Technological Reliability of Recycling Anthropogenic Minerals from the Landfills in the Move Towards Green Energy

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The subject of this paper is electromagnetic activation technology for fly ash, which allows for the transformation of the troublesome energy waste into a valuable market product. The move towards green energy, apart from decarbonisation and reduction of carbon dioxide emissions, is facing additional challenges, i.e. disposal and use of accumulated solid waste, which, due to its specific composition, may soon be considered hazardous. Therefore, it is important to recycle them for sale as a market product. Such opportunities are offered by the technology referred to above, which allows for the use of the above-mentioned anthropogenic minerals in concrete products. As demonstrated in the tests presented in the article, the use of activated fly ash does not deteriorate the resilience of concretes, bringing benefits in the form of avoiding emissions of CO_2 which is the result of limiting the use of cement and reducing emissions and transport costs. The liquidation of landfills also fits perfectly with the key points of the circular economy, at the same time implementing the philosophy of priority for secondary ones. High-efficiency and quick operation of furnace waste landfills has multi-threaded ecological effects, which include emission reductions, protection of natural resources and, finally, restoration and reclamation of devastated areas. The second group includes economic benefits, which will also be the result of many overlapping market effects.

Keywords: *fly ash, electromechanical activation, concrete, move towards green energy*

1. Introduction

The move towards green energy in Europe, including Poland, poses many new challenges, both in terms of the transformation to zero-emission energy, but also in terms of ordering the environment after coal energy. It must be implemented in accordance with sustainable development, keeping up with economic progress and, finally, ensuring an increase in the security of raw materials for both Poland and entire Europe. The reliability of the proposed solutions will be a key element that would not be possible without responsible and effective management of natural and anthropogenic minerals. The shift into circular economy model from the linear one is a necessity, and the existing "take-produce-consume-discard" attitudes should be a thing of the past and should be replaced with the philosophy of "use natural only if there are no secondary ones". The type of waste that still poses a big problem but can also be a chance to implement circular economy, is the constantly produced and collected fly ash from the energy industry. Exploitation of ash accumulated in landfills (25 million Mg) [1, 2] and transforming it into valuable market products should also be one of the goals of the proposed transformation. The move towards green energy should lead to ordering the natural environment with effective and reliable use of accumulated waste in the economy.

It is estimated that approx. 20.7 million tonnes of combustion waste, referred to as combustion by-products, are generated in Poland annually of which:

- approx. 11.8 million Mg – combustion by-product in the form of hard coal,
- approx. 8.9 million Mg – combustion by-product in the form of lignite. [3]

The recycling of combustion by-products in our country in recent years has been at the level of approx. 55–57%, but it is mainly using raw material.[4] In 2018, the document "Circular material use rate" was presented, and describing the methodology of waste circulation in the economy. Combustion waste is assigned to a category and it was included under the number W124. This document presents the methodology of the newly developed CMU indicator. This indicator represents the amount of material reintroduced into the economy, measured as a share of the total material consumption. First, we decided to exclude power recycling and landfilling, so that the CMU only covers material recycling. It is thus clear that the only way is to transfer waste to the economy. Another reason are the changes in the European legislation that force the necessity to revise the national waste catalogue again. As for the literal adoption of the list of the European waste catalogue into Polish legislation, not all waste generated in the systems will be included. In such circumstances, it will be necessary to prove that the above-mentioned waste is recognized as non-hazardous waste. Properties that may classify combustion waste as hazardous waste may result from the content of free calcium oxide CaO or Ca (OH)2 – substances which have been classified as dangerous substances according to Regulation (EC) No 1272/2008 due to proven specific target organ toxicity, skin irritation or serious eye damage. The waste will be considered hazardous if it contains at least one substance in a concentration above the limit value, classified in at least one of the hazard classes. Therefore, the technologies for transforming waste into a safe market product are needed. The use of combustion by-products in the economy

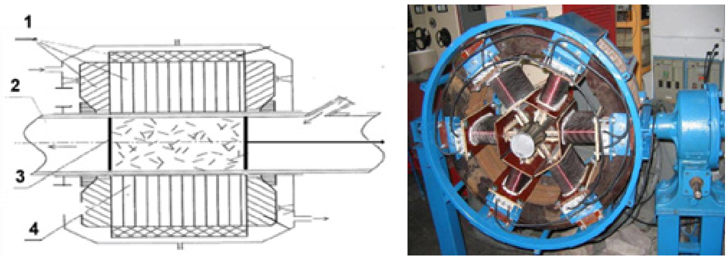

Fig. 1. Simplified diagram of an electromagnetic mill (left) Description: 1 - three-phase inductor, 2 - cylindrical working chamber, 3 - ferromagnetic grinding elements, 4 - inductor pole windings and a physical model of the inductor (on the right) [5,6,7]

