The Study of the Interaction Between Flotation Tailings and Flocculants in Separation Process of Coal

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Summary

The intensification of the mining process is responsible for as much as 45 % of waste rock and tailings getting to the coal preparation plant along with the raw coal. From the chemical point of view, it is material predominantly constituting of silicates (siltstone, conglomerates, kaolins or montmorillonites). The current on-site experience shows that the rising quantities of fine-grained impurities are responsible for protracting the sedimentation processes and thus prolonging the separation of fine-grained fractions. In the past, dewatering of flotation tailings was based on the principle of sedimentation. However, this is becoming very time-consuming due to the colloid character of the suspended particles as the materials get repulsed, thus influencing the sedimentation process. The experiments in this work focus on the verification of the mutual interaction between coal slurries and chemical agents. The aim is to describe the surface properties of tailings particles during their interaction with suitable agents, namely 'Flokor 1.2A' and 'PAX 18', applied for dewatering purposes. At lower doses of Flokor 1,2A or Pax 18 the conditions in the medium got stabilized and the pH did not fall below 6, using the doses of up to 0.4ml.l^{-1} and up to 0.08ml.l^{-1} respectively. The value of 0.05ml.l^{-1} is considered the optimal dose for the agents. The zeta potential shows that the surface charge gets compensated already at low concentrations of both aluminium chloride-hydroxide and polyaluminium chloride. The isoelectric point was reached at the dose interval ranging from 0.02ml.l^{-1} – 0.03ml.l^{-1}. The results were obtained at tailings suspension densities of 10 and 50g.l^{-1}.

Keywords: dewatering, zeta-potential, silicates, flocculation, sedimentation

Introduction

The current coal production is grounded in technologies capable of long-term provision of a number of coal products, ranging from power coal to low-calorific coals. World-wide, the production is increasing due to the higher demand for available electric power from thermal power plants. Quality cokeable coal of high calorific values has contributed to the recent technological development in many industrial branches. Nevertheless, the technological processing of coal of diverse qualities brings along large volumes of wastes. Moreover, apart from lump coal, coal mining is responsible for the production of growing amounts of fine-grained coal materials. The tailings need to be handled, either separately or as parts of other technology steps.

Automation of all the processes is associated with the intensification of the overall mining process, coal preparation and processing of tailings and waste rock. The tailings include waste rock from the bottom, walls or roofs in underground coal mining, and overburden in open cast workings. From the chemical perspective, the material largely comprises of silicate constituents (siltstone, conglomerates, kaolins or montmorillonites). The abundance of such constituents plays an important role, especially in coal flotation, when the finest particles with grain size below 1.5mm are concerned. Extensive amounts of combined water in the coal and also in the slurries require optimal technological procedures to be selected within the preparation process.

The waste rock and tailings are most frequently tipped on predetermined disposal sites. In the case of mining, the waste rock and tailings are dumped on waste rock disposal sites, with the intention of their potential future application, for example in the building industry.

Extensive proportions of fine particles arising from the individual coal preparation steps are loaded with high percentages of retained moisture, unlike the waste rock lumps. Fine tailings must be dewatered and subsequently stored in settling ponds or lagoons. Dewatering of coal tailings brings along technical as well as economic problems, related to their handling, transport, storage and the possible use of their fine-grained fractions.

Dewatering of the tailings is based on the agglomeration of fine particles by means of flocculants and coagulation agents. Flocculation is a complex process dependent on the applied chemical agents (flocculants and coagulation agents), the agent dosing method as well as the reaction of the substances in the given conditions. [1, 2, 3]

The chemism of the tailings and their interaction in the system of solids and the liquid determines the behaviour of the overall suspension. The interaction between the material's colloid particles in the suspension and the chemical agents is a significant technological feature in optimizing the separation of coal tailings and their dewaterEng. The fundamental goal of this optimization is thus the control of this interaction in the medium – stabilization of the multicomponent system when making use of chemical agents. Better understanding of the interaction between the material and a range of flocculation and coagulation agents shall facilitate the description of the connections in the system, suspension dewatering and optimization of the slurry disposal. The aim of the technological flow of the substance separation is the experimental verification of the interaction between the agents and the material, dewatering method, sedimentation, description of the surface properties and behaviour of the final products. Numerous works have dealt with coal preparation and research in the area of flotation regimes in the last twenty years. The research has focused on defining optimal regimes and the efficiency of coal substance separation from suspensions. [4, 5, 6, 7]

From the point of view of the economy of the overall system of the material's preparation and sale, the dewatering process of the colloid fractions predominantly focuses on the coal substance.

However, the research in the domain of effective dewatering of tailings concentrated only on dewatering in sedimentation ponds without any further optimization. At present, the increased proportions of fine-grained fractions are found to be responsible for slowing down the sedimentation processes and prolonging the separation of fine tailings within the current technologies. Dewatering of coal tailings has long been based on the principle of absolute sedimentation and thickening with subsequent transport and storage. Nevertheless, from the point of view of time consumption, this principle is threatened by the colloid character of the suspended particles as the materials are repulsed and the sedimentation is negatively influenced in the sludge lagoons.

