

Technical and scientific review on oil shale upgrading

Abstract

The upgrading of shale oil is one of the new technologies of top industries in the world. The potential scientific and technological treatments of high-grade shale oil production from oil shale is very important in the future industries and is an issue of special interest to refineries. In the present study, we reviewed recent literature on the shale oil upgrading processes. The role of heating rate on the component fraction of shale oil produced was investigated. We determined the influence of supercritical water as a feasible technology that can dissolve polar, semi-polar and non-polar components for the highest rate of light hydrocarbon of shale oil. Ash with transfer heat all over the oil shale and cracking during the retorting process can enhance the quality of shale oil. In the shale oil upgrading treatment, catalyst has a great impact on the final H/C ratio of shale oil and recovered at the end of the retorting process. However, microwave heating is obviously the most successful technique for the upgrading of shale oil implemented to date. Moreover, we studied jet fuel production process from oil shale compared to crude oil. Oil shale has insufficient compounds to produce aviation fuels in which by upgrading process can be produced a high quality of jet fuels.

Keywords: oil shale, upgrading, heating rate, jet fuel, microwave, supercritical

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Ali Shekarifard,^{1,2} Jaber Taheri-Shakib^{1,2}

¹School of Chemical Engineering, University of Tehran, Iran

²Institute of Petroleum Engineering, University of Tehran, Iran

Correspondence: Ali Shekarifard, School of Chemical Engineering, University of Tehran, Iran, Email ashekary@ut.ac.ir

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Introduction

Oil shale is one of the most organic-rich rocks which can be considered as an alternative vast energy resources. Oil shale consisting of more components and materials compared with the true shales. Oil shale Kerogen can be distilled into low to high molecular weight component like sulfur, oxygen and hydrocarbons. The kerogens existing in oil shale are the same type as the organic matter that derived into oil and gas. However, releasing oil shale energy consists as a challenging and not economic in former techniques. Most of Kerogen organic components are insoluble in common solvents, while bitumen can be solved. Oil shale permeability are lower compared with the common hydrocarbon source rocks. Therefore, commercial use can only be achieved by crushing and retorting in surface or in-situ upgrading. Upgrading of oil shale in field application includes in-situ combustion and thermal cracking of organic component.

There are huge oil shale reservoirs in the world. Development of the production technologies of high-quality fuels from oil shale helps economically and technically to provide a confident source of various industries fuels. Oil shale pyrolysis process that requires heating out of contact with air (without oxygen), produces liquid and gas hydrocarbons and ash. The amount, composition and quality of the shale oil generated from oil shale is controlled by pyrolysis conditions. This process may be carried out under prevailing subsurface geological conditions (in situ) or in surface reactors. The difference between surface oil shale operations is based on the percentage yield of gas and shale oil. However, many oil shale in-situ production techniques have some issues which consist of using too much water, environmental effects, inefficient energy balancing and commercial.¹ However, qualification of produced oil shale is another challenge of oil shale industry development.

Recent developments in the oil produced from oil shale with high-quality have been focused on the upgrading techniques. The process used to convert the oil shale in a suitable feedstock called upgrading that uses for the refining industry.

a. The oil shale upgrading process is mainly two methods:²

- i. **Hydrocracking:** Increasing the light compounds by breaking up the large hydrocarbons with adding hydrogen because that increases the proportion of hydrogen in the treatment enabling the formation of newly lighter compounds, e.g., naphtha, heavy gas oil, light gas oil.
- ii. **Thermocracking:** Break up the heavy hydrocarbons using thermal energy (make use of natural gas or coal) to lighter compounds. These processes promote producing undesirable byproducts, e.g., a high concentration of aromatic compounds and coke.

Thermocracking normally yield lower conversions and hydrocracking is more common.