Rys. 1. Uproszczony schemat budowy młyna elektromagnetycznego (po lewej) Opis: 1 –wzbudnik trójfazowy. 2 – cylindryczna komora robocza. 3 – ferromagnetyczne elementy mielące. 4 – uzwojenia biegunów wzbudnika oraz model fizyczny wzbudnika (po prawej) [5,6,7]

Tab. 1. Physical and chemical properties of silica fly ash selected for further research

Tab. 1. Właściwości fizyko-chemiczne krzemionkowego popiołu lotnego wytypowanego do dalszych badań

Properties	**Research result**	**Requirements according to PN-EN 450-1: 2009 standard**
Loss on ignition [% mass]	2.62	≤ 5.0 (cat. A) 2 ÷ 7.0 (cat. B) 4 ÷ 9.0 (cat. C)
Chloride(Cl^-) [% mass]	<0.01	≤ 0.10
Sulfuric anhydride as SO_3 [% mass]	0.34	≤ 3.0
CaO_{free} [% mass]	0.05	≤ 2.5
CaO_{react}[% mass]	3.60	≤ 10.0
$SiO_{2\,react}$ [% mass]	40.78	≥ 25.0
SiO_2 [% mass]	53.28	-
Al_2O_3 [% mass]	25.60	-
Fe_2O_3 [% mass]	6.36	-
$SiO_2 + Al_2O_3 + Fe_2O_3$ [% mass]	85.25	≥ 70.0
MgO [% mass]	2.67	≤ 4.0
Soluble phosphates as P_2O_5 [% mass]	0.0017	≤ 0.01
Na_2O [% mass]	1.054	-
K_2O [% mass]	3.088	-
Alkali as Na_2O_{eq} [% mass]	3.085	≤ 5.0
Specific density [kg/m^3]	2170	-
Blaine specific surface [cm^2/g]	3790	-
Fineness (sieve residue 0.045 mm in wet sieving)	34.35	cat. N ≤ 40 cat. S ≤ 12

has another extremely positive ecological aspect, namely it contributes to the reduction of total carbon dioxide emissions. The use of u combustion by-products as replacements for cements in construction materials will reduce emissions from the thermal treatment of components intended for the production of cement. Thus, the turn to green energy also results in problems with the disposal of waste from long-term production of coal energy. Safe and reliable technologies are needed to turn waste into marketable products, and the benefits of these solutions are multifaceted, from saving deposits of natural resources that we replace by combustion by-products to lower carbon dioxide emissions from cement production. Documentation prepared for the purposes of the registration process of combustion by-products in previous years, carried out at the EU agency ECHA, in accordance with the REACH regulation, proved that there are full grounds to consider that combustion by-products and gypsum from flue-gas desulfurization systems should not be classified as hazardous substances according to CLP Regulation No. 1272/2008 (EU) of the European Parliament and the European Commission of 16 December 2008 on classification, labelling and packaging of substances and mixtures. The hazard analysis and PBT/vPvB assessment showed that the combustion by-products and gypsum from flue-gas desulfurization systems do not meet the criteria for being classified as a hazardous substance (in accordance with Directive 67/548/EEC and 1272/2008/EC and do not pose a threat to humans or the environment. Therefore, there are all grounds to apply innovative high-efficiency technologies of transforming waste into utility products. [5,6,7]