The process of acceleration and optimization of tailings dewatering may be grounded in the principle of chemical destabilization of the medium and the subsequent formation of stable sludge floccules, plus a possible fast separation in the downstream step. The chemical agents impacting the treated material should not further affect the coal substance in the coal preparation process. Therefore, it is important to study the effects of the residual concentrations of the given substances in the process water on the coal flotation process. In coal preparation and separation, closed water circuits require large volumes of water, where residual ions concentrate and may participate in the influence on coal preparation.

Works by Arnold and Aplan imply that a number of clay impurities influence the recovery in coal flotation. The effect of the impurities on the coal preparation is conditioned by the quality of silicates, their quantity and grain sizes. [4]

Also due to their increasing quantities, effective dewatering of clay impurities shall play an important role in the further use of process water.

The major focus of the experiments is measuring the zeta potential in the suspensions, wettability of the material surface and mutual interactions in the medium. The connections among the parameters in various combinations of the conditions shall provide direct evidence on the surface properties of the chemical agents used for fast and effective separation of coal tailings from process water. The results of surface tension, wetting angle and particle sedimentation mutually correlate and offer optimal separation regime options.

Experimental

Basic approach

The basic concept of the experimental work focuses on the verification of the mutual interaction between the flotation tailings and the chemical agents mentioned above and specified below. Making use of the principles of measuring the zeta potential, wetting angle and sedimentation, we acquired a description of the surface properties of the colloid coal tailings in the liquid medium. Last but not least, the following criteria have been taken into consideration:

- formation of as stable floccules as possible,
- as clean and clarified water as possible for its reuse in the preparation technology,
- optimization of doses of the flocculation agents.

Materials

The tested flotation tailings are flotation tailings resulting from coal flotation in the coal preparation at the Paskov Mine, the Ostrava Coal Basin, the Czech Republic (CR). The chemical agents used to destabilize the medium and support the formation of separable floccules are the solutions of PAX 18 and Flokor 1,2A. They are a solution of polyaluminium chloride and a complex solution of aluminium chloride-hydroxide respectively. In the measurements we used water accordant with coal preparation plant process water, whose quality corresponds to service water.

Method of the experiments

The experiment was carried out in laboratories of Institute of Clean Technologies for Mining and Utilization of Raw Materials for Energy Use, at VSB Technical University of Ostrava. The chemism of the tailings was determined by means of WD-XRF spectrometry, making use of Spectron, SpectroscanMakc-GV. The electrokinetic measurements of the zeta potential were carried out using the ZetasizerNano S system from Malvern. The characteristics of the tailings were also studied for their leachability, subject to Waste Leachability Assessment Methodology of the Ministry of the Environment of the CR.

Flocculation tests were carried out in the form of jar tests. The method of the experiments is grounded in the principle of as fast as possible sedimentation of stable floccules. The aim was to optimize the dewatering process in a way so that during sludge treatment there was a high efficiency of floccule formation, they settled fast and dewatered in bucket lifts. The time for the formation of floccules, their stabilization and subsequent sedimentation did not exceed 100 seconds. The feed was determined based on the evaluation of the flotation tailings density at the coal preparation output. The ratio of tailings in experimental suspension corresponded to the long-term mean of the flotation tailings quantities in the sludge, i.e. 10g. The preparation of the samples, agent dosing and evaluations proceeded as follows: - feed of 10g (50g for further extension tests) of solids (dried and homogenized flotation tailings) in 1,000ml of process water,

- agitation of the suspension for perfect homogenization at laboratory temperature of 21°C,
- dose of flocculant at constant stirring at 250rpm,
- 60-second slow phase at 50rpm,
- sedimentation of the suspension.

The procedures above were complemented with an analysis of the suspension to identify pH, conductivity, turbidity and residual concentration of the applied agents. The quality of the medium and its stability was characterized via measuring the zeta potential. Next, viscosity of the medium participating in the zeta potential regime was also determined.

Results and discussion

Characterization of the flotation tailings

The properties of the flotation tailings are, to a certain extent, given by the properties of the mineral phases forming the material in question. The distribution of the individual minerals participates in the formation of colloid suspension, thus influencing the substance ion ratios in water. The chemism of the flotation tailings is also the major parameter which informs us about the potential leachability of elements into the aqueous medium, where flocculation takes place. It is necessary to define the chemism of the treated material in dependence on the choice of the reagents both for flocculation and effective dewaterEng.

The chemical analysis of the phase structure points at the abundance of amorphous, mullitic and silica phases. The material may be characterized as sandstone or siltstone which accompanies the coal mass in the conditions of the black coal basin in the Moravia-Silesian Region. The mineralogical characteristics, along with the chemical composition, indicate SiO2 and other oxides of aluminium, calcium and magnesium as the major components of the coal tailings. The ratios of the individual elements are summarized in Table 1.

Flocculation tests

Tailings suspension is a multicomponent medium, where the solid components in the form of fine mineral particles get in the contact with chemical agents in an aqueous solution. A significant parameter affecting

Tab. 1 Chemism of the flotation sludge

Tab 1 Skład chemiczny metów flotacyjnych

Abundance	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	Cl	K_2O	CaO	TiO_2	MnO	Fe_2O_3
	wt %											
Tailings	< 1.0	2.4	15.0	42.1	0.2	2.1	0.1	3.0	4.4	1.57	0.1	6.5

the suspension properties during flocculation is the pH of the medium. This influence has been reported in a number of works dealing with coal mass flocculation. [8, 9, 10]

The pH value of the medium helps to prepare suitable conditions for the start of flocculation and the conditions for particle sedimentation in the medium. The individual pH values of the medium are also related to the ratio of free H+ and OH- ions which participate in the formation of surface wan der Walls bonds between the fine particles and the wetting solution.

