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Solar pyrolysis of oil shale samples under different operating conditions

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Abstract. *The main objective of this experimental work is to study oil shale pyrolysis by direct heating of solar energy, using a simple concentrated solar system, and a thermogravimetric analyzer (TGA). The tested sample was obtained from a local oil shale deposit, Ellujjun, in Jordan. The TGA test results confirmed that the involved reactions depended on final reactor temperature: the higher the temperature, the greater the weight loss in the sample. A series of experiments using a new design of fixed bed retort powered by solar energy were carried out to study the influence of various operating parameters such as environment inside the reactor and final temperature on the pyrolysis process. The magnitude of the total yield was mainly dependent on temperature and the medium inside the retort. The highest oil yield was witnessed when air was used as gas in the retort, while in subsequent experiments using kerosene the oil yield was much lower. However, this was almost nil in case of using water in the retort. This is the first research of its kind in the Middle East and North Africa (MENA) region, utilizing a solar parabolic dish reflector to heat up the reactor and is deemed to open the way in the future for more detailed research in the field of solar oil shale retorting and/or gasification.*

Keywords: *oil shale pyrolysis, thermogravimetry, Jordan, concentrated solar system, solar dish.*

1. Introduction

1.1. The problem

Jordan is a small country, situated in the most volatile region, the Middle East, in the north-western corner of Asia. Its area is about 90×10^3 km^2, of which more than 80% is as desert with very limited conventional energy and water sources. Its population, almost ten million, including refugees and forging workers from neighboring countries, exerts ever-increasing pressure on scarce resources. According to recent official statistics, nearly 70% is under 34 years of age, while those between the ages of 15 and 24 make up 20% of the population

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[1]. The latter group is mainly comprised of high school and university students who are net consumers of food, water and energy. From the economic point of view, it is a middle-income and open economy country, with very limited natural resources and slow trade flow with neighboring countries due to political instability in these countries. In 2017, most of the consumed energy (i.e. 94%) was imported at a cost of US$ 3.5 billion, approximately 9% of the GDP in that year [2]. Such a high cost of imported primary energy coupled with the lack of resources such as surface water caused double knocking effects on the economy: a very high cost of energy required to support the national economic and social development, as well as a continuous drain of much-needed hard currency. On the other side, there are vast reserves of oil shale (more than 70 billion tons) near the surface in the central region of the country in addition to high solar intensity (on average 5–7 kWh/m^2/day) as well as very good wind regimes in certain locations [3]. Thus, developing such indigenous energy sources would reduce Jordan's dependence on imports and save required hard currency. This research is an attempt to help in developing a safe, low cost and clean method for processing oil shale to yield liquid and gaseous products. It is a new approach and the first of its kind in the Middle East and North Africa (MENA) region and the oil shale industry as a whole. In the following sections the literature review is presented, followed by the characterization of samples and description of the experimental procedure, then the results are analyzed and finally conclusions and recommendations are provided.

1.2. Oil shale in Jordan

The history of oil shale in Jordan is a long story: it started a few thousand years ago when Prophet Moses (peace of God upon him) used the rock to make fire on his trip to the Holy Land. In the early 20th century, during the First World War, it was employed by the Ottomans to fire the steam locomotives of the Hijazi Railway [4, 5]. Oil shale activities carried out in Jordan during the past decade are considered as the most energetic worldwide. Many conferences were organized, and specialized companies and investors signed memoranda of understanding (MoU) and concession agreements (CA) with the Government of Jordan (GoJ), represented by the Natural Resources Authority and later the Ministry of Energy and Mineral Resources. However, few companies have managed to successfully complete the desired feasibility studies as required by GoJ, and signed a CA which is considered a special law by the Jordanian Constitution. These companies are:

- Royal Dutch Shell signed a CA in 2009 to develop an in-situ conversion process (ICP) for deep oil shale deposits to produce light oil.
- Eesti Energia Company, which is fully owned by the Estonian government, signed a CA in 2010 to establish a retorting scheme in Attarat Umm Gudran area, in Central Jordan, using the Enefit 280 surface retorting technology which is based on solid heat carrier technology, Galoter process.

