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Effect of surfactants on the flotation performance of low-rank coal by particle sliding process measurements

Introduction

In the northwest of China, low-rank coals such as lignite, brown coal and sub-bituminous coal reserves are very abundant (Rao et al. 2015), especially, lots of the sub-bituminous coal beds were mined in the Shendong Coalfield. However, the hydrophobicity of low-rank coal particle surfaces containing abundant oxygenated functional groups such as hydroxyl, carbonyl and carboxyl, was generally reduced. Hence, the oxidized surfaces of low-rank coal particle surfaces easily form hydrogen bonds with water molecules. Therefore, the low-rank coal particle surfaces are covered with a stable and thick layer of water film. It is well known the flotation performance of mineral particles is determined by the induction time. At a given hydrodynamic condition in flotation process, shorter induction times indicate higher flotation recoveries (Albijanic et al. 2010; Gu et al. 2003; Sutherland 1948; Yoon and Yordan 1991). Therefore, a high yield of low-rank coal would be impossible to be obtained. This is because a particle would need more induction times (or attachment times) to conquer the stable and thick wetting film on its surface and attach to an air bubble surface in the flotation process.

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In order to improve the flotation performance of low-rank coal, surface modification methods such as chemical reagents adsorption (Chander et al. 1994; Wójcik et al. 1990) and oxygen-containing functional group modification on low-rank coal surfaces (Majka-Myrcha and Girczys 1993; Xia et al. 2012, 2013), were proposed. It is observed that the flotation recoveries of both laboratory-oxidized and naturally weathered coals increased using the nonionic surfactants; however, the oxidized coal particles were difficult to be floated with dodecane usage only (Harris et al. 1995). It has recently been found that a series of non-ionic surfactants, tetrahydrofuryl butyrate, can be used as the more effective collectors than traditional oily collectors, e.g., diesel, kerosene and dodecane for both oxidized and unoxidized coal flotation (Jia et al. 2000). This is because the various functional groups on the nonionic surfactant surfaces will interact with the oxygenated sites on the coal particle surface, orient the hydrocarbon chain to the water interface and spread on the coal particle surfaces more easily than dodecane or kerosene; thereby the flotation performance of oxidized coal could be improved (Aplan 1993; Harris et al. 1995).

In flotation, it was also observed that a high recovery of low-rank or oxidized coals could be obtained using ionic reagents such as: tributyl phosphate, carboxylic acids, and amines with a collector function (Aplan 1993). The ionic fatty acids acting as a collector possessed a better flotation selectivity than nonionic reagents (Sis et al. 2003). Vamvuka and Agridiotis (2001) concluded that cationic surfactants were more favorable to enhancing the hydrophobicity of lignite than anionic and nonionic surfactants. It was also found that the cationic dodecyl amine (DDA) as a collector played a significant role in the flotation response of the lignite than that of kerosene but its flotation selectivity for reducing the coal ash content was not good (Kelebek et al. 2008; Qu et al. 2014). The flotation performance showed that the absorption phenomenon occurs between the negatively charged carbonaceous group of dodecylammonium and mineral matter on the low-rank bituminous coal surface, which increased the hydrophobicity of weathered coal (Bustamante and Woods 1984). Therefore, the interaction between surfactants and coal particle surfaces was investigated by many researchers, which can be applied to explaining the hydrophobicity improvement of a low-rank coal surface.

The non-ionic surfactants with oxygenated functional groups, as a collector, can significantly improve the hydrophobicity of low-rank and oxidized coal surfaces. This is because the oxygenated functional sites of the non-ionic surfactants will interact with the hydrophilic surface of the low-rank coal particles by hydrogen bonding (Jia et al. 2000). The surface hydrophobicity of low-rank and oxidized coal can be significantly improved, which is due to the cationic adsorption on the negatively charged sites of low-rank and oxidized coal surfaces by electrostatic attraction (Bustamante and Woods 1984; Kelebek et al. 2008; Vamvuka and Agridiotis 2001). Due to both hydrogen bonding from the anionic surfactant's polar group and hydrophobic bonding phenomenon with non-polar groups, the flotation collector kerosene shows a strong tendency to spread on the coal surface (Burkin and Bramley 2010a, b; Cebeci 2002). Therefore, the surface hydrophobicity of low-rank and oxidized coal particles can be greatly improved.