2. Electromagnetic activation technology

In order for the material (fly ash) to become a full-value product, e.g. a cement substitute, it must meet a number of properties that are required from such materials, and at the same time be safe for the environment. The improvement of these properties can be achieved by applying, for example, mechanical or electromagnetic activation; by grinding, impact operations, temperature treatment or water treatment. You can combine all these methods with the use of an activator, i.e. an electromagnetic mill. The principle of operation of the electromagnetic mill is to generate a rotating electromagnetic field inside the grinding chamber. Due to magnetic induction, the grinding elements polarize, becoming magnetic dipoles, thanks to which they are attracted with a certain force by this field. As the magnetic induction increases inside the chamber, the chaotic, intense movement of fine ferromagnetic grinding elements is induced. Small size, proper shape and maintaining the appropriate proportions of dimensions allow to quickly achieve high accelerations and a maximum speed of approx. 3000 rpm (50Hz) [8]. The electromagnetic activator installation consists of two basic elements: a working chamber with grinding rods, i.e. magnetic steel rods and an electromagnetic field inducer (stator with salient pole windings). The working chamber is made as a closed non-ferromagnetic tube in which grinding media, excited by the field, move along with the activated material. Inside the working area, the activated material is processed: mechanically – by hitting it with high frequency and speed; thermally – by heating the grinding media under the influence of magnetic induction and mutual collisions,

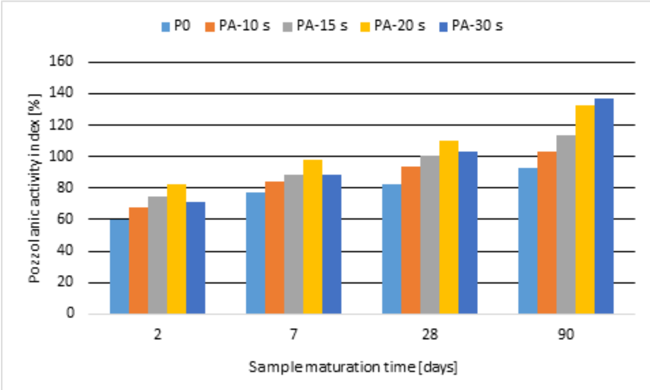

Fig. 2. Index of pozzolanic activity of reference ash (P0) and its modification (PA) after 10; 15; 20 and 30 sec. of mechanical activation
Rys. 2. Wskaźnik aktywności pucolanowej popiołu odniesienia (P0) oraz jego modyfikacji (PA) po 10; 15; 20 i 30 sek. mechanicznej aktywacji

as well as by the influence of the electromagnetic field. The scheme of the construction of the electromagnetic activator and the view of the field inductor are demonstrated in Fig. 1.

Basic technical parameters of the device and the set operating parameters during activation:

- magnetic induction in the central axis of the working chamber zone approx. 0.095T,
- dimensions of the working chamber 80 mm x 140 mm (the volume of the working chamber is about 0.71 dm^3),
- grinding media – steel bars with a diameter of 1 mm and a length of 10 mm,
- degree of filling the volume of the working chamber with grinding media – 10%,
- degree of filling the working chamber volume with feed (fly ash) – 60%,
- fly ash feed weight – 498 g,
- ratio of grinding media mass to feed mass – 0.24.
- activation time: 10 s, 15 s, 20 s, 30 s.

3. Methodology and research material

The primary objective of the research was to carry out electromagnetic activation on fly ash and cement samples, and then to produce concrete with activated fly ash and a reference material based on classic cement formulas. In the next stage, the samples were subject to tests, which are carried out as standard for classic concretes, in order to confirm their usefulness in the economy. The main parameter subject to tests was the durability test, which is decisive when to comes to the possibility of using concretes. These tests were carried out for all produced materials: for activated fly ash and for reference samples. In addition, a number of other studies were performed to describe the effects of the activation process on the end product. Finally, the optimum of activation was found based on the time criterion and increase in specific surface area and reduction of water demand. The subject of the research was silica fly ash subjected to mechanical activation in an electromagnetic mill and reference ash (not activated), which was then used as an additive replacing some Portland cement. The physicochemical properties of the activated ash are presented in Tab. 1.

For comparative tests and for preparing mixtures, CEM I 42.5 R Portland cement was used with a specific surface according to Blaine – 4000 cm^2/g, the content of C3A – 8.78% and Na_2Oeq – 0.87% of the total weight, CEN standard sand according to PN-EN 196-1:2005 standard, tested reference ash and its modifications after the mechanical activation process during the following times: 10; 15; 20 and 30 s. The specific surface area of the reference ash determined by the Blaine method was 3790 cm^2/g, and after its mechanical activation in time 10; 15; 20 and 30 s respectively: 4920; 5490; 6200; 6940 cm^2/g. Standard mortars with a w/s ratio of 0.5 and a binder/sand weight ratio of 1:3 were prepared for the tests. The composition of the mortars for the tested beams is presented below:

- reference mortar (model): 450 g of cement, 1350 g of standard sand, 225 cm^3 water
- mortar after replacing 25% of the cement mass with fly ash: 337.5 g of cement, 112.5 g of ash, 1350 g of standard sand, 225 cm^3 water.