Hydrotreating treatment in the refining industry is one of the most important catalytic processes for decreasing the S, N and O contents in fuels. This treatment is usually the technique of the selection in the fuels production process from alternate hydrocarbon feedstock such as shale oils. The knowledge of the required chemical transformations of the starting molecules is one of the most important factors for extending of catalytic system. Hydrotreating process include the following steps:²

- i. Determination of the various compounds present in oil shale according to their hydrodeoxygenation, hydrodenitrogenation, and hydrodesulfurization reactivities under hydrotreating processing.
- ii. Classification of the least-reactive groups of compounds that cannot be efficiently removed in routine processes.
- iii. Definition of the details of conversion in absence or presence of catalytic.
- iv. Investigation of the latter compounds reactions with catalysts
- v. Definition of the special hydrotreating techniques and catalysts that will produce high quality fuels from oil shale.

- vi. Determining the kinetics of the decomposition of products is important in all of these treatments.

Production of high quality fuels is not the only the purpose in upgrading process, but also is important to maximize shale oil because gas and ash are additionally produced in this process. Several studies have been performed to study the retorting temperature, minerals and particle size on the shale oil yields by using a pyrolysis reactor with a thermogravimetric analyzer.³⁻¹⁰ Willimas et al.¹¹ studied the effects of pyrolysis conditions change on the shale oil properties. Because the properties of product shale oil are inferior to that of light crude oil from a retorting process, additional upgrading treatment is needed.¹¹ Therefore, quality and yield of shale oil should be considered in order to be used as a substitutive crude oil.

In the present study, we investigated upgrading processes of oil shale, which include the effects of retorting temperature rate, supercritical technology, co-pyrolysis processes, catalysts, microwave heating process. Besides, the production of jet fuel technology were investigated and the comparison between shale oil and crude oil process.

Temperature increment effect

One of the most common and efficient techniques in increasing the quality of light hydrocarbon in the shale oil is the thermal heating rate. Early researches indicate that determination of the shale oil temperature increment highly influences the quality and conversion factor of light hydrocarbons. The upgrading experimental results reveal that the fractional composition of extracted oil from oil shale is directly related to the heating rate. Current studies represent that a maximum heating rate which beyond of this value, rapid heating leads to nonqualified shale oil for marketing purpose. Several authors investigated the influence of heating rate on the final composition of cracked crude oil.¹² Conducted a pyrolysis experiment with an oil shale sample in a cylindrical retort chamber by electrical heating for increasing the uniform thermal conduction. Sweeping the generated products was implemented for accurate composition determination by the nitrogen gas. In their experiment, the heating rate can be controlled by the power supply. Condensation system was adjusted at 2°C in order to collect condensable hydrocarbon and release the non-condensable to the atmosphere. Digital weight recording was introduced to measure the retort time and temperature at every increment. The final hydrocarbon component in the upgraded sample was determined at the 550°C with X-ray spectroscopy. Then, treatment process after the upgrading the sample was applied with n-hexane for separation of soluble and insoluble phase. The soluble phase (multenes) was introduced to the chromatograph for further component evaluation. The aliphatic component which extracted from the soluble phase is examined by the gas chromatography for determination of n-paraffin content. The organic component of the shale oil undergo thermal cracking with the temperature increment ranging from 0.3 to 6 °C min⁻¹. From the experimental results, it is concluded that the type and conversion factor of light hydrocarbon is related to the heating rate. However, the H/C ratio has not a direct proportion with the heating rate value. The asphaltene fraction has a slight rising tendency with increasing the temperature increment value. Nevertheless, the paraffin fraction has not followed an increasing trend by decreasing the heating rate which means that at the specific heating rate value, we can obtain the maximum light component. Other authors performed the same studies on that by developing the heating rate into extent range. It can

be concluded that the final hydrocarbon fraction is governed by the peak and increment value of temperature.^{13,14}