- Karak International Oil (KIO) signed a CA in 2011 to implement a surface retorting project in Ellajjun area, employing the Alberta Taciuk Process (ATP) developed in Canada to process tar sand.
- The Saudi Arabian Company for Oil Shale (SACOS) signed a CA in 2014 to implement a surface retorting project in Attarat Umm Gudran area, employing the Russian UTT-3000 technology which is based on solid heat carrier process.

The Estonian-Malaysian-Chinese consortium signed the relevant agreements with GoJ in 2014 to build and operate a new power plant based on direct combustion of oil shale, using fluidized bed technology. However, the financial close was completed and signed in March 2017 with a group of Chinese banks. The 470 MW power plant would cost approximately US$ 2.1 billion [6]. The signed power purchase agreement with the state-owned utility, National Electric Power Company, defined the unit price delivered to the grid at an average cost (levelized tariff) of US$ 0.134 kWh for the first 18 years and then it will be reduced by 30% [7]. It is expected that the project will be completed and become commercially available online by the end of 2020. This is the only oil shale project that is under construction in Jordan.

Unfortunately, all other oil shale projects aiming to retort oil shale are almost stagnant. Shell, after the completion of its exploration program, which included drilling 300 deep wells and carrying out an in-situ oil shale heating field experiment, stopped all activities related to oil shale development in 2016. The decision to stop the development program in Jordan was due to the disappointing results of the field experiments [2]. However, Shell has decided to study the outcomes of field experiments and develop its ICP technology accordingly before entering into the next phase.

Other companies slowed down their schemes in Jordan and the current situation is almost stagnant [8]. This could be due to low oil prices in the international market and securing the huge capital investment required for such developments at current prevailing national and international financial conditions. Equally important are the involved technical and environmental risks which still need more hard work in the near future to overcome. GoJ's oil shale policy regarding helping these companies to surmount their predicaments is not a conducive and proactive one. Without real governmental support to these projects and research and development activities in this field, there is little hope to develop such indigenous huge energy resource.

1.3. Literature review

Oil shale rocks are mainly limestone and marls in which the organic matter is of nearly non-homogeneous nature and finely dispersed in the rock. It is classified among the group of fossil fuels, but it is characterized by its high ash content. The most popular definition of oil shale from the engineering point of view is a fine-grained marine sedimentary rock that contains a complex organic matter called kerogen, whose long chain is broken upon heating, in the absence of oxygen, yielding liquid and gaseous fuels [9].

Until the late 1970s, there was little interest to develop Jordanian oil shale resources, however, during the past three decades, some interest was witnessed especially among researchers to study and investigate such resource. In the year 2006, the situation changed dramatically and there was serious intention of GoJ towards development and utilization of indigenous oil shale resources [10]. Such sudden change could be attributed to many factors: the most important are (i) high bill of imported energy, (ii) increasing national demand, (iii) security of energy supplies into the country and (iv) the increased academic interest worldwide. Historically, on many occasions, the Kingdom has experienced tight and tough conditions due to shortage of supply such as in 2003 as a result of US invasion to Iraq, and the political crisis in Egypt in 2010–2011. Thus, GoJ was forced to look for new sources to import crude oil, petroleum products and gas even at higher costs [3, 9, 11].

Many Jordanian researchers studied the behavior of oil shale pyrolysis under different conditions [3, 9, 12–16]. The characteristics of the produced shale oil were analyzed by Akash and Jaber [17]. The gasification of oil shale, from different deposits in Jordan, was investigated by Sladek and Jaber [16], Jaber et al. [18], Jaber [19] and Jaber et al. [20]. On the international level, a large number of researchers have studied the influence of pyrolysis temperature and heating rate on oil shale decomposition under isothermal and non-isothermal conditions [21–35]. However, only few researchers thought about employing solar energy to directly heat up the retort and pyrolyze/gasify oil shale sample [36]. Thus, there is a dearth of information about using such a clean and free source of energy to convert raw oil shale into liquid and/or gaseous fuel. The present investigation is an experimental study using a concentrated solar power (CSP) system that will collect solar rays and focus them into the reactor, i.e. oil shale retort. This is a new approach and the first of its kind in the MENA region and the world as a whole in the field of oil shale processing. The main objective of this study is to assess the possibility of processing oil shale by using only solar radiation at local prevailing conditions in Jordan. It is also intended to correlate the final yield, i.e. output, under different operating conditions in terms of environment inside the retort and its temperature. At the same time, it should be stressed that geological and mining issues of oil shale are beyond the scope of the current study.

