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Waste from the cement industry – a component of sealing grouts

Introduction

The production of cement produces a byproduct in the form of small amounts of dust captured by the dedusting system of the kiln.

The preferred and most common method of using cement kiln dust (CKD) is its re-use in the cement production process in amounts which do not cause deterioration of the quality of the final product. Complete recycling of CKD into the production process is technologically possible but not practiced due to the strict limits on the permissible amounts of alkali in the mix of raw materials (Maslehuddin *et al.* 2008). Cement kiln dust (CKD) can be used as a raw material in the production of building materials such as bricks, lightweight aggregate, mortar, concrete, and bituminous mixtures. In civil engineering, it can be used to stabilize clay soils (Rahman *et al.* 2007).

A high density of CaO makes it a very effective material for stabilization and hygienization of sewage sludge, while in agriculture it may be used as a component of fertilizers and animal feeds (Bożym *et al.* 2011). Dust from cement kilns like fluidized fly ash can also potentially be used as a material for mineral sequestration of CO₂ (Uliasz-Bocheńczyk 2009; Uliasz-Bocheńczyk and Mokrzycki 2011, 2013).

Grout additives containing CKD are an example of a very interesting application of the aforementioned dust (Stryczek *et al.* 2009). The grouts are widely used in drilling; they can

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be used for sealing the spaces between shroud tubes and for preventing leakage of drilling fluid. In civil engineering they can be used for the construction of municipal and industrial waste landfills, especially in the case of storage of toxic waste. In the hydropower industry, they can be used for minimizing or eliminating flow paths. In communication engineering they can be used for strengthening purposes, and for the demolition of bridge abutments and piers.

Fluidized bed fly ashes are now used mainly in building materials underground mining in the form of aqueous suspensions, as well as in road works (Emitor 2013). These wastes may also be used as a stabilizer for soils (Filipiak 2011, 2013).

This article presents the results of studies on the physicochemical properties of grouts based on fly ash from hard coal combustion in fluidized bed boilers, with the addition of cement kiln dust from a dry process cement plant, specifically as employed in mining technologies.

1. Characteristics of dust from the cement industry

Cement kiln dust (CKD) is a particulate material consisting of particles of clinker, calcium, and other alkaline chemicals. It is very diverse in terms of chemical composition and grain size (Maslehuddin et al. 2008). The share of individual compounds depends largely on the raw materials used in the cement production technology, the fuel type, and the method of dust removal. Most frequently, dust contains unreacted raw material components, clinker dust, ash from fuels containing sulfates, halides, and other volatile compounds.

Table 1. Chemical composition of CKD [%]

Tabela 1. Skład chemiczny pyłów cementowych [%]

Component	Maslehuddin et al. 2008	Aidan and Trevor 1995	Collins and Emery 1983	Peethamparan et al. 2008	Lachemi et al. 2010
SiO ₂	17.1	15.84	14.5	11.91–16.42	13.1
Al ₂ O ₃	4.24	3.57	4.10	2.17–4.66	5.3
Fe ₂ O ₃	2.89	2.76	2.00	1.71–2.34	2.3
CaO	49.3	63.76	40.5	37.35–55.00	58.1
MgO	1.14	1.93	1.55	1.24–2.68	3.3
SO ₃	78.2	1.65	6.50	5.8–14.62	10.6
Na ₂ O	3.84	0.33	0.44	0.17–0.81	0.7
K ₂ O	2.18	2.99	4.66	1.43–7.0	2.8
Cl ⁻	6.9	1.09	–	0.35–3.26	–
L.O.I	15.8	5.38	22.9	3.92–29.63	3.1

(Bożym 2011). CKD from the dry process contains a higher proportion of calcium than dust derived from the wet process (Siddique 2006). The composition of the dust from cement kilns, according to several authors, is presented in Table 1.

CKD contains considerable amounts of alkali and trace metals, such as arsenic, cadmium, lead, mercury, thallium, selenium, zinc, and radionuclides, which are typically at a concentration of less than 0.05 wt. %. This factor is conditioned by the type of pulverized material and the recirculation in the kiln system. These are highly alkaline dusts characterized by a pH of about 12 (Bożym 2011).

