

# Closed Process of Shale Oil Recovery from Circulating Washing Water by Hydrocyclones

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## Abstract

The conventional oil recovery system in the Fushun oil shale retorting plant has a low oil recovery rate. A large quantity of fresh water is used in the system, thereby consuming a considerable amount of water and energy, as well as polluting the environment. This study aims to develop a closed process of shale oil recovery from the circulating washing water for the Fushun oil shale retorting plant. The process would increase oil yield and result in clean production. In this process, oil/water hydrocyclone groups were applied to decrease the oil content in circulating water and to simultaneously increase oil yield. The oil sludge was removed by the solid/liquid hydrocyclone groups effectively, thereby proving the smooth operation of the devices and pipes. As a result, the oil recovery rate has increased by 5.3 %, which corresponds to 230 tonnes a month.

## Keywords

*Oil shale, hydrocyclone, oil recovery, water purification, sludge removal*

## Introduction

Oil shale is one of the abundant energy resources in the world. Global oil shale reserves are estimated at  $10\text{--}15 \cdot 10^{18}$  kJ, which accounts for 35 % of the world's total energy.<sup>1</sup> It has been used for energy technology purposes for hundreds of years. However, the total annual production of shale oil is currently estimated at only approximately  $140 \cdot 10^4$  t in China, Estonia, Brazil, and Australia. That figure is significantly less than the production of petroleum and coal. A definite increase in the production of shale oil is projected in the short term and is likely to keep on increasing in the medium term and eventually after 2020, when conventional oil resources are projected to become scarce.<sup>2</sup>

Current mature oil shale retorting processes are FLQ (China), Petrosix (Brazil), Kiviter (Estonia), ATP (Canada), and Galoter (Russia),<sup>3–5</sup> among others, and their basic characteristic parameters are shown in Table 1. The volume of the gas mixture that exits from retorts that utilize gas as a heat carrier is much larger than those that take solids as a heat carrier. Considering the cost and scale of operation, a water washing system is suited for the retorting process, which utilizes gas as heat carrier, while an oil washing system is mainly applied to the retorts that take solids as heat carrier.

Although oil shale reserves are abundant in China, the oil content is generally low. For example, the oil content of oil shale in Fushun is 2 %–13 %, with an average of 5.5 %, and

belongs to lean ore.<sup>5</sup> The FLQ process is extensively used in China because of its strong lean ore treatment capability. However, some problems exist in the oil recovery process, and these problems have resulted in a low oil yield of 63 %, which is significantly less than that of other processes. In the FLQ process, a large quantity of cooling water is used to condense the pyrolysis gas by direct contacting. Finally, the oil and water are separated through a multistage process of settling and air floating. Gravity sedimentation and a floating pool often need large covered areas. Closed processing has been difficult to achieve and its absence has resulted in air pollution. In addition, the wastewater contains a variety of barely degradable substances, thereby increasing the difficulty of subsequent water treatment and inducing hazards to the surrounding soils and groundwater.<sup>6,7</sup> This problem limits the further development of the FLQ process.

Although oil shale has been identified as a potential source of liquid fuels because of its abundant reserves, the performance of oil shale in the world oil market has historically been economically unfavourable because oil shale is considered a low-grade solid fuel that cannot compete with coal as a solid fuel. Without a low-cost technique to treat the circulating washing water, environmentally innocuous oil shale production is improbable, especially in China. Our proposed process, as discussed in this study, represents an improved method for shale oil recovery through the FLQ process by hydrocyclones.

