Oil Shale, 2026, 43, 1, 104–116 https://doi.org/10.3176/oil.2026.1.05

Ionic liquids, [EMIM]Cl and [BMIM]SCN for sulfur removal from shale oils

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Received 19 September 2025, accepted 23 January 2026, available 29 January 2026

Abstract. *Oil shales from Attarat and Sultani were pyrolyzed at 550 °C to produce shale oils for the present study. The organic sulfur content of the two shale oils was determined to be 9.3 and 10.5 wt.%, respectively. Two ionic liquids (IL), 1-ethyl-3-methylimidazolium chloride ([EMIM]Cl) and 1-butyl-3-methylimidazolium thiocyanate ([BMIM]SCN), were used in liquid–liquid extraction for desulfurization. The extraction process was carried out at room temperature. The liquid–liquid extraction resulted in two-phase formation and redistribution of sulfur compounds into the aqueous IL-rich phase and the shale oil phase. The hydrocarbon sulfur weight percent was determined using a CHNSO analyzer. The removal efficiency for Sultani and Attarat shale oils with [EMIM]Cl was calculated to be 52.4 and 58.1 wt.%, respectively. When [BMIM]SCN was employed for the extraction of sulfur compounds from Sultani and Attarat shale oils, removal efficiencies of 43.8 and 52.4 wt.% were achieved, respectively. When the surfactant T-80 was added to Sultani shale oil and heated to 60 °C, followed by addition of [EMIM]Cl, the extraction efficiency decreased to 40.9 wt.%. On the other hand, when the mixture of shale oil and IL was heated to 60 °C before adding T-80, the weight percent removal increased to 58.1%.*

Keywords: *shale oil, ionic liquid, desulfurization, Attarat, IL/oil ratio.*

1. Introduction

In general, sulfur species in crude oils/fuels are a major issue during refining and cause air pollution, which ultimately endangers public health. Particulate emissions, processing equipment, corrosion of equipment, catalyst poisoning/

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deactivation, transportation, and storage of fuels are affected by the presence of sulfur compounds in their different forms. Hydrodesulfurization (HDS) is the conventional method of sulfur removal from crude oils or distilled fractions [1]. Alternative methods to HDS have been developed, such as oxidation. In oxidative desulfurization, the sulfur compounds in fuel are oxidized to sulfoxides in a slow reaction step, then to sulfones in a fast reaction step, which are then removed through liquid extraction [2, 3], precipitation and adsorption [4], extraction [5], and alkylation, in which the boiling point of the organosulfur compounds is increased by alkylation of thiophene with olefins, thus increasing the molecular weight to allow separation by distillation [6].

Kerogen is the organic-bearing material in oil shale composition that is also known to generate organometallic compounds [7]. Shale oil includes the known classes of hydrocarbons, as well as heteroatoms such as sulfur, nitrogen, and oxygen, in addition to metals [8–11]. The sulfur content of Jordanian shale oil can be as high as 9.0–12.0 wt.% [12]. Baird et al. [13] and Järvik et al. [14] studied the shale oil sulfur content generated from kukersite oil shale and reported sulfur content in the range of 0.4–1.2 wt.%. These authors compiled and summarized works of several researchers who employed standard hydrotreating and more advanced methods to remove sulfur, nitrogen, and oxygen organometallic compounds from shale oil. On the other hand, Rang et al. [15] discussed non-hydrodesulfurization processes, including extraction, oxidation, and adsorption, as advanced methods used to deeply desulfurize liquid fuels, reducing sulfur content to less than 50 ppm.

The sulfur content of Chinese shale oil was reported as high as 2.19 wt.% [16]. Brazilian shale oil from the Irati location contained as low as 1.0 wt.% sulfur [17]. Sulfur in shale oil is present in different forms, such as mercaptans, thiophenes, their derivatives, and also sulfides or disulfides in the lighter fractions of shale oil.

