

KINETICS OF PYROLYSIS OF ATTARAT OIL SHALE BY THERMOGRAVIMETRY

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Oil shale samples from different seems of the Attarat deposit in Jordan have been studied using a thermogravimetric analyzer. The influence of grain size and heating rate (from 3 to 40 °C min⁻¹) on the process of thermal degradation of the shale sample has been determined. The integral method was used to analyze TGA data in order to determine pyrolysis kinetics. The main weight loss of the samples, arising from the conversion of organic matter to oil and gas, occurred within the temperature range from 250 to 550 °C. The magnitude of the total weight loss was mainly dependent on the furnace temperature and, to a lesser extent, on the heating rate employed. The decomposition rate of the studied samples changed significantly at a critical temperature of about 300(±10) °C. The kinetic results obtained are in agreement with those reported for other Jordanian oil shales from different deposits.

Introduction

In Jordan oil shale represents the only available fossil fuel, besides limited reserves of natural gas in the Al-Risha gas field near the Iraqi border. The reserves estimated in the central region of Jordan are huge, i.e. exceeding 4×10^{10} tonnes oil shale, with an average organic content of between 9 and 13% by weight [1–6]. The Attarat oil shale deposit is considered the second largest site in the kingdom in terms of its geological reserves, estimated at about 11×10^9 tonnes, see Table 1 [7]. Yet, only two deposits – El Lajjun and Sultani – were investigated thoroughly [6–8]. Thus, there is a dearth of information available about kinetics and pyrolysis of Jordanian oil shales, because there has been only little interest in developing such local resource. The main reason for this is that the majority of deposits are relatively lean and located in remote regions, where, at best, only a limited industrial

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infrastructure exists. Equally important is poor knowledge of the full extent and exact characteristics of the deposits, since the government has concentrated its efforts, in the past, on the El Lajjun and Sultani deposits. Moreover, the prevailing relatively low unit prices for crude oil on the international market have discouraged developers from making investments to exploit these oil shale resources.

Table 1. Characteristics of Oil Shale Deposits in Jordan

Location	Head area, 10^6 m^2	Average thickness, m	Overburden thickness, m	Oil content, wt. %	Geological reserve, 10^9 tonnes
W. Maghar	650	10–60	41	6.8	31.5
Attarat	290	23–63	53	8.0	11.3
W. Thamad	150	119–196	53	–	10.5
J. Drawish	150	128	48	5.7	8.0
El Lajjun	25	29	29	10.5	1.2
Sultani	24	30	69	9.5	1.1

In order to maximize the output and conversion efficiency of oil shale retorting, optimal process conditions are to be elucidated and applied. Grain size, heating rate employed, and the final pyrolysis temperature are the most important factors, especially the latter influences oil yield and the extent of any oil cracking.

The kinetics of thermal decomposition of various shales from different regions of the world has been investigated, and various suggestions on the decomposition mechanisms have been reported [9–24]. Many thermogravimetric studies have been carried out under isothermal conditions. However, it is more accurate to use the non-isothermal method to determine kinetic parameters of the pyrolysis process, employing a TGA apparatus for heating the sample at a constant rate and recording sample weight change. The experimental times are shorter, and the encountered difficulties, e.g. the initial heating-up period in isothermal methods are fewer. The principal reason for this method popularity in the field of oil shale pyrolysis study is that it more closely simulates the conditions expected in commercial-scale oil shale retorting systems. Many researchers have preferred this technique for determining reaction kinetics, e.g. activation energy [9, 15, 20, 22–28]. Hence, the kinetics of thermal degradation of a small shale particle specimen can be determined using a TGA apparatus, which records the changes occurring with time. The behavior of oil shale during pyrolysis is complicated, because shale is a complex mixture of kerogen and wide range of minerals. Also, shale oil produced by pyrolysis is the result of several physical and chemical reactions occurring simultaneously in series and parallel, while the TGA apparatus measures the overall weight loss caused by these reactions. Hence, TGA provides only general information about the overall reaction kinetics.