Within the flocculation tests making use of chemical reagents, the system of pH values - zeta potential - sedimentation must be complemented by studying the properties and behaviour of the given agents in the conditions of the treated suspension. It is important to define priority requirements for the suspension treatment. Two principles focusing on fast sedimentation at worse turbidity can be applied here. Otherwise, perfect sedimentation with minimum turbidity takes longer. The approaches mentioned above require different conditions depending on selecting optimal flocculation agents.

Used agents

The experiments made use of agents Flokor 1,2A, a solution of aluminium chloride-hydroxide, and PAX 18, a solution of polyaluminium chloride. When applying agents on the base of aluminium ions, the pH of the treated suspension must be taken into consideration. At the pH values ranging around 6, flocculation is not effective enough because of the occurrence of aluminium ions in the form of Al^{3+}.

According to Sabah, in the case of clay suspension of pH 5.5 or pH<6, the edges of the clay particles in the suspension are charged positively and the basal surfaces (alumosilicate layer) are charged negatively. During such conditions floccules of varying sizes are formed due to the interaction between the different particle polarities. [11, 12]

The optimal conditions for fast and efficient flocculation and sedimentation are considered as the medium's pH value around 8. The optimal agent doses were based on the quality of water, tailings suspension and conditions for fast sedimentation of the formed floccules.

The doses of aluminium chloride-hydroxide were selected in the interval from 0.05 to 0.40ml.l^{-1} at constant density of the suspension, i.e. 10g.l^{-1}. In the second stage, the amount of flotation tailing in suspension was gradually increased from 10 to 50g.l^{-1} and the optimal agent dose was determined as 0.05ml.l^{-1}.

An analogous principle was applied during the tests with polyaluminium chloride, when the dose was selected in the interval from 0.02 to 0.2ml.l^{-1} at suspension with 10g.l^{-1} of tailings. At the same time, the dose of 0.05ml.l^{-1} of the agent was tested at ratios gradually amounting to 50g.l^{-1}. Those tests with 50g have been done to prove the flocculation properties of used agents even in higher ratios of tailings suspension.

Interaction of the particles during the flocculation tests

It is clear from comparing the individual flocculation test parameters that the input agent dose and the amount of tailings in suspension influence the variations in the floccules formation and sedimentation. Taking turbidity, residual concentration of aluminium ions and the pH of the medium as controls for selecting the optimal dose for coagulation, the results may be summarized as follows.

As for the variations in the pH values of the medium, it may be stated that at lower Flokor or PAX 18 doses the medium gets stabilized and the pH does not fall below 6. Such values were acquired at doses of up to 0.4ml.l^{-1} of Flokor or of up to 0.08ml.l^{-1} of PAX 18. Dosing both the flocculation agents, the best dose for further optimization tests was determined as 0.05ml.l^{-1} of the flocculant.

The residual concentrations of the aluminium ions in the individual solutions were below the detection limit, i.e. below 0.05mg.l^{-1}.

Chart 1 shows the results of pH and turbidity using the individual doses of Flokor. For turbidity the limit was chosen as L.o.T – 40ZF and for pH the level of L.o.pH – 6. The individual doses demonstrate the gradual decrease in the pH value of the medium all the way to 6 at the top agent dose, i.e. 0.4ml.l^{-1}. Starting at 0.25ml.l^{-1} turbidity rises all the way to the limit value of 40ZF.

As for PAX 18 flocculant, the conditions for effective dewatering were set within identical limits as mentioned above. It proved that doses below 0.1ml.l^{-1} are effective in dewatering flotation sludges at forming stable floccules. The dose of 0.05ml.l^{-1} provides sufficiently stable conditions for the dewatering process in short sedimentation time. Chart 2 shows the variations in pH and turbidity using the PAX 18 flocculant.

Effect of the zeta potential on the stability of the medium

The verification of the medium stability using the individual doses of Flokor and PAX 18 also included the measurements of the zeta potential of the suspension. Both using Flokor and PAX 18 the zeta poten-