2. Research methodology

Grouts must have specific physico-chemical properties dependent on the conditions of use. Selecting the right sealing grout is not an easy task and, therefore, it is not possible to indicate a universal formulation; but grouts for injection should have the following technological parameters (Stryczek and Gonet 2000):

- ◆ suitable rheological properties,
- ◆ suitable mechanical properties,
- ◆ sufficient flexibility under given conditions and over a sufficiently long period,
- ◆ resistance to aggressive waters and micro-organisms,
- ◆ ability to bond in high humidity,
- ◆ adjustable time of injection and binding,
- ◆ no toxic substances,
- ◆ non flammability,
- ◆ the simplicity of the technology of preparing them under difficult field conditions,
- ◆ easily transporting the components to the operation site,
- ◆ relatively low cost of implementation,
- ◆ specified range of penetration of the grout in the rock mass,
- ◆ adequate adhesion for sealing the surface,
- ◆ low shrinkage as well as the possibility of use of expansive material.

Grouts typically consist of the following components (Stryczek and Gonet 2000):

- ◆ binder,
- ◆ solvent,
- ◆ agents modifying the properties of the fresh and hardened sealing grout,
- ◆ fillers.

Common cements, slag-alkali binder, and calcareous fly ash are most frequently used as a binder. Depending on the application, various modifying additives and fillers are used.

The aim of the laboratory tests was to determine the effect of CKD on the selected properties of fresh and hardened grouts made from fluidized bed fly ash. The tested grouts were prepared using a water/cement ratio ranging from 1.1 to 1.4 for the three adopted contents of CKD of 25, 50, and 75%.

Fresh grouts were subjected to basic research; the following parameters were determined:

- ◆ density,
- ◆ fluidity,
- ◆ bleeding rate,
- ◆ setting time.

In order to investigate the effect of CKD on the hardened grout, the compressive strength of the samples with the lowest water/solid ratio was examined. Laboratory tests for the measurement of rheological parameters of fresh sealing grouts were carried out according to the following standards: *PN G-11011:1998 Mining – Materials for Backfilling and Caulking of Cavings – Requirements and Tests* and *PN-EN 196-1 Methods of testing cement*.

X-ray diffraction and thermogravimetric analyses of phase composition were also performed. The measurements were performed using a Netzsch STA 449F3 Jupiter thermal analyzer and a QMS 403C Aëolos for routine analysis of gases. The measurements were performed under a dynamic synthetic air atmosphere with a flow of 40 ml/min. The rate of temperature increase was 10°C/min. Sample weight was approximately 130 mg. X-ray diffraction studies were performed on a Philips PW1050/70 diffractometer in the measuring range $5-65^{\circ}2\Theta\text{CuK}\alpha^1$.

The aqueous extracts of the tested waste were prepared using the procedure described in the Polish standard *PN-EN 12457-2:2006 Characterization of Waste – Leaching – Compliance Test for Leaching of Granular Waste Materials and Sludges – Part 2*. One stage batch test at a liquid to solid ratio of 10 dm³/kg for materials with a particle size below 4 mm (without or with size reduction).

3. Characteristics of materials for the production of sealing grouts

This study used fly ash from hard coal combustion in a atmospheric fluidized bed boiler (AFBCC – fly ashes) (Table 2; Fig. 1) as a grout binder. To determine the effect of dust as a modifier, CKD from a dry process cement plant has been selected (Table 2; Fig. 1).

The CKD used in the study was characterized by a high content of CaO (55.2%) and Cl⁻ (4%), while the fluidized bed fly ash was characterized by a high content of SiO₂ (43.9%) (Table 2).

Fluidized bed combustion fly ash was characterized by a Blaine specific surface area of 5,900 cm²/g, while CKD was 9,300 cm²/g.

¹ The analysis of pollutant leachability, the values of pH, XRD and TGA analysis were conducted in the Department of Building Materials of the Faculty of Materials Engineering and Ceramics at the AGH University of Science and Technology.

