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Separation of <6 mm oil shale using a compound dry separator

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ABSTRACT

A large amount of fine granular materials (<6 mm) are produced during the mining of oil shale. The combustion characteristics of oil shale improve with decreasing size of these materials, for which reason fine-grain oil shale has a high utility value. However, fine oil shale also contains a significant amount of inorganic mineral impurities which can be reduced by physical separation to improve the oil quality. Based on an analysis of the physical properties of oil shale, this paper proposes a compound dry separation process for the cleaning of <6 mm oil shale grains. The effects of the vibration intensity, air velocity, and back angle of the employed separator on the separation results and oil content of the cleaned oil shale were systematically analyzed. Under the optimal vibrational conditions defined by a vibration intensity of 25.76 (amplitude = 4.0 mm, frequency = 40 Hz), air velocity of 0.66 m/s, and back angle of 45°, the yield comprised 35.8% concentrate and 64.2% tailings, with corresponding oil contents of 10.02% and 0.85%, respectively. The probable error of the highest intensity of segregation achieved was 0.155. The proposed compound dry separation of oil shale particles of up to 6 mm was found to be more efficient compared with conventional methods, and the separated fine grade material can be comprehensively utilized by further pyrolysis treatment.

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Compound dry separation;
fine grain; oil content; oil
shale

Introduction

Oil shale is an abundantly available solid source of fossil energy. Based on calorific conversion, oil shale reserves account for the world's second largest source of fossil fuel, second only to coal. On this basis, the global reserve of oil shale is 5.4 times that of crude oil. The oil shale resources are mainly distributed in the United States, Russia, China, Estonia, and other countries. According to EIA statistics, the world's 33 countries' shale oil is up to 410 billion tons. In China, the reserves being twice the nation's oil reserves.^[1–3] The mining of oil shale is accompanied by the production of a large amount of fine-grade oil shale particles measuring <6 mm. The increased level of oil shale mining in recent years has been accompanied by a substantial increase in the production of the fine particles, and this has posed a significant challenge to effective management of the mining process and products. The investigation of these issues are important in view of the important and strategic role that oil shale promises to play in affording a clean and effective alternative source of energy in the face of depleting global oil and gas reserves.^[4]

Oil shale is mainly used for combustion and producing shale oil by pyrolysis. Gasoline, kerosene, diesel oil, wafer, and many other oils and chemical products can also be obtained by the hydrofining of oil shale.^[5] Although the efficiency of the pyrolysis process is not directly affected by the non-uniformity of the oil shale particle size, the pyrolysis effect theoretically increases with increasing uniformity of the feed oil shale. The time and energy requirements of the pyrolysis process also increase with increasing size difference between the coarse and fine particles. Previous studies have shown that the granularity of oil shale affects its combustion characteristics to a certain extent, with the combustion performance increasing with decreasing particle size.^[6–8] Overall, smaller oil shale particles are favorable to both pyrolysis and combustion.

However, large amounts of inorganic mineral impurities are included when oil shale is mined. For oil shale mined in China, the oil content is generally lower than 10%, with ash accounting for 50%–60%.^[9,10] Decreasing this ash content increases the economic value of the oil shale and its comprehensive utilization. It also increases the efficiency of oil shale pyrolysis, and decreases the process cost. In addition, it significantly reduces the environmental pollution due to the

combustion of oil shale.^[11–13] Because of the similarity between some of the physical properties of coal and oil shale, the separation method for coal cleaning can also be applied to the beneficiation of oil shale.^[14,15] In the present study, oil shale particles of sizes <6 mm were separated using a compound dry separation apparatus. The effects of the vibration intensity, air velocity, and back plate angle in the apparatus on the separation process were systematically investigated. In addition, the optimal operation parameters were determined to provide theoretical support for compound dry separation of oil shale.

Experimental

Compound dry separation apparatus

Figure 1 shows the apparatus used for the compound dry separation of oil shale in the present study. The apparatus is composed of a crude ore preparation system, a separator system, and an air supply and dust removal system. The crude ore preparation system comprises a pre-classification screen, crusher, surge bin, and other related elements. The crude ore is pre-classified and crushed before being conveyed to the surge bin for separation. The separator system consists of a compound dry separator. (The particular compound dry separator employed in this study was a laboratory equipment with a right-angled trapezoidal shape.) The air supply and dust removal system includes an air blower, induced draft fan, dust collector, air bag, and valve, and is used for air supply and dust collection. The separation in the compound dry separator is carried out under the coordinative effect of air and vibration. Then the ores are layered by density. The

low-density ores are layered on the upper layer, and high-density ores are in the bottom layer. As a result, the ores are beneficiated by density.

