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Fly ash from sewage sludge as a component of concrete mix resistant to environmental influence

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Abstract
The restrictions on carbon dioxide emissions introduced by the European Union encourage experimental work on new-generation materials containing smaller amounts of clinker. At present, silica fly ashes from the combustion of hard coal are widely used in the technology of cement and concrete in Europe and Poland. This research aims to assess the physical and chemical properties of fly ashes from the thermal treatment of sewage sludge for use in concrete technology in relation to applicable standards and determine their impact on the natural environment. The established concentrations of heavy metals are below the maximum values required when discharging sewage into the ground or waters and also meet the necessary leaching limits when inert waste is allowed to be landfilled and on substances particularly harmful to the aquatic environment. On this basis, it was found that the migration of heavy metals from concrete with the addition of ashes to the water environment is insignificant and should not be a major problem. In addition, the tests showed that the activity index did not exceed the limit value.

Introduction

The use of waste for the production of building materials is becoming increasingly common. Despite being consistent with sustainable development principles, the use of fly ashes from sewage sludge as a concrete modifier requires deep consideration of the impact of such waste products on human and animal health. Another important issue is the determination of the kinetics of the release of selected harmful substances into the environment. This is important both at the exploitation stage of a building object and in the post-exploitation period in which materials from a demolished building object are to be stored or intended for reuse.

Considering that all building materials of mineral origin (including concrete) contain natural radioactive elements and release heavy metals, it is necessary to conduct monitoring studies on the concentration of natural radioactive isotopes and to determine the concentration level of heavy metals. Such research is conducted not only in Poland but also in other countries, including China, Slovakia, Nigeria, Australia, Bangladesh, Tanzania, Pakistan, and Kuwait (Bou-Rabee & Bem, 1996; Msaki & Banzi, 2000; Yang, Wu & Jiang, 2005; Ahmad, 2007; Faheem, Mujahid & Matiullah, 2008; Zapotoczna-Sytek, Mamont-Cieśla & Rybarczyk, 2012; Eštaková & Palaščaková, 2013; Kolapo et al., 2017; Gil & Golewski, 2019). On the basis of these tests, an assessment of the suitability of building materials is carried out. According to the UNSCEAR report (UNSCEAR, 2000), the average annual worldwide effective dose that a person staying in a room receives from building materials is estimated at 0.4 mSv.

It is believed that the migration of heavy metals into the water environment is a potential threat when ashes from the incineration of sewage sludge are used for the production of concrete. The level of heavy metals released to the natural environment from concrete samples containing waste depends primarily on the selection of cement or binder, as well as on the method of production of the concrete. These factors have a decisive influence on the durability and porosity of concrete (Ajdukiewicz, 2011). The leachability of heavy metals from waste-containing concretes is also significantly influenced by the time after which water extracts are made from these concretes. The level of heavy metals released can also be affected by the high temperatures acting on the concrete. The negative effect of high temperature on concrete relates to its impact on its structure, leading to the formation of cracks and even the complete destruction of concrete (Sakr & El-Hakim, 2005).

Fly ashes from the thermal treatment of sewage sludge, in accordance with the applicable Regulation of the Minister of Development of 2016 (Journal of Laws, 2016), can be used for the preparation of concrete mixes for cases when the concentration of heavy metals in the concrete samples for water extracts from the leachability test of these metals does not exceed 10 mg/l in total, which is calculated on the basis of the mass of the elements. So far, studies on water extracts from fly ashes originating from sewage sludge confirm their low susceptibility to leaching (Borowski, Gajewski & Haustein, 2014). Studies indicate a high degree of immobilization of the heavy metals both in the ashes themselves and in solidified ashes (Giergiczny & Król, 2008; Białowiec, Janczukowicz & Krzemieniewski, 2009; Kępys, Pomykała & Pietrzyk, 2013). The migration of heavy metals into the aquatic environment is insignificant and should not be a major problem. However, it is advisable to conduct further research for variable classes and types of concrete and different leaching conditions. Fly ashes from thermal treatment of municipal waste, including sewage sludge, can potentially be used in construction materials (Monzó et al., 2003; Lin & Lin, 2005; Lin, Luo & Sheen, 2005; Hudziak, Gorazda & Wzorek, 2012; Yusuf et al., 2012).

