ЗБОРНИК РАДОВА

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Друштва за заштиту од зрачења
Србије и Црне Горе

04-06. oктобар 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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**XXXII СИМПОЗИЈУМ ДРУШТВА
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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

FLY-ASH FOR USAGE IN THE BUILDING MATERIAL INDUSTRY

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ABSTRACT

In this study, fly ash (FA) from the active and passive cassette TENT A (Nikola Tesla power plant, Obrenovac) is characterized from physicochemical and radiological aspects.Samples of FA consisted of amorphous phase followed by quartz, mullite, plagioclase, K-feldspar, hematite and calcite. As indicated by laser granulometry, the fraction D_{50} of investigated samples are about 80μm, so based on the above, they belong to coarse ashes.Very important parameter which defines chemical composition of the fly ash as precursor material of clinker or alkali activated materialsis presence of organic matter which is expressed by total organic content (TOC).Assessment of radiation exposure during coal combustion depends on the concentration of radioactive elements in the coal and in the resulting fly ash. Fly ash as industrial waste contains certain concentrations of natural radionuclides that are considered naturally occurring radioactive materials (NORM). The results showed that fly ash has satisfactory radiological properties and can be used as an addition to clinkers, but also as a potential precursor of a new class of alkaline activated materials that can be used in the construction sector.

Introduction

Fly ash is a by-product material produced in the combustion process of coal used in power stations. It is a fine grey coloured powder having spherical glassy particles that rise with the flue gases. As pozzolanicmateriala fly ash is used in concrete, mines, landfills and dams. There has been an increasing attempt for fly ash utilization in different sectors. Loya and Rawani [1] identified top areas for the quantity of fly ash utilization as 44.19% in cement and concrete sectors, 15.25% of ash in roads, embankments and ash dyke raising, followed by 12.49% in reclamation of low lying areas and land filling, 8.84% in mine filling, 7.61% in bricks, blocks and tiles, 2.47% in agriculture and 9.14% in others. Since 316 individual minerals and 188 mineral groups are recognized in fly ash, it is one of the most complex materials in terms of characteristics [2]. However, all fly ash includes substantial amounts of silicon dioxide (SiO_2) (both amorphous and crystalline), aluminum oxide (Al_2O_3) and calcium oxide (CaO), the main mineral compounds in coal-bearing rock strata. Fly ash can be classified according to the type of coal from which the ash was derived and depending on pH value and calcium/sulphur ratio, fly ashes are classified as acidic ash (pH 1.2 up to 7), mildly alkaline ash (pH 8–9), and strongly alkaline ash (pH 11–13) [3]. The chemical properties of fly ash depend not only on the type of coal used in a process but also on the techniques used to burn the coal. Specifically, properties of fly ash depend on: (a) boiler configuration, (b) burning condition and temperature of the boiler, (c) the particle size of the coal, and (d) the gas cleaning equipment [4]. The use of industrial by-products, fly ash of the coal combustion process, disposed in landfills near thermal power plants whichrepresent a burden on the environment is nowdays an important task for scientists.

In the thermal power plants (TE) of "ElektroprivredaSrbije" (JP EPS) whose boilers burn lignite, around 6 million tons of fly ash are produced annually as a waste which is deposited in an open waste disposal, taking up an area of around 400 hectares. Depositing ash in open spaces can be very damaging to the environment due to the potentially deleterious effects of ash particles. Dispersal of ash particles by wind and water erosion and the leaching of substances such as salts and heavy metals can be hazardous for the surrounding terrain and underground waters.Fly and bottom ash are produced as a by-product in the process of burning Kolubara lignite (Coal mine basin Kolubara,Serbia) are the basic raw material in the technological process of collecting, preparing, transporting and disposing of ashes by their nature they belong to non-hazardous waste. A potential market for the use of fly ash exists and for now, it is used exclusively by cement factories [5].The production of construction materials using or re-using local industrial waste alumina-silicate materials is a cost-benefit, environmental-friendly and sustainable technology [6, 7].

In order the ashes found in landfills to continue to be used as raw materials in the production of concrete, cement, clinker, and some new materials, for example alkaline-activated materials that would find their application in the construction sector, it is necessary to examine, among others, physico-chemical, mineralogical, and radiological characteristics of FA.The radioactivity of FA can be one of the important reasons against its wider use in the construction industry of Serbia [3]. The presented research is a contribution to the potential solution of environmental protection through the synthesis of potential construction materials based on FA or applications FA as Al-rich by-products in the building industry.

