Flotation Tailings – Intensification of the Dewatering Process by the Aluminium Pre-Hydrolysed Coagulants

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Abstract

Most mineral-separation processes involve the use of substantial quantities of water and a larger portion of it ends up in a tailings stream. Typically, sedimentation and filtration are used to dewater the coal product. Large amount of dilute suspension has to be treated. The main driving force for coal tailing treatment is the elimination of unsustainable disposal of fine coal tailings in large tailings dams. Coagulation/Flocculation is usually a necessary pre-treatment step in dewatering streams containing significant quantities of very fine particles. The effectiveness of the coagulation/flocculation step may determine the performance and, ultimately, the capacity of the dewatering system. The objective of dewatering processes is often to obtain clear water with low percentage of solids. The article presents the results of possibility of intensification of dewatering of the flotation tailing streams from coal preparation Paskov mine (Czech Republic). The results imply that the selected aluminium pre-hydrolysed coagulants (aluminium chlorohydrate – FLOKOR1.2A, FLOKOR D15, polyaluminium chloride – PAX-18) are convenient in dewatering process of tailing slurries and are practicable for further tests and use in industry. It is possible to achieve effective dewatering in short time. Optimal doses of all coagulants do not worsen the conditions in the process water for its hypothetic re-use. Experiments were conducted in temperature interval from 5°C to 20°C. Standard jar tests were performed to evaluate coagulation efficiencies. Different mixing conditions were evaluated. Relation between tailings and addition of coagulant was characterized by zeta-potential. Turbidity, pH values and residual concentrations of aluminium were measured.

Keywords: tailing slurry, flotation, dewatering, coagulation, flocculation, turbidity, zeta-potential

Introduction

Most mineral-separation processes involve the use of substantial quantities of water and a larger portion of it ends up in a tailings stream with a smaller portion in the form of concentrate bearing the target mineral. Water recovery becomes an integral part of the mining operation at the lack of effective water recovery has economic and environmental implications particularly where water is scarce (Castro et al., 1998).

Flotation is widely used to clean fine coal particles by separating them from clay, silt, shale and other ash-producing matter using air bubbles. After flotation either the coal or tailings slurries must be dewatered for practical and economic advantages. Typically, sedimentation and filtration are used to dewater the coal product (Hogg, 2000). Although filtration is expensive and has conventionally been used for dewatering the coal product, in the case of coal tailings dewatering, a large amount of dilute suspension has to be treated and thickening is very cost-effective (Lockhart and Veal, 1996).

The main driving force for coal tailing filtration is the elimination of unsustainable disposal of fine coal tailings in large tailings dams (Scheiner, 1996; Scheiner and Moudgil, 1989).

Coagulation/flocculation (C/F) is usually a necessary pre-treatment step in dewatering streams containing significant quantities of very fine particles. For such systems, the effectiveness of the C/F step may determine the performance and, ultimately, the capacity of the dewatering system.

Effectiveness of the process depends not only on the use of appropriate chemical reagents (coagulants, flocculants, etc.) but also on how they are applied and how the process can be controlled so as to achieve optimum performance in the context of dewatering operations such as sedimentation and filtration. The objective of dewatering processes is often to obtain clear water with low percentage of solids.

Tab. 1. Chemism of the flotation tailings

Tab. 1. Chemizm odpadów 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 %)	< 1.0	2.4	15.0	42.1	0.2	2.1	0.1	3.0	4.4	1.57	0.1	6.5

Tab. 2. Some specifics of coagulants, dosages of coagulants and density of suspension

Tab. 2. Wybrane właściwości koagulantów, dawki koagulantów, gęstość zawiesiny

Coagulant	pH (-)	Ratio OH/Al	Al^{3+} (%)	Al_2O_3 (%)	Cl^- (%)	Specific density (g/cm^3)	State	Suspension density (g/L)	Dosage per one L of suspension (μL/L)
FLOKOR 1.2A	4.2	2.40	11.00	20.79	7.00	1.280	aq. solution	10	50
FLOKOR D15	3.5	2.40	6.00	11.34	6.00	1.200	aq. solution	10	92
PAX-18	1.0	n/a	9.00	17.00	21.00	1.360	aq. solution	10	61

Despite various progressive viewpoints in understanding interactions between particles and coagulants (Dentel and Gosset, 1988; Van Benschoten and Edzwald, 1990; Letterman and Asolekar, 1990; Duan and Gregory, 2003), four primary mechanisms were commonly recognized, i.e. double layer compression, charge-neutralization, sweep-flocculation and bridge-aggregation.