Thermal analysis of oil shale helps in understanding as well as in evaluating the retorting process mechanism, so that the behavior of the shale could be predicted with a high degree of certainty for a wide range of operating conditions. In this study an attempt was made to determine the effect of various retorting conditions (heating rate, furnace temperature and particle size) on the devolatilization profile of the Attarat oil shale.

Experimental

Oil Shale Samples

Only the El Lajjun and Sultani oil shales have been surveyed and analyzed comprehensively for exploitation purposes. Much less attention has been paid on the other known deposits, e.g. at the Attarat site [6–8]. The oil shale obtained from the Attarat field has a similar percentage of organic matter compared to shales from the El Lajjun and Sultani deposits, but it probably represents a resource of greater significance than these two deposits. The oil shale samples investigated in this experimental study were taken from different seams from the Attarat deposit, south of the capital Amman in the central part of Jordan, and supplied by the Natural Resources Authority (Amman, Jordan).

The details of the sampling method used were not provided. The samples of raw oil shale as received were crushed in a jaw crusher and then, without further treatment, sieved in order to obtain the samples of the required particle size. Average main characteristics of the Attarat oil shale are as follows: oil content 8 wt.%, calorific value 5,000 kJ/kg, organic matter in the shale 29 wt.%, density 1,800 kg/m³, ash content 53.2 wt.%, moisture content 3.25 wt.%, sulphur content 2.6 wt.%.

Experimental Apparatus

Kinetic data were obtained using a SETARAM LabsysTM Series TG Analyzer with argon as the sweeping gas supplied at a constant rate of 2 l h⁻¹ at normal conditions. The TGA apparatus permits continuous measurement of sample weight as a function of temperature, or time, and provision is made for an electronic differentiation of the weight signal to give the rate of weight loss, with an accuracy of 0.01 mg. The effects of heating rate, particle size and furnace temperature on the weight loss of the oil shale sample were determined.

Pyrolysis was carried out non-isothermally using a 48-mg sample placed in the alumina crucible, which was then put on the sample pan hanging in the reaction tube with controllable atmosphere. The bodywork was moved upward to close the system, and the start button depressed. The pre-programmed control unit regulates all automatic functions of the recorder, e.g. continuous measurement of the sample mass, as well as the temperature

programming of the furnace. After the furnace temperature had achieved its set value, the sample was allowed to cool to room temperature. The TG analyzer is connected to a PC, which enables to record the signals and store the experimental data, *via* a special cable. The main conditions employed were as follows:

Sample grain size, mm	Powder, <0.5, ≥0.5–<1.0 and ≥3
Heating rate, °C min ⁻¹	3, 5, 10, 20 and 40
Final temperature, °C	600
Gas carrier	Argon

Analysis of the TGA Data

The mathematical procedure used to analyze TGA data and to determine the pyrolysis kinetics is based on the integral method used by many other investigators [13, 22, 29]. Assuming that the mass loss occurring during TGA experiments is a first-order reaction, the rate of decomposition is given by:

$$\frac{dX}{dt} = k(1 - X) \quad (1)$$

where k (the rate constant) is given by the Arrhenius expression.

$$k = A \exp(-E/RT) \quad (2)$$

For non-isothermal kinetic parameters with a constant heating rate ($h = \frac{dT}{dt}$) during the TGA experiment, the integration of Equation (1), assuming a first-order reaction, results in the following equation:

$$\ln[-\ln(1-X)] = \ln \left[\frac{A R T^2}{h E} \left(1 - \frac{2 R T}{E} \right) \right] - \frac{E}{R T} \quad (3)$$

where h is heating rate, °C min⁻¹;

X is the amount of oil shale decomposed during time t , %.