The index of pozzolanic activity of the reference ash and its modification after the mechanical activation process was determined according to the PN-EN 450-1:2009 standard [9]. This indicator is expressed as a percentage ratio of the compressive strength of 40×40×160 mm bars made with a mixture of 75% reference cement and 25% of fly ash, to the strength of standard mortar bars of the same age made with 100% reference cement. The bars in the moulds were stored for 24 hours in humid air, and then, after demoulding, they were stored in water until the resilience tests were carried out.

The samples were subjected to compressive strength tests of 2; 7; 28 and 90 days of maturation in water according to PN-EN 196-1:2005 [10].

The water demand of the reference ash and its modification after the mechanical activation process was determined according to the procedure included in Annex B to the PN-EN-450-1:2009 standard [9].

Studies of the morphology and the texture of fly ash samples and its modifications were carried out using an ISM (IEOL) scanning electron microscope equipped with an energy dispersion spectrometer (EDX), which allowed to determine the chemical composition of selected grains of materials.

4. Results and discussion

The main components of the tested fly ash (Tab. 1.), converted into oxides, are: SiO_2, Al_2O_3, CaO, SO_3, Fe_2O_3, MgO, N_2O and K_2O. They come from the decomposition of clay

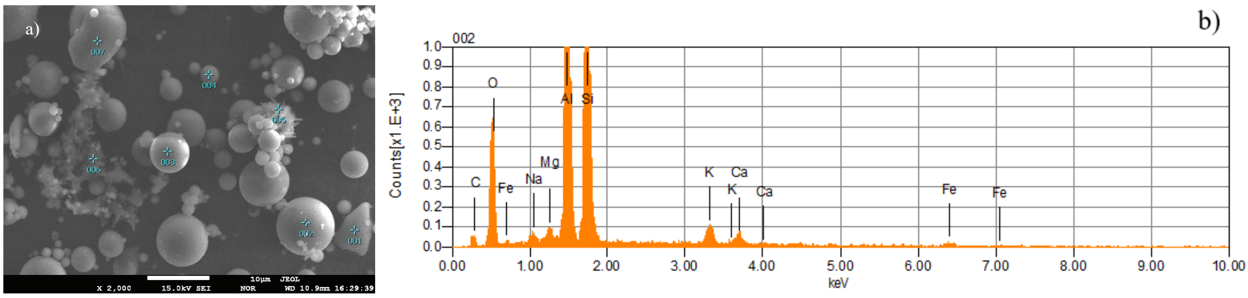

Fig. 3. SEM micrograph and EDS spectrum of reference ash

Rys. 3. Mikrofotografia SEM oraz widmo EDS popiołu odniesienia

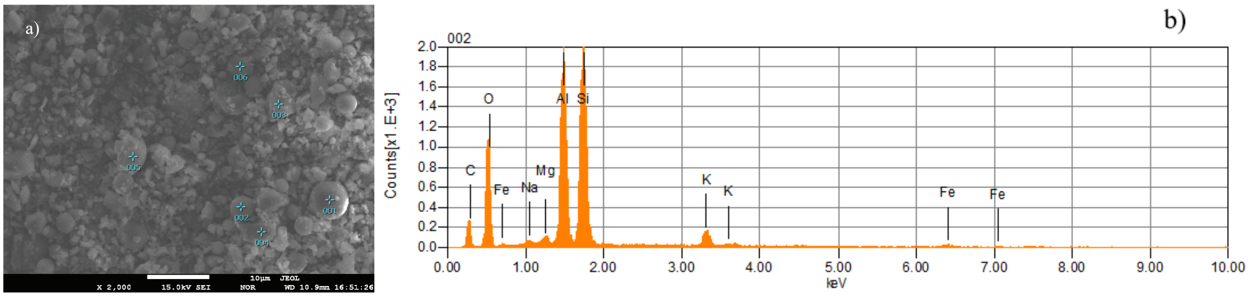

Fig. 4. SEM micrograph and EDS spectrum of ash after activation

Rys. 4. Mikrofotografia SEM oraz widmo EDS popiołu po aktywacji

minerals, pyrite and calcite, which are the inorganic components of coal. The basic chemical composition of this ash is typical for siliceous ashes from coal combustion in pulverized coal boilers without desulphurization. The analysis of the data contained in Tab. 1 shows that the tested silica fly ash meets all the requirements of the PN-EN 450-1:2009 [9] standard, which allows the ash to be used in concrete technology. The relatively low result of the loss on ignition (2.6%) qualifies this ash into category A, and the fineness (34.35%) to category N.