The aim of experiments is testing suitability of the selected coagulants for potable optimization of flotation tailing slurries dewatering. A possibility of the recovery treated water into the flotation process was tested. It was studied an effect of residual coagulants on coal.

Material and methods

All experiments and analyses were 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 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 mineralogical characteristics, along with the chemical composition, indicate SiO2 and other oxides of aluminium, calcium, magnesium as the major components of the coal tailings. The chemism of flotation tailings is summarized in table 1. The chemism of the tailings was determined by means of WD-XRF spectrometry, making use of Spectroscan Makc-GV (Spectron, Ltd. RU).

The coagulants used to destabilize the medium and support the formation of separable floccules were the aquatic solutions of aluminium chlorohydrate – ACH (trade name FLOKOR 1.2A and FLOKOR D15) and polyaluminium chloride – PACl (trade name PAX-18). Coagulants of FLOKOR series were provided by DEMPOL-ECO (PL) and PAX-18 was provided by Kemwater ProChemie (CR). Some characteristics of coagulants are mentioned in table 2. Their mode of action is generally explained in terms of two distinct mechanisms: charge neutralization of negative charged colloids by cationic hydrolysis products and incorporation of impurities in an amorphous hydroxide precipitate ("sweep flocculation"). In the measurements we used water accordant with coal preparation plant process water its quality corresponds to service water.

Standard jar tests were conducted to evaluate coagulation efficiencies. One litre of water and 10g of tailings were feed into a 2-liter glass beaker. Coagulant was added to the beaker while mixing at 250rpm and suspension was well homogenized. Thereafter the mixing speeds were conducted at 250rpm for 30s followed by a slow mixing at 50 rpm for 60s. The residual turbidity was measured via a VIS spectrophotometer DR3900 (Hach Lange, D) after the slow mixing in settling times of 60 s, 240 s and 600 s. The zeta potentials were measured via a laser zeta analyser Zetasizer nano ZS (Malvern Inc., UK) 60 s after the slow mixing. Temperature, pH and conductivity were recorded too.

All coagulants dosages used in this study were calculated by optimal dosage of coagulant FLOKOR 1.2A (optimal dosage of 50 μL/L correlates in amount of 5.5026 mg Al ions/L suspension). It was determined in experiments before (Thomas et al., 2014a; Thomas et al., 2014b), where isoelectric point was reached in pH 7.80

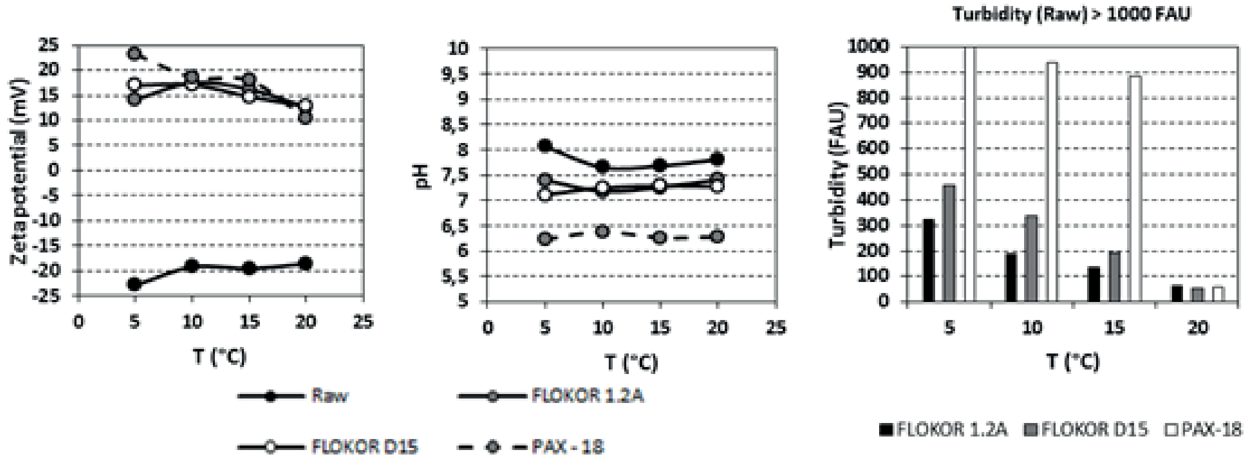

Fig. 1. Effect of coagulant addition on zeta potential, pH and turbidity values of tailing slurries at mixing conditions 30 s/250rpm and 60 s/50rpm in temperature interval from 5°C to 20°C measured after 60 s sedimentation