Supercritical fluid organic solubility power

In upgrading shale oil, two main types of procedure are followed, hydrotreating and carbon removal. Many of studies on upgrading focused on the extraction and enhancing shale oil by using supercritical fluid. Supercritical termed as the fluid above the critical temperature and pressure values which has a special feature. It is evident that for development and increasing the quality of shale oil conversion factor, supercritical fluid such as water can be considered as an efficient organic compound solvent. In addition to this fact, supercritical sweeping raises the marketing quality and not consists of the catalytic and hydrotreating issues. High-density water in supercritical condition has a non-polar characteristic which can be taken into account as a favorable reactive fluid with high solubility power. Moreover, critical fluid has a capability of invasion through small pores because of its non-surface tensioning features. In sweeping process of light hydrocarbon by the supercritical water, cycle stream of soluble through the shale oil can extract low weight molecular hydrocarbon to the condensation system. Regarding this, upgrading shale oil carried out by researchers using supercritical water. Based on the experimental results, water acts as an acid-catalyst at the supercritical condition. Current studies represent that critical water is neutral but above experimental expectation can behave like an acid-catalyst which leads to improving the reaction yield.^{15,16} Temperature conversely correlates with the dielectric property, so that in critical condition, the fluid has the minimum polarity. Decreasing the polarity of water has a drastic effect on the organic solubility feature. However, unlike organic solubility, polar composition solubility power decline in the critical temperature water. At critical temperature, the phase boundaries between organic compound and water diminish which lead to increasing the miscibility of this two phase. In order to sweep higher molecular weight hydrocarbon with super-critical fluid, we have to increase the concentration of H_3O^+ in the Supercritical water. Recent studies indicate that approaching the supercritical water to the bitumen boiling point can assist recovering almost all amounts of this faction. Some authors determined that upgrading of shale oil and extracting light hydrocarbon from oil shale with a mixture of water and same type hydrocarbon can enhance the final conversion products factor. A comparison between extraction with nitrogen gas and supercritical water indicate that supercritical-treating convert dramatically higher rate of asphaltene to multenes. In both of nitrogen and water cycle upgrading, the temperature is the main factor which controls the improvement of reaction. Shale oil upgrading in supercritical water treatment occurs in lower temperature compared with the retorting process.

Solid heat carrier

Besides common retorting shale oil method, co-pyrolysis technique has been proposed by many authors to increase the final yield rate of light hydrocarbon conversion. One of the earliest methods which can be considered is catalytic process by augmenting the oil shale ash to the retort shale oil in order to intensify cracking and upgrading the quality of converted oil shale. Fundamental studies indicate that the possible mechanism of increasing shale oil conversion factor is heat carrier by solid phase and catalytic property. Many studies conducted to oil shale-ash mixture experiment in retorting reactor to

determine the influence of solid particle of ash on the improvement of upgrading process. Pyrolyzed oil shale by augmenting shale oil ash and investigated changing in light hydrocarbon fraction that attributed to the catalytic reactions.¹⁷ Ash-shale oil mixture lab-scale carried out in various shale/ash ratios in order to identify the optimal scale. In the pyrolysis of shale oil with ash, the composition and fraction of upgraded oil shale highly depend on the fixed heating rate, final retort temperature, processing condition of ash catalyst and type of oil shale Kerogen. The overall experimental result shows that catalytically impact of ash both can increase the fraction of light hydrocarbon and also decrease the remained waste component. In this type of ash utilization experiment, the ash particle size is so smaller than the oil shale particles which can enhance the composition of pyrolytic hydrocarbons. The mentioned oil shale ash is obtained from combustion of shale oil at the 850°C temperature and crushed into 0.2 mm. The mixing ratio of ash-oil shale has a great impact on the heating transferring by the solid particle. These co-pyrolysis experiments are the same as common pyrolysis. However, using an intensifying material dramatically enhances the final conversion rate. The co-pyrolysis overall instruction was similar to the common pyrolysis technique. Nevertheless, there is an important technical difference. In co-pyrolysis method, the mixture of ash-oil shale retort in a cylindrical steel stand and increase the average temperature up to the 500°C by electrical heater. Introducing pure non-condensable gas such as argon or nitrogen to the system as a carrier gas can be varied in flow rate at every experiment based on the type of the conversion rate. The volatile and evaporations of shale oil pass into the condenser and non-condensable gases vented through the gas analyzer. Analyzing oil-ash components indicate that they mainly consist of calcium oxide, calcium silicate and aluminum oxide. Based on the results of gas and shale-oil yield evaluation, the rate of conversion is highly related to the primary mixing ratio. The results from final upgraded composition respect the ash/shale ratio reveal that increasing the shale oil from 0:1 to 1:2 range with near to direct proportion. Beyond 1:2 ratio, an opposite trend in both shale oil and gas can be understood. Evaluation of gases at every mixing ratio reveals that above 1:2 portion, the amount of gas mass percent increases extraordinary. The amount of released gas through co-pyrolysis retorting below 1:2 mixing ratio is inversely proportional to the relative ash mass percentage. Mixing oil shale with ash can lead to uniform heating because of filling the void space among oil shale grain. Further pyrolysed oil shale analyzing shows that increasing ash ratio in mixture causes a barrier for escaping the shale oil from the grain element by two mechanism of adsorption and occupying void space. Adhering shale oil to the ash particle surface during upgrading time leads to the cracking reaction and increase the gas yield.¹⁸