2. Experimental equipment and procedure

2.1. Type of oil shale

The tested oil shale samples were obtained from the Ellujjun deposit in the central part of Jordan, near the Karak city. The raw oil shale samples were collected from the open mine and kept in air-tight plastic bags. Details about the samples and their elemental, i.e. ultimate, analyses are given in Table 1 below. It is worth noting that the ash content of oil shale is about 61% by wt. More details about the oil shale deposit can be found elsewhere [37]. The received sample was mixed and milled, then sieved to variant grain size with a mean diameter range of from 100 to 250 μm.

Table 1. Elemental analysis of oil shale sample

Element	Mass, %	Mole, %
C	48.99	4.08
H	5.04	5.00
O	36.82	2.30
N	0.99	0.07
S	8.14	0.25

2.2. Gross calorific value and total carbon

The gross calorific value (GCV) or higher heating value of fuel sample is defined as the amount of heat released when the fuel is completely burned under controlled conditions. The adopted testing procedure is illustrated in the standard ASTM E711-87 [38], and the used device is a Bomb Calorimeter (Parr 1351). The first step in these experiments started with weighing one gram of oil shale, followed by placing it in the crucible that was put in the calorimeter vessel. The latter was fully submerged in an insulated water jacket. By measuring the temperature rise in the water jacket and establishing the energy balance, the heat of combustion was calculated, which equaled the GCV of the sample.

The total carbon content (TOC) of the oil shale sample was determined using an ACE 1100 Elemental Analyzer which determines the carbon isotope composition. The oil shale sample was homogenized after being pulverized, then decarbonized by the carbon isotope analyzer. This elemental analyzer was coupled online via a Thermo-Fisher Delta plus mass spectrometer at the Isotope Laboratory in the Institute of Geological Sciences, University of Erlangen-Nürnberg, Germany. The experiments were performed at high temperature, above 1800 °C. Thus, the tested sample of oil shale was converted into a combination of different gases. The results can be expressed as percentage values versus a standard reference. The accuracy of the analyses was checked by repeating the analyses of the international standard USGS 40. This is a reference material with known ^{2}H, ^{13}C, ^{15}N and ^{18}O isotopic composition and was prepared by Qi et al. [38]. The relative error of this method is small and calculated around 0.49% as shown in Table 2.

Table 2. Total carbon content determined according to the international standard USGS 40

USGS 40	1	2	3	4	5	6	Average	Relative error, %
Total carbon content, wt%	40.9	40.6	40.8	40.7	40.6	40.6	40.7	0.49

2.3. Thermogravimetric analysis

This analytical technique monitors the mass of the sample that is exposed to a controlled temperature program. It is rapid because of the small size of the sample and the high heating rates. The non-isothermal tests with oil shale samples were carried out using a Shimadzu Model-50 Series Thermogravimetric Analyzer (TGA), with N_2, at a constant rate, used as purging gas. In the present investigation, one gram of oil shale was placed inside the TGA furnace and the recorded data were used to determine the effect of temperature (up to 1000 °C), at a constant heating rate of 15 °C/min, on the weight loss and reactivity of the oil shale sample. The pre-programmed control unit regulates all the automatic functions of the recorder. Finally, after the furnace temperature had achieved its set value, the sample was allowed to cool to room temperature.

2.4. Concentrated solar power and oil shale reactor

As stated previously, the main objective of this research is to design and study a new and sustainable method to extract shale oil from raw oil shale, by heating the studied sample inside a solar reactor or gasification bed. To achieve this objective, the first step was designing and constructing a suitable solar test rig that would work under prevailing conditions in Jordan.