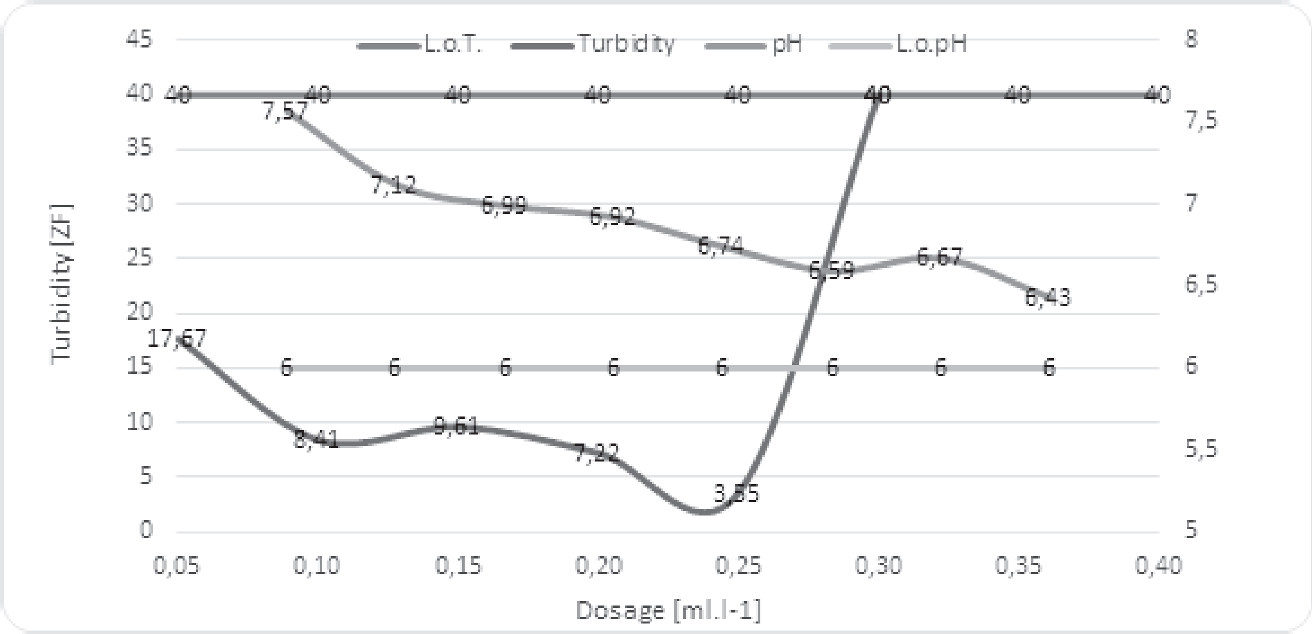

Chart 1 Values of pH and turbidity dosing Flokor

Wyk. 1 Wartość pH i zmętnienie w obecności odczynnika Flokor

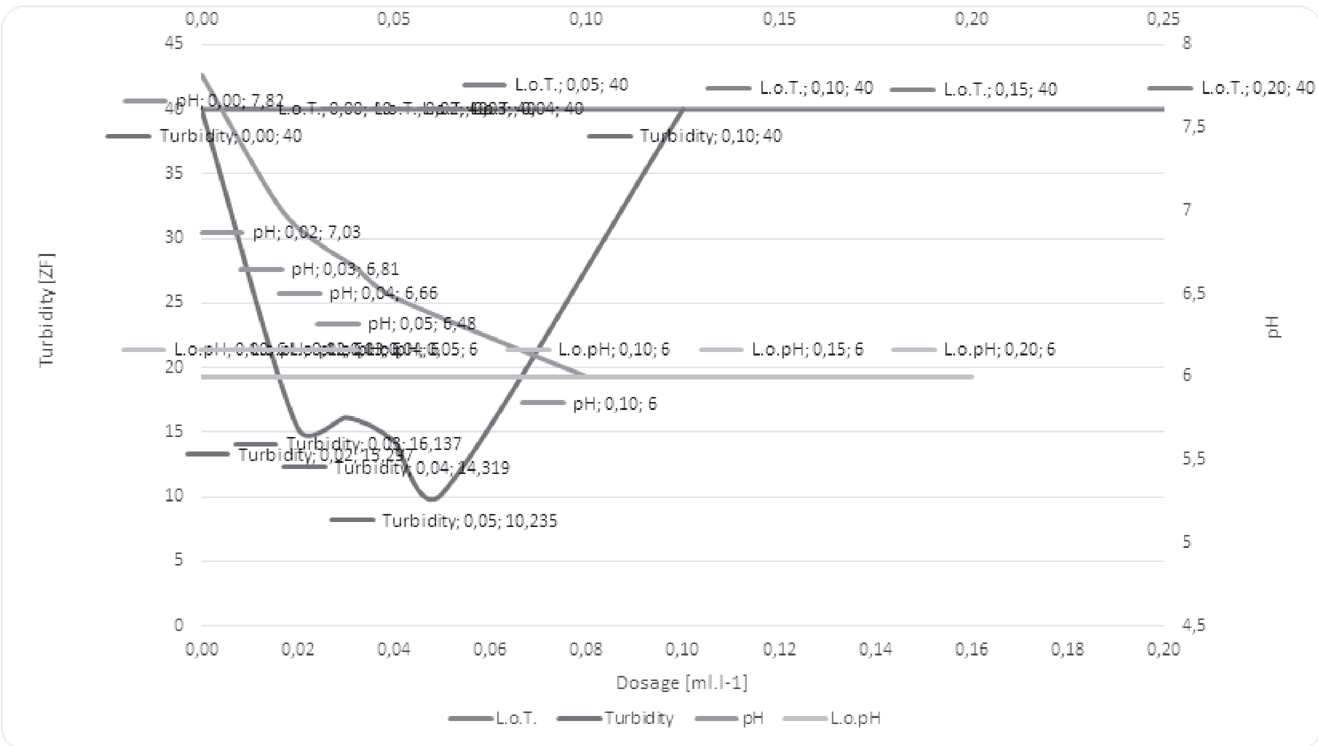