Material properties

The physical properties of oil shale determine its separation efficiency. Oil shale is a particular type of geomaterial, and its density, oil content, and other physical properties are affected by the geological structures at the location where it is found. It was therefore necessary to analyze the geological structures at the origin of the <6 mm oil shale particles employed in the present study in order to establish their physical properties and facilitate the oil shale separation process. The oil content is an important parameter in evaluating the grade of oil shale, and is denoted by ω . Table 1 gives the oil contents of raw ores with different particle sizes. The oil contents are classified into three ranges, namely $\omega > 10\%$, $5\% < \omega < 10\%$, and $\omega < 5\%$. Scanning electron microscopy (SEM) and X-ray diffraction (XRD) have also been used to analyze the surface morphology and density of oil shale with oil contents of $\omega \leq 5\%$, $\omega \geq 10\%$, and $5\% \leq \omega \leq 10\%$. As shown in Fig. 2, the oil content of the oil shale considered in this study was 4.13%. Table 2 gives the results of the sink float test conducted on the oil shale.

Evaluation

The purpose of oil shale separation is to increase the grade and hence the oil content. The combined oil shale separation process performed in the present study using the compound dry separation apparatus took into consideration the differing densities of the

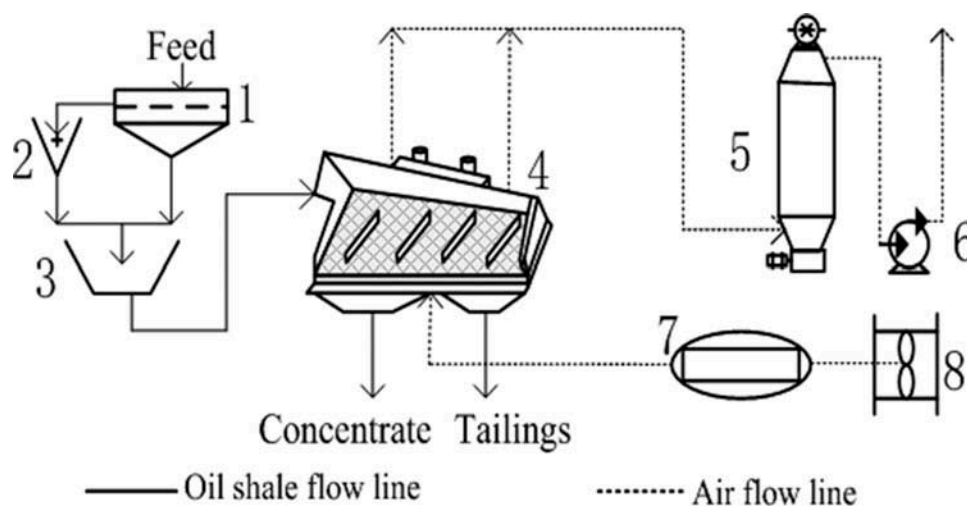


Figure 1. Apparatus for compound dry separation of oil shale. 1, Classification screen; 2, crusher; 3, surge bin; 4, compound dry separator; 5, dust collector; 6, induced draft fan; 7, air bag; 8, air blower.

Table 1. Oil contents of oil shale particles of different sizes, as determined by aluminum retort analysis.

Particle size (mm)	Yield (%)	Oil content (%)	Char content (%)	Moisture content (%)	Gas loss (%)
−6 + 0.5	26.45	6.38	88.61	1.77	4.05
−0.5 + 0.25	24.16	4.32	89.81	2.01	3.42
−0.25 + 0.125	10.14	3.81	90.12	1.98	5.03
−0.125 + 0.075	8.23	3.12	90.65	2.07	5.18
−0.075 + 0.045	7.28	2.87	91.02	2.11	5.25
−0.045	23.74	2.01	91.87	2.13	5.38
Total	100	4.06	91.18	2.09	5.26

impurities. Although the oil content of oil shale decreases with increasing oil shale density, compound separation enables enhancement of the oil content. As shown in Fig. 3, the discharge baffle of the compound dry separator is divided into five parts between the feed end and the tailing discharge end. This enables the derivation of different products, the yields and oil contents of which can be measured.

The oil segregation intensity is a measure of the deviation of the oil content of layer i from the oil content of the feed ore. Using the ash segregation intensity as a reference,^[16] the oil segregation intensity S_{oil} can be calculated as follows:

$$S_{oil} = \sqrt{\frac{1}{n-1} \sum_{i=1}^n \left(\frac{\omega_i}{\omega_o - 1} \right)^2} \quad (1)$$

where ω_i is the oil content of the i th layer (%), ω_o is the oil content of the feed oil shale (%), and n is the sampling number. Better separation results are obtained for higher values of S_{oil} .

