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Removal of Sulfur-Containing Compounds from Shale Oil Using Ionic Liquids

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Highlights

- Oil shale crushed and pyrolyzed at 550 ^{o}C
- Shale oil is treated with ionic liquids
- Oil is separated into two layers
- Rich sulfur compounds and lean phase are tested for sulfur compounds
- Determination of total sulfur in each phase

Abstract

Oil shale is a fine-grained sedimentary rock that contains large quantities of organic materials, mostly kerogen. Jordan has large reserves of oil shale, estimated at more than 70 billion tons, oil shale contains sulfur in the form of pyrite compounds. Organometallic sulfurs are found in shale oil after pyrolysis of the oil shale. The sulfur could be up to 10 wt.% of the total shale oil generated. Oil Shale sample collected from Attarat Um Ghudran mine east of Amman – Aqaba desert highway. In this research, two types of ionic liquid were used to extract sulfur from shale oil: 1-ethyl-3-methylimidazolium chloride ([EMIM][Cl]) (IL-A) and 1-butyl-3-methylimidazolium thiocyanate ([BMIM][SCN]) (IL-B). Ionic liquid was mixed with shale oil in a ratio (1:1) for the extraction of sulfur compounds. Results have shown good ability of ILs to extract sulfur present in shale oil. Results indicate that the removal efficiency of IL-A ranged from 28% to 38.9%, while IL-B efficiency ranged from 30.8% to 41.4%. Ionic liquids have succeeded in extracting organometallic sulfur from shale oil.

Paper type:Research Paper

Keywords: Sulfur, Extraction, Ionic liquid, Shale Oil, Pyrolysis.

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1. Introduction

Oil shale is a fine-grained sedimentary rock that contains large quantities of organic materials, mostly kerogen. When heated, kerogen decomposes into shale oil and flammable gases. Unlike crude oil, which occurs in liquid form in reservoirs, the hydrocarbons in oil shale are buried within the rock matrix and must be removed by heat methods (Dyni, 2006). Oil shale is composed of both inorganic and organic. The inorganic component consists of minerals such as quartz, feldspar, clay, and carbonate, whereas the organic component is largely kerogen (Altun, et al., 2006) **(2)**. Kerogen is a complex combination of hydrocarbons and other factors that influence how effectively shale oil may be produced. Kerogen can constitute between 5% and 40% of the shale weight, contingent upon the deposit, hence influencing the yield and quality of the extracted oil. Jordan is one of the countries with the largest oil shale reserves, estimated at more than 70 billion tons (Abu-Hamatteh, and Al-Shawabkeh, 2008, Al-Ayed and Saadeh, 2021). These have been discovered in

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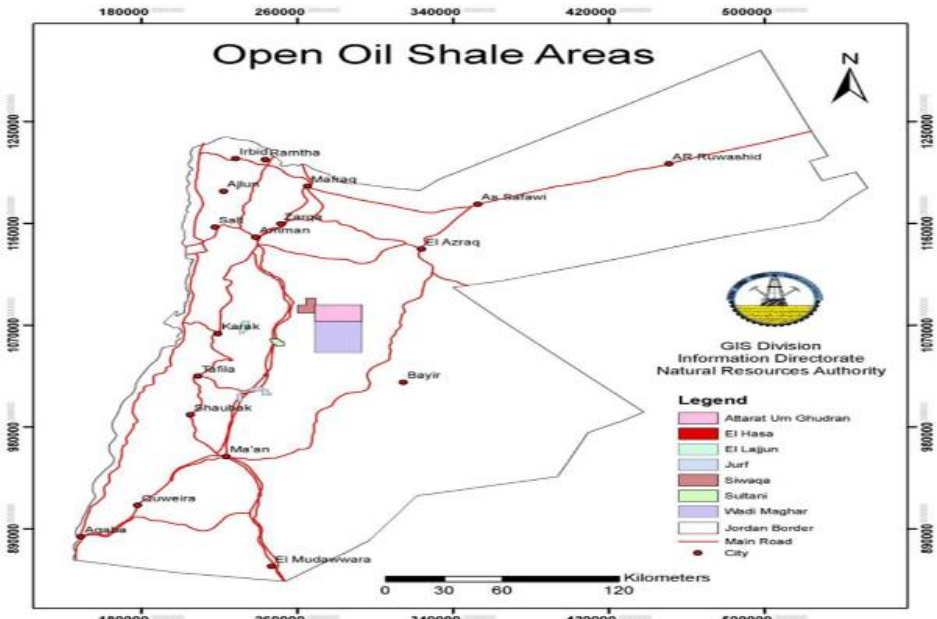
places such as El-Lajjun, Attarat Umm Ghudran, Sultani, and the Yarmouk Valley. The natural character and accessibility of such resources make them suitable for pyrolysis-based total shale oil production. The geological distribution of oil shale in Jordan is presented in **Figure 1**. Pyrolysis is a thermal process that uses heating to break the kerogen of the oil shale in the absence of air (usually at a temperature of 450 − 500 °C) into gases and liquid hydrocarbons. Kerogen, the organic component of oil shale, is converted into organic part of oil shale that is responsible for