Chart 2 Values of pH and turbidity dosing PAX

Wyk. 2 Wartości pH i zmętnienie w obecności odczynnika PAX

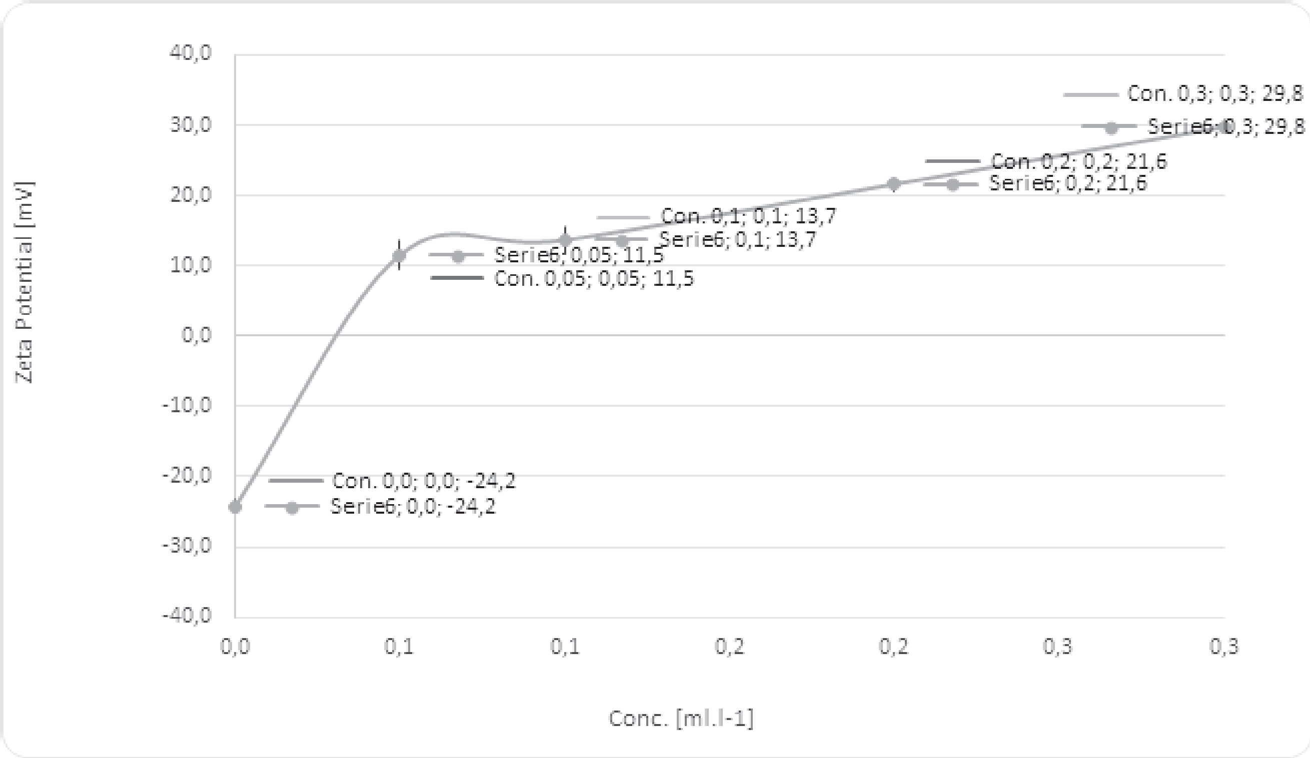

Chart 3 The effect of dosing Flokor on the suspension's zeta potential.