One of the recent advanced methods to remove sulfur from fuels/shale oils is the use of ionic liquids (IL). Some of these ILs have the ability to perform liquid–liquid desulfurization LLD) and oxidative desulfurization, removing the most recalcitrant compounds present in fuels/shale oils. Compounds such as thiophene, benzothiophene, dibenzothiophene, and smaller molecular weight sulfur compounds can leave the oil phase and migrate to the aqueous phase containing ILs [18]. Researchers have reported that the migration of sulfur compounds is achieved through the formation of hydrogen bonding and dipole–dipole interactions, in addition to π–π stacking interactions between the sulfur compounds in fuels and ILs [19]. ILs in the aqueous phase containing the extracted sulfur compounds can be regenerated and recycled, improving the economics of LLD [20].

The role of ILs in removing sulfur compounds from crude oils and/or fractions such as gasoline, kerosene, diesel, and others has been attracting attention for decades. The ease of availability of these solvents to remove sulfur compounds and aromatics has made them popular among researchers.

ILs such as 1-butyl-3-methylimidazolium thiocyanate ([BMIM]SCN) were used to extract dibenzothiophene and aromatics from base oil, in which certain amounts of naphthalene and dibenzothiophene were dissolved [21]. The extractions results showed that 92.0 wt.% of the dibenzothiophene was removed under the experimental optimum extraction conditions, such as temperature and IL/oil ratio. In extractive desulfurization [22], the removal efficiency of dibenzothiophene from n-dodecane model oil was 86.5% at 30 °C, with an IL/oil mass ratio of 1:1 after 30 min. An IL, 1-ethyl-3-methylimidazolium chloride ([EMIM]Cl), was used as an extractant with a model oil (dodecane) containing dibenzothiophene as the sulfur compound [23]. The extraction of the model sulfur compound dibenzothiophene was reported to be 99 wt.%. The researchers also conducted regeneration of the spent IL via toluene back-extraction.

Using ILs in shale oil LLD has not been investigated thoroughly yet. In some cases, the weight percent of sulfur present in shale oil is much higher than that of crude oils or distilled fractions such as gasoline, kerosine, diesel, and fuel oils. Due to the higher sulfur mass percent in shale oils, it is difficult to refine shale oils in ordinary refineries. The shale oil content of heteroatoms/organometallic compounds must be reduced, or the shale oil must be hydrotreated to reduce the share of these compounds to permissible limits for possible distillation in crude oil refineries.

In general, few experimental tests have been performed by mixing ILs with real shale oil to extract sulfur/nitrogen compounds. ILs such as [BMIM]SCN, in addition to others, have been tested for extracting basic and neutral nitrides from Fushun shale oil in China [24]. Recently, in a separate study, ILs, i.e., [EMIM]Cl and [BMIM]SCN, were used to extract sulfur compounds from shale oil generated by pyrolyzing Jordanian Attarat shale oil [25]. The objective of the present work is the extraction of sulfur compounds present in real shale oil obtained by pyrolyzing oil shale at 550 °C from two different locations, Attarat and Sultani. The generated shale oils were tested for sulfur extraction using two ILs.

2. Materials and methods

2.1. Oil shale pyrolysis

The targeted locations were the Attarat Um-Alghudran area (31°16′08″ N 36°26′52″ E) and the Sultani mine in the Al-Hissa location (30.7705° N 35.8761° E). Oil shale samples were crushed and sieved to the desired size, 8 mm, using the British Standard Sieves system. This section of experimental work was initiated by destructive distillation (pyrolysis) of shale oil under inert nitrogen conditions, at temperature up to 550 °C. For each oil shale sample, a mass of 400 g was placed in the retort and pyrolyzed at 550 °C until no more shale oil was condensed. This part of the work generated the shale oil required for IL extraction experiments.

The produced shale oil was separated from the retorted water using a separatory funnel. The bottom water layer was taken and discarded, while the top shale oil layer was used for the liquid–liquid extraction experiments.