In a compound dry separation system, concentrate with a higher oil content usually occurs near the feed end, while gangue with a lower oil content is found close to the tailing discharge end. Figure 3 shows the sampling method employed in the compound dry separator.

The probable error E is used to evaluate the separation efficiency of a compound dry separator, with a lower value indicating higher separation efficiency. The value of E is calculated using

$$E = \frac{1}{2}(\delta_{75} - \delta_{25}) \quad (2)$$

where δ_{75} is the density (g/cm^3) of the feed for a partition coefficient of 75%, and δ_{25} is the density for a partition coefficient of 25%.

The separation process in the compound dry separator is affected by both the density and oil content of the feed. The stability of the oil content of the material in different parts of the bed is also important to maintaining the separation process. The oil content distribution of the material on the bed is a direct indication of the material movement and the progression of the

separation process. The measurement of the oil content on the bed can thus be used to analyze the separation results to optimize the separation conditions. To facilitate the determination of the oil content distribution on the bed, a coordinate system was established, as shown in Fig. 4. The X-axis extends from the feed end to the tailing end, along which the bed is divided into 10 equal parts. The Y-axis extends from the backboard to the discharge end. The material was sampled at the intersections between the X- and Y-axis segmentation lines, and the oil content of each sample was measured.

Results and discussion

Physical properties of oil shale

The XRD pattern in Fig. 2 shows that the main inorganic minerals in <6 mm oil shale are montmorillonite, kaolinite, calcite, pyrite, and carbonates. The figure shows that the mineral contents of the oil shale with $\omega \geq 10\%$ are lower than those for $\omega < 5\%$. The SEM results also show that the microscopic porosity of the oil shale with $\omega \geq 10\%$ is greater, and that the distribution of the organic matter is layered. However, the microscopic porosity of the oil shale with $5\% \leq \omega \leq 10\%$ is lower, and more inorganic minerals are distributed in the layered crack. The microscopic pores in the oil shale with $\omega < 5\%$ are particularly very small, and the XRD pattern also shows higher montmorillonite and calcite contents, indicating a higher ash content, and hence a lower grade.

Effect of vibration intensity on the separation efficiency

Vibration plays an important role in the compound dry separation process. The vibration energy is usually expressed in terms of the vibration intensity Γ . The effects of the vibration intensity on the material density and the efficiency of the segregation are jointly determined by the vibration amplitude A and frequency f . However, a given vibration intensity may be achieved by different combinations of the vibration amplitude and frequency. The determination of the optimal amplitude and frequency combination is significant for improving the separation efficiency. Table 3 gives the oil segregation intensities under different vibration conditions. Figure 6 is a contour map of the intensity of segregation with respect to the vibration intensity.

As can be observed from Fig. 5, S_{oil} varies significantly with the vibration condition. However, there is an optimal vibration range, within which the amplitude and frequency significantly contribute to achieving S_{oil}

