Друштва за заштиту од зрачења представљају прилику да се кроз стручни програм прикажу резултати истраживања у области заштите од зрачења, представе различите области примене извора и генератора зрачења, анализирају актуелна дешавања, размене искуства са колегама из региона, дефинишу проблеми и правци даљег унапређивања наше професионалне заједнице.

Поред тога, Симпозијуми друштва представљају и прилику да у мање формалном маниру сретнемо старе и упознамо нове пријатеље и колеге, обновимо старе и започнемо нове професионалне сарадње.

Ауторима и коауторима научних и стручних радова саопштеним на XXXII Симпозијуму се захваљујемо на уложеном труду и настојању да квалитетним радовима заједно допринесемо остваривању циљева и задатака Друштва и наставимо традицију дугу импозантних 60 година.

Посебно се захваљујемо свима који су подржали одржавање овог Симпозијума.

Свим члановима Друштва, сарадницима и колегама честитамо овај значајан јубилеј!

Организациони одбор XXXII Симпозијума ДЗЗСЦГ

RADIOLOGICAL CHARACTERIZATION OF ALKALI ACTIVATED MATERIALS CONTAINING WOOD AND FLY ASH

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ABSTRACT

Improperly storage of fly ash as industrial wastes has been a cause of land contamination issues. These wastes or by-products have the potential to be used as secondary raw materials in construction, promoting the concept of a circular economy that will avoid land contamination. Here we evaluate radiological environmental impacts when wastes that contain elevated levels of naturally occurring radionuclides (NORs) such as fly ash and wood ash are made into 'green cements' such as geopolymers or alkali-activated materials (AAMs). Alkali-activated materials were formed by alkali-activation reaction of wood and fly ash, as a solid precours, and alkali activator solution, sodium hydroxide and sodium silicate. Three differente concentration of alkali solution were used. Determination of radionuclide content was performed by means of gamma-ray spectrometry. The external absorbed gamma dose rate was 68.6-98.1 nGy/h, and external radiation hazard index for alkali-activated material AAM4, AAM6 and AAM12 were 0.544 Bq/kg, 0.575 Bq/kg and 0.403 Bq/kg, respectively. The results of activity concentration measurements in alkali-activated materials indicate potential of their safe application in building constructions. In terms of some the structural characterizations the obtained alkali activated materials were examined.

Introduction

The production of waste is daily increasing with faster industrial development making environmental control a very important task. Alternative materials which could replace tradicional cements and reduced the environmental pollution, including CO_2 emission, are alkali-activated materials (AAMs) [1, 2]. AAMs are a new class of alumina-silicate materials which are very attractive owing to their excellent mechanical properties, durability and thermal stability. In addition, they are of great interest because of the reduced energy requirement for their manufacture. They poses low thermal conductivity, good mechanical streangth, durability, fire resistance, and the high sustainability [4, 5].

Raw materials, precursors, for alkali-activated materials are solid waste materials, such as fly ash (FA) and wood ash (WA), which are actually by-products of burnig process of coal and wood, respectively. Deposing of these kind of waste materials can caused environmental problems, especially desposing fly ash [3]. Synthesis of AAMs from a solid powder precursors imply dissolving precursors in alkaline solutions. According our previous research

[6] the chemical composition of wood ash shown the high percent of CaO. Incorporation of different forms of calcium during synthesis of alkali-activated materials, will cause the different reaction pathway and increasing sample strengths and thus gives possibilities of application AAMs in construction sectors [7].

Coal mainly contains higher amount of natural radionuclides, and in the burnig process in power plants produce the ash, which represents a risk to the environmental and health for population [8]. According to Mladenović Nikolić et al. [6] using wood ash in production of alkali-activated materials as cementations materials for application in the construction industry, the value of radium equivalent activity for samples which contains wood ash are lower than the recommended maximum level (370 Bq/kg), providing an excess annual effective dose less than 1 mSv, and its use of this is considered safe for the population. Ignjatiović et al. [9] showed in their research the natural radionuclide content, for all concrete samples, were lower than recommended limit. They concluded the fly ashes from Serbia power plants does not require any restrictions on the production from a radiological point of view.

So far, it has been shown in our earlier studies that the alkaline activation of alumino-silicate precursors affects the reduction of radionuclide activity compared to the precursors. The aim of this research is investigate the possibility using of fly and wood ash in construction sector with radiological point of view.