Rys. 1. Wpływ dodatku koagulantu na potencjał zeta, pH, granicę mętności dla warunków mieszania 30 s/250obr/min i 60 s/50obr/min w temperaturze od 5°C do 20°C , czas sedymentacji 60 s

Tab. 3. Turbidity reduction of tailing slurries treated at mixing conditions 30 s/250rpm and 60 s/50rpm in temperature interval from 5°C to 20°C measured after 60 s, 240 s and 600 s sedimentation

Tab. 3. Zmiana mętności zawiesiny odpadów w różnych warunkach mieszania 30 s/250 obr/min i 60 s/50 obr min w temperaturze 5°C do 20°C, mierzone po czasie sedymentacji 60 s, 240 s i 600 s

Coagulant	Turbidity reduction (%)											
	5°C			10°C			15°C			20°C		
	60s	240s	600s	60s	240s	600s	60s	240s	600s	60s	240s	600s
Blank	0	10	15	0	10	22	0	12	22	0	6	10
F1.2A	68	75	86	81	87	91	86	89	92	**94**	**94**	**94**
FD15	55	67	73	66	74	81	81	85	91	**95**	**95**	**96**
PAX - 18	0	18	29	6	23	33	12	28	40	**94**	**95**	**97**

and dosage was in the interval between 20 and 30 μL/L. Suspension density was 10 g/L.

Dosages of all coagulants are summarized in table 2. In first step all experiments were conducted at constant laboratory temperature 20°C. In next steps experiments were conducted at temperatures of 5°C, 10°C and 15°C to simulate full-scale conditions.

Results and discussion

Collected data of turbidity, selected water chemistry parameters (pH, Al^{3+}, conductivity) as well as zeta potential of the tailing slurries particles as a function of coagulant dosage, temperature and utilization mixing properties were examined. Especially turbidity, residual concentration of aluminium ions and pH of treated water were taken as a control of C/F process.

Figure 1 illustrates the effect of FLOKOR 1.2A, FLOKOR D15 and PAX-18 addition on zeta potential, pH and residual turbidity at mixing conditions 250 rpm 30 s and 50 rpm 60 s in temperature interval from 5°C to 20°C after the 60 s sedimentation.

C/F result for aluminium based coagulants indicate that positive trend of zeta potential suggests that charge neutralization by adsorption/deposition of the positively charged coagulants precipitates onto the negatively charged tailing slurries particle surface.

Using of all coagulants it is clear that there is a leap in the tailing slurries suspension's zeta potential from values around -20mV to values around 20mV at 5°C respectively around 10mV at 20°C. In all cases pH values at optimum doses did not fall 7 (FLOKOR 1.2A, FLOKOR D15) respectively 6 (PAX-18). The residual concentrations of the aluminium ions in the individual treated water were below the detection limit, i.e. below 0.05mg/L.

It proved that optimal doses of all coagulants are effective in dewatering flotation tailings at

