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Potential Yield of Oil from Middle Jurassic Sargelu Oil Shale, Zagros Mountains, Southwest Iran: A Pyrolysis Approach

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Abstract

In this study, for the first time, Fischer Assay, thermal dissolution, and direct and indirect hydrogenation methods were used to comparatively evaluate oil (bitumen) yield of the Middle Jurassic Sargelu oil shale. Three oil shale samples in three localities from Sargelu oil shale from Zagros Mountains in southwest Iran were selected. According to the results of oil yield of the samples, the final temperature and residence time from the modified Fischer Assay were not significantly different from those of the standard Fischer Assay. Accordingly, the highest oil yield based on the standard and modified Fischer Assays were 34.29 and 33.7 wt.% from organic matter (OM), respectively. Nevertheless, the modified Fischer Assay could increase the API gravity from 18.30 to 24.82 while reducing the viscosity from 7.34 to 4.13 cP. Based on the results of the thermal dissolution in the presence of different solvents, the highest oil yield was achieved in the presence of methanol at 350°C in 4 hours. Oil yield varied significantly with different solvents (23.1 to 59.4 wt.% from OM). Direct hydrogenation was performed in the presence of hydrogen at two levels of pressure (20 and 30 bar) and a low temperature, resulting in an oil yield of up to 53.68 wt.% from OM. Indirect hydrogenation in the presence of an aqueous solution of 25% sodium formate (HCOONa) at 380°C in 5 hours produced an oil yield of 51.59 wt.% from OM. Indirect hydrogenation method in the presence of water and zinc solution improved the oil yield to 62.93 wt.% from OM. The findings of this study indicated that thermal dissolution and hydrogenation methods can increase oil (bitumen) yield with respect to pyrolysis. Maximum oil yield of the Sargelu oil shale achieved by indirect hydrogenation at 400°C in 2 hours.

Keywords: Sargelu Oil Shale, Pyrolysis, Fischer Assay, Thermal Dissolution, Hydrogenation.

Introduction

Oil shale refers to an immature, unconventional reserve of a non-renewable source of energy, demonstrating large potential for addressing the ever-increasing demand for energy (Shekarifard et al., 2021). Oil shales are fine-grained organic matter (OM)-rich sedimentary rocks that produce, upon destructive distillation (i.e., where oil is produced upon heating the oil shale in the absence of oxygen) (Sinag & Canel, 2010), large amounts of oil and gas (Dyni, 2003). The process of heating the oil shale in the absence of oxygen is referred to as retorting, pyrolysis, or semi-coking (Sinag & Canel, 2010). In the oil shale industry, the so-called Fischer Assay is usually employed to evaluate the oil yield of an oil shale reserve. In this analysis, the studied oil shale is subjected to controlled heating in a small retort, where oil, water, and gas are produced in a neutral atmosphere (Heistand, 1976). Oil shale is composed of kerogen, bitumen, and inorganic minerals

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(Ding et al., 2021). In nature, kerogen is fully solid and cannot be recovered from the reserve in the form of crude oil or natural gas (Kang et al., 2020). Inorganic matter comprises some 50 – 85 wt.% of the oil shale, including silicates, carbonates, quartz, and pyrite (Zhao et al., 2017). With the help of the results of the ultimate analysis, one can determine the composition of the OM in the crude oil shale in terms of percent contents of carbon, hydrogen, nitrogen, and various sulfur species. On the other hand, proximate analysis can properly provide information about the percent contents of moisture, ash, and volatile compounds (Foltin et al., 2017).