In the last decade, the induction time between mineral particle bed and a bubble was investigated using the induction timer setup (Gu et al. 2003; Su et al. 2006). The induction time between a fixed bubble and mineral particle bed is investigated in a breaker with a static measurement environment. Moreover, the generated bubble at the end of a capillary has an immobile surface. The parameters of the induction time tests in each experiment including the initial gap (h_0) between the mineral particle bed and the fixed bubble, the displacement (H_0) and diameter (d_b) of the bubble, and the velocities of the bubble approaching to (u_a) and retraction from (u_r) the mineral particle bed are shown in Fig. 1 (Gu et al. 2003).
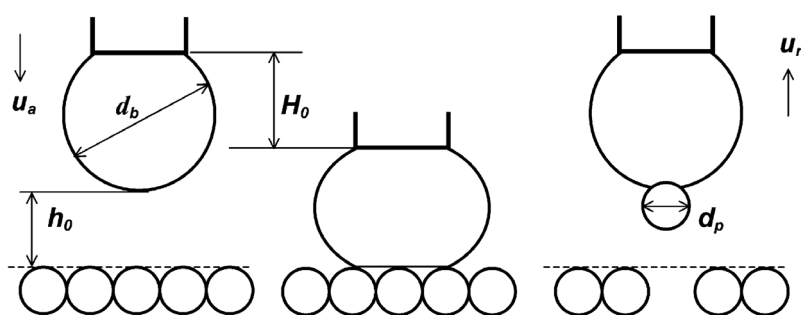

Fig. 1. A schematic of the measurement of induction time (Gu et al. 2003)

Rys. 1. Schemat pomiaru czasu indukcji (Gu et al. 2003)

It was found that the induction time between the bitumen particle bed and the bubble decreased with increasing in the extent of air bubble compression ($H_0 - h_0$) (Gu et al. 2003). Therefore, it indicated that a greater extent of air bubble compression ($H_0 - h_0$) would drive the water film to thin quickly between the bitumen particle bed and the bubble, which corresponds with the theoretical modelling of Jowett (Jowett 1980). However, the impact interaction with a greater extent of air bubble deformation is unfavorable to the attachment of mineral particles to the bubble surface in the real flotation process (Schulze et al. 1989).

During the flotation process, three types of the interaction between particles and bubbles can be observed:
- the thinning process of wetting film between a bubble surface and a particle to a critical thickness,
- the rupture of wetting film at the critical thickness and formation of an initial three phase contact nucleus (Schulze et al. 1989; Sutherland 1948),
- the expansion of initial three phase contact to a stable wetting perimeter (Albijanic et al. 2010; Nguyen et al. 1998; Sven-Nilsson 1934).

In the first step, the drainage time occupied by the thinning process accounts for the predominant part of the induction time, which can reach 76% to 94% of the entire induction time. It is also found that various drainage models of the water film show that the drainage time is affected by the force exerted on the wetting film. So, mineral particles may be

bounced back from the mobile or immobile bubble surface during the impact or collision interaction process and the particles will lose the kinetic energy in the turbulent flotation environment. Therefore, the induction time obtained from the induction time setup with an extent of air bubble deformation cannot be used to effectively reflect the collision and adhesion processes.

