Supporting the Coagulation Process with Shale – Preliminary Studies

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1. Introduction

The amount and variety of anthropogenic pollutants and contaminants in the natural environment have increased in recent years. Searching for new effective materials and technological solutions to prevent the growing threats to the environment is still one of the most important tasks of today's engineering. One of the basic processes used in water and sewage treatment is coagulation (Nawrocki & Biłozor 2000; Girczys & Caban-Pabian 1999; Jiang 2015), which is increasingly improved through the use of various supporting materials (Bina et al. 2009, Aygun & Yilmaz 2010). These include, first of all, various ballasts of floc. They can be minerals such as bentonite (Góra et al. 2016), montmorillonite (Lagaly & Ziesmer 2003) or kaolinite (Li et al. 2016), chitosan (Bhalkaran & Wilson 2016, Zhang et al. 2018), activated silica (Sinha & Mathur 2015), activated carbon (Furlan et al. 2009), organic polyelectrolytes (Kozminykh et al. 2016) or mineral polyelectrolytes (Thakur & Choubey 2014). Adjuvants are used to accelerate floc formation and sedimentation, reduce the negative effects of low temperatures, extend of the optimal pH range for coagulation and lower the dose of coagulant (Nawrocki & Biłozor 2000). Often, these substances also have adsorptive properties. It is significant that most of natural ballasts have a charge of sign opposite to the one of basic coagulant. The beneficial effect of small amounts of dissolved silica as an adjuvant to aluminum sulfate in the coagulation of opacifying pollutants was confirmed in water of low turbidity. The test results demonstrated the participation of silicon complex-

es in modifying the method of colloid charge neutralization by the products of hydrolysis of the coagulant (Duan & Gregory 1996). Moreover, the presence of silica may reduce the bioavailability of aluminum remaining after the coagulation (Birchall et al. 1989).

Thus, substances opacifying water such as bentonites, clays, montomorylonites can be used to enhance the rate of coagulation. They are applied mostly in conjunction with primary coagulants. In such conditions, the presence of fine clay particles increases the number of condensation centers in the gel, which causes a faster adsorption of colloidal particles and the formation of highly loaded and easily sedimenting floc (Nawrocki & Biłozor 2000). The resulting post-coagulation sediment is a complex mixture of pollutants and clayey substances held together by the van der Waals and electrostatic forces (Abdelaal 2004).

The world's attention is mainly focused on the use of bentonites and bentonite clays, the resources of which are unfortunately small in Poland (about 2.9 million Mg), and this is the reason for their import (Burkowicz 2015). However, there are other rocks which could partially replace them. One of them could be gangue (shale, claystone, mudstone), which accompanies the coal beds in Polish mines and is treated as waste during coal extraction. Artificially deposited rocks are mostly clays, shales, mudstones and carbon shales (Skarżyńska 1997). The Central Statistical Office (GUS) gives that 7.66 million Mg of waste rocks from non-metalliferous extraction were generated in Poland in 2015 (Ochrona Środowiska 2016). The greatest amount of gangue in the world are mined in China – around 300-350 million Mg per year (Yu et al. 2016). Presently, the mining waste rocks are used mainly in civil engineering (hydro-engineering, roads) as aggregate, in the restoration of degraded lands and for the production of building materials (Kozioł et al. 2016). In the world, gangue is also used for energy production, in agricultural fertilizer industry and the production of bricks, cement and concrete (Wang et al. 2016). The quality of the extractive wastes, mainly their physical and chemical properties, is largely due to their mineral and petrographic composition. Currently, the waste is increasingly seen as a waste mineral resource. Besides, a comprehensive approach in management of various types of mining waste for a range of applications is observed depending on their quality and taking into account their diverse lithology. This can be done by modifying existing technologies or development of new technologies

aimed in the use and processing of the mining wastes themselves. One of such applications of clayey waste materials could be using them for water treatment in the coagulation process. Their addition during coagulation, particularly in low turbidity water, can cause ballasting the floc and improve the sedimentation properties. Moreover, previous studies have shown that the rock in its natural form can adsorb some organic substances, e.g. phenol (Jabłońska 2012), and inorganic ones, e.g. heavy metals (Jabłońska & Siedlecka 2015, Sikora & Budek 1996).