The hydrocyclone is a typical centrifugal separating device for heterogeneous phases and widely used in mineral processing,<sup>8</sup> petrochemical,<sup>9,10</sup> food,<sup>11</sup> environmental<sup>12</sup> industries, among others. It has various advantages, such

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Table 1 – Main oil shale retorting process

Tablica 1 – Glavne tehnologije ekstrakcije nafte iz naftnih škriljavaca

	Technology Tehnologija				
	FLQ	Petrosix	Kiviter	ATP	Galoter
retort type vrsta retorte	vertical cylinder vertikalni cilindar	vertical cylinder vertikalni cilindar	vertical cylinder vertikalni cilindar	rotating cylinder rotirajući cilindar	rotating cylinder rotirajući cilindar
heat carrier noslač topline	circulating gas cirkulirajući plin	circulating gas cirkulirajući plin	flue gas dimni plin	shale ash pepeo škriljavca	shale ash pepeo škriljavca
feeding size/mm promjer napajanja/ mm	8–75	6–75	25–125	0–25	0–25
single retort capacity /td <sup>-1</sup> kapacitet jedne retorte /td <sup>-1</sup>	100	6000	1500	6000	3000
retort outlet tempera- ture/°C temperatura ispusta retorte/°C	90–110	150	200–250	–	470–490
oil recovery system sustav dobivanja nafte	water washing ispiranje vodom	water washing ispiranje vodom	oil washing ispiranje naftom	oil washing ispiranje naftom	heavy oil washing air cooler ispiranje teškom naftom hladnjak zraka
recovery efficiency/% iscrpak/%	6	90	80	85–90	85–90

as simple structure, high separation efficiency, and low operating cost. The flow field in a hydrocyclone is shown in Fig. 1. A strong vortex flow composed of an outer spiral of downflow and an inner spiral of upflow forms when the fluid is injected tangentially into the hydrocyclone. For the solid/liquid hydrocyclone, the particles are accelerated by centrifugal force toward the wall and migrate downward to the underflow following the outer spiral. Conversely, the light oil droplets are swept into the inner spiral and move upward to the overflow. Thus, immiscible phases with different densities are separated. The driving force for the separation comes from transforming the static energy of fluid (fluid pressure) into dynamic energy (fluid velocity), which typically operates at a low pressure drop of less than 0.3 MPa.<sup>13–15</sup>

This work aims to create a closed shale oil recovery system based on the current circulating water washing system, which increases the oil yield and offers a path toward cleaner production.

## Experimental system

### Closed oil recovery process

The closed process of oil recovery from circulating washing water by hydrocyclone groups is shown in Fig. 2. Oil shale granules with a size of 8–75 mm screened from the crude ore are heated at a temperature of 500 °C in the middle of the retort. The capacity of a single Fushun retort is only 100 tonnes per day, which is substantially less than that of other types of retorts. Thus, a combination of 20 retorts

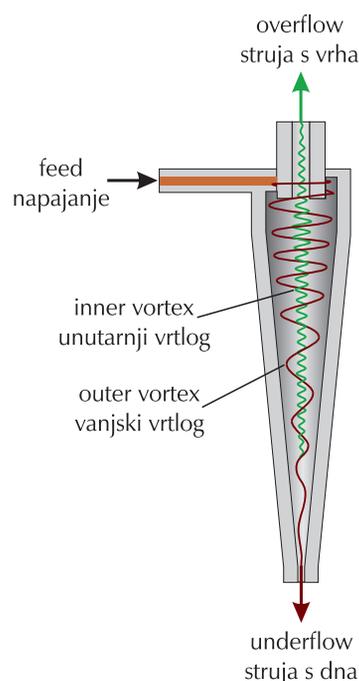


Fig. 1 – Vortex flow in hydrocyclone  
Slika 1 – Vrtložni tok u hidrociklonu

run simultaneously in an FLQ process, which generates a 52250–71250 m<sup>3</sup> gas mixture per hour and shares one set of water washing and oil recovery system. To maximize the shale oil yield and minimize the condensable liquid in the retort gas, the pyrolysis gases that exit from the top of the retorts are washed by four-stage individual circulating