Fly ash, as a waste material and a raw material of mineral origin, also contains natural radioactive elements – mainly thorium and uranium present in hard coal and its combustion products (Olkuski, 2008; Michalik, 2006). Being surrounded by materials that continuously emit harmful radiation can cause many serious human diseases, such as biological changes, cell death, or genetic changes. Considering the unfavorable impact of radioactive elements contained in the ashes, before deciding on their approval for use, tests should be carried out to assess whether this additive can be used. Moreover, it should be checked whether the composite made with the addition of fly ashes meets the requirements for limiting natural radioactivity (Nocuń-Wczelik, 2002; Yu et al., 2005; Zakrzewski, 2005; Giergiczny & Król, 2008).

The basic legal act specifying the requirements for raw materials and building materials used in various types of construction is the Regulation of the Council of Ministers of December 17, 2020, on building materials, for which the radioactive concentration of radioactive isotopes potassium K-40, radium Ra-226, and thorium Th-232 are determined. When exceeding these determinations and the safe value of the radioactivity concentration index, it must be notified to competent authorities (Council of Ministers, 2020). The basis for assessing the activity of natural elements in building materials is the index of the radioactive concentration of radioactive isotopes potassium K-40, radium Ra-226, and thorium Th-232 (index I). Index I (which is dimensionless) considers the content of the naturally radioactive isotopes, and its value informs on the exposure of the body to gamma radiation emitted by natural radionuclides: 40K, 226Ra, and 228Th. The value of the indicator is determined according to the following formula:

$$I = \frac{C_{\mathrm{K-40}}}{3000\,\mathrm{Bq/kg}} + \frac{C_{\mathrm{Ra-226}}}{300\,\mathrm{Bq/kg}} + \frac{C_{\mathrm{Th-232}}}{200\,\mathrm{Bq/kg}}$$

where $C_{\mathrm{K-40}}$, $C_{\mathrm{Ra-226}}$, and $C_{\mathrm{Th-232}}$ are the mean radioactive isotope concentrations of potassium K-40, radium Ra-226, and thorium Th-232, which are expressed in becquerels per kilogram [Bq/kg].

The total uncertainty for determining the value of the indicator, at the confidence level of 0.95, may not exceed 20% of its value if it is not lower than 0.8. The value of the I index greater than 1 means the possibility of exceeding the reference level for external exposure of people to gamma radiation emitted by building materials indoors, amounting to 1 mSv per year, and the need to inform the building supervision authorities about exceeding the value of this index.

As part of the pilot studies, the impact of this additive on the natural environment is found and the leaching values of selected heavy metals from ashes and from ordinary concretes containing fly ashes from thermal treatment of sewage sludge are also determined. This paper also presents the concentrations of natural radioactive isotopes of radium (226Ra), thorium (232Th), and potassium (40K) for ordinary concrete and fly ashes from thermal treatment of sewage sludge originating from sewage treatment plants in Krakow, Łódź, and Warsaw. To determine the possibility of their use in concrete technology, the values of the radioactive concentration index of radioactive isotopes *I* are calculated (for EN 450-1:2012, ASTM-C618-03, and ASTM C379-65T).

Research material and methodology

The averaged samples were used in successively performed tests to verify the basic thesis about the possibility of recovery of fly ashes from thermal processing of sewage sludge in concrete technology. Separate batches of material were collected for the tests during the continuous exploitation of sludge from the selected three wastewater treatment plants: Warsaw, Krakow, and Łódź over three periods. The study of the activity concentration of radioactive gamma isotopes in concrete and fly ash samples was carried out at the Laboratory of Dosimetric Measurements of the National Center for Nuclear Research (LPD NCBJ). Heavy metals leaching test occurred at the Analytical Center of the Warsaw University of Life Sciences. The assessment of color and bulk density was made on the basis of approximately 2 kg of material batches.