Based on the high-speed camera technology, a low-rank coal particle sliding on a mobile surface was shown in Fig 2. It can be found that a complete sliding process of a coal particle on a mobile surface cannot be observed. Therefore, the sliding time of mineral particles on a mobile bubble surface is difficult to be calculated while the coal particle moved from the bottom to top of the mobile bubble surface. Especially since the surfactant concentration is low and the frother was absent in the flotation. Therefore, in order to measure the sliding time of mineral particles at a low or high surfactant concentration, a fixed bubble at the top of a capillary was adopted in this work.
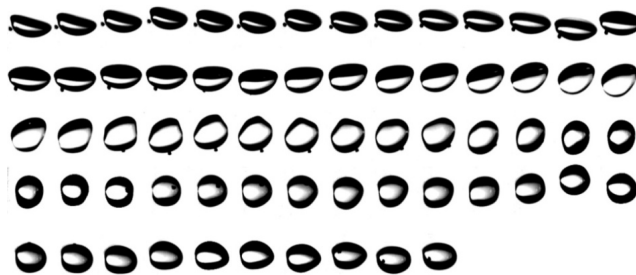

Fig. 2. Sequence of photos illustrating a low-rank coal sliding on a mobile surface

Rys. 2. Kolejność zdjęć ilustrujących przemieszczanie się węgla niskiej jakości na ruchomej powierzchni

1. Experimental method and materials

1.1. Materials

The low-rank coal samples were picked from a coal preparation plant in the Shanxi province of China. In order to eliminate the heterogeneity effect during the sliding measurements, low-rank coal bulk was firstly separated by density. The density fraction of <1.3 g/cm^3 were used as experimental samples, which were crushed by a single toggle jaw crusher. After the crushing process, the low-rank coal samples were screened and the size fraction of −0.500 mm size fraction was obtained. Some of −0.500 mm size fractions were used for the Proximate analysis. The remaining samples of −0.500 mm size fractions were

followed by a grinding process in a laboratory dry rod mill. In order to clearly observe the sliding process of low-rank coal particles on the bubble surface, coarse particles should be obtained. Moreover, the hydrodynamic forces have a great effect on the movement trajectory of the fine particles. Therefore, following the grinding step, the particle materials were wet screened through a 0.250 mm sieve to obtain coal samples 0.500–0.250 mm in size fraction for the flotation and sliding process measurements.

Proximate analysis results of low-rank coal samples selected from the –0.500 mm particle size fraction are shown in Table 1, where M^{ad} is the moisture content, V^{ad} is the volatile matter content, FC^{ad} is the fixed carbon content, and A^{ad} is the ash content. Analytical grade 2-ethyl hexanol, dodecyl amine hydrochloride (DAH), and sodium dodecyl sulfate (SDS) were purchased from Sigma-Aldrich, Australia.

Table 1.   Proximate analysis of low-rank coal sample (air dried, wt.%)

Tabela 1.   Bezpośrednia analiza próbki węgla niskiej jakości z Da Liuta (stan powietrzno-suchy, % wag.)

M^{ad} [%]	V^{ad} [%]	FC^{ad} [%]	A^{ad} [%]
6.07	38.79	53.23	1.91

1.2. XPS measurement

To observe the heterogeneity effect on the low-rank coal surface, 0.500–0.250 mm size fraction for X-Ray Photoelectron Spectrometer (XPS) analyses was carried out at 25°C in an extreme vacuum environment with a solid surface analysis set (ESCALAB 250Xi, America). The peak fitting of data processing was analyzed by the XPS peak fit software (Desimoni et al. 1992; Pietrzak 2009; Xia et al. 2014; Xia and Xie 2014). The surface element binding energies of solid particles were corrected by the C1s hydrocarbon (−CH$_2$−CH$_2$− bond) binding energy at 284.8 eV.

1.3. Flotation tests

Flotation tests of low-rank coal were investigated in the 2-ethyl hexanol, DAH, and SDS solutions. The concentrations of the three surfactants were set to 10^{-3}, 10^{-4}, 10^{-5} and 10^{-6} mol/L. All the flotation experiments were carried out in a 300 mL flotation column. Before the flotation tests, low-rank coal particles were agitated in a 100 mL beaker filled with 80 mL of the corresponding surfactant solution for 5 min. The amount of low-rank coal particles was 3.0 g while the air flow rate was fixed at 500 mL/min in each flotation process.