The first discoveries of probable reserves of oil shales in Iran date back to 1970 when the National Iranian Oil Company (NIOC) explored the Zagros Basin and found a reserve in the Southwest Iran. The two main units of oil shale in this area include the upper part of the Middle Jurassic Sargelu Formation and the lower part of the Late Cretaceous Garau Formation (Shekarifard et al., 2024). Rasouli et al. (2015), investigated OM-rich sediments in five different areas in Southwest Iran, introducing Sargelu and Garau formations as unconventional yet high-potential reserves of hydrocarbon. In this study, the authors referred to oil-producing type-II kerogen as the source of the studied sediments and identified the generated hydrocarbons as mostly oil (Rasouli et al., 2015). Shekarifard (2022) presented a thermochemical study of Sargelu oil shales to characterize their thermos-oxidative decomposition. The thermo-oxidative decomposition was conducted for 13 samples of Sargelu oil shale in three stages: (1) thermos-oxidative decomposition of bitumen, (2) thermos-oxidative decomposition of kerogen, and (3) decomposition of calcite mineral (Shekarifard, 2022). A geochemical study of oil shales usually requires some sort of extraction with organic solvents. Nassef et al. (2015) were found that the type of solvent greatly affects the production efficiency and composition of the produced hydrocarbon from the oil shale in Egypt. They used tetrahydrofuran, toluene, methylene chloride, normal hexane, and chloroform as solvents. Their experimental outcomes showed that the oil yield was maximal when tetrahydrofuran was used as a solvent for extraction (Nassef et al., 2015). Another approach to producing liquid products out of oil shale is hydrogenation, which can be either direct or indirect. In the direct hydrogenation, the pyrolysis is performed at high hydrogen pressure to boost hydrocarbon production (Burnham, 2017). Compared to the thermal extraction method, hydrogenation can lead to higher extraction yields at lower decomposition temperatures. Alternatively, a wide range of hydrogen donor solvents can be used to introduce the hydrogen into the pyrolysis of oil shale indirectly. A common approach to this method is to use a blend of water and sodium formate, which enables a lower decomposition temperature than the thermal extraction method. Experiments by Friedman showed that the hydrogenation of Estonian kukersite oil shales with water over a processing time of 30 minutes at 400 - 430°C can produce liquid and gaseous hydrocarbons at an efficiency of 93%. In contrast to the semi-coking of the kukersite, the adopted method produced hydrocarbons of lower molecular weight and phenol concentration (Klesment and Nappa, 1980). Maloletnev *et al.* (2011) subjected OM-rich shales of the Baltic to hydrogenation at low hydrogen pressure and succeeded in producing liquid (76 – 78%) and gaseous (18 – 20%) products (Maloletnev et al., 2011). Klesment et al. (1980) studied the structure and decomposition mechanism of kukersite oil shales via experiments at sub-industrial temperatures, and further compared the extractions of hydrocarbon via hydrogenation by sodium formate against the standard Fischer Assay (Klesment and Nappa, 1980).

In the present study, standard and modified Fischer Assays thermal dissolution and direct and indirect hydrogenation were performed to compare bitumen (oil) products from Sargelu oil shales, with the focus being on finding the best method for maximum oil yield.

Samples and analytical methods

In this study, three samples from Middle Jurassic Sargelu oil shale from three different locations from Zagros Mountains in southwest Iran were selected. These samples were ground by a

grinder and then sieved by standard meshes into powders of 0.4 to 1 mm in particle size.

Proximate and Ultimate analysis

Selected samples were subjected to proximate analyses. The value of the proximate analysis is that it identifies the fuel value of the as received material; provides an estimate of the fuel quality with indicating contents of organic matter (OM) and moisture. Also, CO₂ released indicates the share of carbonaceous material in mineral matter. Heating value is also determined to indicate energy released in combustion. Ultimate analysis was further performed to characterize the elemental composition of the selected Sargelu oil shales. Results are reported in Table 1.

Standard and modified Fischer Assay

Standard Fischer Assay was practiced on the oil shale samples according to the ISO 647 standard method. To this end, 100 g of the sample with a particle size in the range of 0.4 – 1 mm was heated at 520°C for 80 minutes, and then the final temperature was kept for 10 minutes. Modified Fischer Assay was conducted at different final temperatures in the range of 400 - 480°C. Indeed, the applied final temperature was reduced from 520°C in the standard Fischer method down to 440°C, 480 °C, and 400°C for samples 1, 2, and 3 in the modified Fischer Assay, respectively. The time to reach the final temperature in this analysis was set to 30 – 50 minutes, with the samples subjected to pyrolysis at the final temperature for 3 hours. API degree and viscosity measurements were further done to investigate the quality of the extracted shale oil. (Tables 2 and 3)

Table 1. Results of proximate and ultimate analyses on selected Sargelu oil shales