Fig. 1. Distribution of oil shale in Jordan (Alali, 2006)**.**



hydrocarbons, produces liquid shale oil, gases, and solid residues known as spent shale or coke (Wang, et al., 2021). The produced shale oil requires upgrading to be used as fuels similar to crude oil distillation and hydrotreating. This shows the importance of oil shale as source of energy. The conversion of oil shale into shale oil by providing energy forms that can be transformed into usable products (Guo, et al., 2021). Several important products can be obtained from shale pyrolysis oil such as shale oil (alternative to crude oil), which can be further processed via fractionation into liquid fuels such as gasoline, diesel, and jet fuel (Sakulkit, et al., 2020, Faten et al., 2022). Shale oil obtained through pyrolysis of kerogen in oil shale contains numerous organometallic compounds originating from both organic sulfur in kerogen, and to a lesser extent inorganic sulfur in the mineral matrix. The composition and concentration of these sulfur compounds are different for oil shale of different geological origins, kerogen types, and pyrolysis conditions (Raja, et al., 2017).

The Jordanian oil shales contain organic and inorganic sulfur in which the organic sulfur is mainly incorporated into the kerogen frame. Organosulfur species such as thiophene and alkyl thiophene (C_1-C_4) are generated during kerogen pyrolysis and represent a major part of the sulfur composition in the aromatic fractions of shale oil, and their removal is difficult because of their stable chemical structures (Alharahsheh et al., 2005). Sulfide, disulfide, and thiols are found in small amounts, giving the oil its reactivity and odor. Pyrolysis generates H_2S, leading to environmental and operational problems, a very small amount exists as inorganic sulfur, mainly as pyrite (FeS_2) (Katasonova et al., 2021, Reham et al., 2024). The sulfur content of Jordanian oil shales is generally found to be in the range of 10% to 12% by weight in deposits like El-Lajjun and Attarat Umm Ghudran, and some deposits, including Sultani and Jurf ed-Darawish, have been reported to exhibit increased sulfur contents estimated at 8–10 wt.% (Alnawafleh and Fraige 2015, Alsmadi et al., 2024).

Ionic liquids (ILs) are salts that remain in a liquid state at or near ambient temperature. In contrast to typical salts such as sodium chloride, which possess elevated melting temperatures, ionic liquids frequently have melting points below 100°C. This is due to their unique chemical structure, comprising bulky, asymmetrical organic cations and inorganic or organic anions. The weak intermolecular interactions of these ions reduce lattice energy, resulting in low melting points (Plechkova and Seddon 2008).

They have many advantages such as non-volatility, high thermal stability, a wide range of liquid temperatures, and favorable design characteristics for applications including catalysis, separation, and electrochemistry (Welton 1999). Also, ionic liquids have the disadvantage of high cost (Zhao et al., 2009), environmental impact certain ionic liquids have fluorinated anions (Plechkova and Seddon 2008), and viscosity problems (Freemantle 2010).

Shale oil has embedded Sulfur atoms in its structure as thiophene, benzothiophenes, and dibenzothiophene, the aromatic sulfur compounds that were hard to remove compared with traditional hydrodesulfurization (HDS) reactions (Zhao et al., 2009), ionic liquids facilitate the extraction of these sulfur compounds into the organic phase through hydrogen-bonding and π-π interactions with thiophenes rings (Kulkarni and Afonso 2010).

The main objective of this research is to validate the ability of ionic liquids, 1-Butyl-3-Methylimidazolium Thiocyanate, (IL-A) and 1-Ethyl-3-Methylimidazolium Chloride, (IL-B) to extract organometallic sulfur compounds from shale oil produced from Attarat shale oil. In addition, to test the effect of particle size on the rate of sulfur compounds generated during the pyrolysis process.