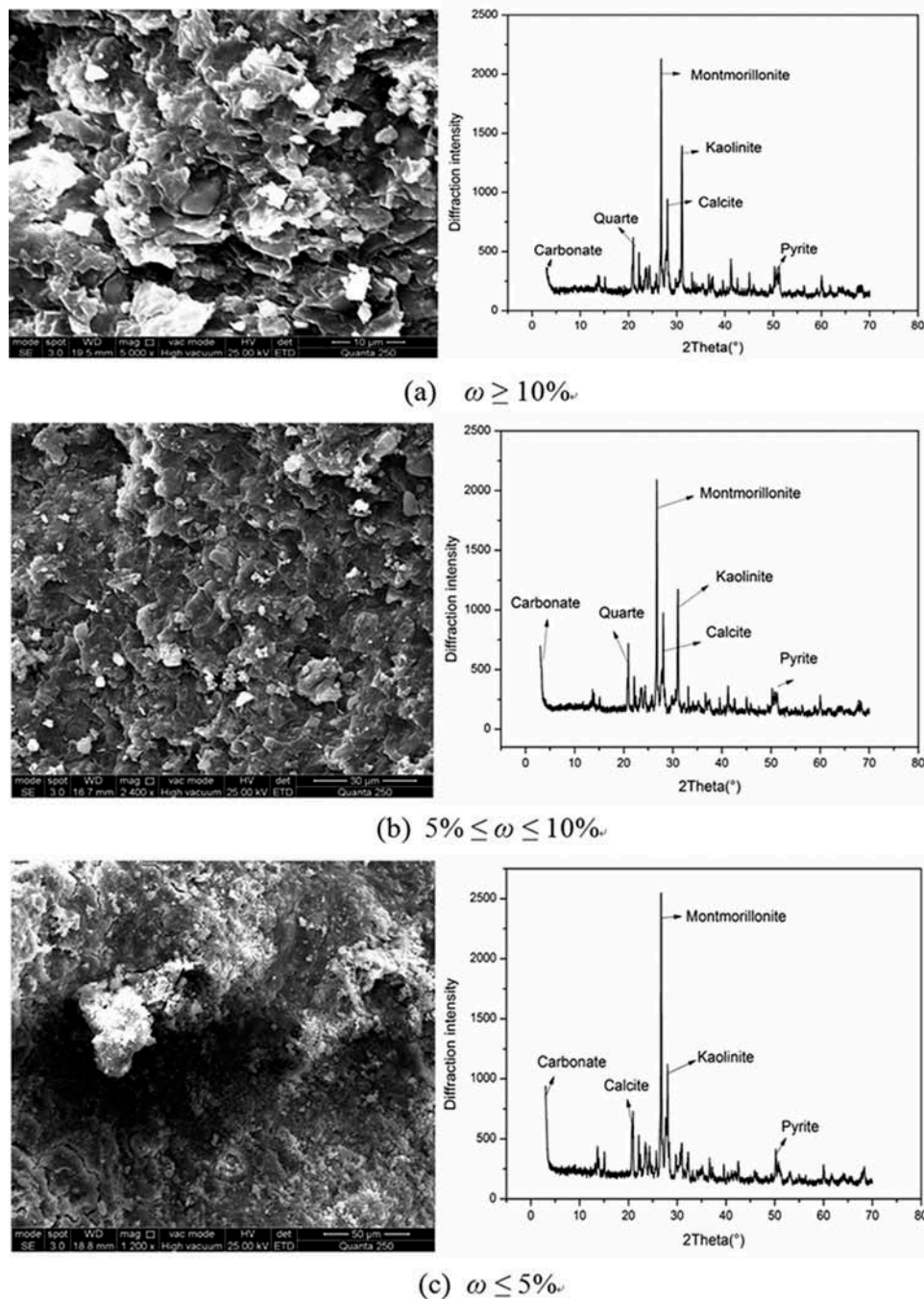


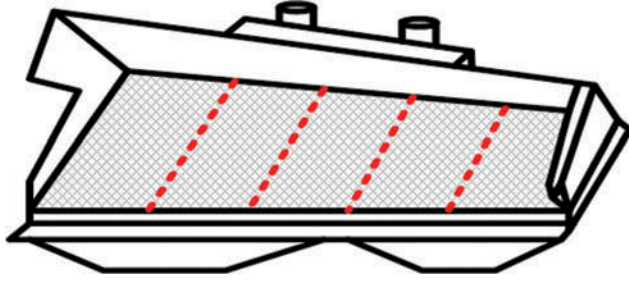
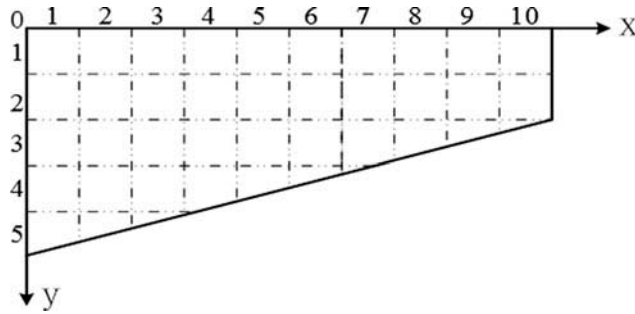
Figure 2. SEM and XRD results for fine-particle oil shale with different oil contents: (a) $\omega \geq 10\%$ (b) $5\% \leq \omega \leq 10\%$ (c) $\omega \leq 5\%$.

of higher than 1 for fine-grain oil shale. This indicates that the relationship between the determinant factors of the vibration intensity and the intensity of segregation for <6 mm oil shale is nonlinear. Better segregation is achieved for a vibration frequency range of 35–40 Hz and amplitude range of 3.8–4.2 mm. More intense vibration increases the energy of the oil shale grains, resulting in the strong chain network among the particles being destroyed. The bed material can then be sufficiently loosened and stratified by density. The