The needed thermal energy to heat the reactor and the oil shale sample was supplied by a special parabolic dish reflector (PDR) combined with a single-axis tracking system as shown in Figure 1a. The parabolic dish, with a diameter D = 2 m, was designed and constructed, in the local market, to work as solar collector. This solar dish was painted with two layers of Zinc Phosphate Primer coating to provide minimum protection from environmental conditions. Then small mirror pieces, about 1500, each approximately 5 cm × 5 cm, were glued on the inner surface of the solar dish, while maintaining the dish curvature. Furthermore, to increase the amount of reflected solar energy and reach the desired temperature, the seven small parabolic solar dishes, with a diameter of d = D/3, were fixed around the main parabolic dish reflector (see Fig. 1b–c).

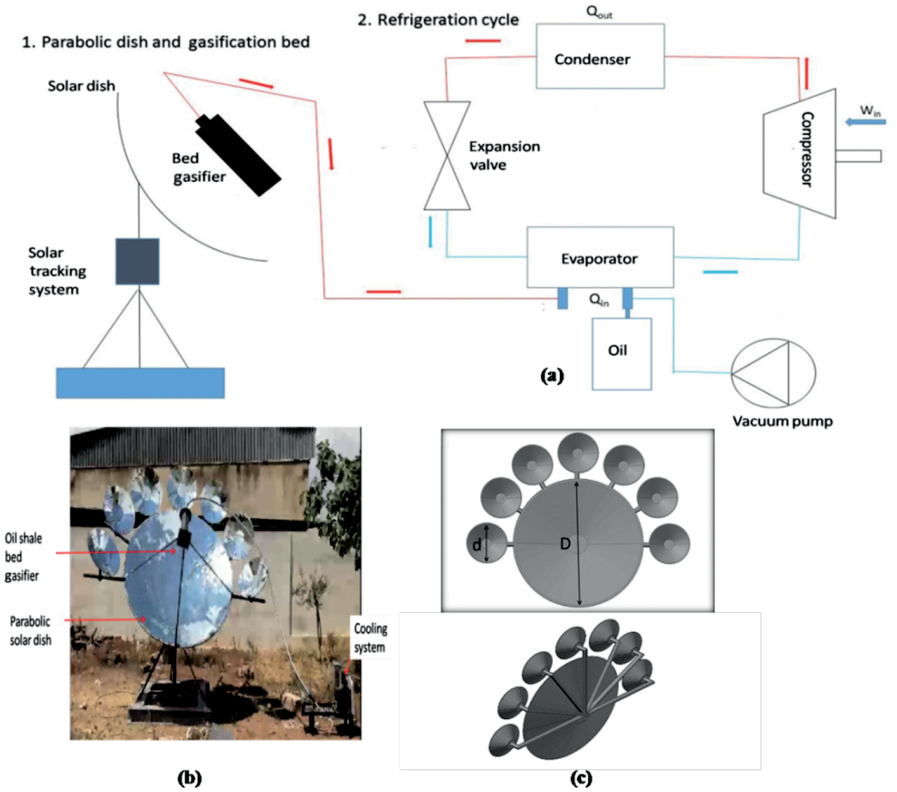

Fig. 1. Testing rig: (a) schematic diagram, (b) experimental setup, (c) design of the parabolic solar dish surrounded by seven small dishes.

As shown in Figure 2, the focal point of the parabolic solar dish was determined by the following equation [39]:

$$F = D^2 /(16*a), \tag{1}$$

where D is the dish diameter, a is the depth of the dish and F is the focal distance (see Fig. 2). In this case for $D = 2$ m and $a = 0.25$ m, the focal distance becomes 1 m. The seven small parabolic dishes were installed around the main dish and adjusted to make one common focal point at the main dish, thus increasing the amount of collected solar energy, and, consequently, enhancing the heat rate into the reactor.