Wyk. 3 Wpływ dawkowania odczynnika Flokor na potencjał dzeta zawiesiny

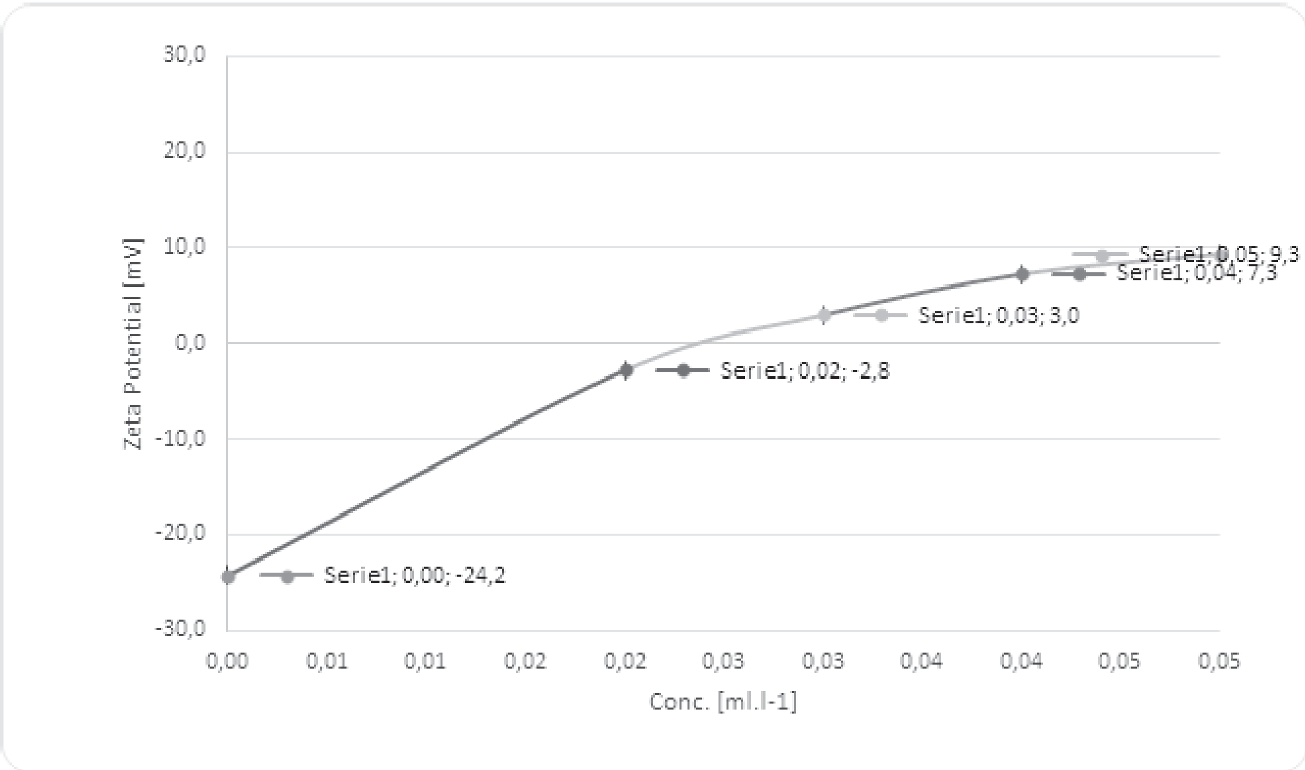

Chart 4 The effect of dosing PAX 18 on the suspension's zeta potential

Wyk. 4 Wpływ dozowania PAX 18 na potencjał dzeta tial was measured at a zero agent dose first and then the doses were increased all the way to the optimal dose ($0.05ml.l^{-1}$). The results imply that already at low agent doses the surface changes occur, which are represented by a rise in the zeta potential values towards the isoelectric point, crossing to positive. Negatively charged surfaces of the tailings particles, or of silicate minerals, are a domain where the charge compensates owing to the effect of functional aluminium ions. It is clear from the results that the compensation of the surface charge occurs already at low concentrations of aluminium chloride-hydroxide or polyaluminium chloride. Chart 3 shows the variations in the zeta potential of the suspension dosing the Flokor agent.

Increasing the doses of Flokor there was a rise in the surface potential of the suspended tailings particles. The optimal limit for doses for good sedimentation appears to be $0.1ml.l^{-1}$, which provides sufficient conditions for the formation of agglomerates and fast sedimentation. A further increase in the dose already brings worse conditions in the process water, in the form of higher turbidity and variations in pH.

Chart 4 reveals the dependence of dosing PAX 18 and the zeta potential values of the suspended particles. In this case the isoelectric point was reached at the dose interval from $0.02ml.l^{-1}$ to $0.03\ ml.l^{-1}$. Dosing the agent up to $0.3ml.l^{-1}$ the conditions for sedimentation already deteriorated and the pH value of the medium fell below 6. It also shows that the hydrolyzing solutions of the applied agents form strong bonds with H^+ and OH^- ions on the surface of the suspended particles and significantly compress the electric double layers. The negatively charged surfaces of the suspended tailings particles give space to the formation of new compensation centres, around which hydrolyzed aluminium ions combine. This bond thus provides the base for forming the agglomerates of suspended tailings particles and their sedimentation.

Observing the effect of an optimal dose on the stability of dewatering the suspension, we also tested the effect of the suspension density on the optimal dose. During the tests the optimal doses of Flokor and PAX 18 ($0.05ml.l^{-1}$) were dosed into the suspension at densities of 10 and $50g.l^{-1}$. The results of measuring the zeta potential of the suspended particles at such doses are in Chart 5. It is clear that there is a leap in the suspension's zeta potential from -20mV to values around 10mV. It is also apparent that the optimal dose of the active agents is sufficient for good sedimentation of the suspension at either density, i.e. 10 or $50g.l^{-1}$. Next, it shows that there is drop in the agent's activity at higher densities. This result correlates with the outcomes when determining the turbidity and sedimentation as surface bonds form between ions, along with enveloping the surface of the suspended particles. This way certain centres appear serving as bases for the formation of larger floccules. The decrease in the zeta potential of the suspended particles is caused by the effect of larger particle surface in thicker medium.