Table 2. Chemical composition of materials used for the research [%]

Tabela 2. Skład chemiczny materiałów wykorzystanych w badaniach [%]

Component	AFBCC – fly ash	CKD
SiO ₂	43.9	16.8
Al ₂ O ₃	12.7	3.9
Fe ₂ O ₃	12.0	1.9
CaO	12.1	55.2
MgO	0.40	1.5
SO ₃	3.99	1.7
Na ₂ O	4.30	0.3
K ₂ O	4.1	4.1
Cl ⁻	0.58	4.0
LOI	b.d.	19.7
Density [Mg/m ³]	2.6	3.1

The results of the grain size distribution analysis of the tested CKD and fluidized bed combustion fly ash showed that the most frequent grain size was in the range of 0–5 μm (Fig. 1).

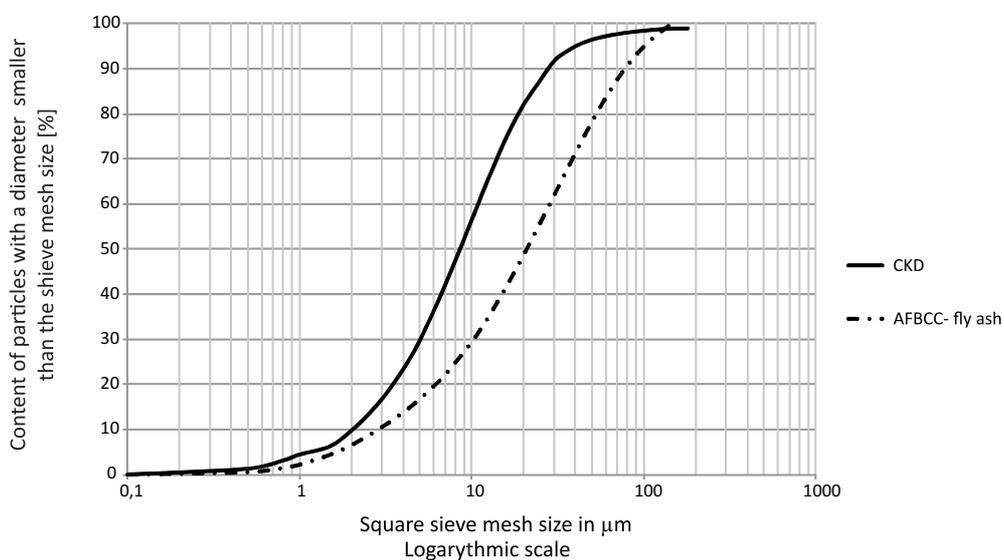


Fig. 1. Size distribution curve of AFBCC – fly ashes and CKD

Rys. 1. Krzywa składu ziarnowego popiołu fluidalnego i pyłu cementowego

Table 3. The results of the studies determining the technological parameters of grouts
 Tabela 3. Wyniki badań określających parametry technologiczne zaczynów

Type of grout	Water/solid ratio	Density [kg/m ³]	Fluidity [mm]	Bleeding rate [%]	Setting time [h]		Compressive strength after 28 days [MPa]	Flexural strength after 28 days [MPa]
					Initial	Final		
100% AFBCC – fly ashes	1.3	1.31	185	0	103	113	0	0
	1.4	1.32	215	0	105	123	–	–
75% AFBCC – fly ashes + 25% CKD	1.2	1.35	180	0	39	49	2.12	6.1
	1.3	1.33	200	0	39	53	–	–
	1.4	1.33	238	3	49	63	–	–
50% AFBCC – fly ashes + 50% CKD	1.1	1.39	200	0	50	62	1.34	4.7
	1.2	1.37	215	0	50	63	–	–
	1.3	1.35	225	2	55	65	–	–
25% AFBCC – fly ashes + 75% CKD	1.2	1.40	230	4	1.2	60	0	2.4

4. Research results

4.1. Technological parameters of cement-fly ash grouts with the addition of CKD

The research showed that the addition of cement kiln dust affects the technological properties of cement-fly ash grouts (Table 3). The most important parameter determining the use of grouts is fluidity. When choosing the water-binder ratios, the study referred to the degree of required fluidity for grouts in the range between 18 and 24 cm (Stryczek and Gonet 2000). The fluidity of the tested grouts increases with the addition of CKD, which may be due to a greater surface area of the dust. The biggest bleeding ratio, 4%, was observed in the grout with the greatest share of CKD, amounting to 75%.

