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STABILIZATION/SOLIDIFICATION OF SLUDGE BY MEANS OF COAL FLY ASH AS A BINDER

The paper focuses on determining the additives being suitable for stabilization/solidification of sludge (produced during wastewater treatment) by fly ash. Fly ash, fly ash with Portland cement, and fly ash with lime were tested as the binders. Different quantities of Portland cement or lime added to fly ash as a binder were mixed with sludge produced during wastewater treatment. Carbon black was added to the mixture of the sludge with a binder in 1, 2, 3 wt% to reduce DOC (dissolve organic carbon) leachability. The prepared mixtures were left in laboratory conditions for 28 days to solidify. Then the effectiveness of sludge stabilization was evaluated by water-leaching test carried out on specimens according to Czech regulations. The specimens in distilled water of the water/solid ratio of 10/1 were mixed together and shaken for 24 hours. Due to high content of organic compounds in the sludge, the DOC content in water leachate was chosen as a relevant criterion. Stabilization/solidification of sludge by means of fly ash decreased the leachability of DOC from 2,320 mg/dm^3 to 164 mg/dm^3 in water leachate of a mixture with 40 wt% of sludge. Addition of a carbon black (1 wt%) decreased the leachability of DOC by almost three times (61 mg/dm^3 for the mixture with 40 wt% of sludge). Addition of Portland cement or lime to fly ash did not give any remarkable effect on the decrease in DOC leachability from wastewater sludge.

1. INTRODUCTION

It is known that the amount of sludge produced during wastewater treatment and discharged in the Czech Republic as well as in other countries still increases. In the Czech Republic, according to the Water Research Institute, 146,4 thousand and 206,7 thousand tons of dry matter were discharged in 1995 and in 2000, respectively. In addition, a large amount of sludge contains high content of heavy metals,

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which poses a serious environmental problem due to their high toxicity. It is necessary to incinerate or to treat this highly toxic sludge by its stabilization/solidification by means of hydraulic binders. Stabilization/solidification is a widely accepted treatment process allowing immobilization of hazardous substances, such as heavy metals, contained in sludge. The principle of this method is based on the formation of strongly impermeable matrix by hydraulic binder in which the pollutants are enclosed. The matrix is formed by hydration of different types of minerals contained in hydraulic binders. Portland cement is the most commonly used hydraulic binder. The hydration of basic minerals in Portland cement is described by the following reaction:

$$n\mathrm{CaO{\cdot}SiO_2} + (3 + m + n)\ \mathrm{H_2O} \rightarrow n\mathrm{CaO{\cdot}SiO_2{\cdot}}m\mathrm{H_2O} + (3 - n)\ \mathrm{Ca(OH)_2}.$$

On the other hand, fly ash coming from power plants is also used to decrease the amount of Portland cement, since they have similar qualitative composition. However, the main oxide in Portland cement is CaO whose content in high-quality cement approaches 60 wt%, while in fly ash, SiO_2 and Al_2O_3 oxides prevail. The different composition of additives leads to formation of different types of minerals (HEWLETT et al. [3]). According to the Czech Bureau of Statistics, almost 50% of this fly ash are disposed without taking advantage of its hydraulics properties. Therefore it is convenient to apply these fly ashes to stabilization/solidification of sludge with hydraulic binder, although it is necessary to increase its ability to harden the sludge by an appropriate activator such as lime. The procedure of stabilization/solidification of the sludge by hydraulic binders such as pulverized fuel ash was used as early as in 1978 by a German company, Rhenus AG. They reported the application of mixtures of pulverized fuel ash and other hydraulic binding agents marked as Rhenipal to conditioning and stabilizing sludge for landfill. Admixtures of Rhenipal and sludge can greatly improve soil stability and also markedly reduce leachability of heavy metals (DIRK [1]).