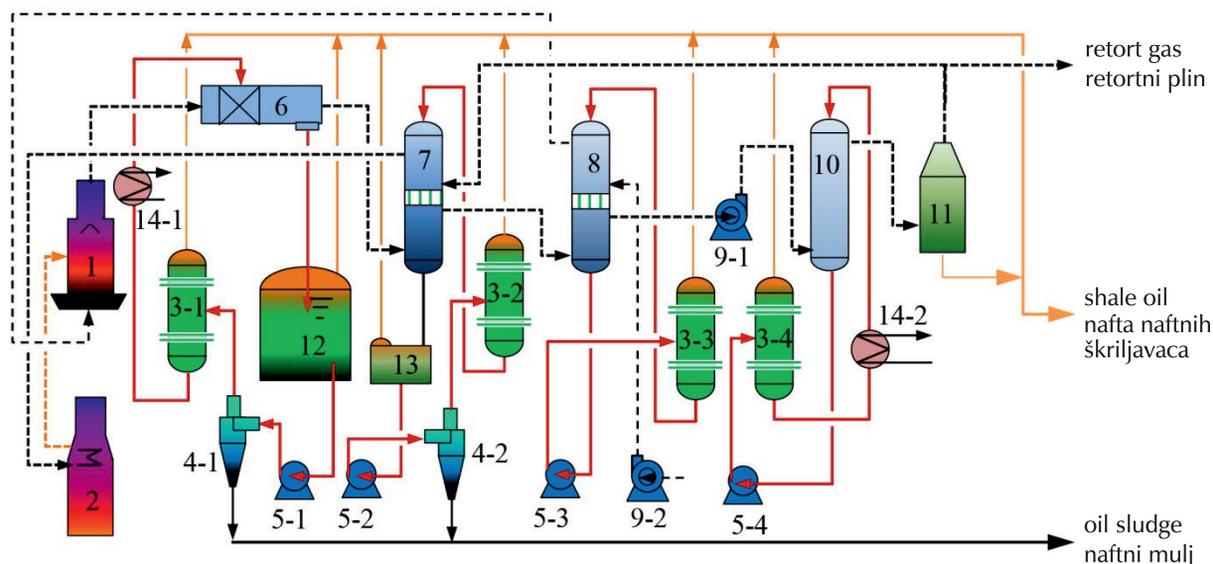


Fig. 2 – Schematic of the closed process of oil recovery from circulating washing water

(1) oil shale retort; (2) regenerative heating furnace; (3) OR-hydrocyclone groups; (4) SR-hydrocyclone groups; (5) water pump; (6) gathering tube; (7) retort gas cooling tower; (8) air cooling tower; (9) air compressor; (10) water cooling tower; (11) electrostatic detarrer; (12), (13) settling tank; (14) heat exchanger

Slika 2 – Shema zatvorenog procesa dobivanja nafte iz vode za ispiranje

(1) retorta za naftne škrljavce; (2) regeneracijska peć; (3) OR-hidrocikloni; (4) hidrocikloni; (5) vodena pumpa; (6) sabirna cijev; (7) rashladni toranj za retortni plin; (8) zračni rashladni toranj; (9) zračni kompresor; (10) vodeni rashladni toranj; (11) elektrostatski precipitator; (12), (13) taložni spremnik; (14) izmjenjivač topline

water washing systems, after which an electrostatic detarrer is used to capture the droplets (Fig. 3). The pyrolysis gases are delivered in the yellow pipelines.

When the pyrolysis gases of 95 °C enter the gathering tube, most of the coarse shale ashes and heavy oil (accounting for 60 % of the total) are washed into the circulating water and then carried into the settling tank (volume of 175 m<sup>3</sup>). The lower level water in the settling tank with a large quantity of suspended oil sludge (a mixture of shale ash, bitumen, and water) is pumped into the sludge removal hydrocyclone (SR-hydrocyclone) group. As most of the oil sludge particles are removed, the overflow of the SR-hydrocyclone is sent to the oil recovery (OR-hydrocyclone) group for further oil/water separation. Thereafter, the purified water that exits from the overflow of OR-hydrocyclone is cooled down to 85 °C by a heat exchanger and returns to the top of the gathering tube.