Materials and methods

Materials

Selected samples of FA were taken from ash dumps TENT A. Location of TENT A is on the right bank of the Sava river, about 40 km upstream of Belgrade between settlements of Krtinska and Urovci, about 3 km west of Obrenovac. Fly ash disposal**:** The cassette 1 of the ash dump of the TENT A in Obrenovac is covered with earth. The cassette 2 is active and the ash dumped into this cassette is mixed with water, while the cassette 3 is passive and recultivated, with planted grass[8].

The sample S1 was taken from the passive cassette number 1 from TENT A. The creation of a passive cassette differs from an active cassette in the part when the subject cassette ceases to be actively used. After the end of its active use, there is a period of aging of the cassette, which implies exposure exclusively to environmental influences.The sample is brownish with some impurities in it. Particle size and granulation are within expectations for the ash sample. Since it is a passive cassette, which has been inactive for several years, sampling was performed by the method of random sampling from several places. The composite sample thus obtained was later mixed, dried and homogenized. The sample S2 was taken from active cassette from TENT A. The sample is dark almost black in color and shows the presence of impurities. The granulation of the powder is within the expected range. Since it was sampled from the active cassette, the reliability of the results may be better than that of the samples from the passive cassettes.

Due to the size of the active cassette, as well as the fact that part of the cassette is covered with water from the sprinklers, for sampling a representative part of the active cassette that can best represent the entire process that takes place in the active cassette was selected[8].

Methods

Among physical characteristics moisture content, granulometry, BET specific surface area, particle density were performed.

The specific surface area of the fly ash samples weredetermined using the BET (Brunauer-Emmet-Teller) methodusing Micromeritics ASAP-2020 analyser, by nitrogenadsorption measurements at 77 K. The samples were pre dried for 60 min at 105 °C. Specific density analysis of flyash samples was determined according to EN 1097–7 [9],by pyknometer method. Moisture measurements were determined by drying sample in an oven SP-440 (max. T 300 °C)on 105 °C for 24 h. Moisture content is given with the relation % $mc_{wb} = (w_w\text{-}w_d/w_w) \times 100$ where mc is expressed onwet basis (w_w is wet weight and w_d is dry weight)

The X-ray powder diffraction (XRD) was performed to determine the phase composition of the FA samples using a PANalytical Empyrean X-ray diffractometer equipped with CuKa radiation with l = 1.54 A. The samples were scanned at 45 kV and a current of 40 mA, over the 2θ range from 4° to 70°, at a scan rate of 0.026° 2θ min^{-1} and step time 172 seconds. The obtained XRD patterns were analysed using X'Pert High Score Plus diffraction software v. 4.8 from PANalytical, using PAN ICSD v. 3.4 powder diffraction files. All Rietveld refinements were performed using the PANalyticalX'Pert High Score Plus diffraction software, using the structures for the phases from ICDD PDF 4+ 2016 RDB powder diffraction files. Amorphous content was determined using the external standard method (NIST SRM 676a) [10]. The chemical composition of FA was determined by X-ray fluorescence (XRF). The XRF analysis was performed with a wavelength dispersion (WD XRF) spectroscope ARL Perform X manufactured by Thermo Scientific with a power of 2500 W, 5 GN Rh X-ray tube, 4 crystals (AX03, PET, LiF200 and LiF220), two detectors (proportional and scintillation), and computer program UniQuant. The samples were quartered, dried at 105 ° C and calcined at 950 °C.

Total organic compound (TOC) analyzed by Analizator CW-800M "Multiphase", ELTRA by dry incineration method, detection of products with IR detector.

The contents of naturally occurring radionuclides in the FA were determined by gamma spectrometry. The samples of FA placed in PVC cylindrical containers (125 ml and 250 ml), sealed with beeswax and left for six weeks.The equilibrium between radon and its progeniesis realized in this way. Radiological analysis was performedby means of a coaxial semiconductor high purity germanium (HPGe) detector (Canberra 7229N-7500-1818 with 20% relative efficiency and 1.8 keV resolution for ^{60}Co at the 1332 keV line) associated with standard beam supply electronics units.The method has been shown previouslyand described by Nenadović, etal. [11] and Mirković et al. [12] Quoted uncertainties (the confidence level of 1σ) were calculated by error propagation calculation. The combined standard uncertainties included the statistical uncertainties of the recorded peaks, efficiency calibration uncertainty and the uncertainty of measured mass.

European Commission [13] recommends that the reference level for building materials should be of the order of 1 mSv/y or less expressed as effective dose caused by external gamma radiation to members of the public. A common screening method the dose caused by building materials is the use of an Activity Concentration Index (I), the value of which is calculated on the basis of the concentrations of Ra-226, Th-232 and K-40. The index ACI is related to the gamma radiation dose in a building and was calculatedaccording to equation 1 [14]:

(1) $$ACI = \frac{Ac_{Ra-226}}{300} + \frac{Ac_{Th-232}}{200} + \frac{Ac_{K-40}}{3000}$$

where $A_{C,Ra-226}$, $A_{C,Th-232}$ and $A_{C,K-40}$ are the activity concentrations in Bq/kg.