	Sample-1	Sample-2	Sample-3
Proximate analysis			
W _a ^d (%)	0.68	1.65	0.57
A _d (%)	59.43	58.57	57.83
OM (%)	17.61	28.39	23.93
CO ₂ of carbonates (%)	22.28	11.38	17.67
Calorific value (MJ/kg)	6260.00	10150.00	8940.00
Ultimate analysis			
C (%)	21.00	26.90	25.4
H (%)	1.24	2.11	1.84
N (%)	0.43	0.70	0.49
S (%)	2.25	3.31	3.40

Table 2. Results of the standard Fischer Assay

	Fischer Assay (Wt%)					Density @15°C (g/cm ³)	API gravity	Viscosity (cP)
	Oil	Semicoke	Pyrogenetic water	Gas+losses	Oil from OM			
Sample-1	9.49	85.89	1.26	3.37	34.29	0.9196	22.40	7.40
Sample-2	9.25	86.12	2.02	2.61	33.49	0.9446	18.30	7.34
Sample-3	3.98	90.77	2.10	3.16	22.27	0.9194	22.41	6.47

Table 3. Results of modified Fischer Assay

	Modified Fischer Assay (Wt%)						Density @15°C (g/cm ³)	API gravity	Viscosity (cP)
	Isother m. 3hr	Oil	Semicoke	Pyrogenetic water	Gas+losses	Oil from OM			
Sample- 1	430- 450	8.74	87.34	1.33	2.60	31.92	0.9111	23.86	3.84
Sample- 2	480	9.24	86.40	1.83	2.53	33.70	0.9052	24.82	4.13
Sample- 3	400	3.12	94.65	1.32	0.91	17.74	0.9080	24.47	3.54

Thermal dissolution

To evaluate the bituminous products from oil shale samples by extraction via thermal dissolution, the selected samples were heated at a fixed temperature of 350°C in small 20-cm³ autoclaves, where they were subjected to thermal dissolution in the presence of different solvents (water, methanol, acetone, hexane, benzene, and 1:1 wt.% water-benzene mixture) for 4 hours (sample to solvent ratio was 5:8 w/w).

Hydrogenation

Direct hydrogenation experiments were performed for samples 1 and 2. In this analysis, sample 1 was mounted into a 500-cm³ autoclave under a pressure of 20 bar at 400°C for different residence times ranging from 1 to 4 hours. To check for the effect of benzene on the performance of direct hydrogenation, a part of the same sample was analyzed in the presence of 100 mL of benzene at 20 bar and 400°C for 2 hours. Sample 2 was further subjected to direct hydrogenation under a pressure of 30 bar and a temperature in the range of 250 - 410°C for 2 hours. To further evaluate the effect of residence time, this sample was hydrogenated directly at fixed temperature and pressure of 410°C and 30 bar for different residence times of 1, 2, and 6 hours. Indirect hydrogenation analysis was conducted by a mixture of hydrogen donors, including an aqueous solution of sodium formate and zinc in water, on the three samples at a temperature of 380°C (up to 5 hours) and 400°C (up to 4 hours) in 20-cm³ autoclaves.

Results and discussion

Characterization of initial oil shale

The heating value of the Sargelu oil shale was different among different samples, ranging from 6260 to 10150 MJ/Kg, which is the result of differences in the OM content. With increasing OM content in the samples proportionally increases the calorific value. Moisture content of the Sargelu oil shale was much lower (on average: 0.96 Wt.%) than that of industrially used oil shales (Shekarifard, 2022). For the Estonian and Chinese oil shales, the moisture and ash contents are reportedly exceeding 5% and 65%, respectively (Qing et al., 2007; Trikkel et al., 2008).

Pyrolysis in Fischer Assay

The results show the oil content of the samples from standard Fischer and modified Assay is 3.98 to 9.49 Wt.%, 3.12 to 9.24 Wt.%, respectively. Oil yield of samples in modified Fischer Assay is a little less than those from standard Fischer Assay.

In order to understand the effects of temperature and residence time on oil yield Sargelu oil shales, standard and modified Fischer Assays were compared with one another. Results show that reducing the final temperature from 520°C in the standard Fischer Assay to 400 - 480°C in the modified Fischer Assay and also extending the residence time from 10 minutes to 3 hours impose

no significant effect on the oil yield of the selected oil shales (Fig. 1a). In the meantime, application of modified, rather than standard Fischer Assay resulted in an increase in API gravity from 22.40, 18.30, and 22.41 to 23.86, 24.82, and 24.47 in samples 1, 2, and 3, respectively. Based on the outcomes, the extracted shale oils could be categorized as heavy to medium crude oils (Fig. 1b). Another effect of the change in the pyrolysis conditions on the quality of the extracted shale oil by modified Fischer Assay could be seen in the face of the significant reduction in the viscosity of the extracted hydrocarbon. For sample 1, 2, and 3 the viscosity reduced from 7.40 to 3.84 cP, 7.34 to 4.13 cP and 6.47 to 3.54 cP, respectively (Table 3, Fig. 1c).