Some of the fine shale ashes pass through the gathering tube. Thus, a certain amount of oil sludge particles form in the circulating washing water in the retort gas cooling tower. A similar treatment process of washing water is applied in the second-stage oil recovery circulation, except the hot water is cooled by the returned retort gas (38 °C) in the upper section of the retort gas cooling tower. In addition, the volume of the settling tank is 65 m<sup>3</sup>, which is not large enough for the sedimentation.

After two steps of water washing, nearly all the shale ashes and most of the heavy oil in pyrolysis gases are removed. Therefore, only liquid/liquid hydrocyclone groups are applied for oil recovery in the last two-stage circulating water systems. With these four steps of water washing, more

than 93 % of the condensable matter is washed into the circulating water. An electrostatic detarrer is used to dry the retort gas because the gases (with a temperature of 38 °C) that exit from the top of the water cooling tower contain a large quantity of oil and water droplets. The designed operating parameters of the four-stage water washing devices are shown in Table 2. The retort gas is then sent to the power station, and the oil sludge is delivered to the filter press for dehydration. All the enriched oil that exits from the settling tank and OR-hydrocyclone groups is gathered for further settling dehydration.



Fig. 3 – Images of cooling towers and electrostatic detarrer. The numbers on the top of the devices correspond to Fig. 2.

Slika 3 – Fotografije rashladnih tornjeva i elektrostatskog precipitatora katrana. Brojevi na vrhu odgovaraju slici 2.

Table 2 – Designed operating parameters of the major devices in the oil recovery system  
 Tablica 2 – Operativni parametri glavnih uređaja u sustavu za izdvajanje nafte

	Gathering tube Sabirna cijev	Retort gas cooling tower Rashladni toranj za retortni plin	Air cooling tower Zračni rashladni toranj	Water cooling tower Vodeni rashladni toranj
gas temperature(in/out)/°C temperatura plina (unutra/van)/°C	95/86	86/79	79/71	76/38
water temperature(in/out)/°C temperatura vode (unutra/van)/°C	85/86	82/84	78/79	40/63
circulating water flux/m <sup>3</sup> h <sup>-1</sup> protok vode u cirkulaciji/m <sup>3</sup> h <sup>-1</sup>	203	500	350	500
head of pump/m visina stupca fluida/m	100	70	60	70
$\Delta p$ of SR-hydrocyclone group/MPa $\Delta p$ na skupini SR-hidrociklona/MPa	0.1	0.1	–	–
$\Delta p$ of OR-hydrocyclone group/MPa $\Delta p$ na skupini OR-hidrociklona/MPa	0.15	0.15	0.15	0.15

## Hydrocyclone separation

As the treatment capacity of a single hydrocyclone is limited by its diameter, several or perhaps hundreds of hydrocyclones are arranged in parallel to meet the large industrial requirement. In order to achieve high separation efficiency, it is necessary to resolved how to ensure uniform pressure drop and flow distribution of each hydrocyclone in the arrangement. According to Wang's theory of flow distribution in manifolds,<sup>16–18</sup> Chen et al.,<sup>19</sup> and Hang et al.<sup>20,21</sup> theoretically and experimentally studied uniform distribution in different types of hydrocyclone groups. In this work, the SR-hydrocyclones were arranged as the Z-Z type [Fig. 4(a)]. However, the OR-hydrocyclones were installed in the shell like the U-U type [Fig. 4(b)]. The density of Fushun shale oil is approximately 900 kg m<sup>-3</sup>, and the kinematic viscosity is 1.03 · 10<sup>-5</sup> m<sup>2</sup> s<sup>-1</sup>.

## Results and discussion

### Sludge removal

The presence of bitumen causes fine sludge particles to form aggregates easily because of their high adhesive ability. Abundant sludge particles cause equipment and pipeline jam, which may result in a serious unit outage or shutdown. Solid/liquid hydrocyclone groups are deployed after the settling tank to remove the fine oil sludge particles. The size distribution of sludge particles is measured by a laser particle size analyser (Mastersize 2000, Malvern Instruments). The particle size distribution of the oil sludge at the inlet of SR-hydrocyclone groups is shown in Fig. 5. The particle size of the oil sludge in the circulating water of a gathering tube is substantially larger than that of a retort gas cooling tower, with the average particle sizes being 75 and 24 μm, respectively.