1.4. Slip angle velocity analyses

The sliding interaction between a particle and a fixed bubble was shown in Fig. 3. The sliding angle (θ) was calculated as the sliding path needed for the particle sliding from the touching point of the bubble surface to the bottom of the bubble. Meanwhile, the time needed for the sliding process of low-rank coal particles was calculated. Thus, the slip angular velocity of low-rank coal moving from the touching point of the bubble surface to the bottom of a fixed bubble was defined as in Equation (1). Moreover, the slip angle velocity was used to evaluate the effect of the surfactant agents on the flotation performance of low-rank coal particles in the absence of collector and frother.

$$\omega = \frac{\theta}{\tau} \tag{1}$$

ω – slip angle velocity [°/ms],
θ – sliding angle [°],
τ – time (ms) needed for the sliding process of low-rank coal particles.

The sliding processes of low-rank coal particles were investigated in the 2-ethyl hexanol, DAH, and SDS solutions with concentrations of 10^{-3}, 10^{-4}, 10^{-5} and 10^{-6} mol/L, respectively. Before the sliding measurements, low-rank coal particles were stirred in a 100 mL beaker filled with 80 mL of the corresponding surfactant solution for 5 minutes.
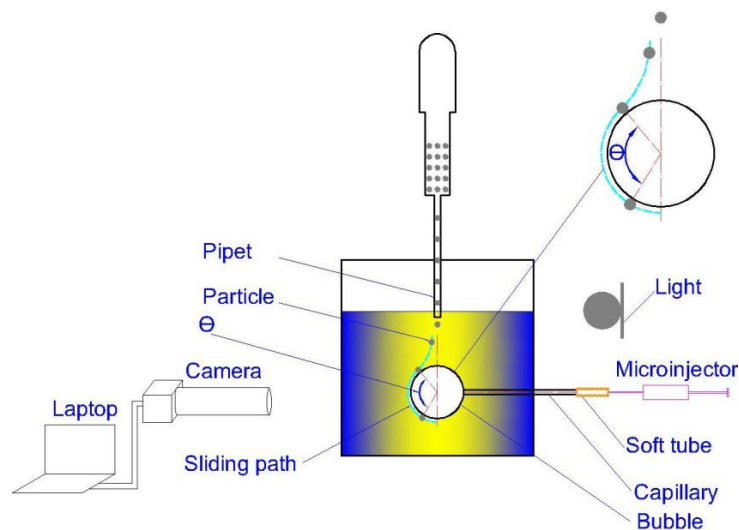

Fig. 3. A schematic of sliding interaction between a particle and a bubble in flotation

Rys. 3. Schemat przemieszczania się interakcji pomiędzy cząstką węgla
a pęcherzykiem powietrza w procesie flotacji

In order to analyze the slip angle velocity, the sliding processes of low-rank coal particles were obtained from a high-speed camera. The frame number of high-speed camera was set to 750 per second. So, the interval time of consecutive photos is 1.33 milliseconds. The inner diameter of the capillary is 1.10 mm. The images of low-rank coal particles sliding on a fixed bubble surface in the surfactant solutions, were manually analyzed by image analysis software (Image-Pro Plus 6.0, Media Cybernetics).

2. Results and discussion

2.1. XPS analysis

Carbon, oxygen, aluminum, and silicium concentrations were determined by XPS wide energy spectra shown in Table 2. The carbon and oxygen contents of 0.500–0.250 mm size fraction were measured to be 82.56% and 15.86%, respectively. The total silicium and aluminum content of 0.500–0.250 mm size fraction was 1.58%. It was also found that the ash content of 0.500–0.250 mm size fraction was 1.91% in Table 1. Therefore, it demonstrated that there were few hydrophilic mineral particles on the coal sample surface. Therefore, the heterogeneity effect of hydrophilic mineral particles during sliding process measurements can be ignored.