Fig. 2 shows the results of the pozzolanic activity of the reference ash and its modifications after mechanical activation, after 2; 7; 28 and 90 days of hardening.

The value of the pozzolanic activated ash index was higher than in the case of the reference ash, regardless of the ash activation time and the maturation time of the mortars. The dynamics of changes in the value of the pozzolanic index was constant for most of the activated ashes, with the exception of the ash activated within 30 s. The ash pozzolanic activity index increased with the increase of activation time (specific surface area) until the optimum for ash was reached after 20s of activation (approx. 6200 cm2/ g). For which, if compared with the reference ash, there was a significant increase in the pozzolanic activity index, which after 2; 7; 28 and 90 days of hardening the mortar reached the following values: 83%, 98%, 110% and 132%. On the other hand, increasing the activation time to 30 s (6940 cm^2/ g) caused the reduction of pozzolanic activity in the first stages of hardening of the mortar. Compared to the reference ash, the pozzolanic activity index, 2 each; 7; 28 and 90 days of hardening the mortar reached the following values: 71%, 88%, 103% and 137%.

Photographic documentation of SEM tests of reference ash (Fig. 3a) indicates high heterogeneity of the grain size within the range of 0 ÷ 170 μm. In the analysed sample of reference ash (Fig. 3a), both single grains and their agglomerates with irregular topography are observed.

In this ash the following morphological types of grains are represented in varying proportions: massive spherical; porous spherical and irregular rounded, irregularly sharp-edged, spongy, sharp-edged massive. Bulky, spherical and irregularly rounded spherical grains are made of glaze (amorphous or containing also slightly crystalline mullite). The chemical composition of the glaze (Fig. 3b) is essentially: Si, Al and K. Some of the grains also contain Fe, which indicates the presence of ferrous phase overgrowths. Fig. 3a shows that the grain composition of the ash is dominated by massive spherical grains with diameters of 1 ÷ 10 μm. The surface of most of these grains is clean, fine (less than 1 μm) crusts or sulphate phase inclusions are less often visible on it. Irregular, sharp-edged and spongy grains are generally found in the ash fraction thicker than 40 μm and are considered as non-melted clay mineral aggregates [11]. The coarse fractions also include massive, sharp-edged quartz grains and skeletal grains that are fragments of unburned carbon in a porous interior with fine spherical enamel grains. The porous, spherical and irregularly rounded grains are hollow (cenospheres) or may be filled with finer spherical grains (plenospheres). The authors of the study [11] believe that porous grains are formed from a viscous alloy with a high proportion of the gas phase, and massive grains – from a liquid alloy. Bulky and porous spherical grains occur essentially individually and form aggregate clusters composed of grains of various sizes (Fig. 3a).

The photographic documentation of the SEM tests of ash after mechanical activation indicates significant differences in the surface texture of the activated ash in comparison with the reference ash (Fig. 3a). Based on a comparative analysis of the photomicrographs from the SEM test of the reference ash (Fig. 3a) and after the modification of the ash (Fig. 4a), it was found that as a result of its mechanical activation, a significant development of its surface occurred, mainly due

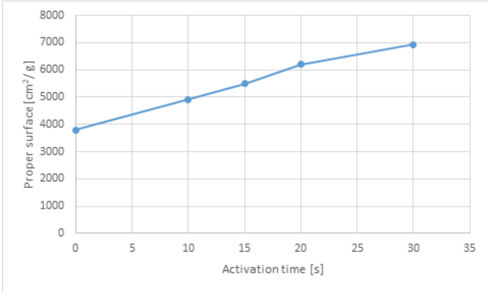

Fig. 5. The effect of activation time on the ash specific surface

Rys. 5. Wpływ czasu aktywacji na powierzchnię właściwą popiołu

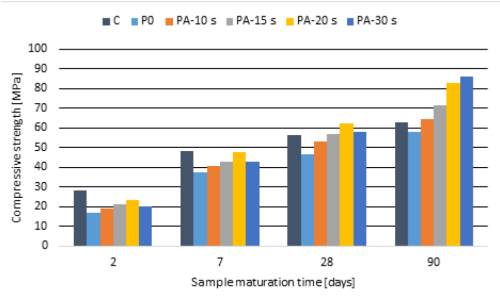

Fig. 6. Compressive strength of mortars: cement C; cement-ash with reference ash (P0); cement-ash with ash activated in 10 s (PA-10), 15 s (PA-15), 20 s (PA-20), 30 s (PA-30)