In theory, the plot of $\ln[-\ln(1-X)]$ against $1/T$ is a straight line with a slope of $-E/R$, the magnitude of which can be used to calculate the activation energy (E). In reality, deviations of the experimental data from the straight-line trend indicate the degree of inaccuracy of the assumption of a first-order reaction. The plots of Equation (3) were made for two samples, and the activation energies were determined from the best-fit lines. The assumption of a first-order reaction appeared reasonable as the correlation coefficient exceeded 0.95. The frequency factor (A) can be calculated from the intercept.

Results and Discussion

Thermal analysis of oil shale helps in understanding as well as in evaluating the retorting process mechanism, so enabling to predict the behavior of the shale with a high degree of certainty for a wide range of operating conditions. Most of the studies proved that the weight loss, as indicated by the TG curves, decreases with decreasing particle size [20, 23, 30]. A typical variation of the percentage conversion with temperature (i.e. the TG curve) is shown in Fig. 1, while Fig. 2 shows the differential weight loss (i.e. the DTG) profile. It is obvious that the decomposition of organic matter of the Attarat oil shale occurred in a single step. The rate of weight loss, due to conversion of the organic matter, is clearly related to the pyrolysis temperature: the higher the temperature, the greater the weight loss.

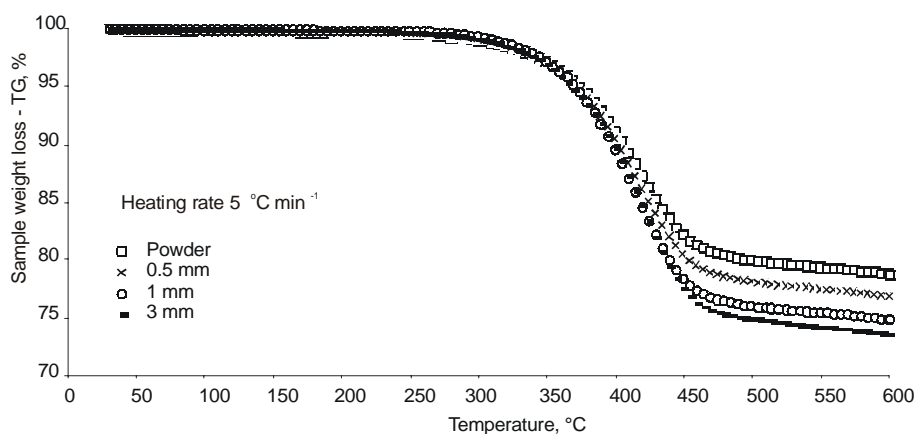


Fig. 1. TG profiles with respect to pyrolysis temperature and particle grain size

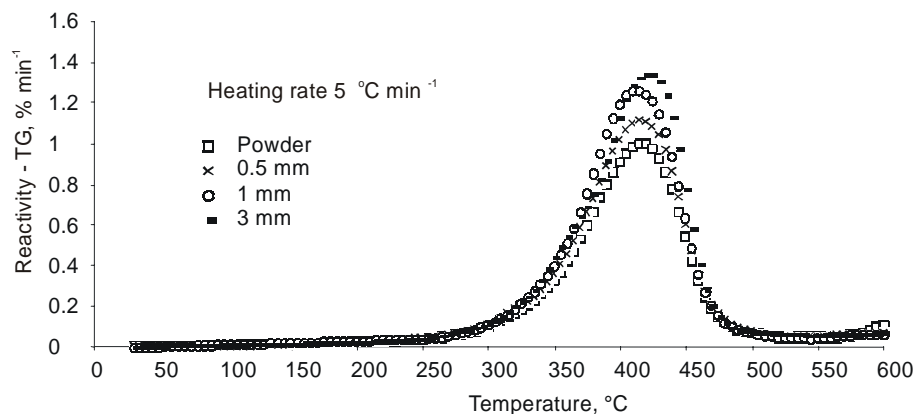


Fig. 2. DTG of Attarat oil shale in relation to pyrolysis temperature and particle grain size

As seen in Figs. 1 and 2, particle size slightly influences the extent and rate of decomposition: larger particles yield higher weight loss under identical operating conditions. The temperature of the maximum weight loss also increases slightly with larger particles: there is a shift towards higher temperatures with increasing particle size. Such an effect may be attributed to the change in the composition of the raw shale due to the grinding process and the following variations in the heat and mass transfer properties within the sample.