Upgrading activator

Adding hydrogen or carbon rejection has generally prevailed until now for enhancing the quality of retorted shale oil. However, using pyrolysis method with contribution of actuator and catalyst is more effective compared with the common hydro treatment process. The main advantages of catalytic contribution in co-pyrolysis process are percentage of conversion, quality of products and reducing residues. The improvement in quality of shale oil during upgrading process can be obtained by augmenting catalyst to the retorting reactor. In catalytic cracking and treating, increasing the light shale oil fraction can be obtained at lower temperature compared to the common retorting process. Many studies investigated the effect of non-acidic and acidic catalyst on the conversion rate of high molecular weight

hydrocarbons. Moreover, recent studies also confirm the contribution of catalyst for removing contaminants from upgraded shale oil. However, catalyst utilization can be applied by all various type of upgrading process such as supercritical water treatment, ash pyrolysis and common retorting reactor. Studied the effect of three types of catalyst on the yield and properties of shale oil during hydrotreating and hydrocracking process. Ni, Co and Mo are introduced to the hydroprocessing technique as catalyst.¹⁹ In hydroprocessing procedure, the cracking and upgrading catalyst augmented to the system after hydro purification step in order to prepare the shale oil for hydrocracking. Hydrotreating reactor was utilized by electrical thermocouple and heater in order to attain constant temperature. Near to 50 g of shale oil and 10% catalyst was placed in the reactor with the exertion of 7.5 MPa hydrogen pressure. By increasing the temperature to 420°C for next to 240 min, the reaction can be observed. Every catalyst has been performed in near to 3 different heating time in order to specify the optimum duration. After the reaction time, the upgraded product was separated from the solid phase by filtration (catalyst). The residuum was dried and weighted for determination of upgrading process yield. Finally, all the converted products such as phenols, asphaltenes and other chemical group are analyzed by the chromatograph. By quality and quantity calculation of upgraded hydrogenated shale oil and gas with respect to the catalyst and heating duration, the researchers can determine the most optimum values. The result of chromatography represents that the universal catalysts DN-3100 Th and GO-30-7 with 60-minute heat exposure duration can have the maximum effect on the quality of hydro-upgraded shale oil and also increasing the hydrogenated shale oil percentage to 93.8. The FTIR spectra indicated that, during catalytic hydro-treating process, the carbonyl and hydroxyl groups converted into methylene groups which lead to increasing the H/C ratio near to 1.06 times higher compare with the initial shale oil.^{20,21}