Rys. 6. Wytrzymałość na ściskanie zapraw: cementowej C; cementowo–popiołowej z popiołem odniesienia (P0); cementowo–popiołowych z popiołem aktywowanym w czasie 10 s (PA-10). 15 s (PA-15). 20 s (PA-20). 30 s (PA-30)

to the grinding of coarse grains, i.e. porous, spherical and irregularly rounded grains with empty interiors (cenospheres) as well as filled with fine (approx. 1 μm) with spherical grains (plenosphere). Dense aggregates of aggregate grains were also disagglomerated.

However, no significant fragmentation of the fine fraction (1÷10 μm) of the massive spherical grains was found, but only the cleaning of their surface of crusts and sulphate phase inclusions. From the analysis of the photomicrograph after mechanical activation (Fig. 4a) it transpires that the most susceptible to destruction are microspheres with empty interiors and small spherical grains inside. This is evidenced by numerous, relatively large, compared to massive spherical grains, fragments of crushed spherical shells of these grains. This is due to their significantly lower compressive strength compared to spherical massive grains, because the thickness of the walls of cenospheres and plenospheres does not exceed 10% and their diameters. Moreover, the fractures of such microspheres reveal the presence of numerous previously closed pores in their walls, which also reduce the mechanical strength of these grains. The deagglomeration of dense clusters of spherical grains and the opening of closed spaces inside the grains containing numerous smaller grains increases the proportion of the activated ash fine fraction of spherical massive grains, which are essentially the amorphous phase of the enamel rich in Si, Al, K. The key element determining the success of the application of fly ash is the increase of the surface area correct after electromagnetic activation. Its increase during activation is shown in Fig. 5.

Along with the extension of the activation time, a systematic increase in the specific surface area can be observed. It is caused by at least two reasons. Firstly, through grain cleaning, which opens the internal pores, and by the fragmentation resulting from the interaction of grinding media and grains with each other. The increase is practically linear and with 30 seconds of activation the value of the specific surface area is in fact doubled. Compressive strength tests performed are shown in Fig. 6. It was found that the compressive strength of mortars with reference ash was lower than in the case of mortars with activated ash, regardless of its activation time or the maturation time of the mortars.

Mortars with an addition of activated fly ash reached the resilience comparable to the resilience of the cement mortar without additives after 28 days of maturation. The analyses carried out demonstrate that the compressive strength of standard mortars with the addition of activated ash gradually increases with the increase of its activation time (an increase in the specific surface area).

As with the poozzolanic activity, until the optimum for ash is reached after 20s of activation (approx. 6200 cm²/g). A further increase of the specific surface results in the reduction of mortar strength in the initial stages of hardening of the mortar.

This effect can be explained by the fact that in the range of ash activation times (up to 20 s), by the number of small spherical particles with an increased content of the amorphous phase (increased pozzolanic reactivity), released as a result of crushing plenospheres coatings, increases significantly. This is also confirmed by the fact that up to a certain level (activation time 20 s), as the specific surface of the activated ash increases, its water demand gradually decreases (Fig. 7) from the level of 96.9% to 88.9% (i.e. by 8.25%), and then for the 30 s activation time this factor increases again.

This can be explained by an increase in the amount of irregularly shaped particles (excessive grinding), which require more water particles to form a shell around the grain. It is also

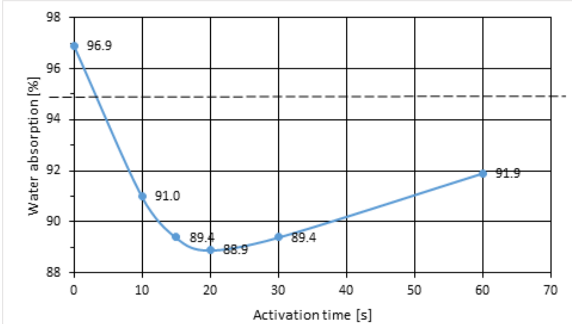

Fig. 7. Changes in fly ash demand depending on the time of its activation
Rys. 7. Zmiany wodożądności popiołu lotnego w zależności od czasu jego aktywacji

worth noting that all the obtained water demand results are much lower than the standard requirements. The tendency to reduce the water demand of ashes in the initial period of their mechanical activation and subsequent growth was also found in other publications [12].