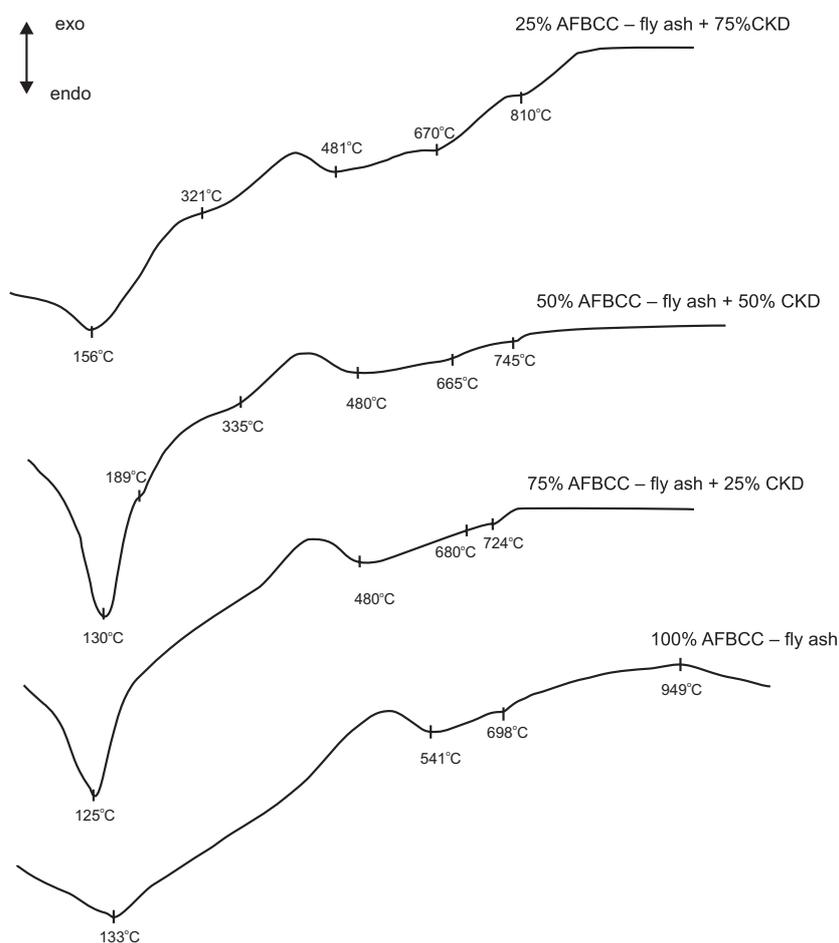


Fig. 2. DTA curves of grout used for the research

Rys. 2. Krzywe DTA badanych zaczynów

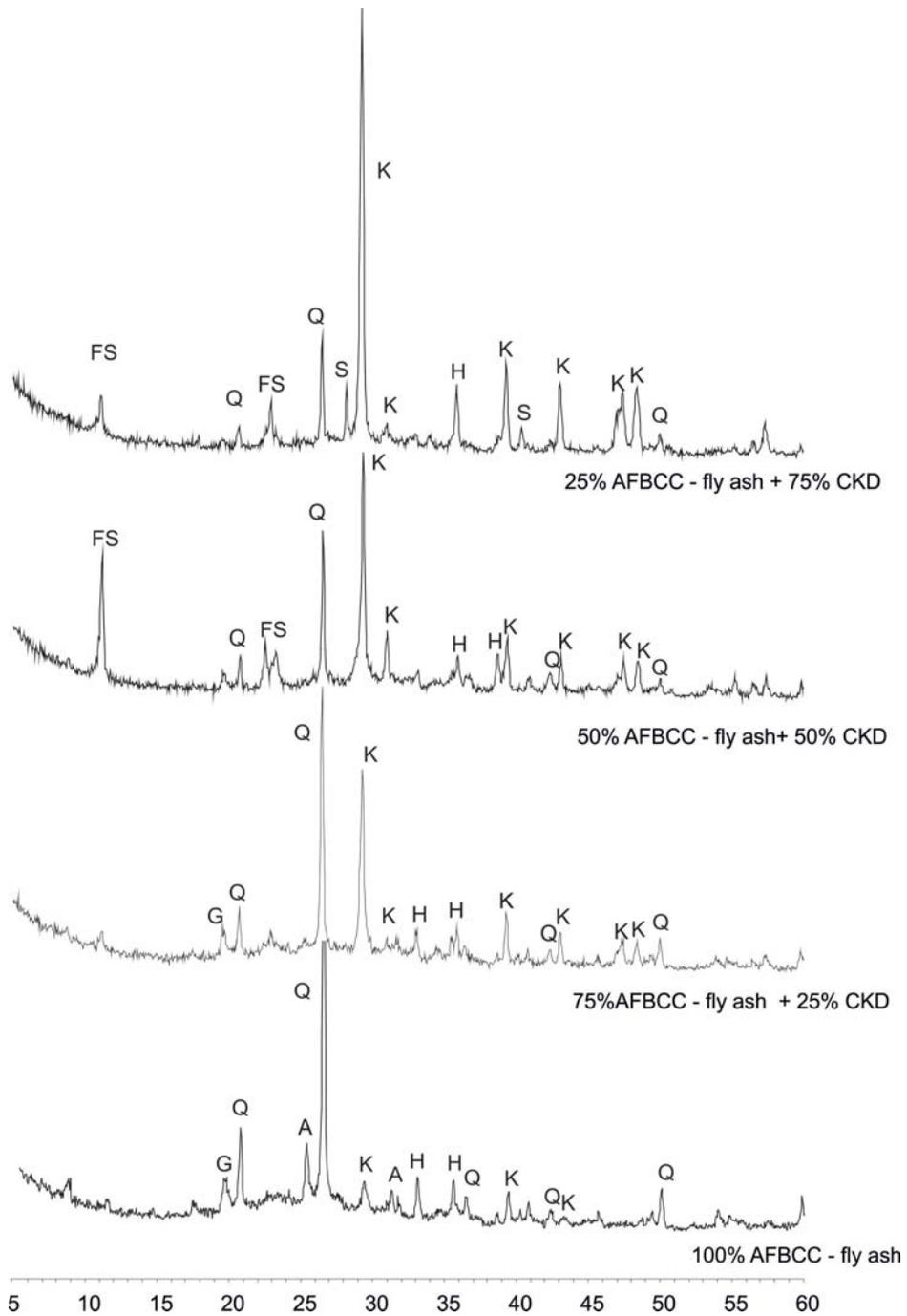


Fig. 3. Diffractograms of grouts used for the research
 K – calcite, Q – quartz, A – anhydrite, H – hematite, G – gypsum, FS – Friedel’s salt, S – sylvite

Rys. 3. Dyfraktogramy badanych zaczynów

Setting time is a very important parameter used in the selection of the proper grout recipe. All of the mixtures containing CKD are characterized by extended setting time. The setting time increases along with increasing amounts of CKD. Lastly, the introduction of dust causes a slight increase in the compressive strength of the tested grouts.

4.2. Phase composition of the grouts

All of the tested samples showed endothermic effects with a maximum at 125–156°C, which can be related to the presence of gypsum or C-S-H (Giergiczny 2006). The grouts with a higher content of particulate matter demonstrated endothermic effects with a maximum at 321°C (grout – fluidized bed combustion ash 50% + CKD 50%) and 335°C (grout – fluidized bed combustion ash 25% + CKD 75%) associated with dehydration of the $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{CaCl}_2 \cdot 10\text{H}_2\text{O}$ phase, which was not present in the grouts with 100% ash content and 25% dust content (Fig. 2).

The endothermic effect observed for all curves with a maximum at about 660°C is associated with decarbonatization of complex compounds with carbonates, which precedes a smaller effect with a maximum around 700–900°C indicating the decomposition of calcium carbonate (Ubbriaco and Calabrese 2000). The endothermic effect with a maximum at about 480–541°C is associated with the presence of portlandite.

The results of X-ray studies (Fig. 3) showed that the main phases in clean grouts prepared with ash from the fluidized bed combustion of coal include calcite, quartz, anhydrite, hematite, and gypsum. This is confirmed by the results of other authors (Giergiczny 2006; Iwanek et al. 2008; Rajczyk et al. 2006).