Sedimentation of the tailings suspension

In order to evaluate the sedimentation properties of the tailings suspensions, sedimentation was tested in measuring cylinders. Optimal doses of Flokor and PAX 18 were selected for the tests and added to the suspensions of two densities, i.e. 10 and $50g.l^{-1}$. The results of the sedimentation tests, as shown in Chart 6, point at a good development of floccule formation and short sedimentation of the flocculating tailings particles. The total sedimentation time to reach the clarification stage and for the floccules to settle on the bottom of the cylinder was 120s. Variations in the size of the floccules are clearly visible at the different densities of the tailings suspension. At the density of $10g.l^{-1}$ larger floccules were observed using PAX 18, while the turbidity of the clarified water was comparable in both the cases. At the higher density, i.e. $50g.l^{-1}$, the situation was reverse as larger floccules were observed when Flokor was applied. It is further apparent from the sedimentation test results that at the higher density of the tailings suspension, very good sedimentation occurred and the process water clarified well. However, when compared to the lower density tests, the turbidity grows in the treated tailings suspension. Turbidity is predominantly caused by very fine particles of the flotation tailings, being uplifted especially due to the circulation, Brownian motion and repulsation. Prolonging the time of sedimentation and the zeta potential around the isoelectric point, the particles tend to form gradually settling floccules. The residual abundance of polyaluminium chloride or aluminium chloride-hydroxide ensures active centres on the surface of the suspended particles for better cohesion of the floccules. Chemical analysis of decanted water from sedimentation process showed that the water could be re-used for further coal (flotation) separation process.

Conclusion

The flocculation tests with subsequent sedimentation manifest that the application of the selected agents on the base of aluminium chloride are practicable and promising for further tests and use in industry. This work points at the fact that there is a close dependence among the choice of the optimal agent, its dosage, character of the suspended particles and sedimentation under required conditions. Combining the above stated parameters, it is possible to achieve ef-