2.2. Liquid–liquid extraction of shale oil

The second part of the experiment was the extraction of sulfur compounds with [EMIM]Cl (purchased from Thermo Fisher Scientific, catalog No. 354091000, CAS No. 79917-90-1, HPLC $\geq 98\%$, water ≤ 1.0, molecular weight = 174.67, solid) and [BMIM]SCN (purchased from Thermo Fisher Scientific, catalog No. H59493.06, CAS No. 331717-63-6, proton NMR 97.5% min., water $< 0.26\%$, molecular weight = 169.25, pale yellow liquid). The extraction of sulfur compounds from shale oil using ILs was also employed at two levels: the stand-alone level and pretreatment of shale oil with a surfactant (Tween-80, nonionic, polyoxyethylene surfactant, Thermo Fisher Scientific, catalog No. 28329, light yellow liquid, concentration 10.3%, pH = 7.59, detergent grade). The treatment steps resulted in two separate phases: a shale oil phase and an aqueous phase containing the ILs and extracted sulf ur compounds.

Both phases were subjected to total sulfur determination. In the present work, only the mass of sulfur in the two phases was of interest, since these represent the total sulfur content of the shale oil.

One gram of shale oil was weighed and mixed with one gram of ILs, resulting in an IL/oil ratio of 1:1. The mixing process was conducted at room temperature; this part of the experiment was performed for both shale oil samples and both types of ILs used. To study the effect of heating, two procedures were applied: heating a mixture of IL and shale oil prior to the addition of surfactant, and heating a mixture of shale oil and surfactant before the addition of ILs. Heating was performed at 60 °C using a water bath. The heated mixture was allowed to cool and separate into two phases. The removed total sulfur was determined using a Euro Vector 3000 CHNSO elemental analyzer.

2.3. Surfactant heating treatment

In this section of the experiment, the effect of mixture heating was investigated. The surfactant was added to the shale oil and IL mixture at less than 0.1 wt.% to avoid emulsion formation. Two types of treatment were tested in this part of the work. In the first trial, the surfactant was added to the mixture before heating in a water bath, i.e., at room temperature, followed by mixing and phase separation. In the second trial, the mixture of shale oil/surfactant and shale oil/IL was placed in the water bath for 3 h at 60 °C before the addition of IL or surfactant, respectively, to induce phase separation. These experiments were conducted in conjunction with distilled water for dilution of additives and facilitation of extract and raffinate separation. This step was performed to investigate the influence of surfactants on the viscosity of shale oil and their role in reducing surface tension during the extraction of sulfur from shale oil in the presence of ILs.

3. Results and discussion

The mass balance of Attarat and Sultani oil shale pyrolysis runs indicated that more than 10% of the oil shale samples by weight were converted to shale oil. Experimental runs were conducted in a stainless steel retort. The produced shale oil was used for sulfur determination before and after IL treatment. The test results showed that the total sulfur in Attarat and Sultani oil shales was 9.3 and 10.5 wt.%, respectively. A single GC–MS analysis run of a shale oil sample from Attarat showed the presence of several sulfur compounds, including, thiophene, 2,5-dimethylthiophene, 3,4-dimethylthiophene, 2,3-dimethylthiophene, 2,3,4-trimethylthiophene, 3,4-diethylthiophene, 2,5-diethylthiophene, benzothiophenes, 2-methylbenzothiophene, and other thiophene derivatives.

Liquid–liquid extraction with ILs allowed the arbitrary redistribution of sulfur compounds from shale oil into two phases. One phase was IL-rich, containing the extracted sulfur compounds, and the other was shale oil-rich. Sulfur content determination was performed for both phases. The efficiency of the extraction process was calculated based on the original sulfur weight measured in the shale oil before liquid–liquid extraction. The weight of sulfur in each phase, i.e., the IL-rich phase (aqueous phase) and the shale oil-rich phase, were determined for efficiency calculations.