It is known that, in addition to heavy metals, sludge contains organic compounds. In evaluating stabilization/solidification of sludge by means of hydraulic binders, dissolved organic carbon (DOC), which reflects the amount of organic compounds being leached into the water, is an important parameter. When organic substances are leached out, they become a substrate for microorganisms coming from other waste in the landfill, which can lead to their excessive growth and hence to additional problems in the landfill. The leachability of organic carbon was measured by other researchers in the mixtures of sludge with Portland cement, sand, coal fly ash and accelerators. The retention or fixing of DOC varied from 70 to 53%, depending on the mixture and these mixtures were supposed to be used as a construction material (VALLS and VAZQUES [12]). The presence of the organic substances generally seems to be a serious problem if hydration of hydraulic binders is taken into account, because even small amounts of organics can alter the process in such a way as to decrease substantially the ability of fixing agents to immobilize the organics and heavy metals contaminated by them. Oil, grease, trichloroethylene (TCE), and phenol decrease the rate of comprehensive strength development, thereby they interfere with a formation of impermeable matrix. The effect of this interference generally increases with an increase in organic substance concentration (MINOCHA et al. [9]). In order to suppress this negative effect, activated carbon is used as an adsorbent of undesirable organic compounds. The effect of activated carbon addition was tested for fixation of phenols in contaminated sands by Portland cement and the results achieved show a strong decrement in phenol leaching in comparison to those obtained for the mixtures without adsorbent (HEBATPURIA et al. [2]).

The purpose of this paper was to investigate the possibilities of using a conventional fly ash as a binder in the field of stabilization/solidification and to replace the Portland cement by this cheap hydraulic binder. This means that fly ash (waste product) is combined with wastewater sludge (also waste product) and the final mixture is disposed into a landfill. The paper is focused on the determination of dissolved organic carbon (DOC) in water leachate in mixtures. Carbon black (Chezacarb SH) in different doses was tested in order to improve fixation of organic substances. Additions of Portland cement and lime were tested in order to increase pH values and to improve stable, solid matrix formed by conventional fly ash.

2. EXPERIMENTAL

2.1. SLUDGE PRODUCED DURING WASTEWATER TREATMENT

The sludge was obtained from a wastewater treatment plant of Aliachem joint stock company, Pardubice, the Czech Republic. This plant is equipped with facilities for mechanical pretreatment and biological treatment of wastewater. After biological treatment, sludge is dewatered in settling tanks (about 5–6 wt% of dry matter), and then subjected to centrifuging with simultaneous dosing the flocculants (about 15 wt% of dry matter). This sludge was a black colored water suspension with 13.60 wt% of dry matter determined at 378 K. The content of organic compounds determined as loss on ignition at 823 K was 69.53 wt% per dry mass.

2.2. FLY ASH

Fly ash was acquired from the heating plant of Aliachem joint stock company, Pardubice, the Czech Republic. In terms of chemical composition, this conventional fly ash can be included in the group of aluminosilicate fly ashes. Pit coal, obtained from the Ostravsko-Karvinska coal basin (the Czech Republic), was pulverized before its burning. The heating plant is equipped with a conventional type of boiler. Fly ash being separated in cyclone separator and cloth filters is the smallest fraction. In this study, the content of fly ash was as follows: 99.95 wt% of the dry matter and 15.05 wt% of unburned coal content determined as loss on ignition at 823 K. Its apparent density was 0.756 g/cm^3.

2.3. CHEZACARB SH, THE CARBON BLACK ADSORBENT

Carbon black (Chezacarb SH), a product of Chemopetrol Litvinov company in the Czech Republic, was used. It is manufactured as a secondary product of the gasification of heavy petroleum residues. The hydrophilic surface of this adsorbent enables its better wettability in water and water solutions (table 1). Carbon black is intended for sorption and decolorization processes in water solutions, and in this work, adsorbs organic compounds.

Table 1

Parameters of carbon black (Chezacarb SH) according to the certificate of inspection

Parameter	Value	Standard
Iodine adsorption number [mg/g]	1199	ASTM D 1510
DBF – absorption [cm^3/100 g]	419	ASTM D 2414
pH	8.8	DIN ISO 787/9
Ash content [%]	0.76	DIN 53 586
Apparent density [g/cm^3]	0.136	ASTM D 1513
Wettability [%]	84	–

2.4. LIME (CAO)

In experiments, fine-ground lime produced by Kotouc Stramberk, the Czech Republic, was used. Its apparent density was 0.845 g/cm^3.

2.5. PORTLAND CEMENT CEM 32.5 (CEM)

Portland cement can play the same role as calcium oxide during hydration of fly ash and also helps to increase pH values. Portland cement, acquired from Cementarny a Vapenky joint-stock company, Prachovice, the Czech Republic, was used.

