



Effect of compound collector and blending frother on froth stability and flotation performance of oxidized coal



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ABSTRACT

The beneficiation of oxidized coal is difficult to achieve by conventional flotation with oily collectors. Oxidized coal samples were prepared by the peroxide solution oxidation and a new type of oxygenated polar compound collector (CC) and a powerful blending frother (BF) were used to intensify oxidized coal flotation in the present study. The effects of the CC and BF on the froth stability and flotation performance (i.e., recovery and selectivity) were investigated. The results show that only 44.51% recovery with 9.71 s dynamic froth stability index (DFS) was obtained for oxidized coal flotation by using conventional diesel and octanol as the collector and frother, respectively. Lots of oxygen functional groups (—OH and C—O) were introduced after peroxide oxidation indicated by Fourier Transform Infrared Spectroscopy (FTIR) and X-ray photoelectron spectroscopy (XPS) analysis, leading to poor hydrophobicity and low flotation recovery. An optimal separation result with 76.24% oxidized coal recovery and 19.43 s DFS was obtained using the CC combined with octanol, which was attributed to the hydrogen bonding between the hydrophilic sites on coal surface and polar oxygen functional groups in CC. An identical DFS (19.43 s) was obtained using conventional diesel combined with the BF, while with only 21.38% recovery. Diesel did not sufficiently render the coal hydrophobicity and as a result only a small amount of coal particles was collected by attaching to bubble surface in pulp phase. The Fuerstenau upgrading curves show that both the maximum recovery and the best flotation selectivity could be obtained using the CC combined with octanol.

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1. Introduction

Coal is the main fossil fuel used in many countries and may be the largest contributor to industrial growth, accounting for almost 70% of primary energy consumption, particularly in China. In recent years, with the depletion of high-quality coal resources and the complication of geological conditions, low-grade coal, such as oxidized coal refuse, has been reconsidered for use and is becoming an important resource. The demineralization upgrading of low-grade coal to obtain available clean fuels has attracted increased attention in the field [1]. A strategy to recover and utilize both the organic matter and mineral matter present in oxidized coal refuse could yield an important energy source offering environmental and economic benefits [2,3].

Froth flotation is an effective separation method for fine coal cleaning and upgrading that is based on the differences in the surface hydrophobicity of organic matter and mineral matter [4–6]. Common oily collectors (diesel and kerosene) are added to the flotation pulp to make coal particles more hydrophobic and separable from the pulp by their attachment to rising air bubbles, which form a particle-rich froth on the suspension surface. However, the beneficiation of oxidized coal

is difficult to achieve by conventional flotation with oily collectors because of the presence of a large quantity of surface oxygen functional groups and poor froth stability [7,8]. The surface physicochemical characteristics, surface functional group composition, and surface roughness are significantly changed by the oxidation process [9,10]. The results of X-ray photoelectron spectroscopy (XPS) analyses have shown that the content of hydrophobic C—C and C—H groups decreases, whereas the content of hydrophilic C—O and C=O groups increases. The surface roughness and cracking also increase after oxidation, according to scanning electron microscopy (SEM) results [9]. A firm hydration film is formed at the hydrophilic sites of oxidized coal surfaces because of hydrogen bonding [11,12]. It is therefore important to develop an enhanced flotation separation process that can optimize the flotation efficiency of oxidized coal, similar to the chemical process of multiphase reactions [13–15].

Numerous methods have been employed to intensify oxidized or difficult-to-float coal flotation, such as the use of new types of collectors; particle surface pre-treatment using grinding, ultrasonic, microwave, and thermal methods; bubble surface modification; and high-efficiency flotation process design. The addition of surfactants or oxygenated functional groups to oily collectors can significantly enhance the oxidized coal flotation yield because of hydrogen bonding between the polar part of the coal surface and the new reagent [16]. A series of

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non-ionic surfactants containing oxygenated and aromatic functional groups, i.e., tetrahydrofurfuryl esters (THF), were used as collectors to enhance low-rank/oxidized-coal flotation by Jia et al. [17]. The members of the THF series were found to be more effective collectors than oily collectors for both oxidized and un-oxidized coals. Xia et al. [18] found that the flotation recovery of oxidized coal was improved by grinding pretreatment, which removed the oxidation layer and exposed some fresh surfaces and hydrophobic sites. Atesok and Celik [19] used bituminous coal pitch in dry grinding, followed by flotation of a difficult-to-float coal, and achieved a remarkable increase in the recovery rate, from 22.1% to 89.9%, and a decrease in the ash content, from 11.8% to 10.1%, because of the higher viscosity of the pitch. The floatability of oxidized coal can also be restored by ultrasonication preconditioning. Feng and Aldrich [20] found that ultrasonication pretreatment had a significant effect on the flotation recovery rate and kinetics of oxidized coal, especially for smaller-size fractions. XPS results indicated that a thin clay layer and oxidized layer were removed using ultrasonication [21]. Buttermore and Slomka [22] reported that the sonication of oxidized coal restored its floatability to a level close to or exceeding that of fresh coal. Microwave and thermal treatments are also common methods for improving the floatability of oxidized coal by removing the pore water and some organic OH functional groups [8]. The characteristics of bubbles, which act as the carriers of particles in flotation, have an important effect on the separation performance. The flotation of hard-to-float coal can be enhanced by suitable bubble surface modification technology. Liu et al. [23] proposed a novel concept for using reactive oily bubbles as the carriers in flotation. Oily bubbles have much higher contact angles than air bubbles, generating a strong collecting power that is conducive to the flotation of both low-rank and oxidized coal [24]. Recently, researchers have found that direct-contact and zero-conditioning flotation process design can make collectors adsorb onto oxidized coal surfaces directly without the prevention of hydration film and can effectively improve the floatability of difficult-to-float oxidized coal [25–27]. Xia and Yang [28] found that reverse flotation was particularly well suitable for the upgrading of Taixi oxidized coal. It is well accepted that oxygenated polar collectors are the most effective method to enhance oxidized coal flotation among above mentioned methods. However, the effect of oxygenated polar collector on oxidized coal flotation performance is complex and required to be further studied.