3.1. Sultani shale oil

The results of sulfur compound extraction using [BMIM]SCN and [EMIM]Cl are depicted in Figure 1. Figure 1 shows three results: the sulfur content of the freshly produced shale oil, shown by the column on the left graph under the title “Shale oil,” is 10.5 wt.%. The weight percent sulfur found in the [BMIM]SCN aqueous extract (AQL) is depicted in the middle column of the upper graph as 4.6 wt.%. The right-hand column indicates the weight percent sulfur remaining in the shale oil phase after extraction, measured as 6.1 wt.%. The lower graph depicts similar results using [EMIM]Cl. As shown, the use of [EMIM]Cl resulted in 5.5 wt.% sulfur. The efficiency of the extraction process was calculated to be 43.8% and 52.4% using [BMIM]SCN and [EMIM]Cl, respectively. As can be seen, [EMIM]Cl is a better extraction agent for sulfur compounds than [BMIM]SCN. It should be noted that the sulfur content of the [BMIM]SCN IL was not deducted from the total sulfur measured in the

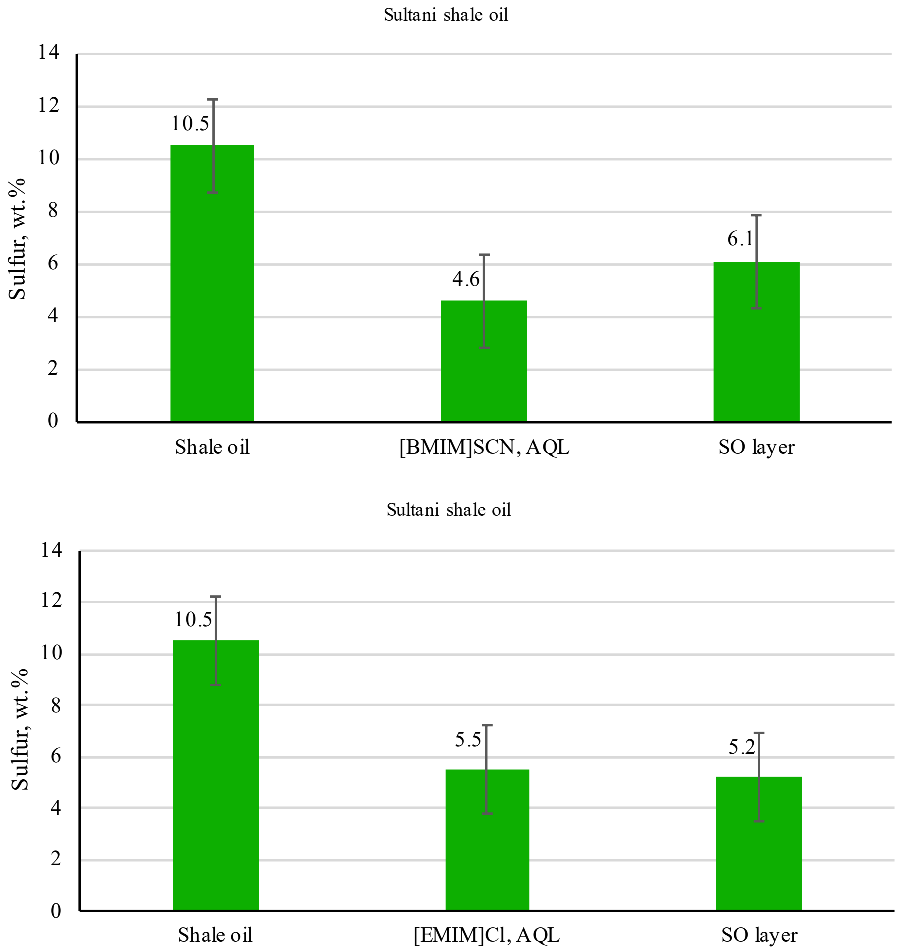

Fig. 1. Sulfur wt.% distribution in [BMIM]SCN aqueous extract (AQL) and shale oil (SO) layer.