2.6. PREPARATION OF SPECIMENS

Figure 1 shows the experimental flow diagram. All components presented in this diagram were weighed accurately (0.1 g) and put directly to a kneader bowl. Sludge was weighed in the range of 40–55 wt%; carbon black, in the range of 1–3 wt%; and binder was calculated to 100 wt%. As binders we tested: fly ash, fly ash with Portland cement addition, or fly ash with lime addition. Lime as well as Portland cement were dosed in the range of 0–20 wt% of binder. At first sludge was blended with carbon black to ensure sufficient disintegration of carbon black in sludge. After a 5-minute blending, the binder was added and the mixture was blended for another 15 minutes. Then, the mixture was compacted into 100 cm^3 PE bottles and left for 28 days to solidify. This period of 28 days was chosen according to the methods for testing concrete materials. After this period, two specimens from each bottle were prepared by cutting the bottles. The specimens were of cylindrical shape with diameter of 4 cm and height of approximately 2.2 cm. The weight of specimens varied from 45 g to 55 g, depending on the mixture composition.

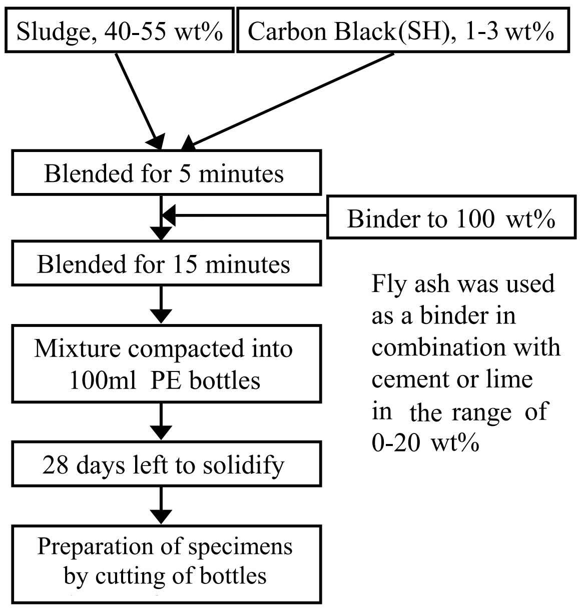

Fig. 1. Treatment procedure

Using this procedure, specimens with different content of sludge, conventional fly ash, lime, Portland cement, and carbon black were prepared. Composition of these mixtures is shown in table 2.

Table 2

Composition of prepared mixtures

Content of sludge [wt%]	Content of carbon black [wt%]	Content of fly ash [wt%]	Lime content in binder [wt%]	Portland cement content in binder [wt%]
40 45 50 55	0 1 2 3	Calculated to 100 wt% for each mixture	–	–
40 45 50 55	3	Calculated to 100 wt% for each mixture	5 10 15 20	–
40 45 50 55*	3*	Calculated to 100 wt% for each mixture	–	5 10 15 20*

Mixtures were prepared and to each sludge sample (varying from 40 wt% to 55 wt%), different doses of carbon black were added (varying from 0 wt% to 3 wt%). Thereafter all these mixtures were blended with a binder. The composition of the binder is also shown. The binder content in each mixture was calculated to 100 wt%. Fly ash alone or with different additions of lime/Portland cement (0–20 wt% of the binder) was used. Example of calculation of each component content: marked mixture (*) content: 55 wt% of sludge, 3 wt% of carbon black and 42 wt% of binder, which consists of fly ash (80 wt%) and Portland cement (20 wt%). The particular number of prepared specimens with a given composition is apparent from the dependence shown (see result section).

2.7. LEACHING TEST

Specimen or appropriate amount of waste (fly ash, raw sludge) was put into a 1.25 dm^3 bottle and distilled water was added. The weight ratio of distilled water to solid waste was 10:1 according to Czech regulations for this test. After 24 hours of continuous shaking, water leachate was filtered through a glass fiber filter with an average pore size of 0.7–1.3 μm.

2.8. ANALYSIS OF WATER LEACHATE

The values of pH, conductivity and DOC of water leachate were measured. For DOC analyses, a total organic carbon analyzer, TOC – 5000 A (Shimadzu corporation, Japan), with automatic control was used. Organic carbon is automatically calculated as a difference between total carbon content and inorganic carbon content. In the water leachate of raw sludge and conventional fly ash, heavy metal content was measured with an atomic absorption spectrometer, GBC 933 AA (GBC Scientific Equipment Pty Ltd., Australia).