Materials and Methods

For the synthesis of the alkali-activated materials, as a precursor, used fly ash (FA) and wood ash (WA). Fly ash originated from power plant „Nikola Tesla“, Obrenovac, Serbia, while the wood ash from wood burning process in individual fireplace. Alkali-actiavated materials were synthesis by mixture fly and wood ash (ratio is 9:1), and the alkali activator solutions (AAS), mixture of sodium hydroxide (Sigma-Aldrich, st. Louis, MO, USA) and sodium silicate solution (Interhem Company, Belgrade, Serbia). The solution of sodium hydroxide was made in three differente concentration (4M, 6M and 12M). The obtained alkali-activated materials labeled as – AAM4 (precursor and AAS- 4M $NaOH/Na_2SiO_3$), AAM6 (precursor and AAS- 6M $NaOH/Na_2SiO_3$) and AAM12 (precursor and AAS - 12M $NaOH/Na_2SiO_3$). The ratio of liquid and solid phase was 1.0. Precursor and AAS were mixed, poured in modls, covered and left ad room temperature for 24 hours. After that the mixture was kept 48 hours at 60 °C, and than left for four weeks at room temperature in controlled conditions. The samples were crushing and sived through a sieve, and prepared for characterization.

Chemical composition was done by x-ray fluorescence spectroscopy–XRF (Li2B4O7/ LiBO2 fusion, measured with XF700 program (XRF-Fusion-Major Oxides). All samples were ignited at 950°C (Loss on ignition (LOI)).

Phase characterization of alkali-activated materials were analyzed using x-ray diffraction (XRD) – Ultima IV Rigaku diffractometer (Rigaku, Tokyo, Japan) equipped with Cu Kα1,2 radiation, with a generator voltage of 40.0 kV and a generator current 40.0 mA. The range of 5-80° 2θ was used for all powders in a continuous scan mode with a scanning step size of 0.02° at a scan rate of 5°/min [10, 11].

Diffused reflectance infrared Fourier transform spectroscopy (DRIFT) was performed by using the Perkin-Elmer FTIR spectrometer, in the spectral region of 400-4000 cm^{-1}.

Activity concentration of naturally occurring radionuclides (uranium and thorium series, ^{40}K and ^{235}U) in alkali-activated materials was determined by the semiconductor high purity germanium (HPGe) detector. Powdered samples were placed in PVC cylindrical containers

(125 mL), sealed, and left in order to reach radioactive equilibrium. Radiological analysis was performed by means of two coaxial spectrometers: AMETEK-ORTEC GEM 30-70, with 37% relative efficiency and 1.8 keV resolution at the 1332.5 keV line 60Co; and Canberra GX5019, with 55% relative efficiency and 1.9 keV resolution at the 1332.5 keV line ^{60}Co. Certified solution of mixed gamma-emitting radionuclides (^{241}Am, ^{109}Cd, ^{139}Ce, ^{57}Co, ^{60}Co, ^{137}Cs, ^{113}Sn, ^{85}Sr, ^{51}Cr, ^{210}Pb, and ^{88}Y), purchased from the Czech Metrology Institute (CMI) [12], was used for the preparation of standards for the energy and efficiency calibration of the spectrometer in accordance with IAEA recommendations [13]. After reaching the radioactive equilibrium, samples were measured, and all spectra were recorded and analyzed using Canberra's Genie 2000 software; net areas of the peaks were corrected for the background, dead time, and coincidence summing effects. All calculations were performed with the Mathematica 5.2 software (Wolfram Research, Inc., Champaign, IL, USA).

Radium equivalent activity (Ra_{eq}) is the index defined to obtain the sum of activities for comparison of specific radioactivity of materials containing different radionuclides ^{226}Ra, ^{232}Th, and ^{40}K, and it is defined as:

$$Ra_{eq} = A_{Ra} + 1.43A_{Th} + 0.077A_K \quad (1)$$

where A_{Ra}, A_{Th}, and A_K are activities in Bq/kg of ^{226}Ra, ^{232}Th, and ^{40}K, respectively [8]. We examine the characteristics of the material in order to assess the possibility of its use as a building material; for limiting the radiation dose from building material, the external hazard index (H_{ex}) is defined as [8].

$$H_{ex} = \frac{A_{Ra}}{370} + \frac{A_{Th}}{259} + \frac{A_K}{4810} \leq 1 \quad (2)$$

The value of this index must be less than unity to keep the radiation hazard insignificant, i.e., to keep the radium equivalent activity and annual dose under the permissible limits of 370 Bq/kg and 1 mSv, respectively [8].