The DTG figure demonstrates clearly that the mass-loss profile has one major stage of oil shale decomposition. However, there is a slight weight loss in the pre-heating phase below 200 °C, which corresponds to the loss of the interlayer water from the clay minerals and from the decomposition of nahcolite, as well as to the physical changes (i.e. softening and molecular rearrangement associated with release of gases) in the kerogen *prior* to its decomposition to bitumen [31]. The loss of hydrocarbon material took place during the major stage (between 200 and 550 °C).

It is evident that the major pyrolysis peak is a singular one, and the extractable organic contents is approximately 21(±2)% of the original sample weight, depending on the particle grain size. TGA experiments show also that oil shale does not devolatilize completely until temperatures of about 540(±10) °C are reached. Different oil shale samples, from the same deposit but of different grain size, exhibited qualitatively the same patterns of thermal degradation when the pyrolysis temperature was raised over the studied range. The increase in the amount of decomposed shale with increasing temperature between 200 and 550 °C is greater than that at temperatures exceeding 550 °C. This is due to the fact that the optimal temperature for maximizing oil shale retorting is around 550 °C.

Table 2. Weight Loss at Different Heating Rates

Heating rate, °C min ⁻¹	Particle weight loss, %			Temperature of rate maxima, °C
	30–250 °C	250–600 °C	Total	
Oil shale sample* A ₁				
3	1.30	23.10	24.40	410
5	1.25	23.75	25.00	419
10	1.04	22.92	23.96	431
20	0.56	24.72	25.28	452
40	1.35	24.93	26.28	460
Oil shale sample* A ₂				
3	0.60	20.61	21.20	411
5	0.40	21.01	21.41	418
10	0.40	21.05	21.45	428
20	0.40	21.87	22.27	444
40	0.13	21.11	21.24	456

* Samples A₁ and A₂ are obtained from different seams of the same deposit at the depth of about 60 and 65 m, respectively.

The behavior of the Attarat oil shale – single-step thermal decomposition – is similar to those observed for the other Jordanian shales, i.e. El Lajjun and Sultani [31, 32]. So behave also various shales from other countries – Colorado [33], Ohio, West Virginia and North Carolina [26] (the USA), Aleksinac and Knjazevac (Yugoslavia) [20], Beypazari (Turkey) [22] and Kark, Dharangi and Malgeen (Pakistan) [23]. The main conclusions derived from our study on the Attarat oil shale are in full agreement with those reported earlier for Jordanian oil shales [9, 31, 32, 34]. As for the shape of the decomposition profile (i.e. single or two-stage), oil shale pyrolysis studies indicate that the conversion of kerogen into shale oil is a two-stage process. At first, decomposition of kerogen to pyrolytic bitumen occurs, and then, decomposition of the bitumen to products takes place [18, 22, 28]. However, the mechanism of oil shale thermal decomposition is complex and involves a series of parallel reactions.

The weight losses during pre-heating and pyrolysis of both oil shale samples having the same grain size, with regard to heating rate and the temperature at which the maximum rate of decomposition occurred, are presented in Table 2. Examination of these values indicates that the heating rate employed has only a negligible effect on the yield or final weight loss (see Fig. 3).