3. RESULTS AND DISCUSSION

3.1. CHARACTERISTICS OF SLUDGE BY WATER LEACHING TEST

Analysis of water leachate of sludge was carried out, and the results are shown in table 3. In water leachate of sludge, a high content of DOC was measured. This leachate was characterized by a intense odour. The parameters of water leachate of sludge did not exceed the limits imposed on them before their disposal into the landfills of non-hazardous waste. However, before being disposed the sludge must be pretreated because of its potential infectivity, odour, and possible water release during land filling.

3.2. CHARACTERISTICS OF FLY ASH BY WATER LEACHING TEST

The fly ash was examined in the leachate test and its results are shown in table 3. The parameters of water leachate of fly ash did not exceed the limits imposed on ash before its disposal into the landfills of non-hazardous waste.

Table 3

Parameters of water leachate of sludge and conventional fly ash

Parameter	Sludge	Fly ash	Limits*
pH	6.69	12.12	5.5-13
Conductivity [mS/m]	332.0	292.8	2000
Dissolved organic carbon (DOC) [mg/dm^3]	2,320	0.599	–
Inorganic carbon (IC) [mg/dm^3]	–	2.897	–
Heavy metals in water leachate [mg/dm^3]			
Cr (total)	0.15	0.054	50
Pb	0.21	< 0.1	10
Ni	<0.1	0.018	50
Cd	< 0.02	< 0.01	0.5
As	<0.05	0.079	5
Al	–	0.306	–
Se	–	< 0.1	5
Sb	–	0.069	–
Ag	–	< 0.05	–

Co	–	< 0.05	–
Fe	–	0.017	–
Mn	–	< 0.01	–
Cu	–	< 0.05	–
Zn	–	< 0.05	–

* Limits for landfill of non-hazardous waste according to Czech standards for waste disposal.

3.3. STABILIZATION/SOLIDIFICATION OF SLUDGE

As mentioned in the experimental section, specimens were prepared after 28 days of hardening and subjected to the water leachate test. The following parameters were measured in water leachate: pH, conductivity and dissolved organic carbon (DOC). The pH values varied from ca. 11.5 for fly ash alone used as a binder to ca. 12.5 for fly ash combined with lime or Portland cement. An increase in pH value has an important contribution to the removal of sludge potential infectivity (WANG and VIRARAGHAVAN [13], KUCHAR et al. [8]).

The effect of carbon black addition on DOC parameter. As mentioned above, sludge has almost organic character, which gives rise to its negative influence on hydration process of conventional fly ash in stabilization/solidification of sludge with fly ash. To improve the hydration process and to reduce leaching of organic compounds into water, additions of carbon black into the mixture of sludge with fly ash were tested.

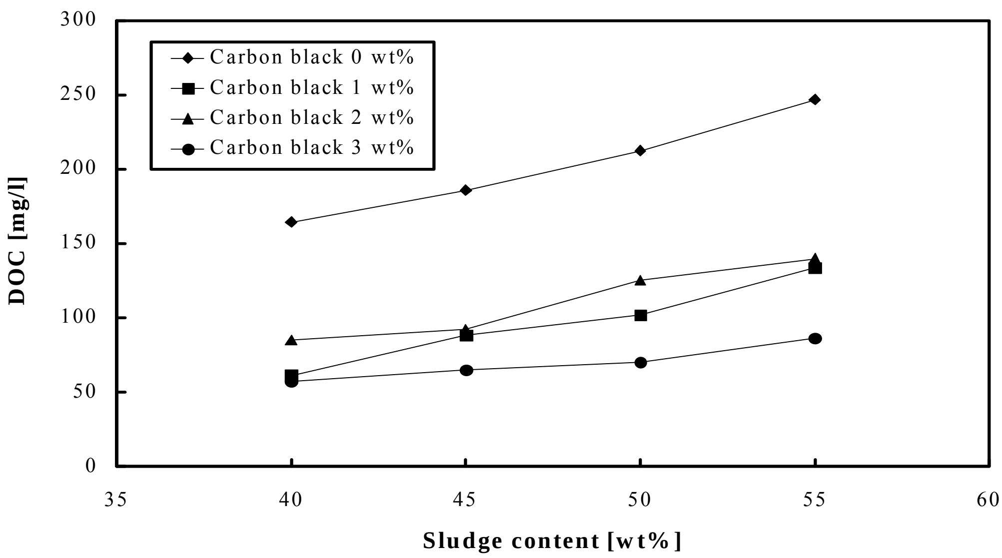

Fig. 2. Relation between DOC content and sludge content in the mixtures with different addition

of carbon black “Chezacarb SH”