To date, little attention has been given to frothers and their potential effect on oxidized coal flotation. Frothers are used for froth generation and to facilitate air dispersion into fine bubbles, which contributes to a high flotation recovery rate and good kinetics [29]. Frothers are commonly characterised as either selective or powerful and chosen on the basis of laboratory and/or pilot plant tests [30]. Low-molecular-weight frothers are more selective but less effective than high-molecular-weight frothers and thus result in the loss of combustible matter in tailings [31]. Tan [32] found that better foaming properties could be obtained using a mixture of selective and powerful polypropylene glycols compared to the use of single frother. Similar findings were reported by Dey et al. [31]. A powerful frother is more effective than selective frother in recovering combustibles in coking coal flotation and it may be well suited to difficult-to-float oxidized coal. Therefore, it is essential to explore the effect of powerful frother on oxidized coal flotation.

Froth structure and stability plays an important role in determining flotation recovery and grade. It is well accepted that poor froth stability is incapable of getting a satisfactory flotation recovery and grade. However, it doesn't mean that a high flotation recovery can be obtained definitely with good froth stability. Froth flotation is a complex system and the final recovery is the result of the interaction of many factors such as properties particles, reagents, bubbles, froth stability, and solution. Based on previously summarised the literature review, a new type of oxygenated polar compound collector (CC), a mixture of diesel and aliphatic acid, and powerful blending frother (BF), a mixture of octanol and aliphatic acid salt, were used to enhance oxidized coal flotation. The effects of the CC and BF on froth stability and flotation performance

were examined in this study. Oxidized coal samples were prepared using the peroxide solution oxidation method and the surface functional groups were characterised using Fourier transform infrared spectroscopy (FTIR) and XPS before and after oxidation.

2. Experimental

2.1. Materials

Fine high-metamorphosed anthracite coal samples were collected from the coal storage yard of the Xuehu Coal Preparation Plant in Yongcheng City, Henan Province, China. Samples that were smaller than 0.25 mm in size were prepared by screening. The particle size distributions are presented in Table 1. Dry coal particles with a size smaller than 0.25 mm were immersed in 30% peroxide solutions in a magnetic stirrer for 24 h at room temperature to obtain the final oxidized coal samples [33]. It should be noted that large amounts of heat and gases were released in the initial stage of oxidation, which indicated that the H₂O₂ solution method was quick and effective in preparing the oxidized coal. Flotation reagents, diesel, octanol, and the CC and BF were purchased from the Zhuzhou Flotation Reagents & Chemicals Co., Ltd.

2.2. Method

2.2.1. FTIR

FTIR (Nicolet is5, Thermo Scientific) was used to identify the functional groups of the coal and flotation reagents in the wave number range of 4000–400 cm⁻¹. A mixture of 2 mg of coal and 300 mg of KBr was prepared and ground to a fineness of 2 μm in an agate mortar. A pressure of 28 MPa was applied to the powder to form thin circular plates, and the influences of the background and vapour were accounted for in the data analysis process. For measurement of the functional groups of the liquid flotation reagents, the painting method, in which a micro-drop of reagent was placed on a KBr pellet, was used to create a homogeneous distribution of reagent film. All of the experiments were conducted at room temperature 20 °C.

2.2.2. XPS

Flotation is a type of interface separation technique. Interfacial properties play a crucial role in bubble–particle interaction sub-processes. Recently, XPS has emerged as an advanced interface chemistry analysis tool and has been used extensively in flotation and energy engineering applications. In the present study, an XPS system (ESCALAB 250Xi, Thermo Scientific, Waltham, MA, America) with Al Kα radiation (hν = 1486.6 eV) and a 900 μm light spot size was used to characterise the surface chemistry changes before and after oxidation. Each XPS spectra data set was fitted using the XPS Peakfit software. It should be noted that binding energy calibration was performed by setting the C1s hydrocarbon peak to 284.6 eV.

2.2.3. Flotation column kinetics experiments

Column flotation tests were carried out at an 80 g/L solid concentration using a traditional laboratory countercurrent flotation column—that

Table 1
Particle size distributions of the coal sample.

Size, mm	Yield, %	Ash, %	Positive cumulative		Negative cumulative	
			Yield, %	Ash, %	Yield, %	Ash, %
0.25–0.125	35.95	18.15	35.95	18.15	100.00	21.18
0.125–0.074	17.44	19.45	53.40	18.57	64.05	22.88
0.074–0.045	11.21	21.66	64.61	19.11	46.60	24.16
–0.045	35.39	24.95	100.00	21.18	35.39	24.95
Total	100	21.18	–	–	–	–

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