The paper shows the preliminary results of research on the assessment of usefulness of waste rock (shale) originating from one of Polish coal mines to support the process of coagulation in low turbidity water. Raw shales as well as modified with simple thermal treatment, which could cause favorable changes in the structure of the materials (Jabłońska et al. 2017), were studied.

2. Materials and methods

The water was taken from the Warta River in Częstochowa on the day of testing. Then it was standing for 30 minutes to remove easily falling suspended matter.

The material supporting the process of coagulation was shale taken from a Polish coal mine extracting the coal in the area of Chief Saddle of the Upper Silesian Coal Basin. According to the Regulation of the Ministry of Environment of 9 December 2014, the waste rock is classified under code 01 01 02 (wastes from extracting non-metalliferous minerals). The waste is collected on dumps as rock material. The material for study was shale in its natural state. Then it was crushed in the vibration mill (TESTCHEM) to the grains less than 0.1 mm and dried to air-dry state. To obtain shale calcined at 600°C and 800°C, the material was introduced into a cold muffle and heated to the desired temperature at a rate of 10°C/min in an electrically heated muffle furnace.

The coagulant was aluminum sulfate – $Al_2(SO_4)_3 \cdot 18H_2O$ p.a. – produced by a chemical plant Kemipol Ltd. as a clear colorless solution. It is characterized by a density of $1\,310 \pm 10$ g/dm^3, pH $= 2.4 \pm 0.5$, with the percentage of Al_2O_3 of 7.9 ± 0.4 (data sheet by Kemipol Ltd.).

The coagulation process was carried out in 2 dm^3 glass beakers, into which a volume of 1 dm^3 of the water was introduced and then the coagulant was added. The coagulant dose of 30 mg/dm^3 was established

based on Stępniak (2006). After addition of the coagulant, a mechanical stirring was applied at 200 r.p.m. for 1 min. followed by 20 r.p.m. for 15 min. After this time, the samples were left for 1 hour sedimentation. Then 100 cm^3 of water were decanted and the turbidity was determined (nephelometric method using HACH 2100 IS turbidimeter according to ISO 7027). The final result was assumed the arithmetic mean of three determinations varying no more than 10% of the highest reading.

The second series of tests was performed as above, except that the raw shale was introduced during the coagulation and rapid stirring. The adjuvant doses ranged from 10 to 80 mg/dm^3. In the third step, the effect of thermal modification was tested by introducing the shale calcined in 600°C and 800°C. The coagulation with use of the material after the thermal modification was performed in the same manner as in the case of raw material. In every case, pH was specified by the natural pH of the treated water and the introduced shale and acidic solution coagulant.

The turbidity removal percentage (efficiency), E, was determined as the percentage change in the turbidity

$$E = \left(1 - \frac{T}{T_0}\right) \cdot 100\%, \tag{1}$$

where T and T_0 are turbidities of water after and before coagulation, respectively.

The chemical composition was determined with use of XRF spectrometry (Philips PW 1404). Trace elements were detected using ICP-AES spectrometry (Thermo Elemental IRIS Intrepid II XSP DUO). The leaching tests were carried out in accordance with PN-EN 12457-4:2006. The following determinations were made in the water filtrate: concentration of sulfate ions – with gravimetric method (PN-ISO 9280:2002), concentration of chlorate ions – with titration method (PN-ISO 9297:1994), concentration of phosphates – with spectrophotometric method with ammonium molybdate (PN-EN ISO 6878:2006), concentration of sodium, potassium and magnesium – using ICP-AES spectrometry in accordance with PN-ISO 9964-2:1994P and PN-EN ISO 7980:2002P. pH was measured using a CPC-401 type Elmetron pH meter. Determination of ash content was made in accordance with PN-ISO 1171: 2002P.

3. Results and discussion

3.1. Characteristics of shale used in tests

Shale in its natural state is characterized by a dark gray tint; it locally shows clear cleavage and is laminated with organic matter (carbon). The mineralogical composition of the rocks is dominated by kaolinite and quartz. The larger amounts of illites, hematite and mica are observed, too. There are also traces of smectites and chlorites. The chemical composition shown in Table 1 reflects the mineralogical composition of the examined rocks. The relatively high content of SiO_2, Al_2O_3 and K_2O indicates mainly the presence of quartz, kaolinite and illite, and sometimes feldspar. A significant part in the composition of the shale belongs to Fe_2O_3, which is attributed to the presence of hematite in the rock. Other elements present in the chemical composition can be associated with clayey minerals such as mica, smectites, chlorites and carbonates: calcite and siderite. The non-combustible constituents of the rock are 84.7% of the mass, and the combustible part is 15.3%.