As shown previously, C1s peak presents at ca. 285 eV and O1s at 533eV (Desimoni et al. 1992; Fiedler and Bendler 1992). In Fig. 4, the XPS wide energy spectra of coal sample

Table 2.   Contents of atomic carbon, atomic oxygen, atomic silicium
and atomic aluminum on low-rank coal surface

Tabela 2.   Zawartość węgla atomowego, tlenu atomowego, krzemionki atomowej
i aluminium atomowego na powierzchni węgla niskiej jakości

Size fraction [mm]	C1s [%]	O1s [%]	Si2p [%]	Al2p [%]
0.500–0.250	82.56	15.86	0.50	1.08

Table 3.   Fraction of C on the surface of low-rank coal (relative % of C1s)

Tabela 3.   Fazy pierwiastka C na powierzchni węgla niskiej jakości z Shendong (% względny C1s)

Size fraction [mm]	C–C, C–H [%]	C–O [%]	C=O [%]	COOH [%]
0.500–0.250	67.35	22.82	9.32	0.51

surface was consistent with literature values (Xia et al. 2014). The C1s for 0.500−0.250 mm size fraction was depicted in Fig. 5. The C1s peaks indicate the presence of C−C or C−H, C−O (alcohol, ether, or phenol), C=O (chinone or carbonyl) or O−C−O, and COOH (carboxyl) groups on the low-rank coal surface (Pietrzak 2009; Xia et al. 2014). As illustrated in Fig. 5, the results indicated that low-rank coal particles contained a certain fraction of hydrophilic functional groups, which may have an effect on the hydrophobicity of low-rank coal particles.
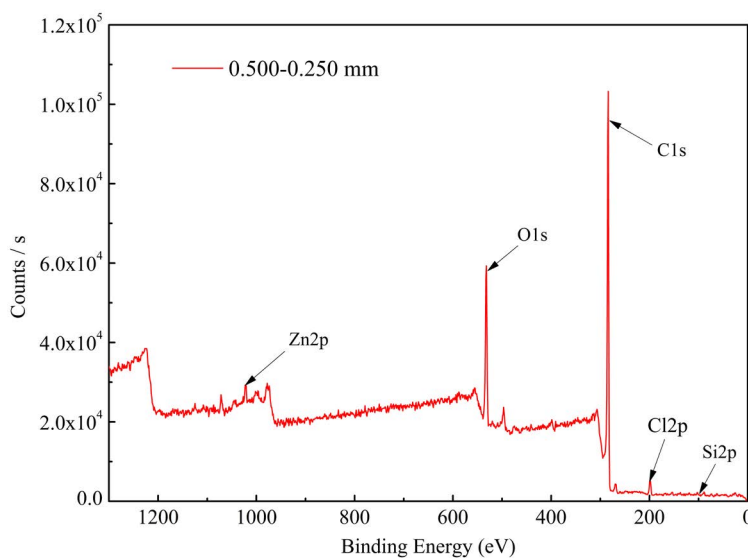

Fig. 4. XPS wide energy spectrums of coal particle surface from 0.500−0.250 mm size fractions

Rys. 4. Szerokie spektrum energii XPS powierzchni cząstek węgla klasy ziarnowej 0,500−0,250 mm
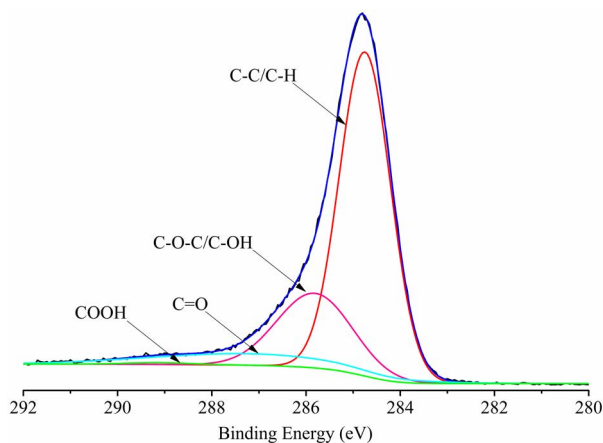