The oil shale fixed bed reactor is made of seamless carbon steel pipe, with an outside diameter of 114 mm, length of about 400 mm and wall thickness of 6 mm. It is painted with black coating and fixed in the focus point to absorb most of the collected and reflected solar rays. The solar system is supported by a stand, which is fixed on a square base plate (200 mm × 200 mm) and

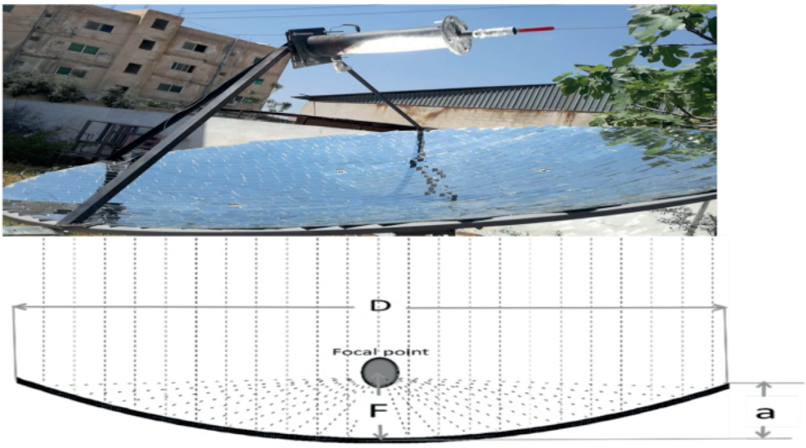

Fig. 2. The reactor placed on the focal point of the parabolic solar dish.

permanently stationed into the ground. The outlet of the reactor, from which the off-gases and vapor come out, is connected to a heat exchanger that is cooled by a small chiller unit with R-134a used as refrigerant fluid. The vapor produced as a result of kerogen changes due to intensive reactions inside the reactor will pass through the heat exchanger and be cooled down by chilled water. The condensed fraction represents the produced shale oil, i.e. liquid fuel, while the other fraction forms non-condensable gases with different chemical composition such as CO, H_2 and C_xH_y known as off-gases.

Fig. 3. Solar single-axis tracking system.

The moving parabolic solar dish is driven by a single-axis tracking system that consists of a DC motor (24 V) and gears as shown in Figure 3. The system is controlled by a micro-switch and two timers. One timer controls the start and stop of rotation in the morning (sunrise) and evening (sunset). The other timer controls the rotation angle tracking the sun: for each hour of the day the motor allows the CSP system to rotate 15°.

2.5. Experimental procedure

All experiments, except TGA, started with weighing one kilogram of milled oil shale sample, followed by placing it inside the reactor in a special steel basket. The reactor was closed and tightened by a set of screws, then positioned in its right place in the CSP system. It was allowed to heat up and the temperature at the center of the bed was measured by a K-type thermocouple. A special small vacuum pump was connected to the reactor outlet and the heat exchanger to allow the reactor to work near atmospheric pressure and let the produced vapor come out of the reactor and through the heat exchanger. This little pump was used instead of a special cylinder and an inert gas flow controller of sweeping gas. Close monitoring of the temperature and general functioning of the CSP system was performed by a trained engineer in all experiments. After reaching maximum temperature depending on conditions on that day, the system was allowed to cool down and the oil shale sample was weighed again to calculate the weight loss. It was noticed during this research work that the surface moisture in the oil shale sample started to evaporate and leave it at slightly above 100 °C. This is in full agreement with findings reported previously by other researchers [40]. As heating by solar energy continued and when the temperature inside the bed reached around 270 °C, the pyrolysis process started, which could be confirmed by a smell nuisance spreading hydrogen sulfide, i.e. rotten egg, as a result of on-going reactions inside the bed. Each run lasted for full day during the summer season when solar intensity exceeds 900 W/m^2, and touching 1000 W/m^2, at noon time for a clear sky during the month of August, as shown in Figure 4 [3].