Na et al. (2012) reported that the yield of shale oil increased as reaction temperature at 500°C increased. Shale oils produced from retorting process could be characterized depending on the reaction temperature. As retorting temperature was increased, the yield of shale oil increased. At high reaction temperature, the portion of gas product increased due to secondary cracking (Na et al., 2012).

Pyrolysis in autoclave in presence of solvent

According to the results obtained from the thermal dissolution, it can be concluded that different solvents lead to different oil yields (Fig. 2). In this respect, outputs of thermal dissolution of Sargelu oil shale samples show that the low oil yields are associated with water, hexane, and benzene as solvents. In general, the highest oil yield for the three samples of Sargelu oil shale was achieved with methanol (59.4 and 54.2 wt.% from OM for samples 1 and 2). Compared to the Fischer Assay, thermal dissolution with methanol led to about 20% higher efficiency at lower temperatures. Shawaqfeh and Al-Harash (2004) have studied the extraction of bitumen from El-Lajjun and Sultani oil shales using different types of solvents.

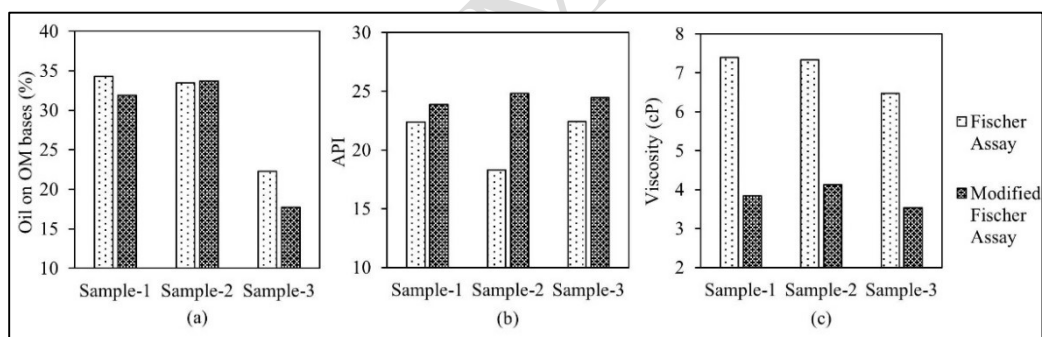


Figure 1. Comparison of oil yield (a), the API (b) and viscosity (c) from standard and modified Fischer Assays

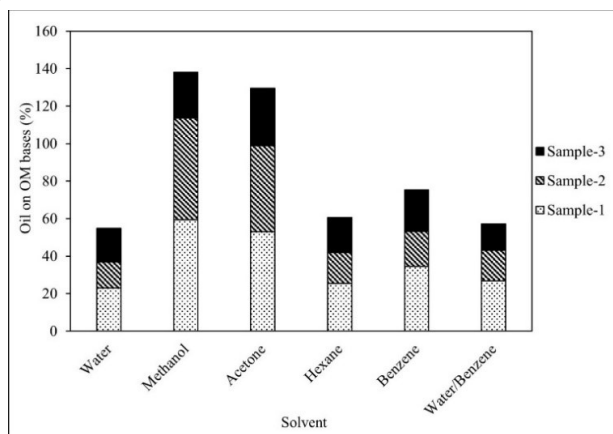


Figure 2. Comparison of the efficiency of bitumen extraction from Sargelu oil shale by thermal dissolution in the presence of different solvents

The results indicated the decrease of solubility index with the increase of solvent's molecular weight as in cases of methanol, ethanol, propanol, and butanol. These trends can be attributed to the mobility of solvent molecules, as it decreases with the increase of molecular weight (Shawaqfeh & Al-Harashah, 2004).