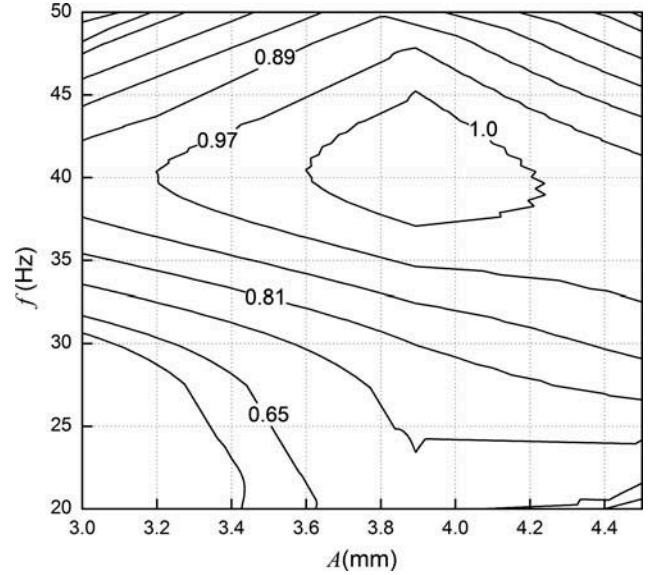
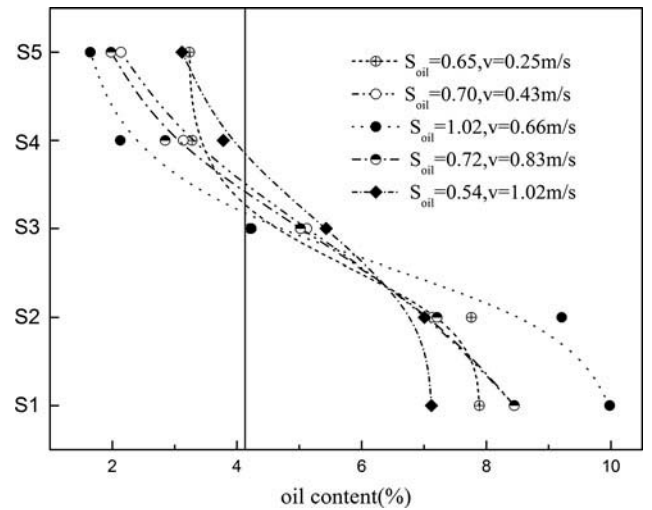
particles cannot be effectively stratified when the amplitude and frequency are too high or too low. This is primarily because, when the amplitude and frequency are inadequate, the strong chain network among the particles is not broken, and there is insufficient fluidization and stratification by density. Moreover, when the amplitude and frequency are excessive, the very high vibration intensity subjects the particles at the bottom of the bed to forced disturbance and violent movement, with the resultant energy transfer

Table 2. Results of sink–float test conducted on <6 mm oil shale particles.

Density (g/cm ³)	Yield (%)	Oil content (%)	Float accumulation		Sink accumulation		$\delta_p \pm 0.1$	
			Yield (%)	Oil content (%)	Yield (%)	Oil content (%)	Density (g/cm ³)	Yield (%)
–1.8	7.08	26.87	7.08	26.87	100.00	4.13	1.8	9.40
1.8–1.9	2.32	16.01	9.40	24.19	92.92	2.39	1.9	5.44
1.9–2.0	3.12	11.89	12.52	21.12	90.60	2.05	2.0	5.10
2.0–2.1	1.98	7.83	14.50	19.31	87.48	1.69	2.1	3.87
2.1–2.2	1.89	5.45	16.39	17.71	85.50	1.55	2.2	4.94
2.2–2.3	3.05	4.87	19.44	15.70	83.61	1.46	2.3	7.28
2.3–2.4	4.23	3.12	23.67	13.45	80.56	1.33	2.4	12.21
2.4–2.5	7.98	2.23	31.65	10.62	76.33	1.24	2.5	68.35
+2.5	68.35	1.12	100.00	4.13	68.35	1.12		
Total	100.00							

**Figure 3.** Schematic illustration of sampling in the compound dry separator.**Figure 4.** Distribution of the sampling points of the material on the compound dry separator bed for measurement of the oil content.**Table 3.** Intensity of segregation (S_{oil}) under different vibration conditions.

f (Hz)	A (mm)	Γ	S_{oil}
20	3.0	4.83	0.53
20	3.5	5.63	0.61
20	4.0	6.44	0.75
20	4.5	7.24	0.59
30	3.0	10.87	0.56
30	3.5	12.68	0.68
30	4.0	14.49	0.81
30	4.5	16.30	0.93
40	3.0	19.32	0.98
40	3.5	22.54	1.06
40	4.0	25.76	1.13
40	4.5	28.98	1.02
50	3.0	30.18	0.49
50	3.5	35.21	0.78
50	4.0	40.24	0.89
50	4.5	45.27	0.57

**Figure 5.** Contour map of intensity of vibration with respect to the vibration condition.**Figure 6.** Intensity of segregation with respect to the air velocity.

substantially interfering with the force characteristics of the particles, resulting in severe back-mixing and deteriorated separation.