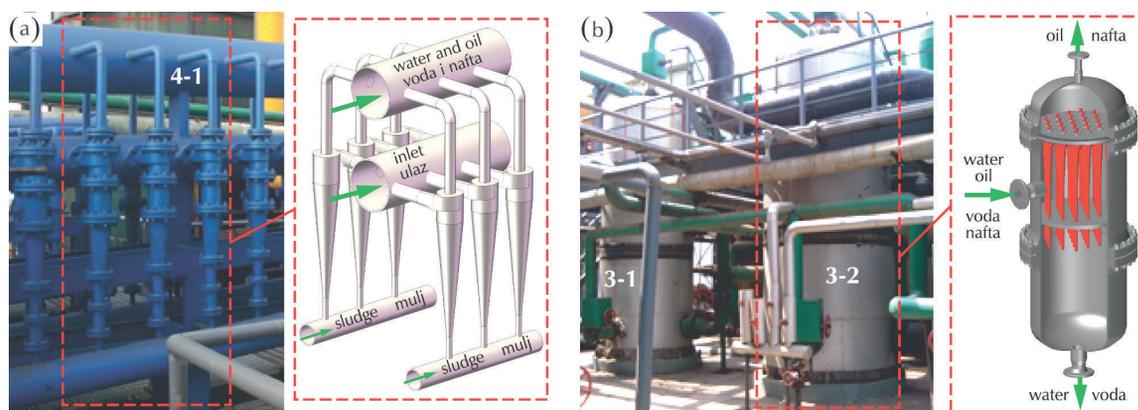


Fig. 4 – Hydrocyclone groups. (a) SR-hydrocyclone group; (b) OR-hydrocyclone groups. The numbers on the devices correspond to those in Fig. 2.

Slika 4 – Skupine hidrociklona: (a) SR-hidrocikloni, (b) OR-hidrocikloni. Brojevi odgovaraju slici 2.

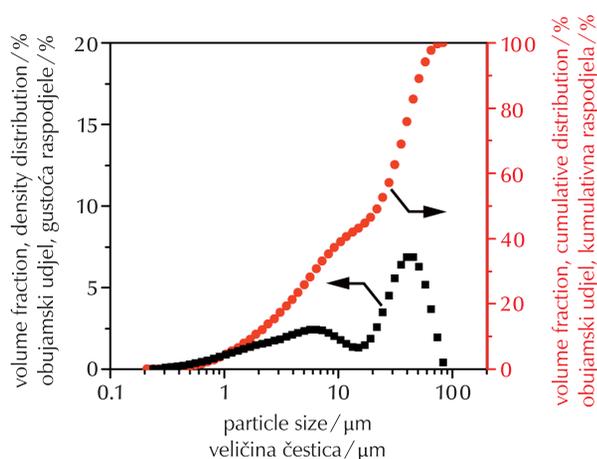
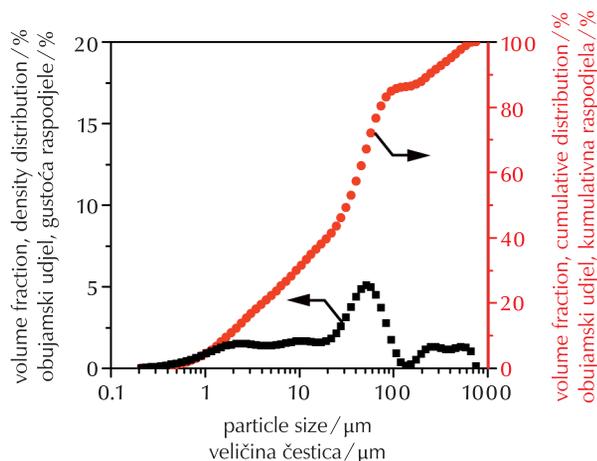


Fig. 5 – Particle size distribution of oil sludge: (a) at the inlet of SR-hydrocyclone for gathering tube, (b) at the inlet of SR-hydrocyclone for retort gas cooling tower