Besides the activity concentration index, in order to estimate a possible health effect due to the exposure to natural radionuclides present in the measured samples, radium equivalent activity, Ra_{eq} [$Bqkg^{-1}$], the external hazard index, *Hex* [$Bqkg^{-1}$], total external absorbed gamma dose rate *D* [$nGyh^{-1}$], and annual effective dose *EDR* [mSv] can be calculated. The radium equivalent activity can be used to estimate the hazard associated with materials that contain 226-Ra, 232-Th, and 40-K. The external radiation hazard index reflects the external radiation hazard due to the emitted gamma radiation. The values of these indicators of exposure can be calculated according to eqs. fromVukanac et *al.[*15].The value of this index must be less than unity to keepthe radium equivalent activity and annual dose underthe permissible limits of 370 $Bqkg^{-1}$ and 1 mSv, respectively.

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 [16]:

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

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 [17]:

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

Results

Physico-chemical properties of FA

Table 1 shows the particle density, bulk density, BET and moisture content of FA samples.

Table4: Particle density, bulk density, BET and moisture content of FA

Sample	Particle density (g/cm^3)	Bulk density (kg/m^3)	BET (m^2/g)	Moisture content (%)
S1	2.20	681	8.0	31.22
S2	2.05	659	17.9	25.92

The characteristic values D_{10}, D_{50} and D_{90} are shown in Table 2 and corresponding PSD curves are presented on Figure 1.

Table 2: Results of laser granulometry

Sample	D_{10} (μm)	D_{50} (μm)	D_{90} (μm)
S1	13.4	78.7	203.4
S2	21.5	85.9	144.1

XRD analysis

Table 3 shows phase composition of investigated samples determined by XRD analysis.

Table 3: Phase composition of ash samples

Sample	Amorphous (%)	Mineral composition (%)						
S1	68.0	Quartz	Mullite	Plagioclase	K-feldspar	Hematite	Calcite	SUM
		14.2	9.2	6.8	1.2	0.2	0.4	100.0
S2	71.6	Quartz	Mulite	Plagioclase	K-feldspar	Hematite	/	SUM
		16.5	6.5	4.6	0.7	0.1	/	100.0

XRF analysis

Table 4 shows chemical composition of all samples determined by XRF analysis.

Table 4: XRF results of chemical composition of all samples

Sample	L.O.I.	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	SO_3	MnO	TiO_2	P_2O_5	Cr_2O_3	SUM
	(%)													
S1	3.00	57.65	23.46	5.28	5.82	1.98	0.32	1.39	0.04	0.07	0.84	0.05	0.05	100.04
S2	5.90	57.92	22.72	6.30	2.99	1.43	0.30	1.29	0.34	0.06	0.72	0.05	0.03	100.10

Table 5 showstotal organic compounds (TOC) in samples of FA.

Table 5: Results of TOC measurements

Sample	TOC (%)
S1	0.56
S2	0.71

Radiological Characterizations

For assessing the specific dose rate, more elaborate methods need to be used in order to consider the actual concentrations and locations of a certain building material in a building. European Commission [13] recommends the model represents by Markkanen in reference [18]. A protection strategy should be established with the aim to promote building materials that do not exceed the reference level.

Radiological characteristics of the ash samples – specific activity (Bq/kg) were presented in Table 6.

Table 6: Activity concentration of natural radionuclides in the investigated samples with associated measurement uncertainties (k = 1).

Sample	Specific activity (Bq/kg)						
	^{210}Pb	^{226}Ra	^{232}Th	^{40}K	^{137}Cs	^{238}U	^{235}U
S1	89±8	72±3	61±4	246±17	<0.1	81±8	5.5±0.4
S2	29±3	51±2	38±3	211±15	<0.1	47±5	2.8±0.2

Table 7 shows radium equivalent activity (Raeq), external radiation hazard index (Hex), the external gamma radiation absorbed dose rate ($\dot{D}$) and effective dose rate (EDR) of fly ashes.