[BMIM]SCN AQL, which may lead to an increase in removal efficiency. The unextracted sulfur remained at 6.1 and 5.2 wt.% in the shale oil layer after extraction with [BMIM]SCN and [EMIM]Cl, respectively.

3.2. Attarat shale oil

One gram of shale oil was weighed and mixed with one gram of IL to give a ratio of 1:1. The mixing process was conducted at room temperature. The formed mixture was mixed thoroughly. This part of the experiment was performed for both shale oil samples and both ILs used. The mixture was allowed 10–15 min to separate into two phases. The results of sulfur extraction from the original shale oil and the layers formed after extraction are depicted in Figure 2. The upper graph represents extraction with [BMIM]SCN, and the lower graph corresponds to [EMIM]Cl results.

The results depicted in Figure 2 show that the extracted sulfur wt.% in the [BMIM]SCN AQL was 4.9 wt.% (left graph), whereas the aqueous layer formed with [EMIM]Cl resulted in 5.4 wt.% sulfur extraction (right graph). These values indicate that 52.3 and 58.1 wt.% removal efficiency were achieved by the [BMIM]SCN and [EMIM]Cl ILs, respectively. These calculated efficiencies are higher than the corresponding removal efficiencies calculated for Sultani shale oil. These differences could be attributed to the nature of the sulfur components present in the shale oils. It should also be noted that the sulfur content of the [BMIM]SCN was not accounted for during sulfur weight percent calculations due to the nature of CHNSO analysis. The sulfur remaining in the shale oil-rich phase (SO layer), as shown in the rightmost column in Figure 2, was 4.6 and 4.3 wt.% for [BMIM]SCN and [EMIM]Cl, respectively.

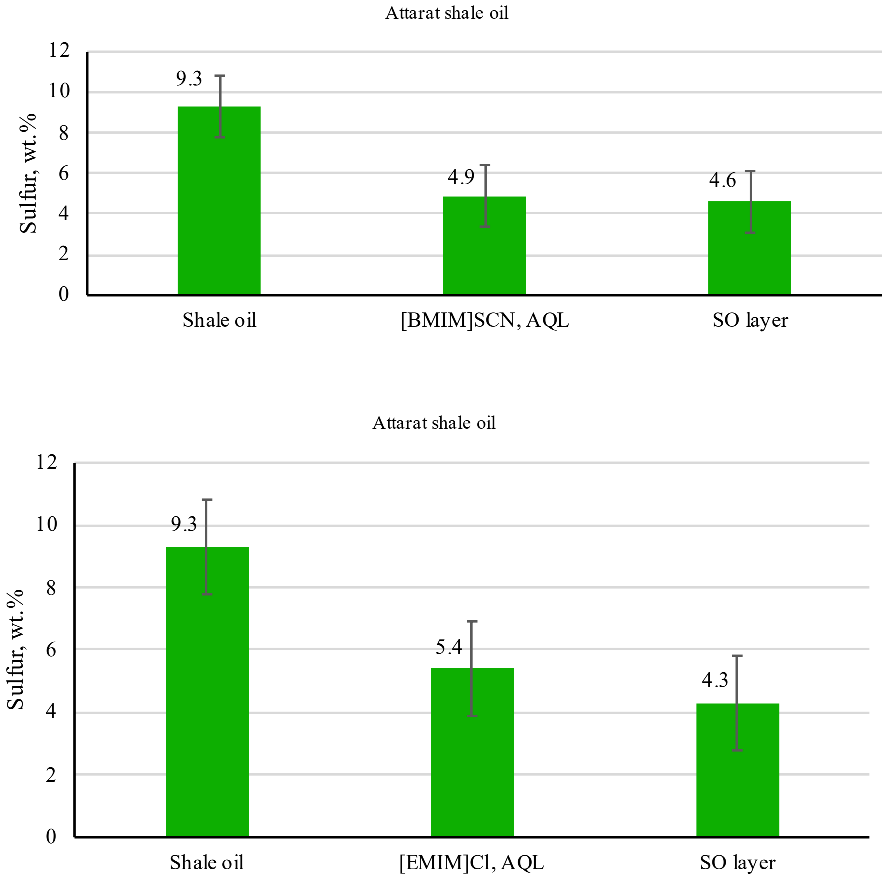

Fig. 2. Sulfur wt.% distribution after treatment with [BMIM]SCN and [EMIM]Cl.