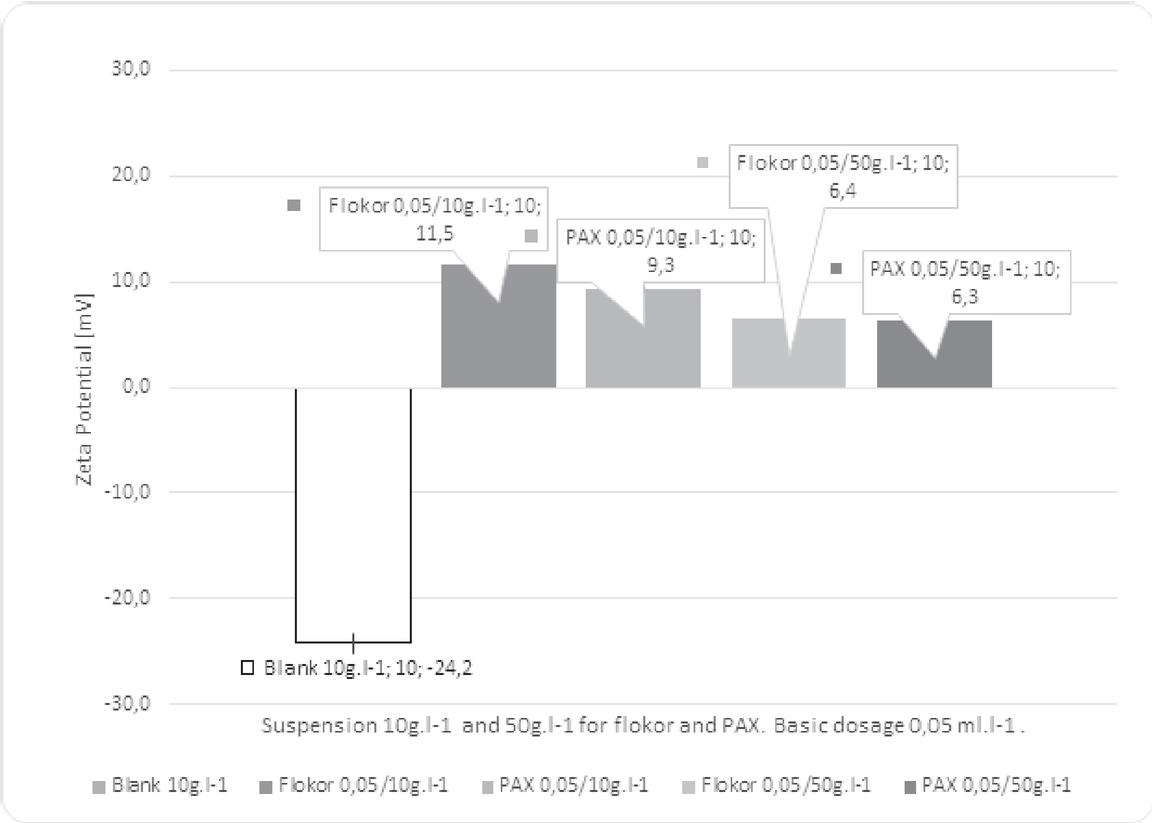

Chart 5 The effect of density and the optimal dose on the zeta potential values

Wyk. 5 Wpływ gęstości i optymalnej dawki w wartości potencjału dzeta

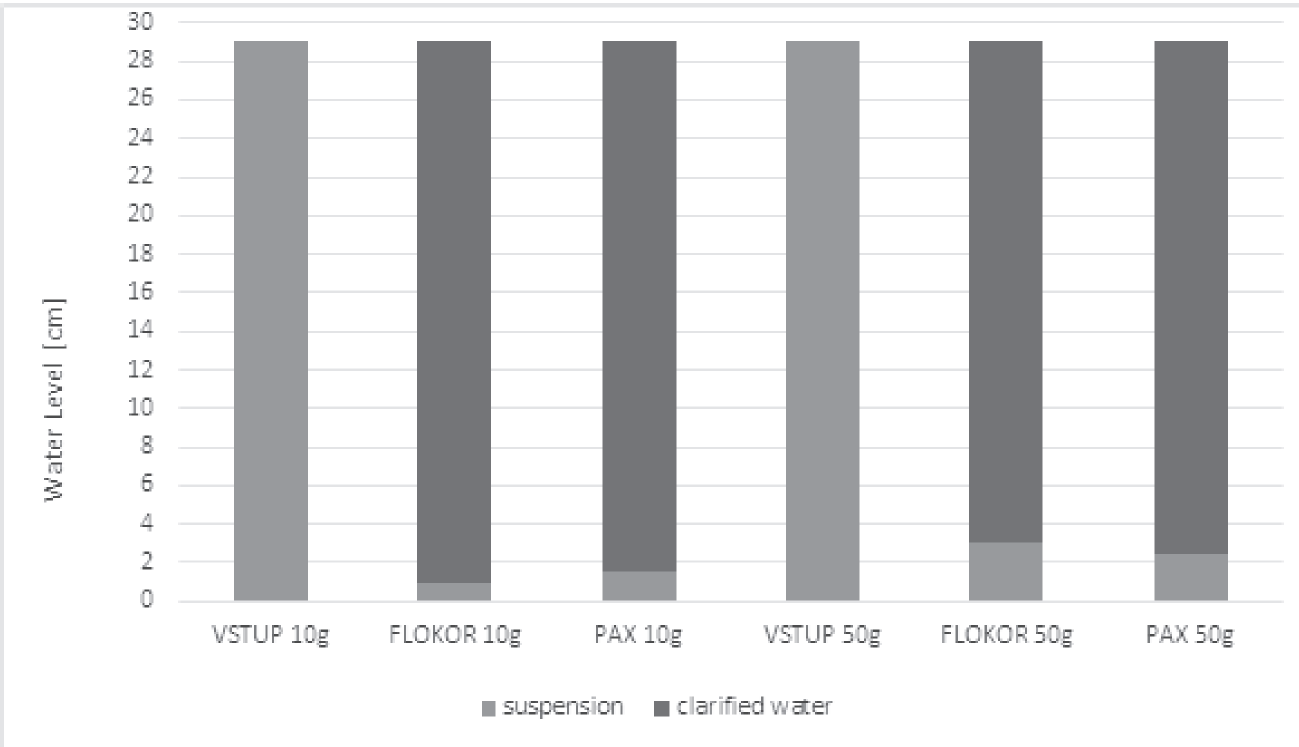

Chart 6 Sedimentation of the tailings suspension at the optimal doses of Flokor and PAX 18

Wyk. 6 . Sedymentacja zawiesiny odpadów górniczych przy optymalnych dawkach Flokor i PAX 18 fective sedimentation in short time. The optimal agent dose accelerates the sedimentation process, and at the same time, it does not worsen the conditions in the process water for its further re-use. Another positive result is also the fact that well settling floccules are formed regardless of the tailings suspension density. An important parameter of the sedimentation quality is also the stability of the floccules and cohesion of the material under sedimentation for further dewatering in bucket lifts. The study of the zeta potential shows good bonding of the agents in the suspended medium and conditions for good flocculation in media whose potential is close the isoelectric point.

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Streszczenie

Intensyfikacja procesów wydobywczycg jest odpowiedzalna za powstanie ponad 45% odpadów górniczych i przeróbczych, które powstaja w procesie wzbogacania węgla. Z chemicznego punktu widzenia, jest to materiał głównie składający się z krzemianów (pyłów, konglomeratów, kaolinu lub montmorylonitów). Współczesne doświadczenia in situ wykazują, że wzrost ilości zanieczyszczeń drobnoziarnistych powoduje trudności w procesie sedymentacji i gorszym wzbogacaniu frakcji drobnouziarnionych. W przeszłości, odwadnianie odpadów flotacyjnych było oparte na procesie sedymentacji. Aktualnie staje sie to bardzo czasochłonne, z uwagi na charakter koloidalny cząstek odwadnianych.W artykule przedstawiono wyniki doświadczeń nad weryfikację wzajemnej interakcji między zawiesiną węglową i odczynnikami chemicznymi (flokulantami) 'Flokor 1.2A' i 'PAX 18' stosowanymi w układzie odwadniania. Przy niższych dawkach flokulantów Flokor 1,2 A i Pax 18, warunki sedymentacji stabilizowały się, wartość pH nie spadała poniżej 6, dla dawek flokulantów 0.4ml/

dm i do 0.08ml/l. Dawkę flokulantów 'Flokor 1.2A' i 'PAX 18' równa 0.05ml/l uważa się za optymalną. Wniki okreslenia potencjału zeta wykazuje, że powierzchnia ładowania zostaje skompensowana już przy niskich stężeniach chlorku zarówno wodorotlenku glinu i chlorku glinu. Osiągnięto punkt izoelektryczny w przedziale dawki w zakresie od 0.02ml.l^{-1} - 0.03ml.l^{-1}. Wyniki zostały uzyskane w stawach zawieszenia gęstości od 10 do 50g.l^{-1}.

Keywords: odwadnianie, potencjału zeta, krzemiany, flokulacji, sedymentacji