The relation between DOC content in water leachate and sludge content in mixtures with conventional fly ash as a binder is displayed in figure 2. A significant decrease in DOC parameter was measured in the mixtures with 1 wt% carbon black in comparison to the mixtures without carbon black. On the other hand, 2 wt% and 3 wt% additions of carbon black did not lead to another significant decrease in DOC content. 1 wt% addition of carbon black is sufficient for adsorption of organics, which can be captured by carbon black in the mixtures containing 40 wt% of sludge and 59 wt% of conventional fly ash, and adsorption equilibrium was adjusted in this mixture. The lowest DOC values were obtained when 3 wt% of carbon black was added to the mixture of sludge and conventional fly ash. Therefore, further experiments were performed with 3 wt% of carbon black.

The effect of Portland cement or lime addition on DOC parameter. Taking account of the hydraulic properties, it is possible to classify fly ash as latent hydraulic binder, whose ability to harden sludge should be developed by an appropriate activator (HEWLETT et al. [3]). In order to improve their hydraulic ability, the mixtures of sludge containing 1 wt% of carbon black with fly ash/Portland cement or fly ash/lime were prepared. Such mixtures are prepared based on the relation between DOC parameter and sludge content. In figures 3 and 4, the relations between DOC content and sludge with addition of carbon black and either Portland cement or lime are shown. Batch additions of Portland cement or lime, 0, 5, 10, 15, 20 wt% of the total weight of binder, were made.

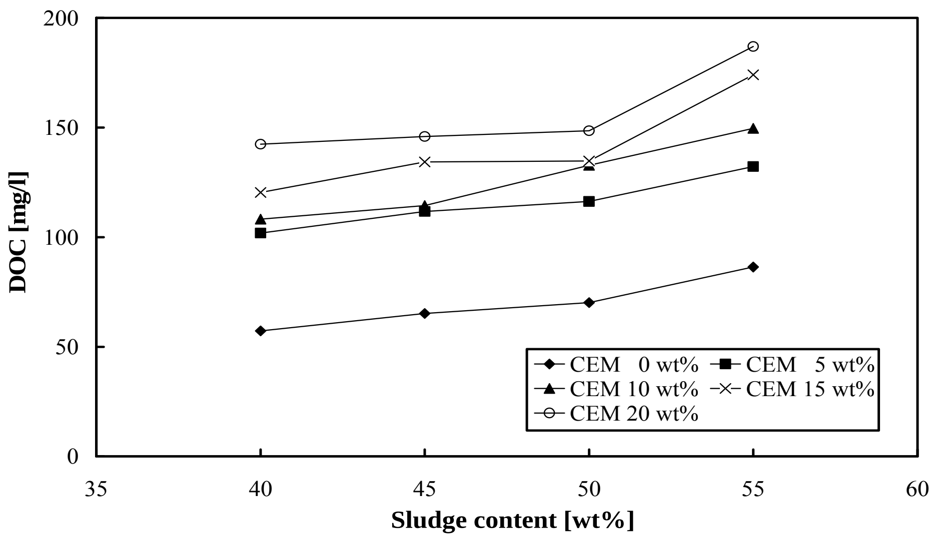

Fig. 3. Relation between DOC content and sludge content in the mixtures with 3 wt% addition of carbon black and different addition of Portland cement