Fig. 5. C1s peaks for low-rank coal of 0.500−0.250 mm size fraction

Rys. 5. Szczyty C1s dla węgla niskiej jakości dla klasy ziarnowej 0,500−0,250 mm

To characterize the effect of different functional groups on the hydrophobicity of low-rank coal, the contents of hydrophilic and hydrophobic functional groups on the particle surface should be demonstrated. The corresponding contents of C−C or C−H, C−O, C=O, and COOH were calculated from the C1s peak intensities in Table 3. The content of C−C and C−H on low-rank coal sample surfaces from 0.500−0.250 mm size fraction was determined to be 67.35% while the content of oxygen-containing functional groups was 32.65%. Furthermore, the contents of C−O, C=O, and COOH groups were found to be 22.82%, 9.32%, and 0.51%, respectively. Therefore, C−O and C=O groups were the majority of hydrophilic functional groups, which might play a significant effect on the slip angle velocity of low-rank coal particles sliding on a fixed bubble surface.

2.2. Flotation results

The flotation results obtained from the 2-ethyl hexanol, DAH, and SDS solutions were shown in Fig. 6. It was found that the flotation recoveries of low-rank coal samples in the three surfactant solutions increased with the concentration increase. Moreover, the highest recovery was 62.84% obtained in 2-ethyl hexanol solution at 10^{-3} mol/L and the lowest recovery was about 3.48% in SDS solution at 10^{-6} mol/L. Furthermore, flotation recoveries in 2-ethyl hexanol and DAH solutions steeply increased from 16.01% and 4.53% to 42.64% and 62.84%, respectively, while flotation recoveries in SDS solutions gradually increased from 3.48% to 18.66%. Meanwhile, it was obviously observed that the difference in the flotation recoveries obtained from the three surfactant solutions increased with the surfactant
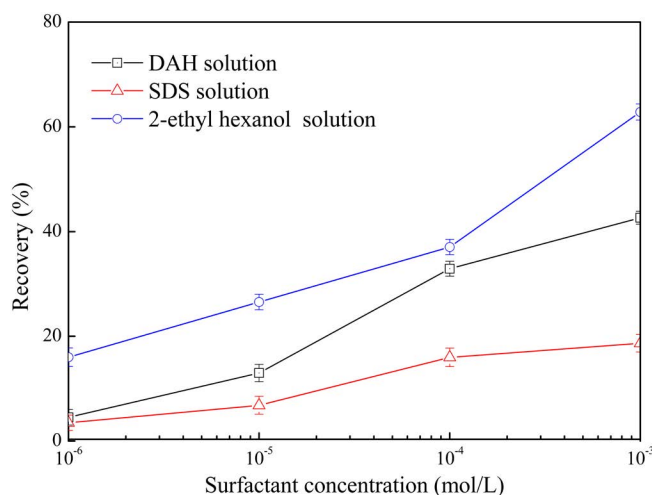

Fig. 6. Recovery of low-rank coal samples as a function of 2-ethyl hexanol, DAH and SDS concentrations

Rys. 6. Odzyskiwanie próbek węgla niskiej jakości w funkcji stężenia 2-etyloheksanolu, DAH i SDS

concentration increase. However, the flotation recovery difference between 2-ethyl hexanol and DAH reached the minimum at 10^{-4} mol/L. From above analyses, it indicated that the hydrophobicity of low-rank coal particles could be greatly improved by nonionic 2-ethyl hexanol and cation DAH surfactant agents.