Leachability tests were carried out for three analyzed fly ashes and for a concrete sample without any additives (BZ). In addition, tests were performed for three samples of concrete with the addition of fly ashes, in which 20% of the cement mass was replaced with individual fly ashes. The tests were carried out for samples with the highest percentage of fly ash (i.e., 20%) according to the EN 12457-2:2006 standard (Wichowski, Rutkowska & Nowak, 2017; Rutkowska et al., 2018). The generated fly ashes are construction materials containing residues from sectors processing that are naturally occurring radioactive material called naturally occurring radioactive material (NORM). The study of the concentration of gamma radioactive isotopes in concrete samples was carried out using the method of high-sensitivity gamma spectrometry using a germanium detector HPGe (high purity germanium) GX3520 Canberra, with 35% efficiency and 1.73 keV resolution for 1332 keV gamma energy. This method consists in recording the energy spectrum of gamma radiation.

Fly ash test results and their analysis

Table 1 summarizes the complete results of the chemical characteristics of the fly ashes recovered from sewage sludge and concrete samples, in which cement was replaced with fly ash in the amount of 20%. The obtained results of the leachability tests were compared with the values constituting the criteria for allowing waste of a given type to be deposited in landfills. Limit

Table 1. Comparison of the leaching values of heavy metals with the values allowing for storage of a given type of waste (in mg/kg dm)

Type samples	Heavy metals (mg/l)											
	Cd	Cr	Cu	Ni	Pb	Zn	As	Sb	Se	Ba	Hg	Mo
BZ	<0.002	0.013	0.013	<0.005	<0.003	<0.030	<0.010	<0.010	<0.010	1.18	<0.005	<0.010
Cracow	<0.002	<0.010	0.008	<0.005	<0.003	<0.030	<0.010	0.019	0.045	0.105	<0.005	0.39
Lodz	<0.002	<0.011	0.009	<0.005	<0.003	<0.030	<0.010	0.022	0.055	0.122	<0.005	0.43
Warsaw	<0.002	<0.010	0.008	<0.005	<0.003	<0.030	<0.010	0.020	0.047	0.112	<0.005	0.40
K – 20%	<0.002	0.016	0.011	<0.005	<0.003	<0.030	<0.010	<0.010	<0.010	1.07	<0.005	<0.010
L – 20%	<0.002	0.018	0.009	<0.005	<0.003	<0.030	<0.010	<0.010	<0.010	1.23	<0.005	<0.010
W – 20%	<0.002	0.019	0.014	<0.005	<0.003	<0.030	<0.010	<0.010	<0.010	1.15	<0.005	<0.010
Limit values of leaching when allowing waste to be landfilled (Journal of Laws, 2015) [1]												
waste indifferent	0.04	0.5	2	0.4	0.5	4	0.5	0.06	0.1	20	0.01	0.5
waste other than inert and hazardous	1	10	50	10	10	50	2	0.7	0.5	100	0.2	10
dangerous waste	5	70	100	40	50	200	25	5	7	300	2	thirty

[1] Bsic test: liquid/solid = 10 L ·kg dm^{-1}.

values were adopted in accordance with the Regulation of the Minister of Economy of 2015 on the admission of waste to landfills (Journal of Laws, 2015).

Fly ashes from the thermal treatment of sewage sludge can be used for the preparation of concrete mixes, when the concentration of heavy metals in water extracts from the leachability test of these metals from concrete samples does not exceed 10 mg/l in total, calculated on the mass of elements (Journal of Laws, 2016). In all tested samples, the total concentrations of heavy metals in terms of the mass of elements did not exceed the limit value. On the basis of the conducted tests, it was observed that the level of leaching of heavy metals from concrete samples containing fly ashes originating from the thermal treatment of sewage sludge was higher than that from fly ash alone.

The lowest leachability of heavy metals was observed for fly ash from the sewage treatment plant in Kraków. The exceptions are Sb, Se, and Ba, whose concentrations in the eluate from ashes from municipal sewage sludge were the highest. The concentrations of heavy metals in the eluates for fly ashes were lower than the limit values for neutral waste. Conducted by Monzo et al., studies on the leachability of trace elements from mature mortars and concretes with the addition of fly ashes have shown that the replacement of Portland cement with ash from thermal treatment of sewage sludge in the amount of 30% does not threaten the safety of the natural environment (Monzo et al., 1999). Chen et al. (Chen et al., 2013) showed that, of the potential impurities, only molybdenum and selenium were leached at concentrations exceeding threshold values. Leaching studies conducted on concrete monoliths showed that concentrations of contaminants, including molybdenum and selenium, did not exceed the threshold values given by the Environmental Protection Agency.

