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Thermogravimetric and Kinetic Analysis on Pyrolysis and Combustion of Oil Shale under Different Oxygen Concentration Atmosphere

G ROLAND NGUIMBI[1], YOU-HONG SUN[*1,2], MINGYI GUO[1], HAN JING[1] AND PHIRICRYTON[3]
[1]*College of Construction Engineering, Jilin University, Changchun 130021 P.R. China*
[2]*Key Laboratory of Ministry of Land and Resources on Complicated Conditions Drilling Technology, Jilin University, Changchun 130021, P.R. China*
[3]*Geology Department, School of Mines, University of Zambia, 10101 LUSAKA ZAMBIA*
Corresponding author: rolandnguimbi@yahoo.fr
***Email:** rolandnguimbi@yahoo.fr; syh@jlu.edu.cn; 23628920@qq.com; 1522345882@qq.com; crytonphiri@yahoo.com*

Abstract: The kinetics of combustion of oil shale by different oxygen contents(11,21,42and 63% Vol.) has been investigated by thermogravimetric analyzer (TGA) at three different heating rates (10,30 and 50°C/min) .The Activation energy E of oil shale thermal decomposition under different oxygen concentrations were determined by iso-conversional method (Kissinger-Akahira-Sunose (KAS) method and Flynn–Wall-Ozawa (FWO) method). The data obtained from TG was used to calculate the Activation energy and discuss the influence of oxygen concentration on kinetic parameters. According to the TG results, the combustion process of oil shale could be divided into three stages, the evaporation of water, the combustion of the decomposition of organic matter and combustion of semicoke. The TG revealed that the oxygen content in atmosphere does not affect the evaporation stage but influence the combustion stage. The results show that the average activation energies were 134.85, 163.41, 211.19 and 242.32 KJ/mol. Activation energy using KAS method were 136.50, 167.37, 216.49 and 225.38 KJ/mol, whereas the FWO method at different oxygen contents were: 11,21,42, 63 vol% ,respectively. The results of kinetic calculation revealed that the activation energy values of the combustion increase with increase of oxygen content.

***Keywords**: Oil shale, Kinetics, Combustion, Oxygen content, Thermogravimetric analysis (TGA)*

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1. Introduction

Oil shale, a fine-grained sedimentary rock with organic matter called kerogen, is rich and widespread throughout the world [1, 2].The retorting process convert oil shale to liquid fuels (shale oil).The shale oil obtained from the oil shale by retorting technology is an ideal substituted for crude oil[3].Due to the growing demand for petroleum commodity, and increase in petroleum prices and limited availability of petroleum reserves, it is particularly important to develop comprehensive technological process of converting oil shale to shale oil, Oil shale is an alternative resource of oil and natural gas.

Thermal decomposition kinetics of fossil fuels is necessary in thermochemical conversion processes, and is aimed at the production of energy and chemical products [4-6].Thermogravimetric (TG) and differential thermogravimetry (DTG) analysis give complete information on the reaction of kinetics. Thermogrametry analysis is the most appropriate method to test the kinetics of thermos-oxidation of solid and it is used to evaluate the thermal characteristics of solid (Coal, Biomass, oil shale..) by isothermal and non-isothermal techniques [6-11]. Thermogravimetric analysis method of oil shale samples has been used to analyze the effects of temperature on thermal decomposition process and to estimate kinetic parameters of complex reactions on oil shale research [11-15].The data obtained from TGA can be used to determine kinetic models of the thermal decomposition process and to determine the kinetic parameter by different kinetic model. Kinetic parameter of thermal analysis for oil shale reactions is being evaluated through several kinetic methods; many kinetic analytical methods have been proposed to estimate kinetic parameters on different oil shale by several researchers. For example, Aboulkas studied the kinetics of Moroccan tarfaya oil shale by Coats-Redfern method [16], Qing estimated the kinetics of oil shale from northern Songliao basin in China using Friedman procedure_[17]. Li, used the regression function and Integral method to calculate the activation energy and apparent frequency factor on Fushun and Maoming oil shale[18]. The Maximum rate method was used by