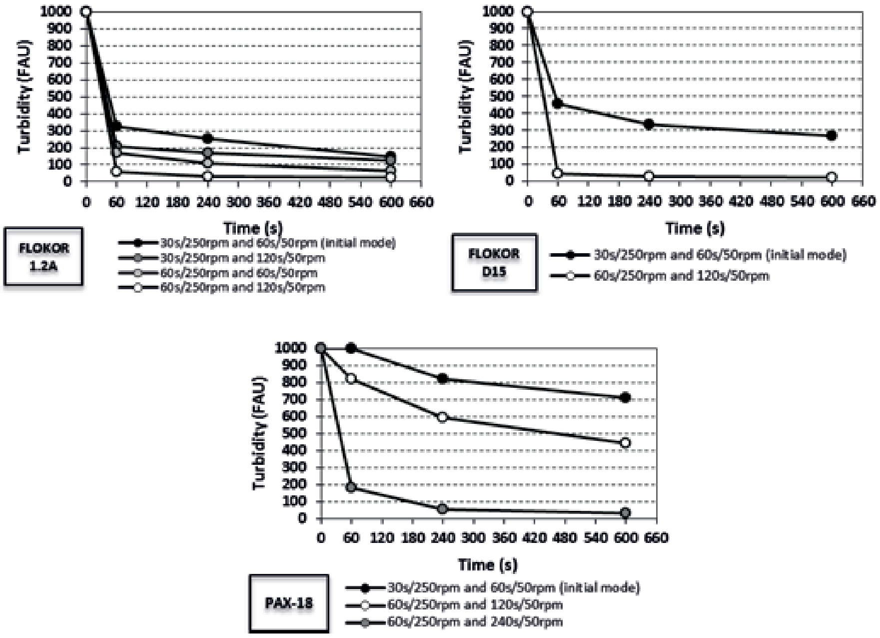

Fig. 2. Turbidity values in settling time for tailing slurries treated at different mixing conditions and at 5°C

Rys. 2. Granica mętności po czasie sedymentacji dla temperatury 5°C

Tab. 4. Turbidity reduction of tailing slurries treated with FLOKOR 1.2A at different mixing conditions and at 5°C measured after 60 s, 240 s and 600 s sedimentation

Tab. 4. Zmiana mętności zawiesiny po zastosowaniu koagulantu FLOKOR 1.2A dla temperatury 5°C, czas sedymentacji 60 s, 240 s i 600 s

Mixing conditions at 5°C	Turbidity reduction (%)		
	60s	240s	600s
30s/250rpm and 60s/50rpm*	68	75	86
30s/250rpm and 120s/50rpm	79	83	88
60s/250rpm and 60s/50rpm	83	89	94
60s/250rpm and 120s/50rpm	**94**	**97**	**98**

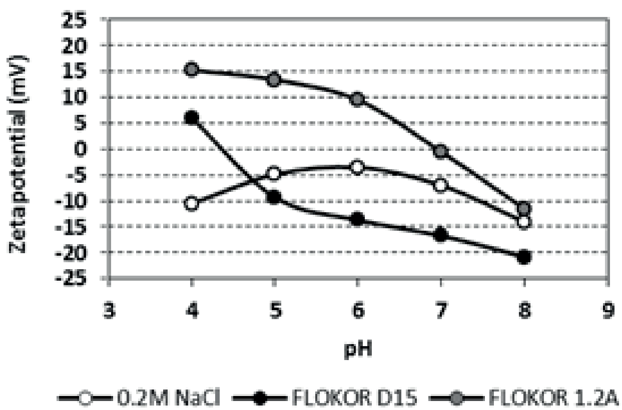

Fig. 3. Zeta potential and pH values of the coal sample in treated water from dewatering process of tailing slurries and 0.2M NaCl solution

Rys. 3. Potencjał zeta I pH próbek węgla w wodzie z procesów odwadniania i roztworze 0.2M NaCl forming stable floccules in short sedimentation time at 20°C. Effectiveness of turbidity reduction was around 95±2% in all cases at 20°C. Initial mixing mode was 250rpm for 30 s and 50 rpm for 60 s. Experiments conducted in temperature interval from 5°C to 15°C were not so effective like at 20°C. It is summarised in table 3. PAX-18 did not work in temperatures from 5°C to 15°C so different mixing conditions were tested.

From the first the changed mixing conditions were tested with coagulant agent FLOKOR 1.2A at 5°C. The effect of changed mixing conditions corresponds with turbidity reduction. Efficiency of the changed and initial mixing conditions at 5°C for FLOKOR 1.2A is summarized in table 4. The best results were achieved in double time of mixing conditions, i.e. 250 rpm for 60 s and 50 rpm for 120 s. The higher dosage was tested too but the effect on turbidity reduction was same as with optimal dosage.

Remaining two coagulants were tested with changed mixing conditions. Like changed mixing conditions were selected 250 rpm for 60 s and 50 rpm for 120 s. Turbidity reduction at 5°C with FLOKOR D15 was comparable to results of FLOKOR 1.2A. PAX-18 is applicable at 5°C but the times of slow mixing conditions must be longer four times. Compared mixing conditions and trends of turbidity reduction, of all coagulants at 5°C, are illustrated in figure 2.