Considering environmental standards and technical specifications, it was found that the use of sewage sludge ash in building materials seems possible (Chen et al., 2013). Wichowski et al. (Wichowski, Rutkowska & Nowak, 2017) conducted research on the leachability of heavy metals from three different ashes: limestone, silica, and sewage sludge. On the basis of the conducted tests, it was observed that the level of leaching of heavy metals from concrete samples containing lime and silica ashes was lower than that from the ashes alone. For fly ash from the thermal treatment of municipal sewage sludge, leachability from a concrete sample containing this additive was higher than that from ashes. Leachability did not exceed the limit values adopted for inert waste. In the eluate for the analyzed ashes, the presence of barium, chromium, and copper was measured; additionally, the molybdenum content was measured for the calcareous ash. The leachability of heavy metals from samples containing fly ashes is lower or comparable to that in concrete without the addition of fly ashes. For the thermal treatment of sewage sludge and lime ash, the leachability of metals was at a comparable level. The smallest leachability was observed for silica fly ash. The exceptions are antimony and selenium, whose concentration in the eluate from sewage sludge ashes was higher, and barium and chromium, whose leachability was higher for limestone ash. Concentrations in the eluates of heavy metals for all fly ashes were lower than the limit values for inert waste (Wichowski, Rutkowska & Nowak, 2017; Rutkowska et al., 2018).

All eluates of the analyzed samples were characterized by high pH values, which may relate to the presence of oxides in the composition of the tested material. The highest pH value was recorded for reference concrete samples, 12.29, and the lowest value, equal to 9.12, for a sample of ash from the combustion of municipal sewage sludge from Lodz. In addition, the lowest values for other physicochemical parameters were also observed for this material, such as conductivity (2.2234 mS/cm), chloride concentration (7.6 mg Cl/l), color (23.45 mg Pt), turbidity (13.43 NTU – Nephelometric Turbidity Unit), phenolphthalein alkalinity (1.05), salinity (1.08 (sal) (o /oo)), and TDS (total dissolved solids) dry matter content (1.0915 g/l). Barbosa et al. (Barbosa et al., 2009) showed that the COD (chemical oxygen demand) in the eluate from the ashes from the combustion of conventional fuels had higher values. The observed differences may relate to the combustion technology used and, above all, to the temperature and the composition of the sewage sludge. Replacing 15% of the Portland cement in the concrete with fly ash did not significantly change the pH of the eluate, but it resulted in a weakening of the color intensity and a decrease in turbidity, as well as an increase in phenolphthalein alkalinity, hardness, and COD.

In addition, higher values of salinity, electrical conductivity, and TDS dry matter were observed in all eluates with a higher concentration of chlorides in the samples of 20%. The highest values of electrical conductivity (7.8575 mS/cm), phenolphthalein alkalinity (39.24), hardness (70.42 °T), and salinity (4.25 (sal) (o/oo)) were shown for reference concrete samples without additives. Wichowski et al. observed that the highest pH values were recorded for concrete samples with silica fly ash, equal to 12.26, and the lowest value, equal to 9.75, for a sample of ash from sludge incineration. In the silica fly ash eluate, the highest turbidity (50.80 NTU), chloride concentration (90.60 mg Cl/l), and color (143.00 mg Pt) were observed (Wichowski, Rutkowska & Nowak, 2017). Table 2 shows the concentrations of natural radioactive isotopes of potassium ^{40}K, radium ^{226}Ra, and thorium ^{232}Th of the tested samples of concrete and fly ashes from sewage sludge.