Table 7: 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 fly ash samples

Sample	Ra_{eq} (Bq/kg)	H_{ex} (Bq/kg)	$\dot{D}$ (nGy/h)	*EDR* (mSv/y)	*ACI*
S1	348.65	0.4813	80.4	0.394	0.627
S2	267.81	0.328	55.3	0.271	0.430

Discusion

Mineral composition of the samples was determined. Samples consisted of amorphous phase followed by quartz, mullite, plagioclase,K-feldspar, hematite, calcite. Quartz amount in both sample of FA from passive and active casete is approximately the same. Also, amount of amorphous phase is slightly higher for S2 sample.Such a high % of the amorphous phase indicates a good reactivity of FA in further processes of use as a component in the production of clinker or alkali-activated materials.During the burning process, the mineral substances from the coal change into the crystalline and amorphous phases that make up the ash.For example, calcite is thought to mostly transition to CaO, which in some cases can react with SO_2 and CO_2 to form anhydrite and again calcite.Clay matter, like feldspar, melts and transforms into glass, sometimes with the action of solvents such as FeO and CaO.Kaolinite usually passes into mullite, glass and sometimes cristobalite, while the other types of clay and feldspar transition to glass[19].Quartz partially melts, but some quartz grains remain.In this way, in addition to the amorphous phase that is characteristic of fly ash, crystalline mineral species mainly include quartz, mullite, K- and Ca-type feldspars, calcite, gelenite, hematite. The spherical particles that make up the glassy (amorphous) part of the ash are mostly thin, hollow, ceramic microspheres and are called cenospheres.These spheres are created under specific conditions, as molten drops of clay minerals, mica, feldspar and quartz.Spheres are usually charged gases resulting from combustion of organic matter, decomposition of carbonates, dehydration clay minerals and pore water evaporation.These are mainly CO_2, N_2, O_2 and H_2O.It is assumed that these spheres are formed at temperatures between 1230 and 1400°C[20].

According to particle size, ashes are divided into fine and coarse fraction.The fine fraction includes ash whose particle size is below 45 µm, while ashes with particle sizes above that belong to coarse ash.Ashes from bituminous coals have finer particles compared to ashes produced by burning lignite coals[19].As a result of laser granulometry, the fraction D_{50} of both samples are about 80µm, so based on the above, they belong to coarse ashes.

Among chemical parameters which are analyzed SiO_2 amount is almost the same(~58%) for the S1 and S2 sample. The values for CaOis lower for the S2–2.99 %-almoust twice value of S1.

Very important parameter, which defines chemical composition of the fly ash as precursor material of clinker or alkali activated materials, is presence of organic matter. It is expressed by total organic content (TOC). It effects mainly emissions, as in unstable operating conditions the presence of TOC in clinker raw mixture can contribute to the CO_2 emissions.

The behavior of natural radionuclides in the process of burning coal depends on a number of factors, such as the type and characteristics of coal, ash content in coal, calorific value of coal, combustion temperature, chemical and physical form in which the radionuclides are found coal and others. In the combustion process, the organic component is eliminated thus that there is an increase in the concentration of radionuclides in ash compared to coal. Change in natural radioactivity as a consequence of the operation of thermal power plants it can also affect the food chain, soil-plant-animal-human. Considering that *EDR,* $H_{ex}$and *ACI* are less than 1,FAs are safe from the aspect of external exposure with the limitation of the duration of exposure to less than 20% of hours per year.

Conclusion

Physico-chemical characteristics of samples led to conclusion that these ashesare heterogeneous materials.

Accordingly, the criteria $Ra_{eq} < 370$, $H_{ex} < 1$, FA from bouthcasete can be freely re-used as raw materials for building materials.

This researchingoffer a good basis for further investigations, consideringpossible utilization of these ash in production new materialswhich can be applied in the construction sector.

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UPOTREBA LETEĆEG PEPELA U INDUSTRIJI GRAĐEVINSKOG MATERIJALA

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REZIME

Procena izloženosti zračenju pri sagorevanju uglja zavisi od koncentracije radioaktivnih elemenata u uglju i u letećem pepelu koji nastaje. Uzorci letećeg pepela se sastoje od amorfne faze praćene kvarcom, mulitom, plagioklasom, K-feldspatom, hematinom, kalcitom. Kao rezultat laserske granulometrije, frakcija D50 za ispitivane uzorke je približno 80 μm, tako da na osnovu navedenog, leteći pepeli predstavljaju pepele krupnih čestica. Veoma važan parametar je prisustvo organske materije koje se izražava ukupnim organskim sadržajem. Leteći pepeli kao industrijski ostaci sadrže određene koncentracije prirodnih radionuklida koji se smatraju prirodnim radioaktivnim materijalima (NORM). U ovojm istraživanju, leteći pepeo iz aktivne i pasivne kasete TENT A (termoelektrana Nikola Tesla, Obrenovac) karakteriše se sa radiološkog i fizičko-hemijskog aspekta. Rezultati su pokazali da leteći pepeo ima zadovoljavajuća radiološka svojstva i da se može koristiti kao dodatak klinkerima, ali i kao potencijalni precursor nove klase alkalno aktiviranih materijala koji se mogu primeniti u građevinskom sektoru.

САДРЖАЈ