Based on the above results, it can be concluded that the optimum fly ash activation time is 20 s, after exceeding this time, the demand for water increases, and the pozzolanic activity and compressive strength index decreased or slightly increased in the last stage of maturation.

The results of experimental studies have demonstrated that there is a relationship between the pozzolanic activity of ashes and the degree of their fragmentation. An increase in pozzolanic activity was found to reach the value of the Blaine specific surface area of 6200 cm^2/g, followed by its gradual reduction due to the progressive increase in the water demand of the activated ash due to an increase in porosity. A gradual increase in the proper surface area of the modified fly ash improves its pozzolanic properties by increasing the ability to react with $Ca(OH)_2$ and reducing water demand. It also positively influences the quick increase of the early strength, increases the strength parameters of the finished products, while reducing the water demand and improving the liquefaction of the mixture. Replacing 25% of cement with it in the mortar only slightly reduces the early strength, while in the further maturation period, the strength increases exceed the results obtained for pure cement.

5. Summary

The research carried out with the use of waste fly ash and electromagnetic activation technology indicates that there is a possibility of obtaining a valuable product from fly ash, which today is a kind of ecological and financial problem for the energy sector. The Green Deal provides for a gradual decarbonisation of the energy sector and the transition to renewable energy sources. On the other hand, apart from the elements strictly related to energy conversion and emissions, which are commonly discussed, there is also the issue of solid waste. In order to be effective, green transformation must be comprehensive, safe and reliable, so today it is necessary to indicate technologies that can turn troublesome waste into a market product. The presented electromagnetic activation technology shows the potential possibilities of treated waste. It indicates that the green transformation can also be an opportunity to protect valuable raw materials by using secondary anthropogenic minerals already extracted, which pose a problem, with an additional positive effect of avoided CO emissions$_2$ in cement production. A comprehensive approach to the issue of the Green Deal governance will create a safe system of transformation and will select reliable technologies for the exploitation of anthropogenic resources. Fly ash is a valuable material that, after appropriate treatment, can be used locally in road construction, which will again result in the protection of natural deposits and the avoided emissions from transporting aggregates from longer distances. The old slogan "grey to gold" is still valid and how eloquent it sounds in the new version, namely "grey to green".

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Niezawodność technologiczna odzysku minerałów antropogenicznych ze składowisk w zielonym zwrocie energetycznym

Przedmiotem artykułu jest technologia aktywacji elektromagnetycznej dla popiołów lotnych, pozwalająca kłopotliwe odpady energetyczne przekształcić w wartościowy produkt rynkowy. Przed zielonym zwrotem energetycznym poza samą dekarbonizacją i redukcją emisji ditlenku węgla stoją wyzwania dodatkowe, utylizacji i wykorzystania nagromadzonych odpadów stałych. które ze względu na swój specyficzny skład w niedługim czasie mogą być uznane za niebezpieczne. Dlatego też ważne jest aby zawracać je do gospodarki w postaci produktu rynkowego. Takie szanse daje opisana technologia, która pozwala na stosowanie z powodzeniem ww. minerałów antropogenicznych w wyrobach betonowych. Jak wykazały przedstawione w artkule badania, zastosowanie aktywowanych popiołów lotnych nie pogarsza właściwości wytrzymałościowych betonów, przynosząc korzyści w postaci unikniętej emisji CO_2 wynikającej z ograniczenia stosowania cementu oraz redukcji emisji i kosztów transportowych. Likwidacja składowisk wpisuje się również doskonale w założenia gospodarki obiegu zamkniętego realizując jednocześnie filozofię pierwszeństwa dla wtórnych. Wysokosprawna i szybka eksploatacja składowisk odpadów paleniskowych przynosi wielowątkowe skutki ekologiczne, na które składają się redukcje emisji, ochrona zasobów naturalnych i wreszcie przywracanie i rekultywacja terenów zdewastowanych. Drugą grupa, są korzyści ekonomiczne, które również będą wypadkową nakładających się na siebie wielu efektów rynkowych.

Słowa kluczowe: *popiół lotny, aktywacja elektromechaniczna, beton. zielony zwrot energetyczny*