The external absorbed gamma dose rate, $\dot{D}$ (nGy/h), in air 1m above the ground due to radionuclides ^{226}Ra, ^{232}Th, and ^{40}K in measured samples was calculated [8]:

$$\dot{D} = 0.462A_{Ra} + 0.604A_{Th} + 0.0417A_K \quad (3)$$

In order to estimate the health risks, the annual effective dose rates was calculated using the conversion coefficient from the absorbed dose in air to the effective dose (0.7 Sv/Gy), the indoor occupancy factor (0.8) - assuming that people spend approximately 80% of the time indoors, and 8760 h (1 year) annual exposure time as proposed by UNSCEAR (1993). The annual effective dose (EDR) was calculated from the formula [8, 14]:

$$EDR(\text{mSv}) = \dot{D}(\text{nGy/h}) \text{ x } 8760\cdot(\text{h/y}) \text{ x } 0.8 \text{ x } 0.7 \text{ (Sv/Gy)} \ 10^{-6} \quad (4)$$

Results

XRF analysis

Table 1 shows chemical composition of raw materials, WA and FA.

Table 1. Chemical composition of WA and FA.

Chemical	WA (wt.%)	FA (wt.%)
Na_2O	0,51	0,32
MgO	4,25	2,07
Al_2O_3	4,06	27,36
SiO_2	4,07	55,90
P_2O_5	1,92	0,07
SO_3	1,18	0,18
K_2O	11,16	1,49
CaO	38,76	3,69
TiO_2	0,11	0,67
MnO	1,43	0,07
Fe_2O_3	0,72	5,93
ZnO	0,193	0,013
As_2O_3	0,14	0,15
BaO	0,26	0,08
L.O.I.*	31,06	1,79

*Loss on ignition 950 °C

Radiological characterization of AAM

Results of activity concentration of gamma-emitting radionuclides in alkali-activated materials AAM4, AAM6 and AAM12 shown in the Table 2.

Table 2: Activity concentration (in Bq/kg) of gamma-emitting radionuclides in alkali-activated materials

Radionuclides	Activity concentration (in Bq/kg) of gamma-emitting radionuclides		
	Samples of alkali activated materials		
	AAM4	AAM6	AAM12
^{137}Cs	3,82±0,37	4,19±0,47	2,80±0,31
^{210}Pb	59,3±3,9	58,6±5,7	37,3±2,6
^{235}U	5,07±0,76	4,56±0,93	2,7±0,54
^{226}Ra	**94,2±8,6**	**95,9±8,7**	**63,3±5,8**
^{238}U	100,4±11	93,4±8,2	63,2±6,7
$^{235}U/^{238}U$	0,0505	0,0488	0,0427
$^{234}Th(^{228}Ac)$	**53,1±3,8**	**55,6±3,8**	**42,3±2,9**
^{40}K	**408±21**	**485±26**	**332±17**

Table 3 shows results of radium equivalent activity (Ra_{eq}), external radiation hazard index (H_{ex}), the external gamma radiation absorbed dose rate ($\dot{D}$) and effective dose rate (EDR) calculated on the basis Equation (1), (2), (3) ,(4), respectively.

Table 3. Radium equivalent activity (Ra_{eq}), external radiation hazard index (H_{ex}), the external gamma radiation absorbed dose rate ($\dot{D}$) and effective dose rate (EDR) of alkali-activated wood and fly ash

Samples	Ra_{eq} (Bq/kg)	H_{ex} (Bq/kg)	$\dot{D}$ (nGy/h)	EDR (mSv/y)
AAM4	201,55	0,544	92,6	0,454
AAM6	212,75	0,575	98,1	0,481
AAM12	149,35	0,403	68,6	0,337

Structural characterization of AAMs

XRD analysis

Figure 1 shows the diffractogram of alkali-activated materials.