2.3. Slip angle velocity results

In Fig. 7, the slip angle velocities obtained from 2-ethyl hexanol, DAH and SDS concentrations were depicted. It should state that the slip angle velocity measured in deionized water was set to 10^{-7} mol/L in Fig. 7. It was observed that the angle velocity greatly increased from 0.59°/ms and 0.60°/ms to 0.85°/ms and 0.75°/ms, respectively, with the concentration increase in 2-ethyl hexanol and DAH solutions. On the one hand, it may be because that the non-ionic 2-ethyl hexanol with oxygenated functional groups, as the collector, markedly increase the hydrophobicity of low-rank coal particles by hydrogen bonding with surface oxygenated sites and through hydrophobic bonding of the hydrocarbon chain of the collector with hydrophobic carbonaceous sites on the low-rank coal surface. On the other hand, the adsorption of surfactants on both the low-rank coal particle and air bubble surfaces would reduce the surface free energy, which may facilitate low-rank coal particles sliding on the bubble surface. Furthermore, the stability of wetting films can be reduced by the adsorption of non-ionic, anionic and cationic surface-active substances on the surface of solid and the concentration of surfactant solutions (Kosior et al. 2014, 2015; Krasowska et al. 2007).
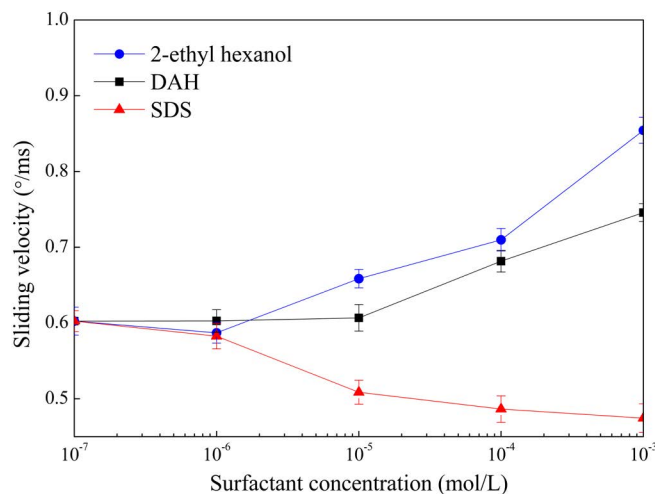

Fig. 7. Slip angle velocity of low-rank coal samples as a function of 2-ethyl hexanol,
DAH and SDS concentrations

Rys. 7. Prędkość kąta poślizgu próbek węgla niskiej jakości w zależności od stężenia 2-etyloheksanolu,
DAH i SDS

Therefore, slip angle velocities in 2-ethyl hexanol and DAH solutions increased with their flotation recovery increase.

However, the angle velocity dramatically decreased from 0.58º/ms to 0.47º/ms with the concentration increase in SDS solutions although the flotation recoveries in SDS solutions gradually increased from 3.48% to 18.66%. This may be due presumably to the polar groups in SDS orientating outward, which would increase the stability of wetting films on the surface of low-rank coal (Aktas and Woodburn 1994; Aplan 1993; Brennan 2011; Ceylan and Küçük 2004). Moreover, Qu (Qu et al. 2014) indicated that flotation performances were depressed at a high dosage of the SDS agent. Furthermore, this may be due to the stability of flotation froth in high concentration of SDS solutions increase, which would cause the foam entrainment increase. Therefore, flotation recoveries in SDS solutions gradually increased. It was also found that the slip angle velocities of low-rank coal particles in 2-ethyl hexanol, DAH and SDS solutions were very close at 10^{-6} mol/L although the flotation recoveries were obviously different. So, the slip angle velocity cannot be used to evaluate the effect of surfactants on the flotation performance of low-rank coal while the surfactant concentration was no more than 10^{-6} mol/L.

From the above analyses, it was demonstrated that the hydrophobicity of low-rank coal particles could be improved by nonionic 2-ethyl hexanol and cation DAH surfactants and depressed by anionic SDS. This is because a complete sliding process of a coal particle on a mobile surface cannot be observed. So, the sliding time of mineral particles on a mobile bubble surface is difficult to calculate. Especially, the surfactant concentration is low and the frother was absent in flotation. However, the slip angle velocity can be used to evaluate the effect of surfactants on the flotation performance of low-rank coal while the surfactant concentration was more than 10^{-6} mol/L in this work.

Conclusion

In this paper, flotation performances were investigated in the 2-ethyl hexanol, DAH, and SDS solutions, respectively. The flotation results indicated that the hydrophobicity of low-rank coal particles could be improved by nonionic 2-ethyl hexanol and cation DAH surfactants. Moreover, from the analysis of slip angle velocity, it demonstrated that the flotation responses of low-rank coal were depressed by anionic SDS. Furthermore, it was found that the slip angle velocity can be used to evaluate the effect of surfactants on the flotation performance of low-rank coal while the surfactant concentration was more than 10^{-6} mol/L.