As shown in figures 3 and 4, DOC increased with an increase in sludge content in mixtures. It was found that either Portland cement addition or lime addition did not lead to lower content of DOC in water leachate. These results seem to indicate that fly ash stabilizes/solidifies the sludge in two ways. In the first one, it forms a matrix and keeps pollutant inside this matrix, and in the second one, it exhibits adsorption ability. The formation of the strong matrix may be based on hydration of minerals similar to Portland cement. However, the fly ash contains different types of minerals from Portland cement, hence the minerals in fly ash may lead to the formation of the matrix that is different from those in Portland cement. Moreover, the formation of this matrix in fly ash is much slower than that in Portland cement. It is reported that approximate hydration of Portland cement ranges from 70 to 80% after 28 days, while hydration of fly ash is lower than 10% (HEWLETT et al. [3], POON et al. [11]). This may result not only from different composition of fly ash and Portland cement, but also from the phase change of components contained in the fly ash during coal combustion at high temperature. This high temperature causes melting of SiO_2, which creates a quite strong glass phase on the surface of fly ash, consequently, this glass phase hinders dissolution of minerals. The hydration rate can be increased by lime or Portland cement, because the possibility of fly ash activation mainly lies in the breaking down of its glass phases, which can be achieved due to high pH and high temperature. The best results are achieved when pH is higher than 13 (DONGXU et al. [4]). However, the results showing increase in DOC parameter with addition of lime or Portland cement did not confirm this presupposition. Therefore, as shown in figures 3 and 4, DOC parameter significantly increased with 5 wt% addition of lime or Portland cement in the mixture because they replace the fly ash as an adsorbent. The DOC reduction was not achieved even if the amount of lime or Portland cement was further increased to 10, 15, 20 wt%. Thus, it seems that the adsorption ability of fly ash is more significant in this short period of time and the organic substances are adsorbed not only by carbon black, but also by fly ash. Recently, the ability of fly ash to adsorb organics was confirmed by adsorption of dyes from aqueous solutions (JANOS et al. [6]) and adsorption of PCBs from wastewater (NOLLET et al. [10]). It was considered that the replacement of fly ash by Portland cement or lime could lead to activation of fly ash used as a binder, while a decrease in the content of fly ash as an adsorbent might give rise to a decrease in the amount of organics adsorbed by fly ash. Moreover, GLASSER [5] reported that hydraulic binders such as Portland cement interact with organic compounds to form soluble organics of lower molecular weight, which can penetrate easily through calcium silicate aluminate matrix formed by hydraulic binder. The increase in DOC value is therefore attributable to leaching of decomposed organics from the sludge during hardening process.

The addition of Portland cement or lime also led to a significant increment in conductivity of water leachate. This may be due to better solubility of lime or better solubility of minerals contained in Portland cement in comparison with the solubility of minerals contained in fly ash. When Portland cement is mixed with pure water only, the compounds comprised in it start to dissolve which leads to the increase in conductivity together with continuation of hydration; dissolved minerals are transformed into strong matrix and conductivity decreases. It is possible to predict that with continuation of hydration, the values of conductivity will decrease.

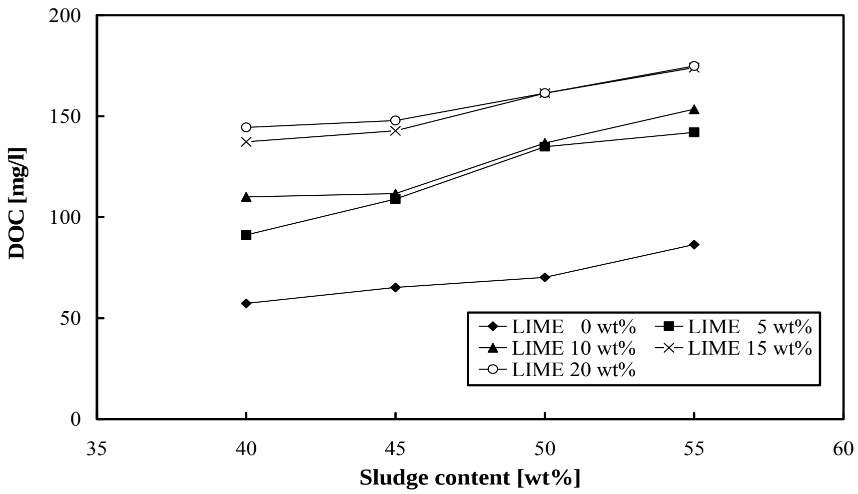

Fig. 4. Relation between DOC content and sludge content in the mixtures with 3 wt% addition of carbon black and different addition of lime