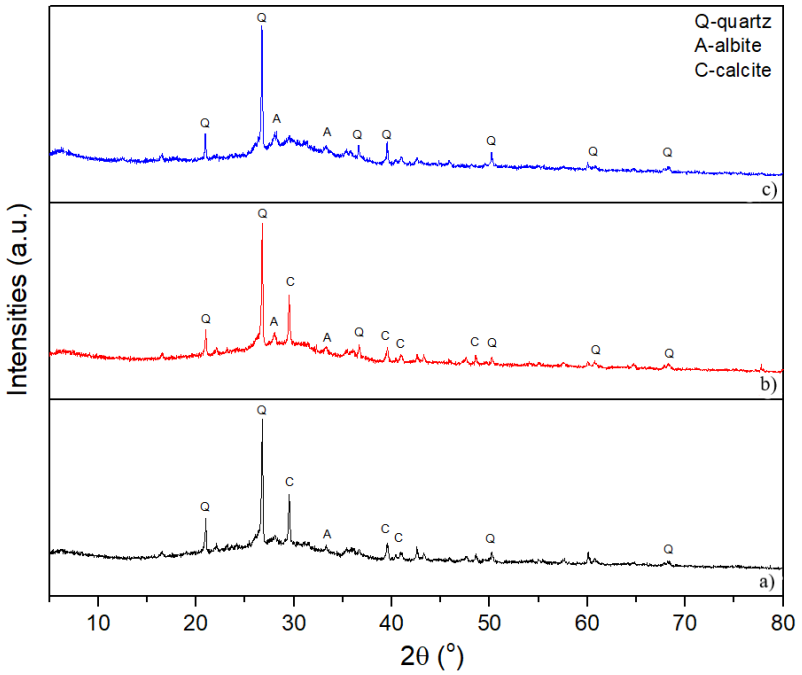

Figure 1. XRD diffractogram of alkali-activated materials: a) AAM4, b) AAM6 and c) AAM12

DRIFT analysis

Diffused reflectance infrared Fourier transform spectroscopy was shown on the Figure 2.

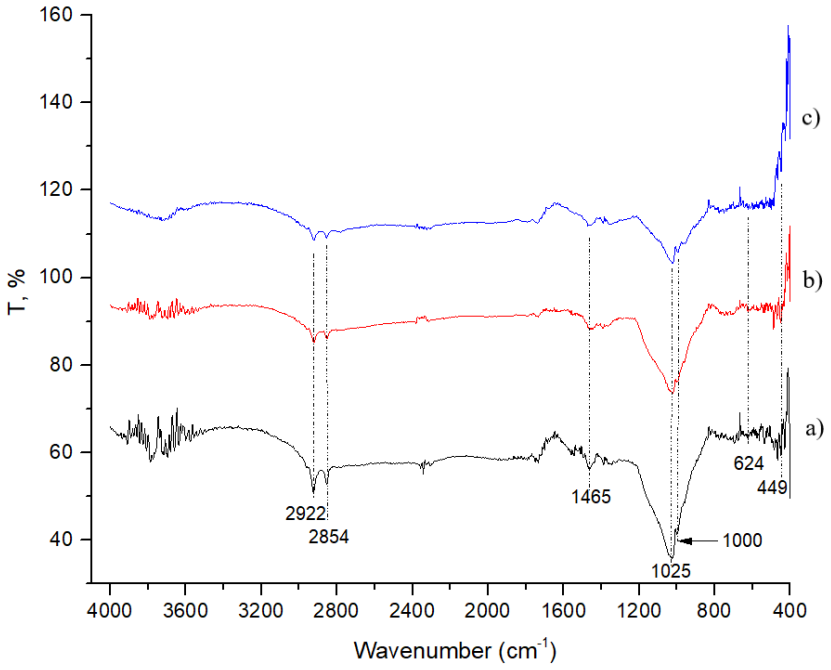

Figure 2. DRIFT spectrum of alkali-activated materials: a) AAM4, b) AAM6 and c) AAM12

Discusion

Chemical analysis of WA showed that the highest content of CaO, and K_2O, while the percentages of Al_2O_3 and SiO_2 are quite small and approximately the same, about of 4%. The content of Al_2O_3 is twice time lower than percent of SiO_2 for the fly ash originated during combustion process of coal in thermal power plant and their values are 55.9 % and 27.36 %, respectively. The content of CaO presented in fly ash is 3.69 %. The presence of the mentioned oxides is significant from the aspect of the alkaline activation process, that is, the polymerization that occurs during alkali activation.