In cases of recovery treated water into the flotation process it was needed to test effect of residual coagulants on coal. It was proved by measuring zeta potential and wetting angle black coal in laboratory conditions.

As process water was used treated water from dewatering process comments before (used coagulant FLOKOR 1.2A and FLOKOR D15) and 0.2M NaCl solution. The pH values were adjusted by molar solutions of NaOH and HCl. Measured zeta potential values are shown in figure 3.

Wetting angle was measured in the apparatus Tensiometer Attension Theta. Contact angle was evaluated directly by measuring of the angle formed between the solid particle and the tangent to the surface of the test fluid droplets. Contact angle was measured on polished samples of coal. Coal samples have been taken from active part of Paskov Mine coal district with technological granulation 0–0.5 mm.

The results of the surface properties of the materials show a strong dependence of electro-kinetic to pH solutions and mineralized water. Measured values of electro-kinetic potentials of a relatively low ashes coal samples indicate the suitability of a non-polar hydrophobic agents for treatment. Determination of wetting angle results of surface electro-kinetic properties of real value added wettability with a visual assessment of the three-phase contact. Measurement results show sufficient wetting angle of the surface hydrophobicity of particles of coal samples with values between 70° to 77° and it is good for flotation process.

With increasing in pH values of the coal slurries is increased negative zeta potential, probably within changes in adsorption of OH^- ions. At neutral pH of technological (operational) water in flotation process are the values of measured zeta potential in the range from 0mV to -20mV. The value of 0mV (isoelectric point) indicates, that there are no space repulsions – particles are neutral.

Conclusion

Coagulation/flocculation (C/F) tests manifest that the selected aluminium based coagulants are convenient in dewatering process of tailing slurries and are practicable for further test and use in industry. There is a close dependence among the choice of the optimal coagulant agent, its dosage, temperature, mixing conditions, character of the suspended particles and sedimentation under required conditions. Combining the above stated parameters, it is possible to achieve effective dewatering in short time. Optimal doses of all coagulants do not worsen the conditions in the process water for its hypothetic re-use. An important parameter of the C/F process is also the stability of the floccules and cohesion of the material under sedimentation. The study of the zeta potential shows good bonding of the agents in the suspended medium and conditions for good C/F in media whose potential is close the isoelectric point.

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Odpady flotacyjne – intensyfikacja procesu odwadniania koagulantem glinowym

Większość procesów separacji minerałów wiąże się z zużyciem znaczących ilości wody, której większość trafia do strumienia odpadów. Zazwyczaj w celu odwodnienia węgla wykorzystuje się procesy sedymentacji i filtracji. W efekcie trzeba zagospodarować duże ilości rozrzedzonej zawiesiny. Największym wyzwaniem w przetwarzaniu odpadów węgla jest eliminacja nieopłacalnego składowania odpadów drobnego węgla w ogromnych stawach osadowych. Koagulacja/flokulacja jest często ważnym wstępnym krokiem w procesie odwadniania strumieni zawierających znaczące ilości bardzo drobnych cząsteczek. Skuteczność koagulacji/flokulacji może określić skuteczność, oraz ostatecznie, przepustowość systemu odwadniającego. Częstym celem procesu odwadniającego jest uzyskanie czystej wody z niską zawartości fazy stałej.

Artykuł przedstawia wyniki badania możliwości intensyfikacji odwodnienia strumieni odpadów flotacyjnych z kopalni węgla Paskov (Republika Czeska). Wyniki wskazują, że wybrane hydrolizowane koagulanty glonowe (aluminiumchlorohydrate – FLOKOR1.2A, FLOKOR D15, polyaluminium chloride – PAX-18) są odpowiednie do procesu odwadniania zawiesin w praktyce przemysłowej. Możliwe jest osiągnięcie skutecznego odwodnienia w krótkim czasie.

Optymalne dawki koagulantów nie pogarszają jakości wody do hipotetycznego ponownego użycia. Eksperymenty zostały przeprowadzone w przedziale temperatury od 5°C do 20°C. Standardowe testy metodą sedymentacji zostały przeprowadzone w celu określenia skuteczności koagulacji. Różne warunki mieszania zostały zbadane. Określono potencjał zeta, zmierzono mętność, wartości pH oraz stężenia osadów koagulantu.

Słowa kluczowe: zawiesina odpadowa, flotacja, odwodnienie, koagulacja, flokulacja, mętność, potencjał zeta