The introduction of dust resulted in the appearance of phases containing chlorine – the $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{CaCl}_2 \cdot 10\text{H}_2\text{O}$ phase – Friedel's salt, and KCl – sylvite, which is due to its high content in CKD.

4.3. Leachability

The leachability of impurities from grouts is a very important factor in the possibility of their use. To determine the effect of the addition of CKD on the leachability of chemical impurities, the composition and characteristics of the water eluate of grouts were examined. The results were compared with the requirements of the Polish standard PN-G-11011:1998 (Table 4).

The leachability of impurities of the tested grouts meets the requirements of the standards for As, Cd, Cu, Hg, Pb, Cr(III), and sulfides. When it comes to sulphates, the standard is met by the grouts containing dusts. Increasing the dust content increased the leachability of chloride until the permissible value of the Polish standard PN-G-11011:1998 was exceeded by the grouts containing 50 and 75% dust. The pH value in all cases exceeded the value permissible by the standard.

Table 4. Leaching of chemical impurities from grouts used for the research

Tabela 4. Wymywalność zanieczyszczeń z badanych zaczynów

Type of designation	AFBCC – fly ash	Type of grout			Limit values of leachability in the PN-G-11011 standard
		75% AFBCC – fly ash + 25% CKD	50% AFBCC – fly ash + 50% CKD	25% AFBCC – fly ash + 75% CKD	
As [mg/dm ³]	<0.05	<0.0001	<0.0001	<0.0001	0.1
Cd [mg/ dm ³]	<0.005	<0.00022	0.00003	<0.00003	0.02
Cu [mg/ dm ³]	0.06	0.00012	0.00011	0.0009	0.5
Hg [mg/ dm ³]	<0.05	<0.00003	0.000044	<0.00003	0.05
Pb [mg/ dm ³]	<0.05	0.0001	0.0034	0.0144	0.5
Cr(VI) [mg/ dm ³]	0.71	0.10	0.25	0.24	0.5
Cr(III) [mg/ dm ³]	0.04	0.08	0.00	0.02	0.1
Sulphates [mg/ dm ³]	1 589	206	272	259	500
Chloride [mg/ dm ³]	35	798	1 981	1 950	1 000
Sulfides [mg/ dm ³]	<0.1	<0.1	<0.1	<0.1	<0.1
pH	12.5	12.3	12.5	13.1	6.0–12.0

The addition of dust had little impact on the leachability of impurities. A reduction in leachability as a result of the addition of dust was identified for Cd, Cu, Hg, Cr(V), and Cr(III), which can be explained by immobilization of C-S-H (Deja 2002). The addition of dust caused an increase in the leachability of chlorides related to their high content in the studied dust, despite part of them binding in the new phases – the $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{CaCl}_2 \cdot 10\text{H}_2\text{O}$ phase – Friedel's salt, and sylvite – KCl.

Conclusions

Grouts used as a binder, or as an additive or filler, may include various kinds of mineral wastes, making the economic use of certain mineral wastes possible. AFBCC – fly ash, thanks to their high reactivity, can be used as a binder in grouts in which CKD can be used as an additive. A wide range of tests were performed for a complete analysis of the impact of the addition of dust on the technological and physico-chemical characteristics of grouts based on fluidized bed combustion fly ashes.

On the basis of laboratory testing and analysis of the obtained results it can be concluded that an increased concentration of CKD in grouts based on fluidized bed combustion fly ash favors the following:

- ◆ increased fluidity,

- ◆ extended setting time,
- ◆ a slight increase in the strength of the tested materials.

The analyses indicated the presence of calcite, quartz, anhydrite, hematite, portlandite and gypsum and probably C-S-H in the phase compositions of the tested grouts. The grouts containing 50 and 75% dust showed the presence of the $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{CaCl}_2 \cdot 10\text{H}_2\text{O}$ phase, which was not found in grouts based on fluidized bed combustion fly ash and those containing 25% dust. The grouts containing 75% dust showed the presence of sylvite.

A problem encountered was the high pH, which – if not reduced – rules out use in mining technologies. The increased leachability of chlorides can be regulated by using lower amounts of dust.