4. CONCLUSIONS

The ability of fly ash and fly ash with addition of lime or Portland cement to be used as a binder allowing stabilization/solidification of sludge produced during wastewater treatment was investigated by mixing sludge, carbon black and fly ash (with and without Portland cement or lime). Specimens prepared from these mixtures were left to harden for 28 days and then their leachability in water was tested. A substantial decrease in DOC leachability, i.e. from 2.320 mg/dm^3 for untreated sludge to 164 mg/dm^3 for specimens with 40 wt% of sludge, was achieved by stabilization/solidification of sludge with fly ash only as a binder. The addition of carbon black as an adsorbent was investigated and the results obtained showed another significant decrease in DOC, even if only 1 wt% of carbon black adsorbent was added (61 mg/dm^3

in the mixture with 40 wt% of sludge). Further, the addition of carbon black (3 wt%) did not cause another important decrement of parameter DOC in the mixtures with 40 wt% of sludge, but had a positive effect on DOC leachability in the mixtures with the sludge content ranging from 45 to 55 wt%. Addition of Portland cement or lime showed an increase in DOC. It is possible that organic substances are adsorbed on the surface of fly ash and when it is partially replaced by lime or Portland cement, its adsorption capacity decreases. Furthermore, the interactions between cement and organics could lead to the decomposition of these organics, thereby to an increase in DOC parameter. Higher conductivity and pH values were measured in the mixtures with lime or Portland cement. Higher pH can reduce potential infectivity of sludge.

Hydration is a time-consuming process which is well described for Portland cement, but its run is not clear for latent hydraulic binder like fly ash. The period required for fly ash hydration is supposed to be much longer than that for hydration of Portland cement. Also the presence of organic compound negatively affects hydration process, because it inhibits the dissolution of minerals. Based on these facts, it is possible to suppose that DOC parameter will decrease for long with the increment of hydration ratio. Portland cement or lime addition could be characterised by higher hydration rate in comparison with fly ash alone and plays important role in a decrease in leachability of pollutants in long period of time.

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STABILIZACJA/ZESTALANIE OSADÓW ŚCIEKOWYCH ZA POMOCĄ WĘGLOWEGO POPIOŁU LOTNEGO JAKO SUBSTANCJI WIĄŻĄCEJ

Określono modyfikatory służące do stabilizacji/zestalania osadów ściekowych, które powstały podczas oczyszczania ścieków, za pomocą popiołu lotnego. Popiół lotny, popiół lotny z domieszką cementu portlandzkiego i popiół lotny z domieszką wapna palonego były testowane jako substancje wiążące. Różne ilości cementu portlandzkiego lub wapna palonego dodawano do popiołu lotnego, a następnie mieszano z osadem ściekowym. Aby zmniejszyć wymywalność rozpuszczonego organicznego węgla z mieszaniny osadów ściekowych z substancją wiążącą (w ilości 1, 2, 3 procenta wagowego), dodano do niej sadzy. Tak przygotowaną mieszaninę trzymano przez 28 dni w warunkach laboratoryjnych, aby zestaliła się. Następnie oceniano efektywność stabilizacji osadu na podstawie testu ługowania przeprowadzonego zgodnie z czeską normą. Próbki mieszano z wodą destylowaną (stosunek wody do ciała stałego wynosił 10/1) i wytrząsano przez 24 godziny. Ponieważ osad zawierał znaczną ilość substancji organicznych, więc zawartość rozpuszczonego węgla organicznego w odcieku po ługowaniu przyjęto za miarodajny wskaźnik. Stabilizacja/zestalenie osadu za pomocą popiołu lotnego zmniejszała wymywalność rozpuszczonego węgla z 2320 mg/dm^3 do 164 mg/dm^3 w wodnym odcieku po ługowaniu mieszaniny,
w której osad stanowił 40 procent wagowych. Dodatek sadzy (1 procent wagowy) zmniejszał prawie trzykrotnie wymywalność rozpuszczanego węgla organicznego (61 mg/dm^3 w mieszaninie z 40-procentowym udziałem osadu). Dodatek cementu portlandzkiego lub wapna gaszonego do popiołu lotnego nie miał istotnego wpływu na obniżenie wymywalności rozpuszczalnego węgla orgnicznego z osadu.