2. Materials and Methods

2.1 Reagents

In this research, two types of ionic liquids were used as reagents 1-Butyl-3-Methylimidazolium Thiocyanate, and 1-Ethyl-3-Methylimidazolium Chloride, **Table 1.** Show some properties of reagents. Ionic Liquids (IL) are promising options to develop extractive processes that replace conventional Hydrodesulfurization (HDS). Traditional HDS process employ Cobalt and/or nickel molybdenum catalysts at elevated temperatures and pressures in the ranges of 300 – 500 ºC and 10 -210 bar respectively (Haifa et al., 2023). Several workers (Welton, 2009, Saleh et al., 2021, Butt et al, 2020) are working to develop industrial processes to replace HDS using Ionic Liquids. The high thermal stability, low melting point, high polarity, non-volatility, non-flammability, immiscibility with water in addition to ease of recycling and regeneration, in addition to other physical and chemical unique properties that makes the ILs an attractive material despite their high cost (Waseware, 2013). The 1-butyl-3-methylimidazoliumthiocyanate, [BMIM]SCN, (IL-B) displays a high S-removal capability from gasoline and diesel in single-stage extraction (Dharaskar, et al., 2014) in addition to high thermal stability and acceptable viscosity values are behind the choice of using it in the extractive desulfurization (EDS).

Table 1. Major properties of ILs reagents.

Ionic Liquid Name	**Company**	**Molecular Formula**	**Molecular weight**	**Assay %**	**Physical State**
1-Butyl-3-Methylimidazolium Thiocyanate, (IL-A)	Chem Cruz	$C_9H_{15}N_3S$	197.30	>95%	Liquid
1-Ethyl-3-Methylimidazolium Chloride, (IL-B)	Thermo Scientific	$C_6H_{11}ClN_2$	146.62	98%	Solid

2.2 Sample Collection and Preparation

Study samples were crushed, sieved using BSS standard sieving system. Since, large quantity of shale oil is needed, desired sizes were selected based on the quantity collected on the different sieves.
The first step was to crush oil shale into small pieces using a jaw crusher. Sieving the crushed oil shale using special vibratory sieves of different sizes such as BSS sieving system. During sieving three particle sizes, 2.8, 6.7, and 13.2 mm were selected for the study. The third step was the pyrolysis process of oil shale using a controlled stainless steel retort where the oil shale particles were heated to temperatures ranging from 500°C to 550 °C, at the rate of 10 °C/min under controlled nitrogen flow rate.

2.2.1 Procedure for Sulfur Extraction from Shale Oil

The process of extraction of sulfur-containing compounds from shale oil consists of two steps:

a. The solution for the extraction of the sulfur compounds from shale oil was prepared by mixing 1 ml liquid or 1 gram solid (depending on the physical state of the IL) of the desired ionic liquid (either IL-A or IL-B) with 2 ml deionized water. The mixture was stirred for 1-2 hours at room temperature, then allowed to separate for 15 minutes. After decantation, the water layer was removed, and the ionic liquid rich phase is used for the extraction process.
b. The IL rich phase is mixed shale oil in a ratio of 1:1. The mixture was stirred for about 60 min using a magnetic stirring plate at 500 rpm and room temperature. The resultant solution is separated into a separatory funnel to isolate the IL-sulfur rich from the shale oil. The three samples, i.e., the original shale oil, the IL-sulfur rich phase (solution) and the treated shale oil were subjected to sulfur measurement and analysis.

3. Results and Discussion

Organometallic compounds present in shale oil are produced as a result of pyrolyzing oil shale to produce petroleum like-material called shale oil. During processing of crude oil, the weight percent of sulfur should not exceed 4 wt.% to be refined without special attention sulfur content. The impact of sulfur on the catalyst and environmental pollution is tremendous and needs attention. Sulfur bonded to hydrocarbons poses operational problems during refining and product specification. Removal of these organometallic sulfur containing compounds is important in shale oil industry.

3.1 Particle Size Effect on the Quantity of Sulfur in Shale Oil

Particle size is an important factor influencing the yield of shale oil during the pyrolysis process. In catalytic reactions, the diffusion of reactants, products across (mass transfer) the solid material of the catalyst plays an important role in the product distribution of the reaction. In addition, heat transfer has an additional role in affecting the reaction kinetics. To study the influence of particle size on the amount of the extracted sulfur-containing compounds from oil shale, different particle sizes are tested. As reported in Table 2, in general, increase the particle size decreases, the sulfur content measured in weight percent of the total sulfur content in sample. It's clear from **Table 2,** that increasing particle size from 2.8 mm to 6.7 mm, the sulfur decreased from 11.76 %wt. to 9.93 % wt. It's clear from these data that when the particle size is increased by a factor of 2.4, the reduction in sulfur content is decreased by 15 % wt. This indicates that less sulfur is extracted from the original oil shale sample into the shale oil liquid. On the other hand, when the particle size is increased by a factor 4.7 times with respect to the smallest particle size used in this work, the sulfur content is decreased by a factor of 25% wt. As a result, there is no direct relationship between the size and the amount of sulfur extracted from the oil shale.