Pyrolysis in autoclave in presence of hydrogen

The effect of the presence of hydrogen in improving the oil yield of Sargelu oil shale was investigated in two stages. In the first stage, the oil shale samples 1 and 2 were directly subjected to hydrogenation under an operating pressure of 20 and 30 bar, respectively. In this stage, we comparatively studied the effects of temperature and residence time on the pyrolysis process. In the second stage, the oil shale sample 3 was subjected to indirect hydrogenation in the presence of hydrogen donors (an aqueous solution of 25% sodium formate and zinc in water) to produce hydrocarbon and other pyrolysis products. Fig. 3 shows the results of direct hydrogenation on sample 1 at a fixed temperature of 400°C. According to this figure, the highest oil yield with direct hydrogenation at 20 bar and 400°C was achieved after 1 hour of residence time (38.16 wt.% from OM). By further extending the hydrogenation process time, heavier hydrocarbons were decomposed in the presence of hydrogen, leading to increased production of the pyrolysis gas. Compared to hydrogenation in the absence of a catalyst, introduction of benzene further increased the gas production (from 32.59 to 52.88 wt.% from OM) – that is, introduction of benzene reduced the solid residue and the extracted shale oil.

Investigation of the effect of temperature on the oil yield by hydrogenation on sample 2 indicates that, similar to sample 1, extending the hydrogenation process time tends to reduce the shale oil yield while increasing the gas production. Accordingly, for this sample, with increasing the hydrogenation time from 2 hour to 6 hour (while fixing the hydrogenation pressure and temperature at 30 bar and 410°C), the oil yield decreased from 53.68 wt.% to 17.34 wt.% from OM (Fig. 4).

Evaluation of the effect of hydrogenation temperature on this sample showed that an increase in the final temperature of hydrogenation tends to increase the amounts of shale gas and oil produced. This depicts that the required activation energy for the pyrolysis is not available at 250°C, and the weight of solid residue (91.35 wt.%) shows that the majority of the OM content of the sample has not even engaged in the pyrolysis process. At 410°C, however, more shale oil was extracted, leaving smaller solid residue (Fig. 5). According to the results of the direct hydrogenation at 30 bar, it was found that the highest shale oil yield of sample 2 is associated with a processing temperature of 410°C for 2 hours.

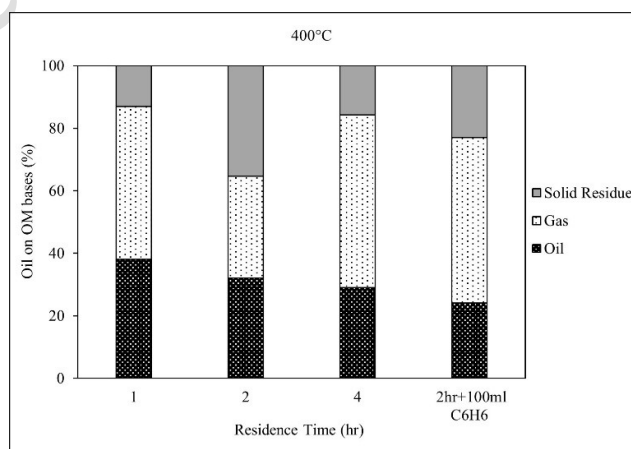


Figure 3. oil yield of the Sargelu oil shale sample 1 upon extraction in the presence of hydrogen at 20 bar and 400°C for different residence times in the range of 1 to 4 hours

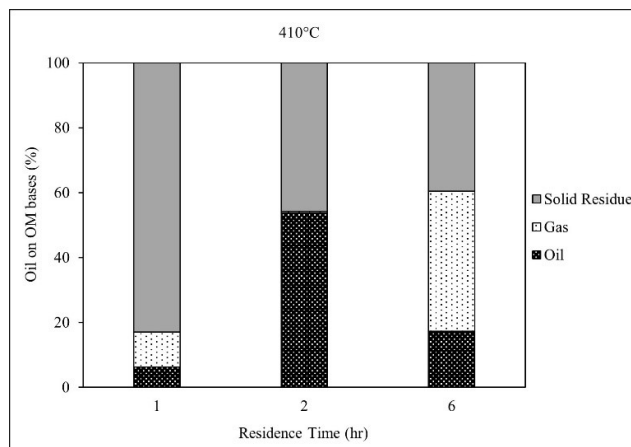


Figure 4. Comparison of the produced amounts of pyrolysis products via direct hydrogenation on the Sargelu oil shale sample 2 at 30 bar and 410°C for different residence times in the range of 1 to 6 hours