As can be seen from the data presented in Table 2, the concentrations of potassium, radium, and thorium isotopes in the samples are within the following limits: 197–526 Bq/kg, 18–110 Bq/kg, and 13.8–75.9 Bq/kg, respectively. The mean crustal concentrations of potassium, radium, and thorium reported in the UNSCEAR report are 400 Bq/kg, 35 Bq/kg, and 30 Bq/kg, respectively. For fly ash samples, considering the measurement uncertainty, the concentrations of potassium, radium, and thorium isotopes were higher than the average concentration of this isotope in the earth's crust. Such a wide dispersion of concentrations of natural radioactive isotopes in fly ashes is primarily caused by the different composition of sewage sludge supplied to the sewage treatment plant, which may also contain natural radioactive isotopes. The lowest concentrations of natural radioactive isotopes were obtained for ordinary concrete and fly ashes from the sewage treatment plant in Warsaw. The highest concentrations of natural radioactive isotopes of potassium, radium, and thorium were found in a sample of fly ash from the sewage treatment plant in Łódź. Table 3 presents the activity index *I* calculated on the basis of the determined concentrations of potassium, thorium, and radium.

Table 2. Concentrations of natural ^{40}K isotopes, ^{226}Ra, and ^{232}Th in the samples

Designation	^{40}K [Bq/kg]	^{226}Ra [Bq/kg]	^{232}Th [Bq/kg]
BZ concrete	197.0 ± 31.0	18.0 ± 2.8	13.8 ± 2.2
Ash from Krakow (K)	474.0 ± 50.0	54.9 ± 5.0	52.7 ± 4.6
Ash from Lodz (L)	526.0 ± 56.0	110.0 ± 10.0	75.9 ± 6.7
Ash from Warsaw (W)	388.0 ± 41.0	84.7 ± 7.7	64.9 ± 5.7

Table 3. Activity index *I* of the tested samples

Sample type	Activity *I* indicator
BZ	0.19 ± 0.05
K	0.60 ± 0.04
L	0.92 ± 0.02
IN	0.73 ± 0.06

Index *I* informs about the exposure of the whole body to gamma radiation from natural radionuclides, i.e., potassium, radium, and thorium. As seen in Table 3, the value of the index *I* is in the range of 0.19–0.92 Bq/kg. The highest value was obtained for fly ash samples from Łódź, and the lowest for ordinary concrete samples. The limit value of 1.0 Bq/kg was not exceeded for any of the tested samples. Analyzing the content of ^{226}Ra (according to the previous Regulation of the Council of Ministers), the parent isotope of radium, it was found that its values are

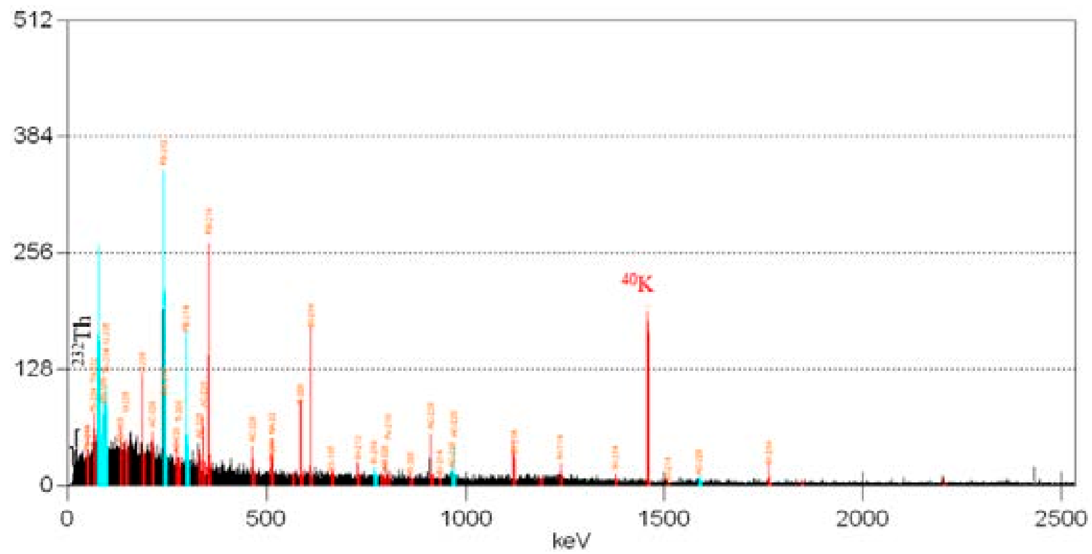

Figure 1. Gamma radiation energy spectrum recorded during the testing of the BZ concrete samples