Jet fuel

Complex combination of treatments takes place in refineries, converting the petroleum feedstock into jet fuels which requires higher energy contents. Currently, the jet fuels are mainly produced from refining of crude oil distillation for the purpose of aviation fuels (e.g. powering aircrafts) which is suitable for most turbine engines.²² The jet fuels are also suitable for use in both low and high pressure and temperature engines such as large piston engines.²³ Jet fuels are kerosene type fuels that being used in turbine engines aircraft and another grade of jet fuels are a wide cut kerosene (a blend of gasoline and kerosene) but it is rarely used except in very cold climates. This type of jet fuels has a flash point above 38°C and a freeze point maximum of -47°C.²⁴⁻²⁶ Therefore, jet fuels must be the quality and specification properties such as: high calorific efficiency, low viscosity in low temperature conditions, ability to burn without danger and cleanly and chemical stability. Main components of Jet fuels are composed of:²⁷⁻³⁰

1. Saturated straight and cyclic alkanes (>85%)
2. Aromatics especially benzene
3. Olefins (Low concentrations: <1%).

The straight paraffin compounds are required in large quantities, because boost to higher production of heat per unit mass due to high ratio of H/C compared to the other hydrocarbons. Therefore, these components of jet fuels are capable of burning cleanly. The cyclic

paraffin compounds are characterized in the jet fuels by reduced freezing points and suitable density. The low concentration of aromatics and olefins compositions are important because they are high reactivity and produce rapid smoke.

Oil shale compared to the conventional crude oil is a more appropriate choice in the jet fuels production. These compositions and features of aviation fuels with light upgrading of oil shale can be converted to a high yield of superior quality jet fuel.³¹ Shale oils compared to the conventional crude oil generally contains:³²⁻³⁴

- i. low naphtha and residue fractions
- ii. Large amounts of hydrogen
- iii. High paraffinic components
- iv. Large portion of kerosene

Also the portion of distillates in shale oils is relatively large. Therefore, it is suitable to be used for jet fuel. studied the nitrogen and quinolines compositions of the jet fuel cuts of Occidental shale oil compared to the heavy gas-oil cuts.³⁵ They show that heavy shale oil fractions by special upgrading process can be the best selection for production of jet fuels. Several studies have been performed to investigate the application of oil shale to jet fuel production that show aviation fuels refined from oil shale resource have the highest n-alkane content.³⁶⁻³⁸

Now, shale oil is a conventional resource of aviation fuels that would be fully compatible with current aviation fuels systems.³⁹ Technical treatments of jet fuel production by oil shale:⁴⁰⁻⁴³

1. Grinding the large amounts of oil shale down to a suitable particle size
2. Retorting process for separating the shale oil or extract kerogen from oil shale by a high temperature
3. Production of oil shale: maximum oil yield by inputs of significant energy
4. Upgrading processes:^{44,45}
 - I. Hydroprocessing: primary upgrading the shale oil to different fuels
 - II. Supercritical treatment: based on the use of solvents under supercritical conditions (methanol toluene and water can be used as a solvent in this new technology)
 - III. Special additives as follows:
 - i. Tetra-ethyl lead: reduce the tendency of jet fuels to detonate.
 - ii. Anti-oxidants: inhibit and prevent the forming of peroxide and gum deposits compositions in jet fuels system.
 - iii. Static dissipater: lowering the dangerous effects of static electricity in high flow rate fuel transfer systems.
 - iv. Anti-icing: prevent the formation of ice crystals and sometimes used to combat microbiological growths at high altitudes.
 - v. Corrosion inhibitors: protect ferrous metals and improve the lubricating properties of aviation fuel system.
 - vi. Thermal Stability Improver: in the high temperature areas

prevent the deposit formation in the aircraft fuel system.

- vii. Metal de-activators: in fuel oxidation suppress copper by catalytic effect.