Table 1. Chemical composition of shale used in tests
Tabela 1. Skład chemiczny badanego łupka ilastego

Constituent	Content, mass %	Constituent	Content, mass %
SiO_2	53.4	K_2O	2.7
Al_2O_3	20.1	TiO_2	0.6
Fe_2O_3	4.5	P_2O_5	0.05
CaO	1.1	SO_3	0.1
MgO	1.8	Loss on ignition	15.3
Na_2O	0.2	Others	0.05

The content of trace elements in the shale and permissible limits in soils and land surface (at a depth of 0 to 0.3 m below the surface) are shown in Table 2. The levels of trace metals are much lower than the limit values permissible in surface soils belonging to the group B areas (i.e. areas classified as agricultural land, forest land and wooded shrub, and built and urbanized areas with the exception of industrial areas).

Table 2. The content of trace elements in the tested shale and their permissible limits in soils of group B as specified in the Regulation of the Ministry of Environment of 9 September 2002

Tabela 2. Zawartość pierwiastków śladowych w badanym łupku i ich dopuszczalne wartości w gruntach grupy B podanych w Rozporządzeniu Min. Środ. z dnia 9 września 2002

Element	Content, mg/kg of dry mass									
Element	As	Cd	Co	Cr	Cu	Mo	Ni	Pb	Sn	Zn
Determined	12	<0.5	4.5	33	44	<2	19	<0.5	2	171
Limit	20	4	20	150	150	10	100	100	20	300

The tested rocks characterize a low level of leaching of constituents soluble in water (Tables 3 and 4). Among leached anions, SO_4^{2-} and Cl^- can be found, whereas the leached cations were mainly Na^+, K^+ and Mg^{2+}. Among detected heavy metals, the highest mobility was observed for iron ions. Concentrations of Cd(II), Cr(III) and Ni(II) were below the limits of quantification. pH was weakly alkaline. The determined values were below the permissible limits and pH was in the permissible range.

Table 3. The concentration of heavy metals in water extract from shale and limits given in the Regulation of the Minister of Environment of 18 November 2014

Tabela 3. Stężenia metali ciężkich w wyciągu wodnym z łupka ilastego I wartości dopuszczalne wg Rozporządzenia Min. Środ. z 18 listopada 2014

Element	Concentration, mg/dm³							
Element	Cd	Cr	Cu	Fe	Mn	Ni	Pb	Zn
Determined	<0.007	<0.004	0.02	0.28	0.09	<0.003	0.015	0.05
Limit	0.4	0.5	0.5	10	–	0.5	0.5	2.0

Table 4. Selected parameters of aqueous extract of shale and limits given in the Regulation of the Minister of Environment of 18 November 2014

Tabela 4. Wybrane parametry wyciągu wodnego z łupka ilastego i wartości dopuszczalne wg Rozporządzenia Min. Środ. z 18 listopada 2014

Parameter	pH	Concentration, mg/dm³					
Parameter	pH	Na^+	K^+	Mg^{2+}	Cl^-	SO_4^{2-}	PO_4^{3-}
Determined	7.5	46	12	14	31	24	0.14
Limit	6.5-9	800	80	–	1000	500	–

3.2. Effect of shale dose and calcination

The effect of shale dose on decreasing the turbidity in coagulation is shown in Table 5.

Table 5. Effects of coagulation with use of aluminum sulfate aided with raw and calcined shale
Tabela 5. Efekty koagulacji siarczanem glinu wspomaganej materiałem ilastym surowym i kalcynowanym

Shale dose, mg/dm^3	Turbidity, NTU			
	Series 1		Series 2	
Raw water	9.9 (pH = 7.32)		11.8 (pH = 7.53)	
Water and coagulant	4.68 (pH = 4.69)		6.65 (pH = 4.89)	
	Water with coagulant and shale			
	Raw shale	Shale calcined at 600°C	Raw shale	Shale calcined at 800°C
10	3.09	2.69	2.61	4.94
20	4.06	2.11	5.62	4.66
30	4.09	3.07	5.55	4.09
40	4.69	3.55	5.51	4.22
50	3.91	2.87	4.92	6.10
80	7.11	3.47	5.81	5.75
pH range	4.62-4.78	4.68-4.93	4.68-4.95	5.07-5.53