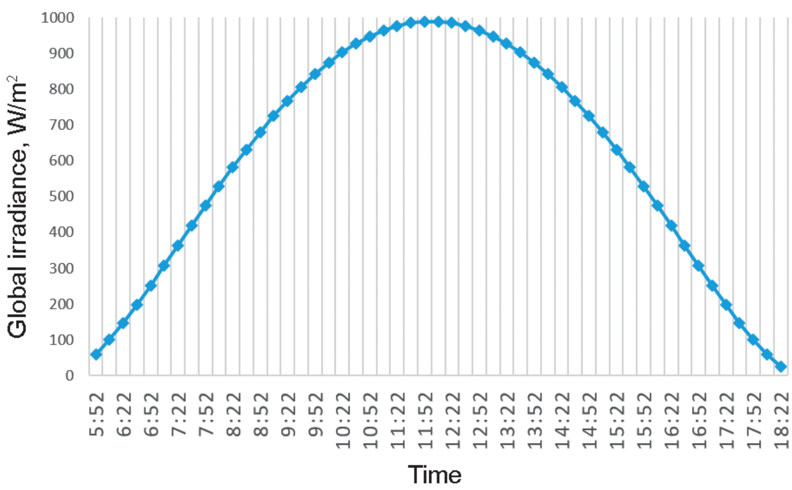

Fig. 4. Typical daily global solar irradiance in Amman in August.

3. Results and discussion

Figure 5a displays the thermogravimetric analysis profile during the conducted experiments for Ellujjun oil shale. It is clear from this Figure that the rate of weight loss is related directly to the bed temperature: the higher the temperature, the greater the weight loss. This is because at high temperatures, the chemical reactions proceed at higher rates [36]. The weight loss could be summarized in three steps. The first one occurred at about 100 °C and involved evaporation of surface moisture which was approximately 1% by wt and continued until a temperature of about 270 °C. The weight loss here could be attributed to the liberation of the interlayer water from the clay minerals and the decomposition of nahcolite as well as physical changes of kerogen prior to its conversion to bitumen and gases [14, 19, 41]. The second step started at a higher temperature, but below 300 °C, and lasted up to 525 °C. This is the most important step of oil shale pyrolysis in which conversion of organic matter occurred. The recorded weight loss during this stage was about 18% by wt and was referred to as the loss of volatile matter in the shale. The third step of weight loss occurred in the gasification stage above 625 °C and lasted up to 850 °C, in which calcium carbonate in the inorganic part decomposed, followed by the loss of fixed carbon. The final step, i.e. gasification, exhibited maximum weight loss of around 24% by wt of the oil shale sample. The total weight loss of the tested oil shale sample, from the Ellujjun deposit, during the TGA test was about 43%. These results fully comply with data obtained by other researchers in pyrolysis and gasification studies for the same type of oil shale [12, 14, 20].

Figure 5b shows the derivative thermogravimetry (DTG) curve for the oil shale sample. Three peaks are observed in this Figure. The first one appears at 101 °C, the second is around 450 °C and the third one at 775 °C. The

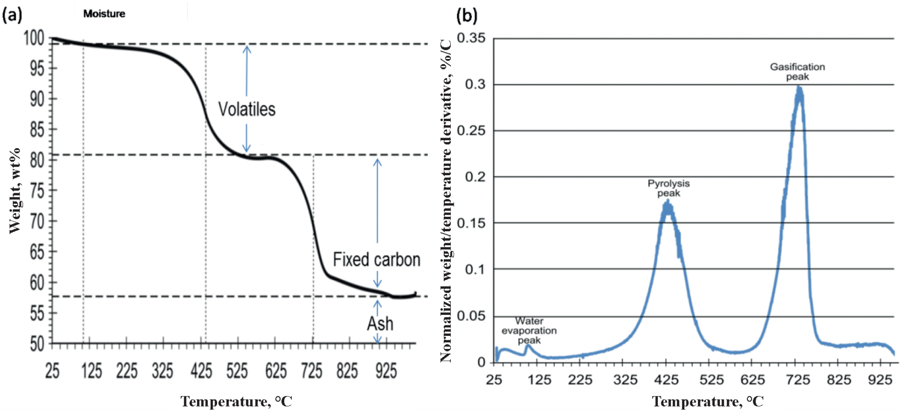

Fig. 5. The results of thermogravimetric analysis: a) TGA profile, b) TGA results showing the moisture, pyrolysis and gasification peaks.