3.3. Surfactants and heating roles

After discussing the findings of mixing ILs with shale oils, the effect of adding surfactants such as Tween-80 (T-80) was investigated. The importance of the surfactant lies in its impact on surface tension forces [26]. Shale oil is more viscous than normal crude oils; unfortunately, no reliable experimental data on viscosity measurements are reported in the literature, and it was not possible to measure viscosity in the present work.

The effect of surfactant addition to Sultani shale oil in the presence of different ILs is depicted by comparing Figures 1 and 3. As can be calculated from both figures, the addition of T-80 in the presence of [BMIM]SCN as an extraction agent increased the removal weight percent from 4.6 wt.% (Fig. 1)

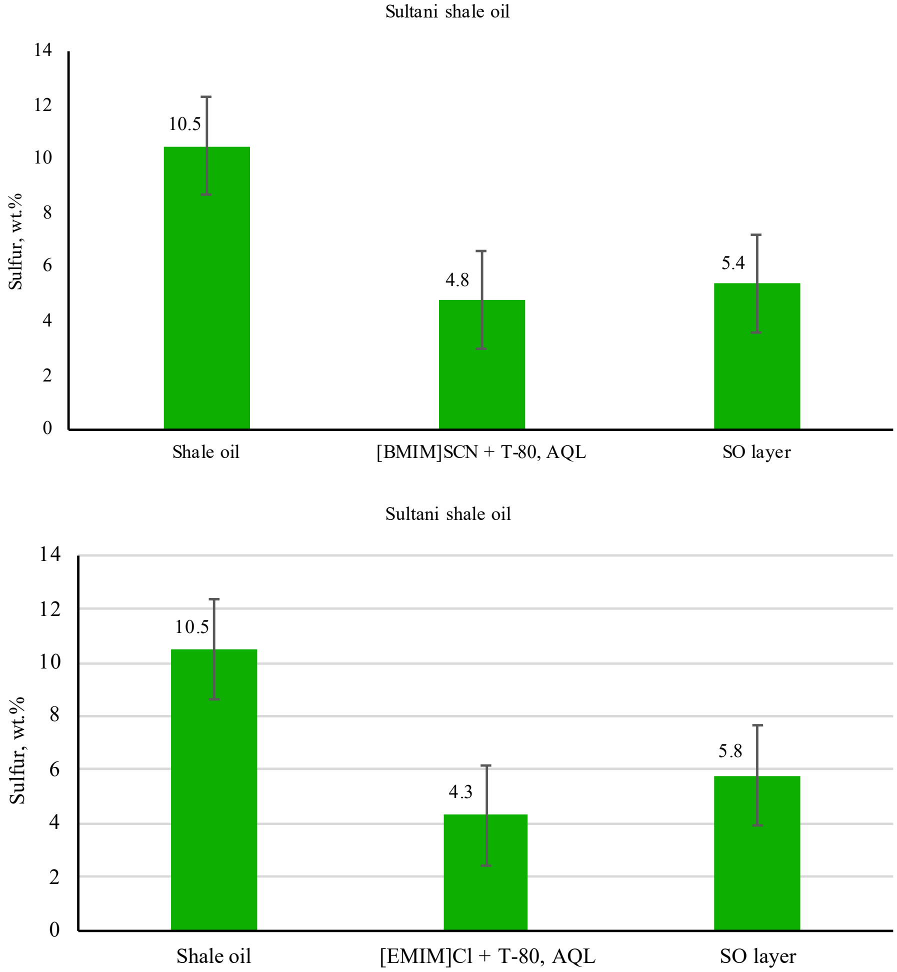

Fig. 3. Sulfur content in AQL and SO layers after adding surfactant T-80 to shale oil and ILs mixture.

to 4.8 wt.% (Fig. 3), i.e., a 4.3% increase in removal efficiency. Similarly, when the surfactant aided the extraction process in the case of [EMIM]Cl, the weight percent of sulfur removal decreased from 5.5 wt.% (Fig. 1) to 4.3 wt.% (Fig. 3), i.e., a 21.8% decrease. This indicates interactions between shale oil, the type of IL, and T-80 during the extraction process. Further investigations are required to explain the nature and type of these interactions.