Slika 5 – Raspodjela veličine čestica u naftnom mulju: (a) na izlazu iz SR-hidrociklona u sabirnu cijev, (b) na izlazu iz SR-hidrociklona u radhladni toranj za retortni plin

The oil sludge particles were removed by the solid/liquid hydrocyclone having a cylindrical diameter of 100 mm, and working at the pressure drop  $\Delta p$  of 0.1 MPa (between the inlet and the overflow of the hydrocyclone), which was in accordance with the research of Yang et al.<sup>22</sup> The grade efficiencies for SR-hydrocyclone groups are shown in Fig. 6. Satisfactory separation efficiency for the circulating washing water is achieved when the cut size  $d_{50}$  of the SR-hydrocyclone is approximately 20–25  $\mu\text{m}$ .

The gravimetric method is applied to determine the solids content in the water in accordance with the Chinese national standards GB 11901-89 and GB 511-88. After the gravity settling tank, the oil sludge at the inlets of SR-hydrocyclone groups of the gathering tube is approximately 416  $\text{mg l}^{-1}$  (dry weight), and that in the retort gas cooling tower is 195  $\text{mg l}^{-1}$ . However, through the SR-hydrocy-

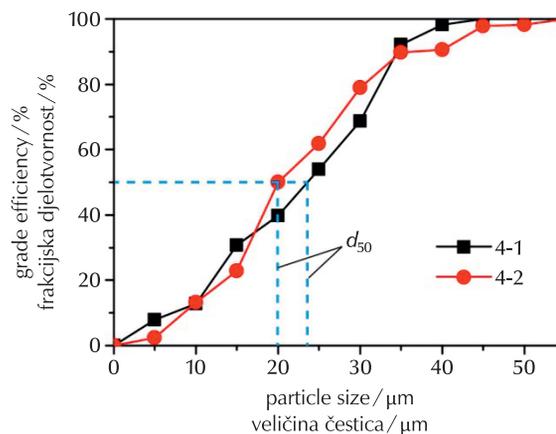


Fig. 6 – Grade efficiency of SR-hydrocyclones: (4-1) grade efficiency of the gathering tube SR-hydrocyclone; (4-2) grade efficiency of the retort gas cooling tower SR-hydrocyclone

Slika 6 – Frakcijska djelotvornost SR-hidrociklona: (4-1) SR-hidrociklon sabirne cijevi, (4-2) SR-hidrociklon rashladnog tornja retortnog plina

clone, the sludge dry weight is reduced to 47 and 27  $\text{mg l}^{-1}$ , respectively.

### Oil recovery

A key component of the closed process is the OR-hydrocyclone, which has a cylindrical diameter of 35 mm. To treat the large volume of circulating water, hundreds of de-oiling hydrocyclones were installed in parallel in a shell [Fig. 4(b)]. As the oil further separated from the circulating washing water, two benefits were obtained, namely, increased oil yield and enhanced condensation efficiency with the increase in the specific heat of water.

The total shale oil that condensed from the pyrolysis gas at the gathering tube and retort gas cooling tower was approximately 90 %. The operational states of OR-hydrocyclone groups are shown in Table 3. The hydrocyclone groups had a low pressure drop of 0.12–0.15 MPa, indicating low energy consumption. The residence times of the settling tank in the gathering tube and retort gas cooling tower were 52 and 7 minutes, respectively. After exiting the settling tank, the water still had a lot of oil, especially the water from the retort gas cooling tower. As most of the shale oil was recovered in the gathering tube and the retort gas cooling tower, the low oil contents in the washing water of the last two-stage cooling towers rendered the recovered shale oil uneconomical for use as settling devices for oil/water separation. With the assistance of OR-hydrocyclone, the oil content in the circulating water was restricted to a low level. Moreover, the oil yield had increased.