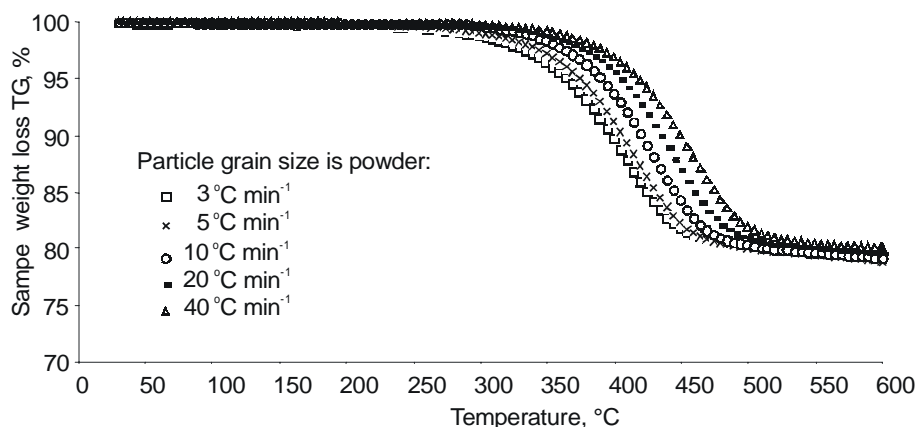


Fig. 3. TG profiles with respect to heating rate

When the heating rate was increased, the maximum rate of decomposition shifted to higher temperatures. The shift in the DTG peak, which is a measure of relative reactivity (i.e. rate of kerogen degradation), is illustrated in Fig. 4 and can be attributed to the variations in the rate of heat transfer with the change in the heating rate and to short exposure time to a particular temperature at higher heating rates [32, 35], as well as to the effect of the kinetics of decomposition [36]. At higher heating rates, the temperature on the surface of each shale particle will be higher than in its core. The reactions occurring inside the particle at relatively low

temperatures yield the products, which, passing through the higher temperature region, undergo secondary reactions before emission from the particle. These secondary reactions reduce the yield of shale oil, the required product from oil shale retorting [28]. At lower heating rates decomposition of kerogen is almost complete, whereas, at high heating rates (e.g. $40\text{ }^{\circ}\text{C min}^{-1}$), complete decomposition would occur at a higher temperature compared to the original of about $550\text{ }^{\circ}\text{C}$. At lower heating rates longer exposure time to a particular temperature is needed. Increasing the heating rate from $3\text{ to }40\text{ }^{\circ}\text{C min}^{-1}$ significantly (by about 8 times) raised the reactivity (defined as the ratio of the rate of weight loss per unit of time) of the studied shale samples.