Relationship of products with upgrading methods

As each upgrading technique has its own advantageous with respect to the preferable final products, thus it is necessary to compare these methods based on the enhanced components. As mentioned before many methods have been investigated in order to upgrade the shale oil products based on their component properties. However, from the final upgraded components ratio of oil shale, each method can yield different efficiency. In microwave heating upgrading technique, the final upgraded shale oil products are produced according to their dielectric property.⁴⁶ So, extracting the upgraded shale oil conforming to the polarity and dielectric property of component can provide a broad range of products during the exposure time. In electromagnetic wave technique, the component molecule with a higher dielectric property will be extracted at the earlier treatment time. Supercritical final products have similarly enhanced products at every stage of treatment compared with the microwave heating technique. However, the percentage of pyrolysed nonpolar component is higher than electromagnetic upgrading method due to high solubility power of supercritical fluid at every stage of processing. In addition, in microwave heating method, the final products have higher paraffin component ratio compared with the upgraded products from other relevant pyrolysis methods. Therefore, in demanding for higher paraffin content products, using microwave upgrading technique can be preferable respect to the others. Oil shale products are naphtha, diesel and jet fuel which can be extracted from the ratio of polar and non-polar composition regarding the mentioned upgrading methods. Naphtha is one of the most achieving products of oil shale upgrading technique which can be extracted at the earliest stage of retorting because of its low boiling point. Naphtha mainly contains paraffin and aromatic which has almost a non-polar behavior. One of the best methods for extracting naphtha from the treated oil shale is using of supercritical fluid in order to dissolve non-polar component with the polar.⁴⁷ However, another advantageous of supercritical water for enhancing the quality of naphtha (light hydrocarbon) is its low ability in the extraction of high molecular weight hydrocarbon during the upgrading process. However, the most efficient method for recovering high molecular weight hydrocarbon products such as heavy and vacuum gas oil from the shale oil is conventional pyrolysis method by combining with shale oil ash for increasing the overall heat transfer. Nevertheless, the combination of any mentioned technique with microwave heating can increase the final yield percentage and reduce the amount of residue in conventional or catalytic pyrolysis.⁴⁸

Conclusion

Main conclusions for this study are summarized following:

- i. The magnitude of temperature increment has a great impact on the final quality of retorted shale oil. Determination of heating rate effect can be evaluated based on the upgraded shale oil and light component fraction. The maximum conversion factor has a near direct relation with a low heating rate. Increasing the heating rate to the maximum value can increase the possibility of residue generation, however, very low-temperature increment

cannot guarantee yielding higher H/C ratio composition.

- ii. Supercritical fluid treating can be considered as a commercially feasible technology in enhancing the quality of shale oil. Polar properties of water can be diminished by increasing the pressure and temperature which turn into a super solvent. Supercritical water has special features to dissolve polar, semi-polar and non-polar component. Supercritical water extraction of organic compound and solubility sweeping power can yield the highest rate of light hydrocarbon.
- iii. Co-pyrolysis process is similar to the common pyrolysis methods with a higher yield of light hydrocarbons. Ash can contribute into common retorting process by two main mechanism heat carrier and catalytic effect. Ash can act as a solid heat carrier which can transfer heat all over the oil shale sample. In addition, ash can intensify the cracking and treatment reaction during retorting process. However, the ash-shale oil mixture ratio has to be specified in order to have maximum upgraded shale oil and minimum gas.
- iv. Catalysts have undeniable influences on the improvement of reactions. Analysis catalytic retorting methods indicated that augmenting catalyst can have a great impact on the final H/C ratio of shale oil. In addition to the mentioned beneficent effect of catalyst, they can be recovered at the end of the retorting process which can increase their demanding. Moreover, the catalyst can have a great impact on the contaminant reduction of upgraded shale oil.
- v. Using microwave heating in oil shale pyrolysis method can have a great deal in economic attention. Microwave heating has been proposed as a good heat exposure because of their feasibility, uniform heating and intensify reaction effect. The Combination of ordinary pyrolysis method with electromagnetic wave heating technology can increase the yield and quality of upgraded products.
- vi. The shale oil conventional treatment is the dominant technology for the production of jet fuels in the developed aviation industries. Jet fuel production from the upgrading of oil shale has a high quality and best compatible with current aviation fuels systems compared to the crude oil-based conventional process.

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Conflict of interest

The author declares no conflict of interest.

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