Effect of the air velocity on the separation efficiency

The effect of the air flow on the separation efficiency was investigated using a vibration intensity Γ of 25.76 ($A = 4.0$ mm, $f = 40$ mm), bed lateral angle of -2° , longitudinal angle of 7° , and back angle of 45° . The results are presented in Fig. 6.

As can be observed from Fig. 6, for $S_{oil} = 0.65$, the oil content of the material on the separation bed deviates from 4.13%, indicating that the oil contents of the different layers are similar, which implies poor separation. The deviation of curves from the straight line of raw ore oil contents expresses the separation efficiency of ores. The layer is better when the deviation is greater. The deviation is largest for $S_{oil} = 1.02$ and an air velocity of 0.66 m/s. With further increase of the air velocity, the curve deviation decreases, indicating deteriorating stratification. The oil content initially increases with increasing air velocity, then begins to decrease. These observations indicate that the air velocity affects the material distribution on the bed with respect to the oil content. Figure 7 shows the oil content distribution on the bed with respect to the air velocity, together with the corresponding contour maps.

As shown in Fig. 7, the oil content of the material on the bed decreases from the feed end to the tailing end for an air velocity of 0.25 m/s. However, within 100–400 and 700–800 mm along the bed, there are irregularities in the oil content distribution. This is

because a lower air velocity decreases the force exerted on the material, resulting in reduced porosity and degree of loosening of the material. There is thus reduced activity of the particles, with insufficient energy to break their powerful chain network, resulting in nonstratification by density. The optimal air velocity is 0.66 m/s, with further increase generating too much particle activity and excessive loosening, which cause intense random motion of the particles on the bed. In addition, shortening the bed length along the X-axis reduces the particle motion time, resulting in increased back-mixing and reduced separation efficiency.

Effect of back plate angle on separation efficiency

The tests that were used to investigate the effect of the back plate angle on the separation efficiency were conducted using a vibration intensity Γ of 25.76 ($A = 4.0$ mm, $f = 40$ mm), bed lateral angle of -2° , longitudinal angle of 7° , and air velocity of 0.66 m/s. The results are presented in Fig. 8. Figure 9 shows the oil content distribution with respect to the back plate angle, together with the corresponding contour maps. Figure 10 shows the back plate angle adjustment diagram.

As shown in Fig. 8, when $S_{oil} = 0.35$, the oil content of the bed material deviates from that of the ore near a value of 4.13% of the latter, indicating poor separation. The deviation of curves from the straight line of raw ore oil contents expresses the separation efficiency of ores. The layer is better when the deviation is greater. With increasing back angle and oil content, the intensity of segregation and the deviation of the bed material oil content from the ore oil content gradually increase. The degree of

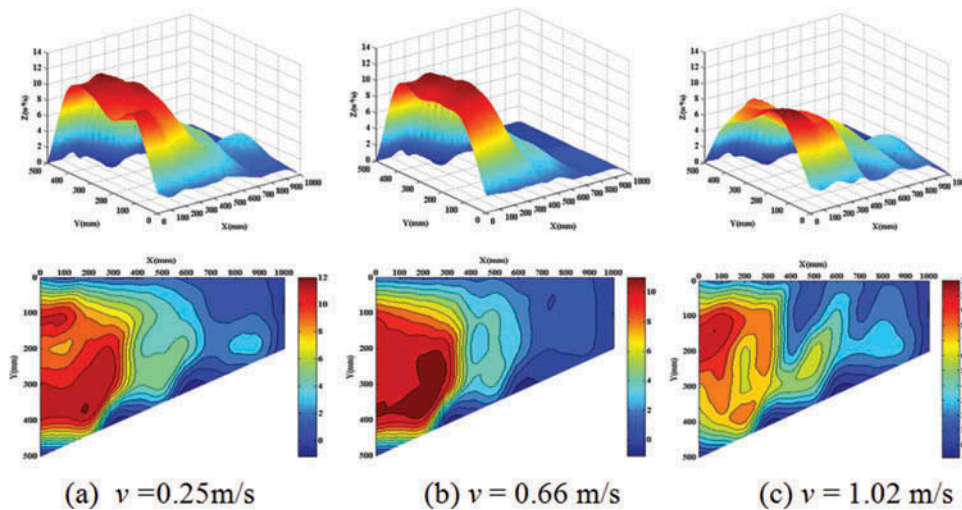


Figure 7. Oil content distribution of the material on the bed with respect to the air velocity.