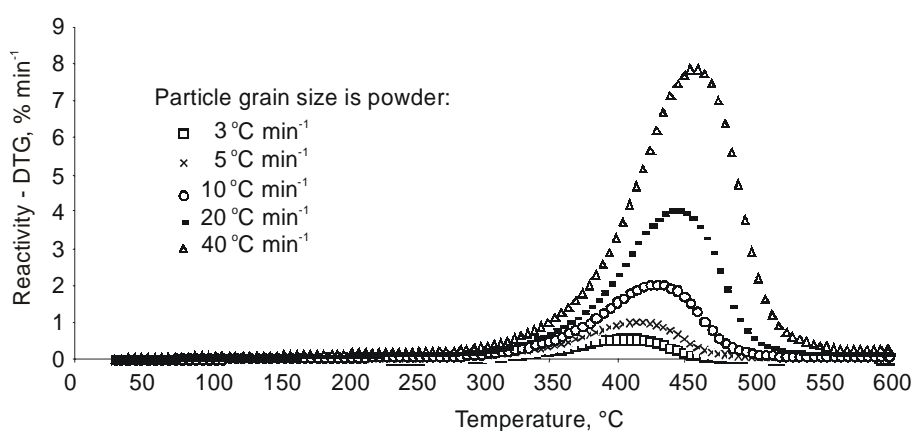


Fig. 4. DTG profiles in relation to heating rate

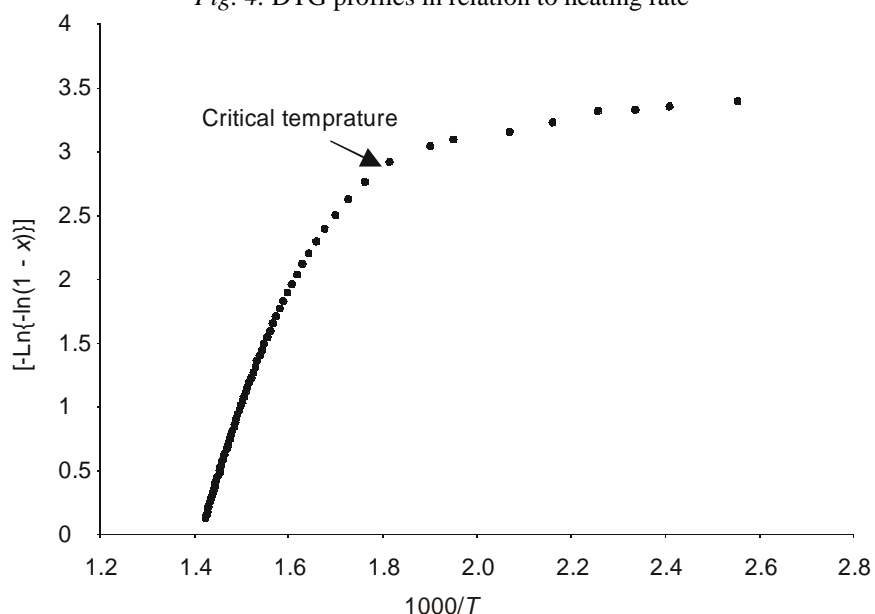


Fig. 5. Determination of apparent activation energy from TGA data

The Attarat oil shale has two kinetic expressions (defining thermal decomposition process) within the temperature range covered while heating the specimen. It means that the rates of the Attarat shale decomposition reactions change at some critical temperature (i.e. the plot of Eq. (3) gives two straight lines of different slopes over the studied temperature range, and cross at a critical temperature as shown in Fig. 5).

In this study, it has been found that the average value of the critical temperature is around $300(\pm 10)$ °C (see Table 3). Below this temperature, the heat absorbed softens the kerogen, and a small mass loss takes place. Above the critical temperature, destructive distillation accompanied by cracking (at higher temperatures) occurs. The same (i.e. the existence of two temperature regions between 290 and 425 °C) has also been reported for El Lajjun and Sultani oil shales [31, 32].

A critical temperature of between 325 and 425 °C was reported for Yugoslavian Aleksinac and Knjazevac shales [14], as well as for the British Kimmeridge clay oil shale [15]. An average of between 300 and 350 °C was found for Turkish Seyitomer and Himmetoglu shales [22]. Such findings corroborate a serial reaction model, which assumes that kerogen is first converted to bitumen, which then cracks to release oil and gas leaving char. However, oil generation may occur at the beginning of the pyrolysis process. So, a thermal degradation mechanism describing better the behavior of the investigated shales includes two stages: kerogen is initially converted to bitumen, oil, gas and residue, and, in the second stage, the bitumen is converted to oil, gas and residue.

Table 3. Kinetic Parameters of Tested Oil Shale Samples

Heating rate, °C min ⁻¹	Activation energy, kJ mol ⁻¹		T_c , °C
	E_1	E_2	
Oil shale sample* A ₁			
3	12.96	77.48	298.0
5	11.46	78.29	296.0
10	12.90	85.25	302.8
20	14.48	79.29	298.4
40	15.79	75.59	311.0
Oil shale sample* A ₂			
3	16.85	86.66	305.1
5	16.22	93.78	297.6
10	15.63	97.55	300.9
20	17.79	94.69	303.0
40	16.00	85.91	307.2

* Samples A₁ and A₂ are obtained from different seems of the same deposit at the depth of about 60 and 65 m, respectively.