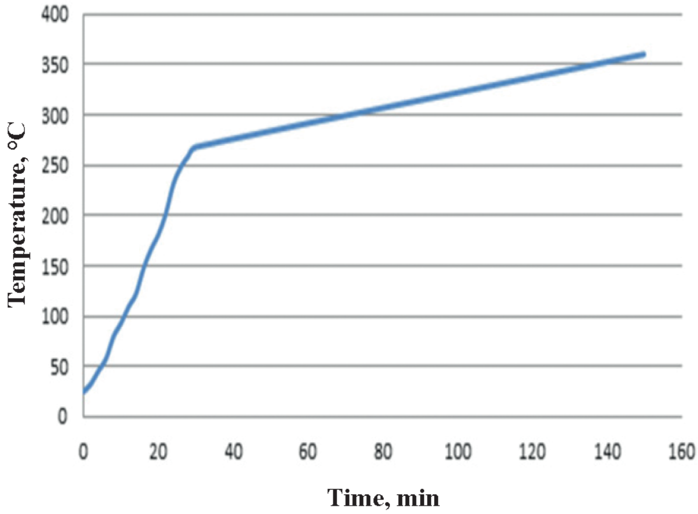

Fig. 6. Typical daily measured temperature in the center of the oil shale bed.

second peak corresponds to kerogen pyrolysis and the third is attributed to the decomposition of carbonates. Again, these findings are comparable with results obtained by other investigators in previous studies [13, 14, 19, 20]. In the solar pyrolysis experimental work, three different experiments were conducted to understand the influence of environment inside the reactor on the yield. These included (i) air, (ii) fresh tap water to stimulate wet oil shale sample and (iii) kerosene. The temperature at the center of the oil shale sample inside the reactor, i.e. gasifier, was monitored and measured as a function of time (hour in the day). It was noticed that the time required to heat up the reactor starting from ambient temperature to reach a temperature of about 360 °C in the center of the bed was around three consecutive hours. Figure 6 shows the temperature rise of the oil shale sample inside the reactor against time. It is clear that the initial heating-up took nearly 30 minutes to reach a temperature of approximately 270 °C, then the temperature continued to increase but at a lower rate as shown in Figure 6.

The results of solar pyrolysis process are summarized in Table 3. The maximum oil yield, after five consecutive hours, of the heating process was about 6.8% by wt, as an average of three experiments, when air was used as medium inside the reactor (see Fig. 7). It is clear from Figure 7 that pyrolysis reactions did not take place until the temperature of the oil shale sample reached approximately 250–270 °C, or after about 60 minutes from starting up the CSP system. A similar pattern was observed during the TGA and fixed bed retort tests for an oil shale sample obtained from the same deposit in Jordan [13]. In the other two tests the yield was lower:

6.1% by wt and almost zero for kerosene and water, respectively. Such results could be expected due to the fact that pyrolysis was conducted in the presence of inert gas such as nitrogen, which is the major constituent of air, and this should yield maximum shale oil. When water was added into the reactor, to represent wet oil shale sample, all the energy supplied by the CSP system went for converting liquid water into vapor. This was confirmed by the continuous measurement of temperature of the oil shale sample inside the reactor, which did not exceed 100 °C: saturation temperature under normal pressure. In other words, all solar heat was absorbed by water to convert to steam. To confirm this, an energy balance was established based on basic thermodynamic calculations of total heat required to convert the introduced mass of water (i.e. 0.1 kg) to vapor, under constant pressure, using the following equation:

$$Q_{\mathrm{Total}} = Q_{\mathrm{Sensible}} + Q_{\mathrm{Latent}}. \quad (2)$$

Table 3. Summary of oil shale solar pyrolysis

Medium	Air	Water	Kerosene
Mass of oil shale sample, kg	1.0	1.0	1.0
Mass used, kg	0.0	0.1	0.1
Heating time, h	5.0	5.0	5.0
Oil yield, wt%	6.8	0.0	6.1
Maximum temperature, °C	370	100	252

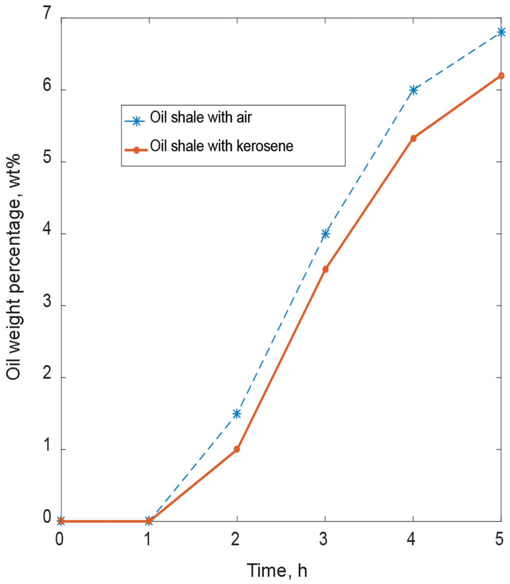

Fig. 7. Solar pyrolysis in the fixed bed reactor.