Wang on North-Korean Oil shale [19], Coats-Redfern (C-R) method and Pre-exponential factor method was used by Bai [20,21], Kissinger–Akahira–Sunose method (KAS) was used by Aboulkasa on Moroccan oil shale [22] and Arash estimated activation energy by Flynn–Wall-Ozawa (FWO) method [23]. Many analytical methods are available to calculate the kinetic parameters of oil shale. All these studies have been focused on pyrolysis reaction in nitrogen atmosphere but there have been no studies on the influence of atmospheric oxygen content on the combustion process, while the combustion process depends on the consumption of oxygen and the quantity of oxygen on combustion in which atmosphere play an important role during the combustion process. The oxygen in the atmosphere influences the position and amplitude of the DTG curve [24].The thermal decomposition of oil shale under different oxygen content atmosphere condition has not been investigated. However the combustion on solid material under different oxygen concentration has been studied using biomass [25-28]. The combustion behavior of oil shale under different oxygen atmosphere concentration has not been explored yet and it needs to be carried out and would be of interest in oil shale research.

The present study investigated the kinetics of thermal decomposition of oil shale in atmospheric mixtures of O_2/N_2. The pyrolysis and combustion under different oxygen concentration atmosphere was carried out using thermogravimetric analyzer (TGA) and data from TGA have been used to determine the value of activation energy through two iso-conversional methods, Flynn–Wall-Ozawa (FWO) method and Kissinger-Akahira-Sunose method (KAS) at three different heating rates (10, 30 and 50°C/min) and the optimal oxygen content for combustion of oil shale was determined.

2. Experiment:

2.1. Materials:

The oil shale sample used in this work was obtained from Huadian basin Northeastern China. Table1 shows the data on proximate, ultimate and Fischer assay analysis data results, conducted in accordance with the national standard of China. The samples of raw oil shale were crushed and sieved to particles size less than 0.9mm in diameter_according to The ASTM D2013-07and GB 474-2008 standards

Table 1: *Properties of Huadian Oil Shale*

Proximate Analysis (wt. %)	
Volatiles	39.34
Ash	56.91
Moisture	3.26
Fixed carbon	3.75
HHV (MJ /kg)	13.07
Elemental Analysis (wt.%)	
Carbon	29.23
Nitrogen	0.61
Hydrogen	4.28
Sulfur	4.92
Fischer Assay Analysis (wt.%)	
Shale oil	19.69
Gas	6.38
Water	4.98
Char	68.95

2.2. Thermogravimetric Analysis (TGA):

Thermogrametry analysis (TGA) was performed using Germany NETZSCH ST449F3thermal analyzer. Temperature, gas flow and mass loss of the TG analyzer was calibrated according to the experimental condition.5-10mg of oil shale samples were heated from room temperature to 750°C at different heating rates of 10, 30 and 50°C/min in each run. The combustion was carried out in non-isothermal condition under N_2/O_2 atmosphere with the content ($11\%O_2/89\%N_2$, $21\%O_2/79\%N_2$, $42\%O_2/58\%N_2$ and $63\%O_2/37\%N_2$). With gas flow rate maintained at 60ml/min, the sample mass loss and the rate of mass loss were recorded continuously as function of temperature and time. The analysis was performed at least three times for each atmosphere and heating rate in order to eliminate mistake and assure reproducibility of results. The experimental conditions were as follow:

Combustion gas : N_2/O_2 ($11\%O2/89\%N_2$, $21\%O_2/79\%N_2$, $42\%O_2/58\%N_2$ and $63\%O_2/37\%N_2$)
Heating rate: 10°C/min; 30°C/min and50°C/min,

Flow rate of carrier gas: 60ml.min$^{-1,\backslash}$
Combustion temperature: 25 to 750°C,
Initial weight of the sample: 5-10±0.5 mg

3. Mathematical Model Kinetic Analysis:

The combustion process reaction of oil shale can be written as the follows [29]:

Oil shale → shale oil + gas + char

The thermogravimetric data analysis was used to determine the kinetic parameters of the thermal decomposition of the oil shale; however, solid state reactions are more complex processes. The thermal decomposition of solid-state (oil shale) is a function which depends on the temperature (T) and on the conversion function (α).The kinetic analysis equation is described as the basic equation below:

$$\frac{d\alpha}{dt} = k(T)\,f(\alpha) \tag{1}$$

The temperature dependence on the function *k*(T) given by the Arrhenius equation and the Eq.1it can be expressed as:

$$k(T) = A\exp\left(-\frac{E}{RT}\right) \quad (2)$$

Where *f*(α) is the conversion function which is the time (min) , α represented the degree of conversion ($0 \le \alpha \le 1$) ,T is the absolute temperature (k), *k* is the reaction rate, A is the pre-exponential factor (min^{-1}), E is the Activation energy ($KJ.mol^{-1}$),R is the universal gas constant ($KJ.mol^{-1}.K^{-1}$).Where α is the degree of advance defined by [30, 31]:

$$\alpha = \frac{Mo - Mt}{Mo - Mf} \quad (3)$$

With M_o being the initial mass at t=0, M_t is the mass at time t, and M_f the final mass at the end of the combustion. During the combustion the heating rate β is a constant

$$\beta = \frac{dT}{dt} \quad (4)$$

Inserting Eq.2 and Eq.4 in Eq.1, the kinetic equation changes as follows:

$$\frac{d\alpha}{dT} = \frac{A}{\beta}\exp\left(-\frac{E}{RT}\right) f(\alpha) \quad (5)$$

Integrating Eq.5 gives:

$$g(\alpha) = \int_0^{\alpha} \frac{d\alpha}{f(\alpha)} = \frac{A}{\beta}\int_{To}^{T} \exp\left(-\frac{E}{RT}\right) dT \quad (6)$$

Where g(α) is the integral function of conversion , T_0 (k) is the initial temperature.

According to recommendations by the Kinetics Committee of International Confederation for Thermal Analysis and Calorimetry (ICTAC)[32] proposed that the iso-conversional methods are very appropriate to describe the complex kinetics of fossil fuel process because the results is in close proximity to practical and depends only on the conversion degree and temperature. In this work, activation energy from conversion was determined by thermogrametry analyzer data(α ,T),at different heating rates (β) by using Kissinger-Akahira-Sunose (KAS) method and Flynn–Wall-Ozawa (FWO) method by Integration of Eq.6 according to each method.

3.1. Kissinger-Akahira-Sunose (KAS) method:

Kissinger-Akahira-Sunose method has been developed for non-isothermal analysis. In the KAS method, the relation between the temperature T and the heating rate β is given by the following equation:

$$\ln\left(\frac{\beta}{T^2}\right) = \ln\left[\frac{AR}{g(\alpha)E}\right] - \frac{E}{RT} \quad (7)$$

The activation energy for different conversion values can be calculated by the slopes of these graphs at different conversion degrees from the linear plots of $\ln\left(\frac{\beta}{T^2}\right)$ versus1/T. Using this expression, it is possible to calculate the values of the activation energy.

3.2 Flynn–Wall-Ozawa (FWO) Method:

The Flynn–Wall–Ozawa method was based on following equation.

$$\ln(\beta) = \ln\left[\frac{0.0084AE}{g(\alpha)R}\right] - 1.0516\frac{E}{RT} \quad (8)$$

The plots of the straight line, $\ln(\beta)$ versus1/T obtained at different heating rates give the straight line whose slope determines the activation energy E.

4. Results and Discussion:

4.1. Thermal decomposition of oil shale:

Figure 1 and Figure 2 respectively shows the weight loss (TG curves) and weight loss rate (DTG curves) of the thermal decomposition of oil shale under O_2/ N_2 mixtures at different O_2 concentrations atmospheres. The TG curves show the percentage mass loss of an oil shale sample over the range of temperature from 25°Cto 750°C.As can be seen in the figure1 and figure 2, the combustion process of oil shale under (O_2/N_2) atmosphere could be divided into three individual stages: the first stage from25°C to 160°Cattributed to the moisture removal weight loss occurring in this stage around 2.9wt%.The influence of environment was negligible, temperature was the main factor influencing the evaporation.

The second stage occurred in the temperature range of 328 -540°Cwhich is related to the main decomposition stage attributed to the thermal decomposition of kerogen and bitumen present in the oil shale. The weight loss of the second stage was mainly due to their lease and combustion of volatile matter and combustion of fixed carbon, the mass loss mainly occurred in this stage of the process. The temperature and the oxygen concentration were the main factors influencing the thermal decomposition at this stage.