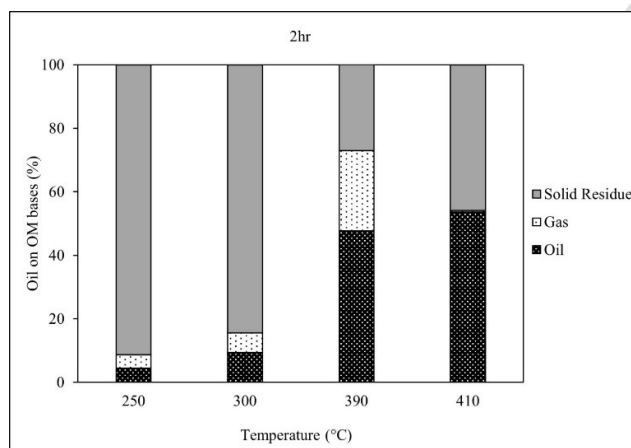


Figure 5. Comparison of the produced amounts of pyrolysis products via direct hydrogenation on the Sargelu oil shale sample 2 at 30 bar and temperatures in the range of 250 - 410°C for a residence time of 2 hours

Focusing on the indirect hydrogenation process, Sargelu oil shale sample 3 was studied with water and sodium formate as hydrogen donors. In this approach, the sodium formate was decomposed at the hydrogenation temperature (Eq. 1), and the produced carbon monoxide reacts with water to release hydrogen (Eq. 2). Results show that the shale oil yield increases with the hydrogenation time at constant temperature, reaching a maximum of 51.59 wt.% from OM in 5 hours (Fig. 6). The significant effect of sodium formate as a hydrogen donor turns a large portion of the OM content of the shale sample into hydrocarbon (extracted oil and gas). Indeed, the gas and oil yields become equal after 1 and 3 hours, while the semi-coke content is minimized. Compared to direct hydrogenation, the introduction of sodium formate in the indirect hydrogenation enables higher oil yields at lower processing temperatures.



The introduction of zinc into water (Eq. 3) releases hydrogen content in the water, indirectly contributing to the hydrogenation reaction. Results show that the indirect hydrogenation by means of aqueous zinc solution over extended residence time at constant process temperature can reduce the weight of solid residue. That is, the released hydrogen reacts with the carbon content of the oil shale, turning relatively heavier compounds into bituminous liquid and lighter

ones into pyrolysis gas. The reaction time imposes a significant impact on the oil yield. Indeed, with extending the reaction time to up to 2 hours, the oil yield was maximized at 62.93 wt.% from OM, while the gas yield was reportedly 25.03 wt.%. Further extension of the reaction time increased the gas yield (33.47 wt.%) while minimizing the solid residue (6.61 wt.%) (Fig. 7). Indirect hydrogenation with zinc and water, rather than sodium formate, enabled maximum shale oil yield at 400°C within a shorter reaction time.



In general, the results of the direct and indirect hydrogenation analyses on the selected Sargelu oil shale samples showed that the indirect hydrogenation is a more effective approach to pyrolysis-based extraction at lower temperatures.

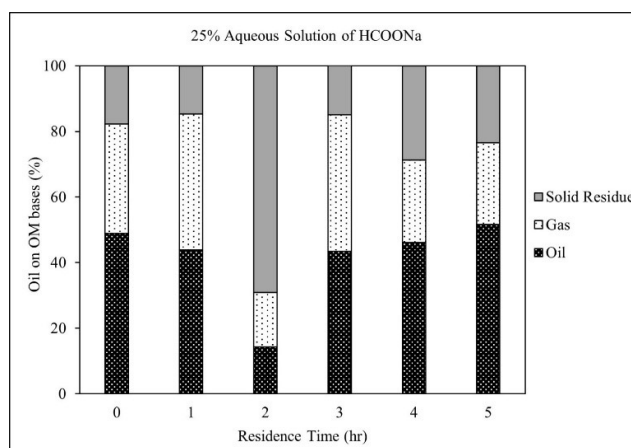


Figure 6. Comparison of the produced amounts of pyrolysis products via indirect hydrogenation on the Sargelu oil shale sample 3 in the presence of an aqueous solution of 25% sodium formate at 380°C for different residence times in the range of 0 – 5 hours