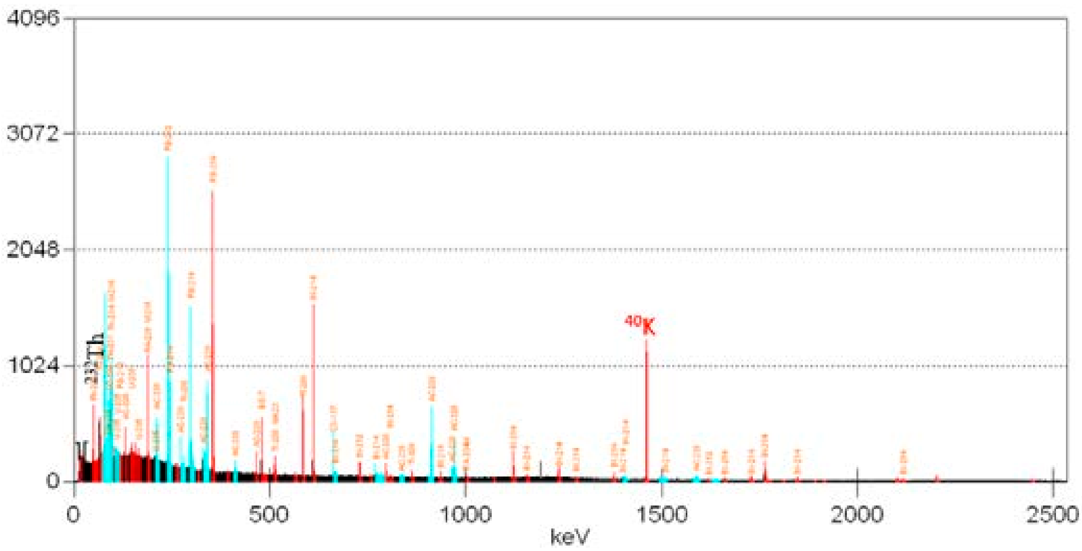

Figure 2. Gamma radiation energy spectrum recorded during the examination of the ash samples from Kraków

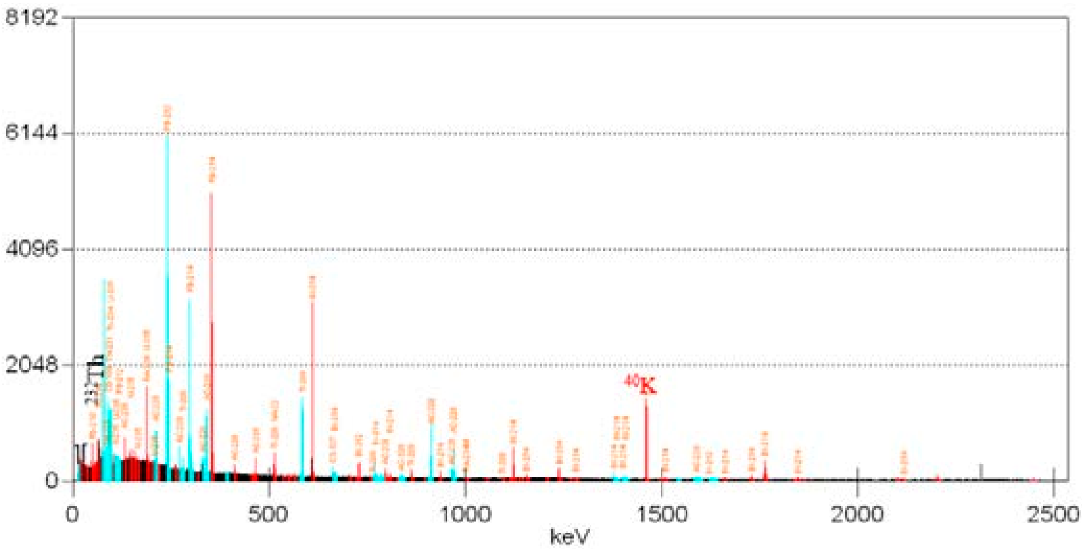

Figure 3. Gamma radiation energy spectrum recorded during the examination of the ash samples from Łódź