Table 2. The concentration of sulfur in pyrolyzed shale oil as a function of particle size.

Oil Shale, Particle size, mm	Sulfur Concentration ($\times 10^4$ppm)
2.8	11.76
6.7	9.93
13.2	8.76

Plotting the data of Table 2 on excel sheet as depicted in **figure 2,** The weight of sulfur-containing components of the shale oil extracted from shale oil is inversely proportional to oil shale particle size. It's clear that the generated trendline exhibits a good fit to the experimental data with coefficient of determination (R^2) equals to 0.93. These findings confirm that the particle size affects the rate of sulfur compounds generated from oil shale.

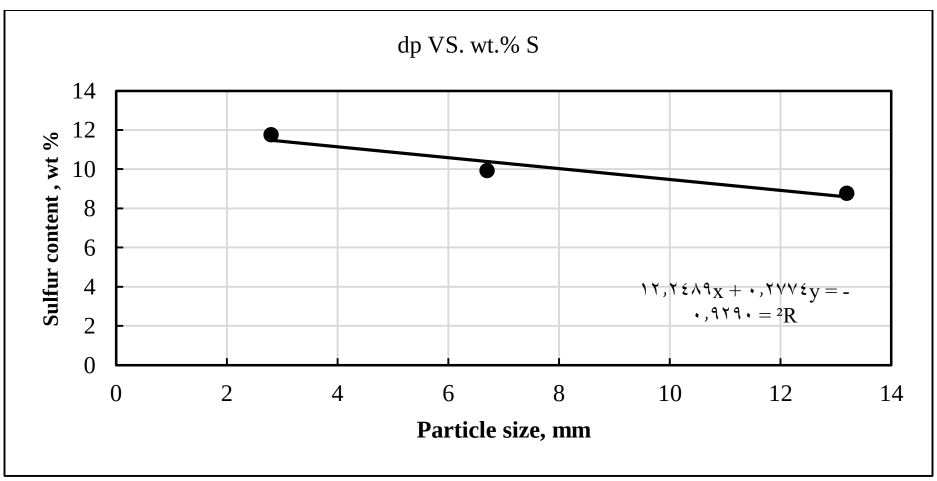


Fig. 2. sulfur weight percentage in shale oil as function of particle size.

3.2 Extraction with 1-Ethyl-3-Methylimidazolium Chloride ([EMIM][Cl]) (IL-A)

In this research,1-Ethyl-3-Methylimidazolium Chloride (IL-A) ionic liquid is used to extract sulfur containing compounds from shale oil. The IL-A compound has demonstrated extracting ability to sulfur. The experimentally measured extracted sulfur ranged between 2.63 to 3.53 $\times 10^4$ppm in the IL-A rich phase in the studied range of particle sizes. On the other hand, the wt. % sulfur containing compounds remain in the shale oil rich phase fluctuated between 6.06 – 8.46 $\times 10^4$ppm.

Results of **Table 3** indicate that the original generated shale oil sample shale oil 1(2.8) contained 11.76 $\times 10^4$ppm sulfur before extraction which decreased to 8.46$\times 10^4$ppm after extraction. This result shows that a 28% reduction in sulfur content of the sample is achieved using IL-A extracting agent. The sulfur wt. % in IL-A rich phase after extraction was 2.76 $\times 10^4$ppm, whereas the sulfur remains in the shale oil after extraction is 8.46$\times 10^4$ppm. Analyzing test sample oil 2(6.7), it is clear from the table that the IL-A could extract 3.53 $\times 10^4$ppm from 9.93 $\times 10^4$ppm. The content of the IL-A rich phase contained 35.5 % of the total sulfur in the original shale oil sample. On the other hand, the left sulfur wt.% in shale oil sample is 64.5%. Comparing the extraction results of the two samples, namely, oil 1(2.8) and oil 2(6.7), it's clear that the extraction efficiency is not the same.

Table 3. Results of Sulfur extraction using ([BMIM][SCN]), IL-A.