Table 3 – Operational states of OR-hydrocyclone groups  
 Tablica 3 – Osobine radnih skupina OR-hidrociklona

Device number Broj uređaja	$\Delta p$ MPa	Circulating water flux Protok vode u cirkulaciji $m^3 h^{-1}$	Oil mass fraction Maseni udjel nafte $mg l^{-1}$	
			inlet ulaz	outlet izlaz
3-1	0.15	200	784	114
3-2	0.15	500	3733	445
3-3	0.12	350	770	188
3-4	0.13	500	284	110

In comparison with the conventional process, production had improved with the newly developed oil recovery process (Table 4). As the oil content of the raw material declined slightly, the monthly treatment of oil shale increased at an average of 4487 t to maintain productivity. With the application of the OR-hydrocyclone groups, the shale oil recovery rate increased by 5.3 %. The monthly increase of shale oil is 230 t with the same weight of the raw material.

Table 4 – Comparison with conventional and new processes  
 Tablica 4 – Usporedba konvencionalnog i novog procesa

	Conventional process Konvencionalni proces	New process Novi proces
average mass fraction of oil in raw material/% prosječni maseni udjel nafte u sirovini/%	6.85	6.69
monthly treatment of oil shale/t mjesečna prerada naftnog škrljavca/t	60760	65247
monthly shale oil production/t mjesečna proizvodnja nafte iz škrljavca/t	2621	2980
oil recovery rate/% iscrpak nafte/%	62.97	68.27

## Conclusion

The closed process of oil recovery from the washing water was successively applied in an industrial retorting plant. With the application of hydrocyclones, this process presents various advantages as follows:

- (1) The shale oil in the circulating water is further recovered by the oil/water hydrocyclone groups, thereby improving the oil yield by 5.3 %. Given their high processing capability, hydrocyclones cover a smaller area than do settling devices. As the oil sludge is mostly removed by the SR-hydrocyclone groups, blockage of devices and pipes, which seriously affects production stability, is prevented effectively.
- (2) This process is closed such that the emission of pollutants in the circulating water is reduced and the condition of the surrounding air is improved.

## Summary

This paper introduces an improved process of shale oil recovery from washing water in the Fushun oil shale retorting unit. Solid/liquid hydrocyclone groups were used to remove the oil sludge, which avoided the blockage of devices and pipes. In order to increase the oil yield, liquid/liquid hydrocyclone groups were used to recover the oil in the washing water. In addition, with the application of hydrocyclone groups, the circulating water systems were closed, thus decreasing the pollutant emissions to the air. According to the operating results, the oil yield increased 5.3 %. This new process has good prospects in oil shale retorting industries.

## ACKNOWLEDGEMENTS

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## List of abbreviations and symbols Popis kratica i simbola

- OR – oil recovery  
– izdvajanje nafte
- SR – sludge removal  
– uklanjanje mulja
- $d_{50}$  – cut size of hydrocyclone,  $\mu m$   
– veličina razdvajanja hidrociklona,  $\mu m$
- $\Delta p$  – pressure drop, MPa  
– pad tlaka, MPa

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## SAŽETAK

### Izdvajanje nafte naftnih škriljavaca hidrociklonima iz vode za ispiranje u cirkulaciji

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Konvencionalni postupak za dobivanje nafte iz naftnih škriljavaca u pogonu Fushun ima nizak iscrpak. Sustavu upotrebljava mnogo svježe vode te se troši mnogo energije, vode i onečišćuje okoliš. U radu se razvija zatvoreni sustav za dobivanje nafte iz naftnih škriljavaca s cirkuliranjem vode za ispiranje namijenjen retornom postrojenju Fushun. U procesu su primijenjene skupine hidrociklona za smanjenje količine nafte u vodi za ispiranje istodobno povećavajući iskorištenje. Naftni mulj uklonjen je skupinom hidrociklona čvrsto/tekuće što osigurava miran rad uređaja i cijevi. Iscrpak nafte povećao se za 5,3 %, odnosno 230 tona mjesečno.

#### Ključne riječi

Naftni škriljavac, hidrociklon, proizvodnja nafte, pročišćivanje vode, uklanjanje mulja

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