Artificial radionuclide ^{137}Cs (Table 2) was detected in low concentration in all AAM samples. Decrease of specific activity was detected for 40K, 232Th, and 226Ra obtained for all AAM samples related to values of specific activity was detected for 40K, 232Th, and ^{226}Ra of FA investigated by Nenadović et al, 2021 [15]. Table 3 presents the total values of the *Raeq*, *Hex*, *D*, and annual effective dose *EDR* originated from AAMs. The smalest values of calculated parameters were mainly observed for AAM12 sample. The value of external radiation hazard index -*Hex* must be less than unity to keep the radiation hazard insignificant, i. e., to keep the radium equivalent activity and annual dose under the permissible limits of 370 Bqkg^{-1} and 1 mSv, respectively [15].

The mineralogical composition of the present phases was determined by X-ray analysis (Fig 1). The obtained data confirmed the presence of some main crystal phases. In the diffractogram of all three samples, the presence of an amorphous phase, which characterizes some higher background on the diffractogram in the range of 15–35° 2θ, was noticeable, and the presence of dominanted crystalline phase of quartz—SiO_2 in all sample detected. Crystalline phase calcite -$CaCO_3$ was observed in AAM4 and AAM6, followed by smaller amounts of albite, while quartz and albite presented in AAM12.

The DRIFT spectra all of AAMs are very similar. The presence of two bands in the range of OH-group vibrations was noted in the DRIFT spectrum. The band at ~3700 cm^{-1} was derived from the regularly distributed group OH in the structure. The wide band ~3450 cm^{-1} was related to a randomly placed hydroxyl group in the structure. Vibrations associated with symmetric and asymmetric stretches of C-H in the methyl and methylene groups, present in the spectrum were found at wavelengths 2922 cm^{-1} and 2854 cm^{-1} [16, 17], respectively. The characteristic stretching asymmetric vibrations C=O and carbonate vibrations at 1465 cm^{-1} were expected due to presence of CaO in wood ash (Table 1) and the possibility of carbon dioxide formation in highly alkaline wood and fly ash [18]. The peak at 624 cm^{-1} assignable to Mg–O– or K–O bonds were observed in alakli-activated material. A vibration at 449 cm^{-1} indicated Si–O bonds, while vibrations at 1025 cm^{-1} were the main characteristic of the alkali-activated materials which represented the Al–O and Si–O bonds [19].

Conclusion

On the basis of the obtained results, it can be concluded that fly and wood ash are suitable for obtaining alkali-activated materials with satisfactory radiological and structural characteristics. The calculated maximum Ra_{eq} of all AAMs, lower than the recommended maximum and investigated materials do not cause a significant radiation hazard. The main structural characteristic of the AAM is observed at 1025 cm^{-1}, which represents the Al–O and stretching Si–O bonds, and the Si-O-Al polymeric framework. XRD analysis showed that AAMs consists predominantly of phase of quartz, calcite and albite.

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RADIOLOŠKA KARAKTERIZACIJA ALKALNO AKTIVNIH MATERIJALA KOJI SADRŽE DRVENI I LETEĆI PEPEO

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SAŽETAK

Nepravilno skladištenje letećeg pepela kao industrijskog otpada je uzrok problema kontaminacije zemljišta. Ovaj otpad ili nusproizvodi imaju potencijal da se koriste kao sekundarne sirovine u građevinarstvu, promovišući koncept kružne ekonomije kako bi se izbegla kontaminaciju zemljišta. U ovom radu procenjuje se radiološki uticaj na životnu sredinu kada se otpad koji sadrži povišene nivoe prirodnih radionuklida kao što su leteći pepeo i drveni pepeo pretvara u ″zelene cemente″ kao što su geopolimeri ili alkalno aktivirani materijali (AAM). Alkalno-aktivirani materijali nastali su reakcijom alkalne aktivacije drvenog i elektrofilterskog pepela, kao čvrstih prekursora, i rastvora alkalnog aktivatora, natrijum hidroksida i natrijum silikata. Korišćene su tri različite koncentracije ratvora alkalnog aktivatora. Određivanje sadržaja radionuklida urađeno je spektrometrijom gama zraka. Jačina apsorbovane doze bila je 68,6-98,1 nGy/h, a H_{ex} (Bq/kg) za alkalno aktivirane materijale AAM4, AAM6 i AAM12 je bio 0,544 Bq/kg, 0,575 Bq/kg i 0,403 Bq/kg, respektivno. Rezultati merenja koncentracije aktivnosti u alkalno aktiviranim materijalima ukazuju na potencijal njihove bezbedne primene u građevinskim konstrukcijama. Alkalno aktivirani materijali su ispitani i u pogledu nekih strukturnih karakteristika.

САДРЖАЈ