Aiding the coagulation with the raw shale decreased the turbidity of the water compared to the treatment without the shale. Typically, the turbidity was reduced to about 30-40% of the initial value, and the highest reduction was obtained for the lowest tested doses of the shale (10 mg/dm^3). Greater doses of shale did not caused a further reduction in turbidity. Using the shale calcined at 600°C gave usually better results than the raw shale – the turbidity was decreased to about 20-30% of its initial value, with the highest reduction for shale dose 10-20 mg/dm^3. Introducing the shale calcined at 800°C did not improve the reduction, and even sometimes worsened it.

The percentage turbidity removal is shown in Figure 1 and summarized in Table 6. Introducing the coagulant alone resulted in reduction of the turbidity by about 40-50%. Addition a portion of raw shale gave

a further decrease in turbidity with the exception of one case (shale dose 80 mg/dm^3). The highest efficiency (78%) was obtained for 10 mg/dm^3, i.e. for the smallest of the doses tested. A similar percentage turbidity removal (74.3-98.2%) was obtained by Bina et al. (2009), who used alum as a coagulant combined with chitosan as an adjuvant. The colloidal particles of shale are endowed with a negative charge, which is opposite to the charge of Al(III) hydroxides. During the mutual coagulation, the shale fragments are centers of condensation, which accelerate the flocculation and the formation of heavy, complex and rapidly falling floc. However, larger shale doses resulted in lower turbidity removal efficiency, although usually slightly higher than in the absence of the shale. Higher doses of the adjuvant increase the stability of the colloidal particles. Most likely, they cause the formation of a mesh of associated particles that interacts with the colloidal particles and impedes their coagulation. Comparison of Figures 1a and 1b for raw shale shows a significantly higher turbidity removal in the first sample than in the second. This indicates additional substances having an effect on the reduction of turbidity were present in the samples. Nevertheless, the results of the study showed the ability of shales to improve the effect of aluminum sulfate as an agent reducing water turbidity.

The use of clay calcined at 600°C gave better results than using the raw shale – the turbidity removal efficiency was 65-79%, the highest for 20 mg/dm^3. The results obtained for shale calcined at 800°C are not clear – the turbidity removal efficiency increased in some cases, but decreased in other (compared to the raw shale efficiency of turbidity removal). Shale calcination is likely to cause favorable shifts in the crystalline shale structure, resulting in additional active centers (Heller-Kallai 2006), thus increasing the sorption surface of the sediment flocs. However, too high temperature of calcination can again weaken the shale properties to support of coagulation.

To summarize, the highest turbidity removal was obtained for shale calcined at 600°C in small doses. It seems that the ratio of coagulant and adjuvant doses should be not less than 3:1, and the adjuvant dose should not exceed 10-20 mg/dm^3.

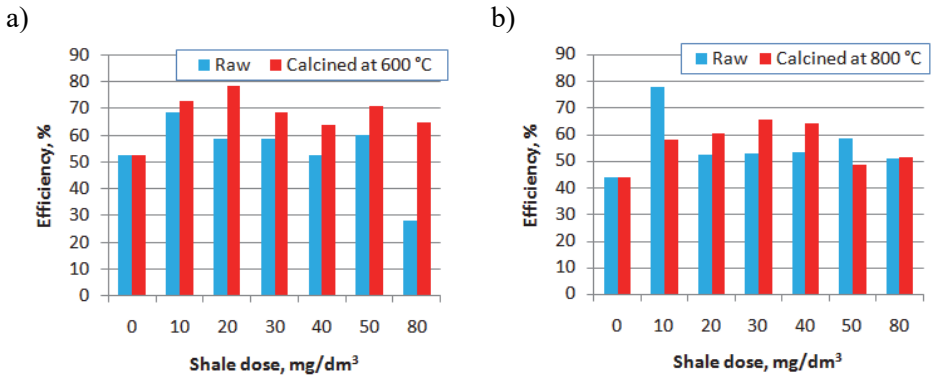a)   b)