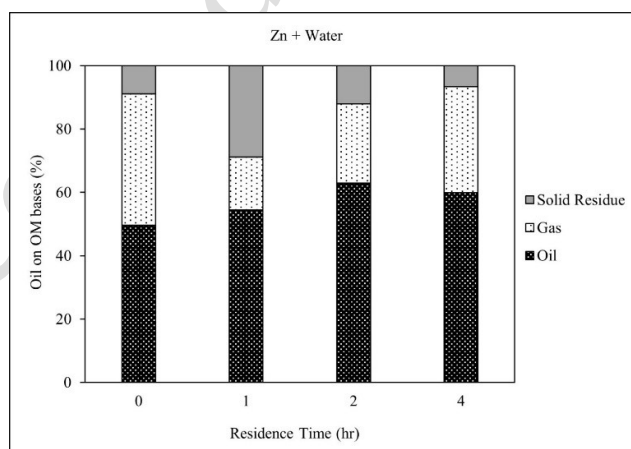


Figure 7. Comparison of the produced amounts of pyrolysis products via indirect hydrogenation on the Sargelu oil shale sample 3 in the presence of zinc-water solution at 400°C for different residence times in the range of 0 – 4 hours

The increase in the yield of shale oil is attributed to three reasons: the higher pyrolysis conversion rates, the addition of external hydrogen sources, and the displacement effect of steam. The hydrogen free radicals provided by steam can induce the breaking of chemical bonds in oil shale, promote the cracking and conversion of organic matter, leading to the release of more shale oil product. Meanwhile, steam provides an external hydrogen source for cracked oil

fragments and participates in the formation of shale oil during pyrolysis of oil shale. Hydrogen in steam enters into shale oil in different forms and inhibits the polymerization of cracked oil fragments into shale char. Furthermore, steam also exhibits a positive displacement effect and can carry shale oil to the pores and fractures, making it easier to discharge during pyrolysis (Kang et al., 2025).

Maloletnev et al. (2011) reported the degree of organic matter of shale liquefaction 96–98% at a hydrogen consumption of 1.5–2.0% for the reaction, and the yield of liquid products 76–78% on a basis of the organic matter of shale paste, including 40–45% products with boiling point to 320°C, 18–20% gas, and 8–9% water. Molecular hydrogen, which was introduced into the process in an amount of 1.5–2.0%, was mainly consumed for the formation of C₁–C₄ hydrocarbon gases (8–10% yield), and it can be returned to the process after their conversion with water vapor (Maloletnev et al., 2011).

Conclusion

Results of analyzing the oil yield of Sargelu oil shale showed that this reserve exhibits promising hydrocarbon generation potentials, with the actual oil yield varying with the applied experiment conditions.

Results of Fischer Assays showed that the final temperature and residence time cannot affect oil yield significantly although they can improve the quality of the extracted shale oil in terms of higher API and lower viscosity.

Proper choice of solvent in thermal dissolution can improve oil yield, thereby increasing the oil yield over the Fischer Assay. Thermal dissolution in the presence of methanol led to an oil yield of 59.4 wt.% from OM.

In direct hydrogenation, extending the residence time at a constant temperature of 410°C increased the gas production while reducing oil yield. On the other hand, rising the reaction temperature in direct hydrogenation over a fixed reaction time of 2 hours improved oil yield.

For the studied samples, direct hydrogenation enabled a maximum oil yield of 53.68 wt.% from OM, which was significantly higher than the best outputs of the Fischer Assay (33.49 wt.% from OM). In a particular case, indirect hydrogenation in an aqueous solution of sodium formate at 380°C resulted in an oil yield of 51.59 wt.% from OM, while the corresponding figure for aqueous zinc solution at 400°C was as high as 62.93 wt.% from OM. The same sample exhibited an oil yield of only 22.27 wt.% from OM when subjected to the Fischer Assay at 520°C.

As a results, it can be concluded that one can adjust the pyrolysis conditions in the presence of an appropriate solvent to hydrogenate Sargelu oil shale at lower temperatures and hence obtain shale oil of oil shale by consuming less amounts of thermal energy. In the meantime, it should be noted that changes in the extraction and experiment conditions can affect the quality of the produced shale oil.

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