During the experiments, the temperature of water was increased from the initial temperature T_1 = 25 °C to the final temperature of about T_2 = 100 °C, thus, the sensible heat was calculated to be 31.5 kJ:

$$Q_{\text{Sensible}} = m_{\text{water}} C_p (T_2 - T_1). \quad (3)$$

The latent heat at atmospheric pressure 101.42 kPa was obtained from saturated water tables [42] and equaled 225.7 kJ:

$$Q_{\text{Latent}} = m_{\text{water}} \, (h_{fg}). \quad (4)$$

This brings the total heat to equal 257.18 kJ. In other words, the heat of vaporization in this particular case is far much, i.e. at least seven times, higher than the sensible heat. This explains why oil shale should be dried from surface moisture before being processed using this new system powered by solar energy. This applies to the kerosene experiments, and most of the heat supplied by the CSP system was enough to evaporate the kerosene with a higher boiling point of about 250 °C, which had been added to the oil shale sample as solvent. Hence, few pyrolysis reactions occurred inside the reactor. In the future and in order to be able to raise the temperature inside the bed, a more powerful CSP system will be required.

The results of bomb calorimeter tests, which represent the gross heat of combustion, showed the average high heating value of raw oil shale to be about 6950 kJ/kg and for the produced shale oil 41200 kJ/kg. Similar results have been reported earlier also by other researchers for oil shale from the same deposit [17, 43]. It is obvious that removing ash from oil shale and concentrating organic matter as a result of the pyrolysis process increased the energy density of the new synthetic fuel, i.e. shale oil, by six times or more.

The experiments for determination of total carbon content in the oil shale sample were done using the carbon isotope composition. The chromatographic analyzer was used to separate these gases and determine the elemental composition of each component. The total elemental composition of the Ellujun oil shale oil can be written as: $C_{4.078}H_{4.99}O_{2.30}N_{0.07}S_{0.25}\mathit{Ash}$.

The content of sulphur in the oil shale sample is high. This is true for almost all Jordanian oil shales, which represents a serious problem for retorting projects. Hence, post-treatment of the produced shale oil is a must to remove sulphur or reduce its content to acceptable limits [17].

The kinetic results obtained from the study in hand corroborate those reported for the same oil shale, i.e. Ellujjun, previously [12, 13, 41]. However, some slight differences between the results were found and this could be attributed to different reasons. But the most important is that oil shale has a complex heterogeneous nature, especially the kerogen; hence, it would be difficult to obtain the same experimental results. Equally important are variances in sample preparation procedure, amount of sample tested, grain size and analysis method adopted. In the future, a more powerful CSP system should be designed and constructed to be able to concentrate and supply required heat and increase the temperature inside the reactor up to the pyrolysis stage (about 500 °C) or even gasification stage (above 800 °C).

4. Conclusions

This experimental study is the first of its kind in Jordan and the Middle East and North Africa region. It was undertaken to investigate solar pyrolysis potential of an oil shale sample from Jordan. The fixed bed retort heated directly by a solar dish was used to convert the oil shale organic matter into liquid and gaseous fuel. The results of the thermogravimetric analysis and fixed bed retort tests are encouraging and proved that the studied sample can be successfully processed to yield oil and gas. This, however, is the first reported experimental study using a concentrated solar system to heat up the oil shale inside the retort. Although constructing the retort does not need great knowledge of the reaction mechanism, the optimization of solar dish design requires a deep understanding of variables governing the rate of collected and concentrated solar energy that should be enough to bring the temperature up to desired limits. It is deemed that this research will be continued in the future to design and construct a more powerful CSP system to explore production of synthetic fuels from oil shale. Such information is still highly required to provide a full understanding of oil shale conversion and processing using solar energy.

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