It is noteworthy that the critical temperatures recorded are comparable with the literature data on the temperature of formation of the maximum amount of bitumen fractions during non-isothermal pyrolysis [37]. Miknis and Turner reported theoretical predictions concerning Wyoming oil shale, USA, and assumed that at the heating rate of $10\text{ }^{\circ}\text{C min}^{-1}$ maximum bitumen fractions are expected at a temperature of about $375\text{ }^{\circ}\text{C}$. Differences in the values of critical temperature can be attributed to higher heating rates as well as different types of oil shale used.

As seen from Table 3, there are two kinetic expressions for each heating rate, representing two stages of decomposition during the pyrolysis process. The influence of heating rate on activation energy, which is inversely related to the heating rate, is clear: there is a decrease in the apparent activation energy upon increasing the heating rate. This could be attributed to greater rates of heat transfer. In the first stage, the reactions proceed at relatively low temperature with average activation energy (E_1) between 13.5 and 16.5 kJ mol^{-1} for the two samples (A_1 and A_2), respectively. Such a low value of E_1 confirms that only physical changes and/or breaking of weak chemical bonds take place during the first stage of kerogen decomposition [12, 22, 32, 38]. In the second stage, the reactions have much higher values for activation energy (E_2), within the range of 79.2 – 91.7 kJ mol^{-1} for both samples. The slight difference in activation energies and in weight loss is due to the fact that in the TGA experiments only a small amount of kerogen was tested, compared with the total mass of the oil shale sample (which itself is minute), so any heterogeneity or non-uniformity in the distribution of organic matter will manifest itself in the final results. In addition to the complications caused by simultaneously occurring mineral decomposition reactions, noises in the experimental system, due to particle cracking and condensation, may affect the final conclusions. It should be stressed here that the obtained activation energy (from TGA data) characterizing the decomposition process are only representative values for a complicated series of the reaction system. These values are, however, considered to be good enough from the engineering point of view of oil shale retorting, and can be still used for the design of practical retorting systems.

Again, the kinetic results obtained from the present study for the Attarat oil shale, almost corroborate those reported for the El Lajjun and Sultani oil shales [9, 31, 32, 34, 39]. However, it should be remembered that the difference between the results of this study and those reported for El Lajjun and Sultani oil shales is due to the fact that the samples were taken from different deposits having dissimilar composition, and the influence of the process parameters, such as heating rate and particle size, was different. Oil shale has a complex heterogeneous nature, especially the kerogen: hence, it would be difficult to achieve the same experimental results, even for nominally the same sample (in this study the key tests were replicated). Therefore, the same experimental technique, including sample preparation

procedure, analysis method and the kinetic model for the analysis should be employed in order to achieve a reasonable comparison.

Conclusion

Oil shale is by far the largest fossil fuel resource in Jordan, with reserves totaling approximately 4×10^{10} tonnes. However, as yet, there is a little information available concerning the pyrolysis of Jordanian oil shales, and most of the pertinent published information is concerned either with the economics (e.g. financial pre-feasibility studies) of utilizing such a resource, or geological surveying for two deposits (i.e. El Lajjun and Sultani). This is most probably because there has been only little interest in developing this resource due to the prevailed low crude oil unit prices, during the last twenty years, compared with those for the final energy recovered from oil shale.

All samples of the Attarat oil shale, tested using TGA apparatus showed that the conversion of kerogen is totally dependent on the pyrolysis temperature: the higher the temperature, the greater the weight loss of both samples. The behavior of Attarat oil shale can be described by two kinetic expressions valid in the studied temperature range, the values of the apparent activation energy are consistent with a first order reaction occurring. The critical temperature, at which the rate of kerogen decomposition changed significantly, was $300(\pm 10)$ °C. Comparing the results of this study with previous investigations on other Jordanian oil shales showed that kinetics and decomposition behavior of the Attarat shale are almost similar to those determined for both El Lajjun and Sultani shales.

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