The third stage was extended up to 600°C which corresponded to the decomposition of carbonate minerals

The TG and DTG profile showed that shapes of the curves are relatively similar to each other. The different oxygen content has not affect the evaporation but hasa remarkably influence on thermal decomposition on organic matter of the oil shale(kerogen and bitumen).The rising oxygencontent, accelerate the burning of organic matter as suggested by some literatures [24,33,34] theses researches shows that higher oxygen content is necessary in combustion process. However, our study showed that, increasing the oxygen content from 21% to 63%, and increased the conversion of oil shale from 7% to 26%.This weight loss corresponded to the decomposition of the organic matter.

The total weight losses mass loss (Table 2) under different oxygen contents at the end of the reaction were; 29.09; 30.38; 32.13 and 31.32% at the heating rate of 10 °C/min, 29.87, 30.76; 31.60% and 32.28 % at the heating rate of 30°C/min and 30.43; 31.56; 33.24% and 32.56 % at the heating rate of 50°C/min, then with the oxygen content of 11, 21, 42, and 63% vol.%, respectively.

The DTG curves show three peaks occurring between 90–160°C; 359–547 °C and 625–757°C (depending on the oxygen concentration).Table 2 and 3show that at the oxygen content of 11; 21;42 and 63 vol.% the maximum reaction rates of mass loss at peak temperature were 0.09%/s; 0.28%/s; 0.85%/s and 0.55%/s respectively.

The result indicates that the oxygen promoted the decomposition of oil shale and the release of the volatile matter leading to the increase in the mass loss rate. The optimal oxygen concentration for combustion during our work was; 42-63%vol.

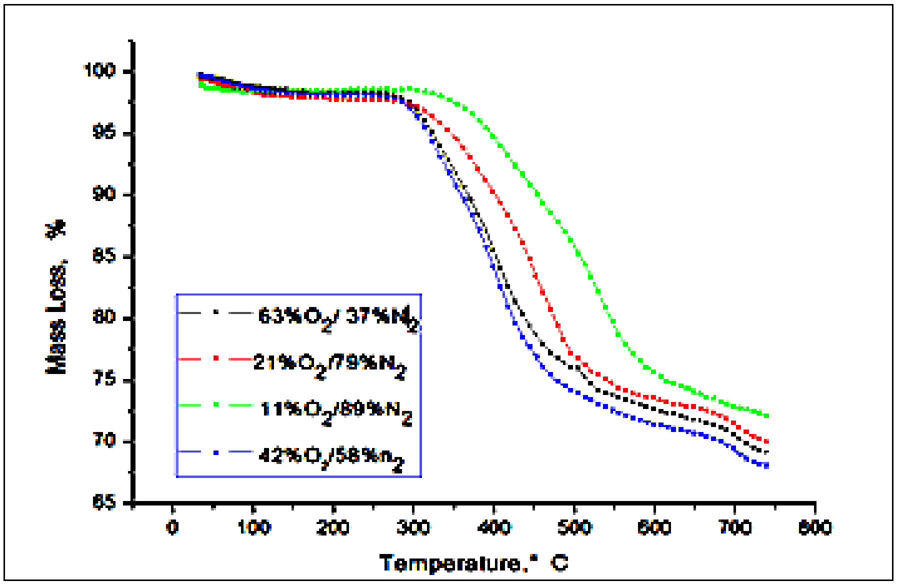

Figure 1: *TG curves of combustion of oil shale under different O_2/N_2 atmospheres at the heating rates of 30°C/min*