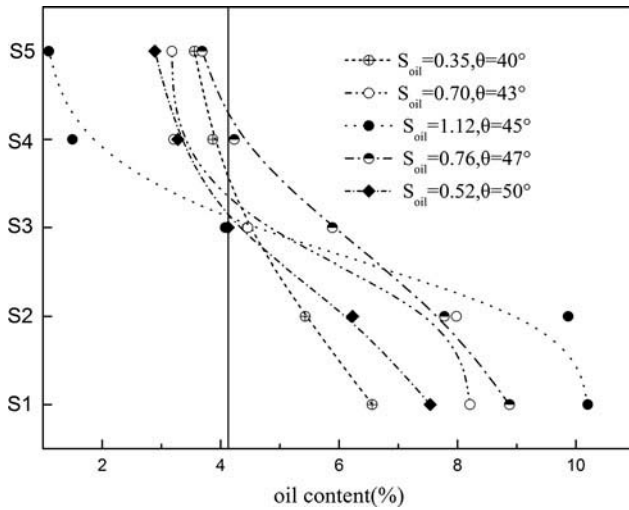


Figure 8. Intensity of segregation of the oil content with respect to the back plate angle.

deviation is highest for $S_{oil} = 1.12$ and $\theta = 45^\circ$. With further increase of the back angle, the degree of deviation begins to decrease, indicating decreased separation efficiency. The intensity of segregation of the oil content increases with increasing back angle until a maximum value is attained, and thereafter begins to decrease. Hence, in the case of extreme variations of the oil content on the bed, the back plate angle can be adjusted to improve the segregation efficiency.

As shown in Fig. 9, for a back plate angle of 40° , the oil content generally decreases along the X-axis from the feed end of the bed to the tailing end. However, there is an increase in the material content within 750–900 mm. Along the Y-axis, the oil content increases gradually from the back plate to the discharge end, with a random

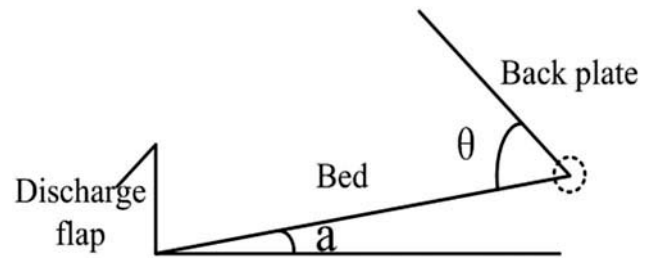


Figure 10. Back plate angle adjustment diagram.

distribution occurring within 150–250 mm. These observations can be explained by the fact that a smaller back plate angle decreases the force exerted on the material by the back plate, resulting in reduced energy and activity of the particles. There is thus lower energy transfer among the particles, resulting in poor loosening of the material and nonstratification by density. When the back plate angle is increased, the force exerted on the particles increases, resulting in increased activity. This enables the breaking of the strong chain network among the particles, leading to higher energy transfer, accelerated material movement, and enhanced sorting. The optimal back plate angle is 45° . A larger angle weakens the force exerted on the particles, resulting in decreased activity and kinetic energy. There is also an increase in the potential energy at the tailing end, causing the particles to move in that direction, where their kinetic energy is used up in overcoming the potential energy. This leads to intense back-mixing of the particles and deterioration of the separation efficiency. When the back plate angle is 50° , there is severe back-mixing and deterioration of the separation efficiency within 150–300 and 450–600 mm along the X-axis and 200–450 mm along the Y-axis.

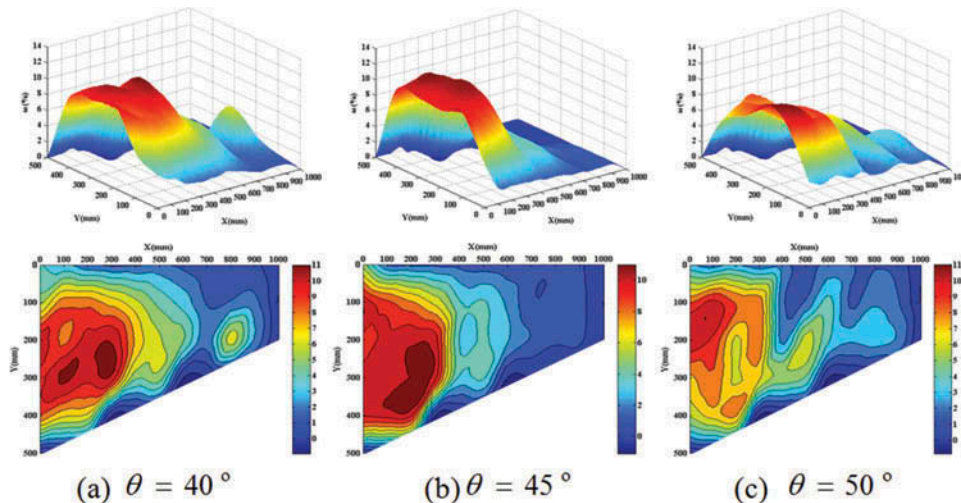


Figure 9. Oil content distribution on the bed with respect to the back plate angle.