The interactions of adding T-80 to shale oil and to the shale oil–IL mixture is presented in Figure 4. The T-80 effect was investigated in two cases: in the first case, T-80 was added to shale oil, followed by heating at 60 °C for 2.5 h in a water bath before adding the IL; in the second case, IL was added to shale oil, heated at 60 °C in a water bath for 2.5 h before adding T-80. The first case represents the interactions of T-80 with shale oil alone at 60 °C before IL intervention, while the second case represents the interactions of IL with shale oil at 60 °C before the addition of T-80. It should be noted here that there is a combined effect of heating and extraction at higher temperature compared with room-temperature extraction. Further investigations into these interactions are recommended.

The effect of [EMIM]Cl and T-80 was tested in the present work. As shown in Figure 4, heating shale oil with T-80 at 60 °C before adding [EMIM]Cl to the mixture resulted in 4.3 wt.% out of 10.5 wt.% from the original shale oil (AQL phase), i.e., 40.9% extraction efficiency compared with 5.5 wt.% extraction before heating and without the addition of T-80. This amounts to a reduction of 11.48% in removal efficiency. This behavior is likely due to T-80 being a nonionic surfactant with strong emulsifying and wetting ability. Unfortunately, we cannot ascribe this result to either the effect of temperature or surfactant addition. Similar studies [27] on crude oil and different ILs showed a decrease in extraction efficiency, when the temperature increased from room temperature to 60 °C, from 21% to 15%. These findings clearly support the possibility of decreased extraction efficiency due to increased temperature. This observation is in agreement with the findings of Mohammed et al. [27].

On the other hand, when shale oil and IL were mixed and heated, followed by the addition of T-80, the sulfur removed was 6.1 wt.%, corresponding to 58.1% removal efficiency. This must be compared with the results without heating and without the addition of T-80, in which 5.5 wt.% of sulfur was removed from 10.5 wt.% shale oil, indicating a removal efficiency of 52.38%, i.e., a 5.7% decrease. However, the change in removal efficiency results from the combination of heating, surfactant addition, and extraction at higher temperature. More work is needed to isolate the individual effects of temperature increase and surfactant addition on sulfur removal efficiency. Accordingly, no clear effect can be deduced in this study.

The interaction of surfactants (T-80) indicates direct interactions between IL and sulfur-containing hydrocarbons of shale oil under the influence of temperature. The decrease in extraction percentage in the presence of T-80 could be due to interactions between the IL and the surfactant and/or the effect