These examination will be repeated using ash other than AFBC – fly ash in order to obtain the proper pH and leachability of impurities to meet the requirements of the Polish standard *PN G-11011:1998 Mining – Materials for Backfilling and Caulking of Cavings – Requirements and Tests*. These results will also be the basis for further examinations of grouts using other AFBC – fly ash and dust for a better understanding of their properties and to obtain mixtures that meet the aforementioned standard.

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ODPADY Z PRZEMYSŁU CEMENTOWEGO – SKŁADNIK ZACZYŃÓW INIEKCYJNYCH

Słowa kluczowe

zaczyny iniekcyjne, pyły z produkcji cementu, popioły fluidalne

Streszczenie

Pył cementowy jest odpadem z procesu produkcji cementu. Część powstałych w trakcie produkcji cementu pyłów trafia do powtórnego wykorzystania w procesie, jednak pod warunkiem, że nie zawierają one składników mogących obniżyć jakość końcowego produktu. Z uwagi na znaczny udział tlenku wapnia, pyły mogą być stosowane także w ochronie środowiska do: neutralizacji kwaśnych ścieków, stabilizacji i nawożenia gleb czy do unieszkodliwiania osadów ściekowych. Jednym z kierunków gospodarczego wykorzystania tego typu odpadów jest stosowanie ich jako dodatków do zaczynów iniekcyjnych.

W pracy przedstawiono wyniki wstępnych badań świeżych i stwardniałych zawiesin wykonanych na bazie pyłu cementowego oraz popiołu fluidalnego ze spalania węgla kamiennego w aspekcie zastosowania ich jako materiału do sporządzania zaczynów iniekcyjnych. Badaniom poddano zaczyny sporządzone z popiołów fluidalnych z wprowadzonymi pyłami cementowymi w ilości: 25, 50 i 57%.

Wprowadzenie pyłów cementowych do zaczynów sporządzonych z popiołów fluidalnych wpłynęło na ich podstawowe parametry technologiczne poprzez: wzrost rozlewności, wydłużenie początku i końca czasu wiązania oraz wzrost wytrzymałości.

Badane zaczyny charakteryzują się podobnymi składami fazowymi, jednak dodatek pyłów spowodował pojawienie się fazy $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{CaCl}_2 \cdot 10\text{H}_2\text{O}$ oraz KCl.

Przeprowadzone oznaczanie wymywalności wykazało, że badane zaczyny nie spełniają wymagań normy PN-G-11011:1998 *Górnictwo - Materiały do podsadzki zastalanej i doszczelniania zrobów – Wymagania i badania w zakresie wymagań dla pH oraz zawartości chlorków po dodaniu 50 i 75% pyłów.*

WASTE FROM THE CEMENT INDUSTRY – A COMPONENT OF SEALING GROUTS

Key words

sealing grouts, dust from cement production, fluidized bed fly ashes

Abstract

Cement kiln dust is a waste product of the cement production process. Part of the dust generated during the burning of Portland cement clinker may be reused in the process, provided it does not contain components that might reduce the quality of the final product. Given the large proportion of calcium oxide, the dust can also be used for purposes of environmental protection in neutralization of acid wastewater, stabilization and fertilization of soil, or disposal of sewage sludge. One of the methods of the economic utilization of this type of waste is its use as a grout additive.

This paper presents the preliminary results of research into fresh and hardened grouts made on the basis of cement kiln dust – as well as ash from fluidized bed combustion of hard coal – used as a material for the preparation of sealing grouts. The grouts prepared from fluidized bed fly ash and cement kiln dust, in the proportions of 25, 50, and 57%, were examined.

The introduction of cement kiln dust into grouts prepared from fluidized bed fly ashes affected the grouts' basic technological parameters, increasing their fluidity, lengthening the setting time, and increasing their strength.

The studied grouts are characterized by similar phase compositions; however, the addition of dust has resulted in the formation of the $3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{CaCl}_2 \cdot 10\text{H}_2\text{O}$ phase – Friedel's salt, and sylvite – KCl.

A leachability determination has shown that the tested grouts do not meet the requirements of the Polish standard PN G-11011:1998 *Mining – Materials for Backfilling and Caulking of Cavings – Requirements and Tests* regarding the requirements for pH and chloride content after adding 50 and 75% dust content.