Oil Sample	Sulfur Concentration in Original Oil ($\times 10^4$ppm)	Sulfur Concentration in Oil After using IL-A ($\times 10^4$ppm)	Sulfur Concentration in IL-A after Extraction ($\times 10^4$ppm)
Oil 1 (2.8)	11.76	8.46	2.76
Oil 2 (6.7)	9.93	6.06	3.53
Oil 3 (13.2)	8.76	6.26	2.63

which could be attributed to the nature of sulfur extracted compounds which were influenced by the size of sample used to generate the shale oil sample. Since the organometallic sulfur in shale oil could be either sulfide, disulfide, thiophene, mercaptans in which the wt.% of these components differ depending upon the size used which reflected on the amount extracted. Analyzing the extraction results of oil 3(13.2) sample, and according to Table 3 results, sulfur concentration in original oil was 8.76 $\times 10^4$ppm reduced to 6.26 $\times 10^4$ppm, using IL-A component. The IL-A rich phase contained 2.63 $\times 10^4$ppm after extraction process whereas the oil rich phase results in 30 wt.% reduction.

As can be concluded from the extraction results, using IL-A has succeeded in extracting an average of 30 wt.% of sulfur from shale oil without clear trend. The complexity of types of sulfur components generated during oil shale pyrolysis could be the source of unclearness of the trend. In addition, the reaction conditions such as particle size, heterogeneity of oil shale, reaction time and pyrolysis temperature could play a vital role on the nature and type of sulfur organometallic compounds generated.

3.3 Extraction with 1-Butyl-3-Methylimidazolium Thiocyanate ([BMIM][SCN]) (IL-B).

The second ionic liquid used in this research is 1-Butyl-3-Methylimidazolium Thiocyanate, (IL-B). The extracted sulfur using IL-B ranged between 4.1 – 2.43 $\times 10^4$ppm in the ionic liquid rich phase. The average extracted amount in the rich-IL-B phase is 33wt% of the original sulfur content in sample. Investigating the individual extraction samples, it can be seen from **Table 4** that for the sample oil 1(2.8) and IL-B extracting agent, the extracting efficiency is 30 wt.%. It is apparent that 3.46$\times 10^4$ppm is extracted from 11.76$\times 10^4$ppm from the original sulfur content, whereas 70wt. % remain in the shale oil rich sample. Similar results are found for samples oil 2(6.7) and oil 3(13.2). The ionic liquid, IL-B is capable of extraction 41.3 and 27.8 wt. % of sulfur from the original oil content of sample respectively. The sulfur compounds are distributed between the IL-B rich phase and shale oil rich phase during the extraction. Again, here, no clear trend could be found regarding the effect of particle size and amount extracted ascribing that to type and nature of the sulfur compounds generated under reaction conditions. As a result, the ionic liquid IL-B type is also efficient in extracting sulfur compound similar to the IL_A reported earlier with varying degree of efficiency

Table 4. Results of Sulfur extraction using ([BMIM][SCN]) IL-B.

Oil Sample	Sulfur Concentration in Original Oil ($\times 10^4$ppm)	Sulfur Concentration in Oil using IL-B (10^4ppm)	Sulfur Concentration in IL-B after Extraction (10^4ppm)
Oil 1 (2.8)	11.76	8.13	3.56
Oil 2 (6.7)	9.93	5.81	4.1
Oil 3 (13.2)	8.76	5.93	2.43

4. Conclusions

This study focuses on the extraction of organometallic sulfur from shale oil using different ionic liquids. Two types of ionic liquids used, IL-A and IL-B, have been used. The results have shown the ability of ionic liquids to extract sulfur from shale oil. IL-A extracted more than 30wt.% of the total sulfur present in the shale oil generated by pyrolysis of oil shale particles with 2.8, 6.7 and 13.2 mm particle sizes. On the other hand, IL-B also demonstrated an ability to extract organometallic sulfur compounds from shale oil with 35 wt.% removal efficiency.

It is well known that there are different types of sulfur components that are present in shale oil. Unfortunately, in this work, the type of sulfur compound in shale oil was not identified before the extraction process. It is recommended that a simulated shale oil mixture should be prepared to identify the selective extraction by using ionic liquids. Also, it is recommended to identify the type of sulfur compound extracted to the ionic liquid rich phase by analyzing the extracts.

Credit Author Contribution Statement

All authors must specify their roles using the CRediT taxonomy. Use clear role labels and multiple roles per author if applicable.
Example:
O. Al-Ayed: Conceptualization, Supervision, Writing – Review & Editing
Eyad Abu-Nameh: Investigation Project Administration.
M. Daradkeh: Methodology, Validation, Data Curation, Writing-Original Draft

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