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**WPŁYW SURFAKTANTÓW NA SPRAWNOŚĆ FLOTACJI WĘGLA NISKIEJ JAKOŚCI
W POMIARACH PROCESU PRZEMIESZCZANIA SIĘ CZĄSTEK**

Słowa kluczowe

węgiel o niskiej wartości, prędkość kątowa poślizgu, hydrofobowość,
środek powierzchniowo czynny, XPS

Streszczenie

W niniejszym artykule zbadano właściwości flotacyjne węgla niskiej jakości w roztworach 2-etyloheksanolu, DAH (chlorowodorku dodecylu) i SDS (dodecylosiarczanu sodu). W celu zmniejszenia niejednorodności i skutków hydrodynamicznych hydrofobowości i trajektorii ruchu cząstek węgla niskiej jakości przyjęto do doświadczeń próbki węgla o niskiej zawartości popiołu z klasy

ziarnowej 0,500−0,250 mm. Wynik XPS wykazał, że całkowita zawartość krzemu i glinu w klasie ziarnowej 0,500−0,250 mm wynosiła 1,58%. Stwierdzono również, że zawartość popiołu w klasie ziarnowej 0,500−0,250 mm wynosiła 1,91%. W związku z tym wykazano, że na powierzchni próbki węgla znajduje się niewiele hydrofilowych cząstek mineralnych. W ten sposób można pominąć efekt heterogeniczności hydrofilowych cząstek mineralnych podczas pomiarów kąta poślizgu. Wynik XPS wskazał również, że po procesie mielenia zawartość minerałów na powierzchni węgla niskiej jakości była bardzo mała, dlatego też występuje słabe oddziaływanie na hydrofobowość tych próbek. Wyniki flotacji wskazują, że hydrofobowość cząstek węgla niskiej jakości można poprawić za pomocą niejonowego 2-etyloheksanolu i kationowych środków powierzchniowo czynnych DAH. Analiza prędkości kąta poślizgu wykazuje, że flotacja węgla niskiej jakości została obniżona przez SDS. Ponadto zaobserwowano, że prędkość kąta poślizgu może być wykorzystana do oceny wpływu środków powierzchniowo czynnych na sprawność flotacji węgla niskiej jakości, gdy ich stężenie jest większe niż 10^{-6} mol/L.

EFFECT OF SURFACTANTS ON THE FLOTATION PERFORMANCE OF LOW-RANK COAL BY PARTICLE SLIDING PROCESS MEASUREMENTS

Keywords

Abstract

In this paper, flotation performances of low-rank coal were investigated in the 2-ethyl hexanol, DAH (dodecyl amine hydrochloride), and SDS (sodium dodecyl sulfate) solutions, respectively. In order to reduce the heterogeneity and hydrodynamic effects on the hydrophobicity and movement trajectory of low-rank coal particles, respectively, experimental coal samples with low ash content and 0.500−0.250 mm in size fraction were adopted. The XPS result demonstrated that the total silicium and aluminum content of 0.500−0.250 mm size fraction was 1.58%. It was also found that the ash content of the 0.500−0.250 mm size fraction was 1.91%. Therefore, it demonstrated that there were few hydrophilic mineral particles on the coal sample surface. Thus, the heterogeneity effect of hydrophilic mineral particles during sliding process measurements can be ignored. The XPS result also indicated that after the grinding process, the mineral content on the low-rank coal surface was very small, which would play a small role in the hydrophobicity of low-rank coal samples. The flotation results indicated that the hydrophobicity of the low-rank coal particles could be improved by nonionic 2-ethyl hexanol and cation DAH surfactants. Moreover, from the analysis of slip angle velocity, it demonstrated that the flotation responses of low-rank coal were depressed by anionic SDS. Furthermore, it was observed that the slip angle velocity can be used to evaluate the effect of surfactant agents on the flotation performance of low-rank coal while the surfactant concentration was more than 10^{-6} mol/L.