Fig. 1. Turbidity removal vs. shale dose for initial turbidity 9.9 NTU (a)
and 11.8 NTU (b)
Rys. 1. Procent usunięcia mętności w funkcji dawki łupka ilastego dla mętności
początkowej 9,9 NTU (a) i 11.8 NTU (b)

Table 6. Turbidity removal efficiency with aluminum sulfate (30 mg/dm^3)
aided with shale (10-80 mg/dm^3)
Tabela 6. Skuteczność obniżania mętności siarczanem glinu
w dawce 30 mg/dm^{3}wspomaganej materiałem ilastym (10-80 mg/dm^3)

Initial turbidity, NTU	Turbidity removal efficiency, %			
	Coagulant	Coagulant + raw shale	Coagulant + shale calcined at 600°C	Coagulant + shale calcined at 800°C
9.9	53	28-69	65-79	–
11.8	44	51-78	–	48-65

4. Conclusions

Based on carried out experiments and achieved results it was pos-
sible to draw the following conclusions:
- The use of shale as a substance aiding the process of coagulation with
  aluminum salts improves the quality of treated water of low turbidity.
  Supporting the coagulation process with raw shale resulted in higher
  turbidity removal than the use of coagulant alone.
- Using the shale calcined at 600°C improved the turbidity removal
  efficiency. The shale calcined at 800°C gave ambiguous results.
- The highest turbidity removal was obtained for low doses of shale
  (10-20 mg/dm^3).

- The shale could be used as an adjuvant in coagulation, especially in low turbidity waters. Its grains are crystalline nuclei for the flocs, they increase the number of contacts, accelerate the formation of flocs, ballasting them and facilitating sedimentation.

However, the assessment of the efficiency of supporting the co-agulation process with shale requires further research on removing other impurities (color, organic substances and heavy metals) from surface water depending on the order of introduction of the coagulant, shale and their doses.

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Wspomaganie koagulacji łupkiem ilastym – badania wstępne

Streszczenie

W pracy rozważono możliwości zastosowania płonnych skał ilastych (łupków ilastych) z kopalni węgla kamiennego do wspomagania procesu koagulacji. Badano surowe i kalcynowane w temperaturze 600°C i 800°C łupki ilaste towarzyszące pokładom węgla kamiennego z kopalni położonej w południowej części Polski. Jako koagulantu użyto siarczanu glinu. Analizowano efekty obni-

żenia mętności w próbkach wody pobranej z rzeki Warty. Wspomaganie procesu koagulacji łupkiem surowym przyniosło większą efektywność zmniejszenia mętności w wodzie niż w przypadku zastosowania samego tylko koagulantu. Efektywność obniżania mętności była najwyższa w przypadku zastosowania łupka kalcynowanego w temperaturze 600°C. Użycie łupka kalcynowanego w temperaturze 800°C przynosiło nieco gorsze rezultaty, w niektórych przypadkach efektywność obniżania mętności była niższa niż z użyciem łupka surowego. Wyniki badań wskazują, że proces koagulacji może być efektywnie wspomagany łupkami ilastymi. Wcześniejsze badania (Jabłońska, B., Siedlecka, E., Removing heavy metals from wastewaters with use of shales accompanying the coal beds, Journal of Environmental Management, 155 (2015), 58-66) wykazały, że łupki ilaste mają stosunkowo dobre właściwości sorpcyjne, co może dodatkowo pozytywnie wpływać na jakość oczyszczanej wody.

Abstract

In the paper, clayey gangue (shale) accompanying coal beds was considered to support the coagulation process. The raw shale from a mine located in the southern part of Poland as well as the shale calcined at temperatures of 600°C and 800°C were tested. The coagulant was aluminum sulfate. The effects of turbidity reduction in water samples taken from the river Warta were analyzed. Aiding the process of coagulation with the raw shale increased the efficiency of reducing turbidity in water in comparison with the use of coagulant alone. The turbidity reduction was highest when using the shale calcined at 600°C. Applying the shale calcined at 800°C brought slightly worse results, and in some cases the turbidity reduction was lower than for the raw shale. The results indicate that the shale could be an effective adjuvant in the coagulation process. In addition, previous research (Jabłońska, B., Siedlecka, E., Removing heavy metals from wastewaters with use of shales accompanying the coal beds, Journal of Environmental Management, 155 (2015), 58-66) has shown that shale has relatively good adsorptive properties, which may further contribute to the quality of the treated water.