Table 4. Partition coefficients for compound dry separation of <6 mm feed oil shale.

Density (g/cm ³)	Average density (g/cm ³)	Feedstock sink–float result (%)	Tailings sink–float results (%)		Concentrate sink–float results (%)		Calculated feed stock sink–float results (%)	Partition coefficient (%)
–1.9	1.75	9.40	1.49	1.21	44.32	8.31	9.52	12.71
1.9–2.0	1.95	3.12	0.59	0.48	13.12	2.46	2.94	16.33
2.0–2.1	2.05	1.98	0.52	0.42	8.69	1.63	2.05	20.49
2.1–2.2	2.15	1.89	0.73	0.59	7.09	1.33	1.92	30.73
2.2–2.3	2.25	3.05	1.49	1.21	7.47	1.40	2.61	46.36
2.3–2.5	2.40	12.21	13.80	11.21	8.00	1.50	12.71	88.20
+2.5	2.60	68.35	81.39	66.13	11.31	2.12	68.25	96.89
Total			100	81.25	100	18.75	100.00	

Separation performance

The <6 mm oil shale separated by the compound dry separator yielded 35.8% and 64.2% concentrate and tailings with oil contents of 10.02% and 0.85%, respectively. The partition coefficients and partition curve are presented in Table 4 and Fig. 11, respectively. The cleaned oil shale ($\omega > 10\%$) can be pyrolyzed to generate shale oil, gasoline, kerosene, diesel, and many other chemical products are obtained when the shale oil is hydro-cracked. Then the products could be used for power generation, heating, building, and diesel for comprehensive utilization of the fine-grain oil shale.

Conclusions

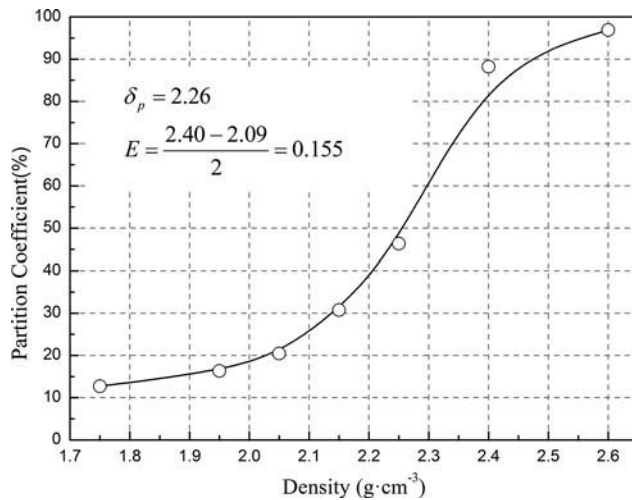
A compound dry separator was used to separate <6 mm oil shale. The effects of the vibration intensity, air flow velocity, and back angle on the separation efficiency were investigated. Following is a summary of the findings:

- (1) Investigation of the oil shale microstructure and composition by SEM and XRD revealed the presence of inorganic minerals, namely montmorillonite, kaolinite, and calcite. This decreased the microscopic porosity, with the oil content decreasing with increasing amount of inorganic minerals. Conversely, the microscopic porosity increased with increasing amount of organic matter.
- (2) The intensity of segregation S_{oil} of the oil content by compound dry separation initially increases with increasing vibration intensity, but begins to decrease when the vibration intensity exceeds an optimal level. There are similar relationships between the intensity of segregation and the air flow velocity and back angle. The intensity of segregation is maximum for vibration intensity, air flow velocity, and back angle values of 25.76, 0.66 m/s, and 45°, respectively. The oil content decreases from the feed end to the tailing end along the X-axis of the separator bed, while it increases from the feed end to the discharge end along the Y-axis.
- (3) The compound dry separator yield from the <6 mm oil shale in this study comprised 35.8% and 64.2% concentrate and tailings with oil contents of 10.02% and 0.85%, respectively.

The results of the present study indicate that <6 mm oil shale can be efficiently cleaned by a compound dry separator.

Funding

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**Figure 11.** Partition curve for compound dry separation of <6 mm oil shale.

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