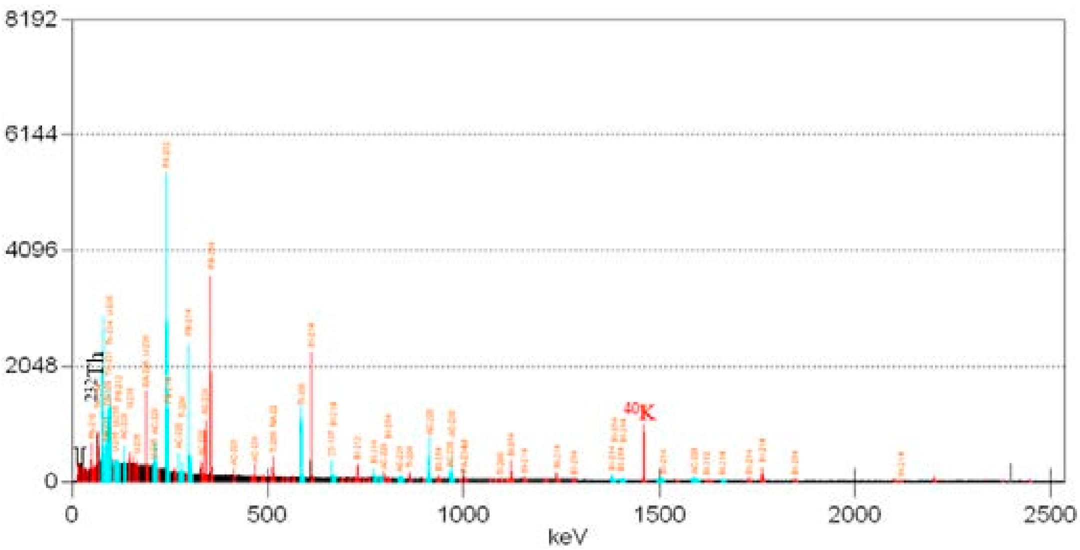

Figure 4. Gamma radiation energy spectrum recorded during the examination of the ash samples from Warsaw

in the range of 18.0–110.0 Bq/kg. The lowest values were obtained for ordinary concrete samples, and the highest for ash samples from Łódź. For none of the tested samples, the limit value of the indicator was not exceeded equal to 240 Bq/kg. Therefore, it can be assumed that the fly ashes from the thermal treatment of sewage sludge used in the research are radiologically safe and can be used in buildings intended for people and animals. Figures 1–4 show the gamma radiation energy spectra recorded during the examination of individual samples with marked isotopes ^{40}K and ^{232}Th.

Summary of studies evaluating properties of fly ashes

Fly ash from the thermal treatment of sewage sludge does not meet the requirements of EN 450-1:2012, not only by definition but also due to its physical and chemical properties. This makes it impossible to use the material as a type II additive to concrete. The use of such ash in concrete technology will be necessary to obtain European technical approval.

Based on the conducted research, it was found that fly ash from the thermal treatment of sewage sludge is a safe and valuable concrete modifier, and the following conclusions were formulated:

1. The leachability from the concrete sample with the addition of fly ash from the thermal transformation of sewage sludge was higher than that from the fly ash alone, but did not exceed the limit values adopted for neutral waste. The leachability of heavy metals from samples containing fly ashes is comparable to or lower than that of concrete without the addition of fly ashes. The migration of heavy metals into the aquatic environment is insignificant and should not be a major problem. However, it is advisable to conduct further research for variable classes and types of concrete and different leaching conditions.
2. None of the fly ashes tested exceeded the limit value of the radioactive isotope concentration index of radioactive potassium K-40, radium Ra-226, and thorium Th-232. Fly ash from the sewage treatment plant in Lodz was the least safe in terms of radiological protection because it had the highest value of *I* index (i.e., 0.92 Bq /kg).
3. Fly ashes from the thermal treatment of sewage sludge from three different sewage treatment plants used for this study are radiologically safe and can be used in buildings intended for people and animals.

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