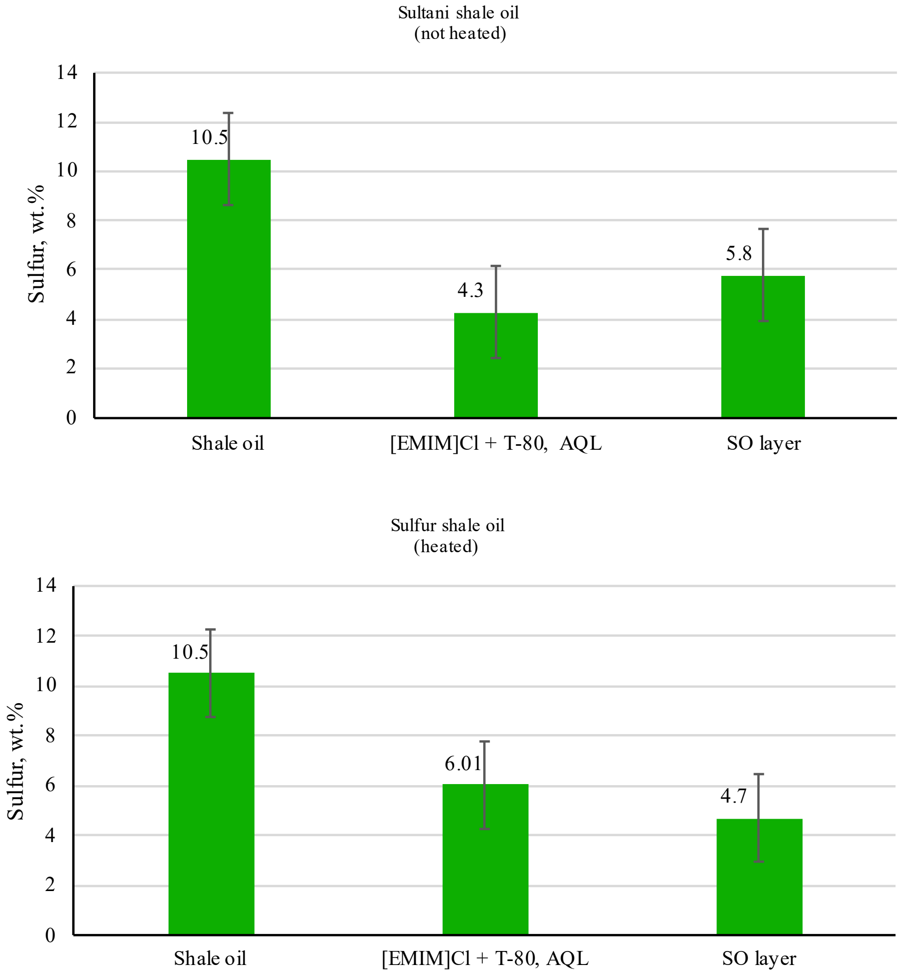

Fig. 4. Effect of heating on sulfur content in AQL with T-80 and mixture of shale oil and ILs.

of increased extraction temperature. Extraction with [EMIM]Cl in the presence of T-80 shows a decrease of sulfur in the AQL phase from 5.5 to 4.3 wt.%, i.e., a 21.8% reduction. This result could be ascribed to interactions between T-80 and [EMIM]Cl at higher temperatures than room temperature. T-80 can hydrogen-bond to Cl^- or coordinate to $EMIM^+$, leading to breaking ion–ion contacts and loosening the ionic network, which lowers viscosity [24] and can improve extraction mechanism and process efficiency, while temperature has the opposite effect [27]. Gao et al. [24] reported that denitrogenation of shale oil decreased with temperatures above 50 °C. This result aligns with the present findings. More work is needed on the effect of temperature on the extraction efficiency, equilibrium values, and the recyclability of ILs.

4. Conclusions

The extraction of sulfur compounds from shale oil is feasible using the two tested ionic liquids. 1-ethyl-3-methylimidazolium chloride ([EMIM]Cl) and 1-butyl-3-methylimidazolium thiocyanate ([BMIM]SCN) were found to be effective in sulfur removal. The extraction ability of [BMIM]SCN is less pronounced than that of [EMIM]Cl. More than 50% removal efficiency can be achieved. Extraction temperature plays a significant role in the extraction process. The Tween-80 surfactant contributes to the extraction, but a thorough investigation is required to understand the mechanism and the different interactions between sulfur components in shale oil, ionic liquids, and surfactants such as Triton-X100. Further research will focus on using other ionic liquids under different operation conditions and evaluating the recyclability of the ionic liquids for extraction.

Data availability statement

Data are contained within the article.

Acknowledgments

The authors gratefully acknowledge the Jordan Atomic Energy Commission, Analytical Chemistry Laboratories, for their assistance with experimental analyses. The publication costs of this article were covered by the Estonian Academy of Sciences.

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