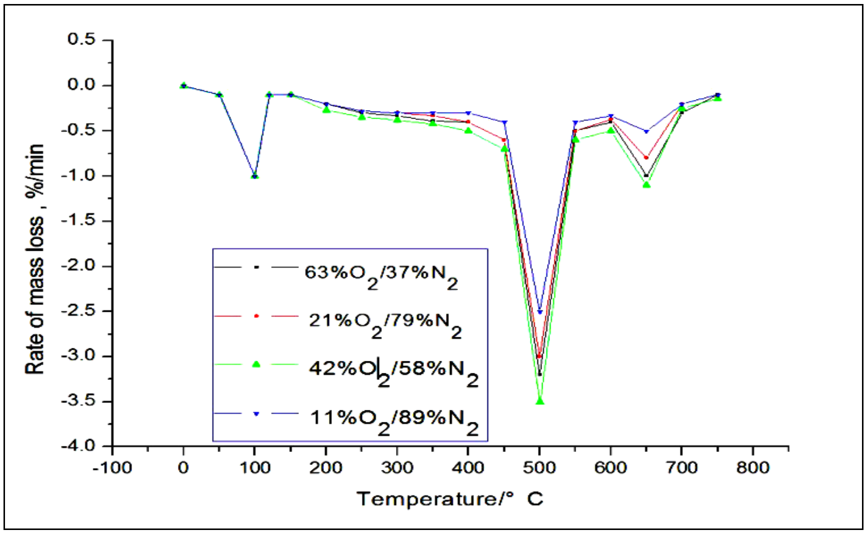

Figure 2: *DTG curves of combustion of oil shale under different O_2/N_2 atmospheres at the heating rates of 30°C*

***Table 2**: Characteristic Parameters from TG and DTG of Oil Shale under Different Oxygen Concentration*

Atmosphere	Temperature Range for main Decomposition (°C)	Average Rate of Mass Loss dw/dt (%s^{-1})	T_{peak}(°C)	Residue mass (%)
11%O_2/89% N_2	383-460	-0.0009	438	69.22785
21% O_2/79% N_2	407-472	-0.0028	446	69.05769
42% O_2/58% N_2	420-494	-0.0085	486	67.3963
63% O_2/37% N_2	425-519	-0.0055	498	68.14865

***Table 3:** Characteristic Parameters of the Combustion Profiles of Oil Shale in 41%O_2/59%N_2 Atmosphere at Different Heating Rates (10, 30 and 50°C/min)*

Heating rate(°C/min)	Second Stage		Third Stage			
	T_2	W_2	T_3	W_3	T_{final}	Loss (%)
10	424	0.82	640	-	767.626	29.09589
30	459	0.85	668	-	738.491	31.60377
50	471	0.88	692	-	703.085	32.95135

***Table 4**: Characteristic Parameters of the Combustion Profiles of Oil Shale at Heating Rate 30°C/min at Different Combustion Atmosphere*

Atmosphere	Second Stage		Third Stage			
	T_2	W_2	T_3	W_3	T_{final}	Loss (%)
11%O_2/89% N_2	431	0.81	616	-	757.2043	30.77215
21% O_2/79% N_2	443	0.83	632	-	741.20	30.94231
42% O_2/58% N_2	459	0.85	668	-	738.491	32.6037
63% O_2/37% N_2	468	0.83	689	-	702.431	31.85135

T_2 and T_3 are the temperature range for the second and third peak in DTG (°C).
W_2 and W_3 are the average mass loss rate of the second and third stage respectively (dw/dt; %/s)
T_{max} is the temperature for maximum reaction rate (°C)
T_{final} is the temperature in the end of reaction (°C)

4.2. Kinetic Characteristics under Different Atmospheres:

The Kinetic analysis of oil shale combustion at different oxygen concentration atmospheres were studied and understand the details of the combustion process suggested by Flynn–Wall-Ozawa (FWO) and Kissinger-Akahira–Sunose (KAS) methods. In our study three different heating rates were used to calculate the kinetic parameter and seven different percentages of conversion rates were pointed out in each curve, (α: 0.20, 0.30, 0.40, 0.50, 0.60, 0.70 and 0.80)

4.2.1. Flynn–Wall-Ozawa (FWO) methods:

Eq.8 is one of the FWO methods that can be used to calculate the kinetic parameter directly from the TG data analysis, conversion (α), temperature (T) obtained at three heating rates value (ß), a plot of ln β versus 1/T should be a straight line. This slope of the linear plot of ln β versus (1/T) is used to determine the kinetic parameter.

The slopes of linear fittings are presented in Fig 3.the values of activation energy calculated by the FWO methods are listed in Table 5.The activation energies under different oxygen levels increased from 136.50 to 225.38 KJ/moles with increase in oxygen content and the increase of mass conversion rate.

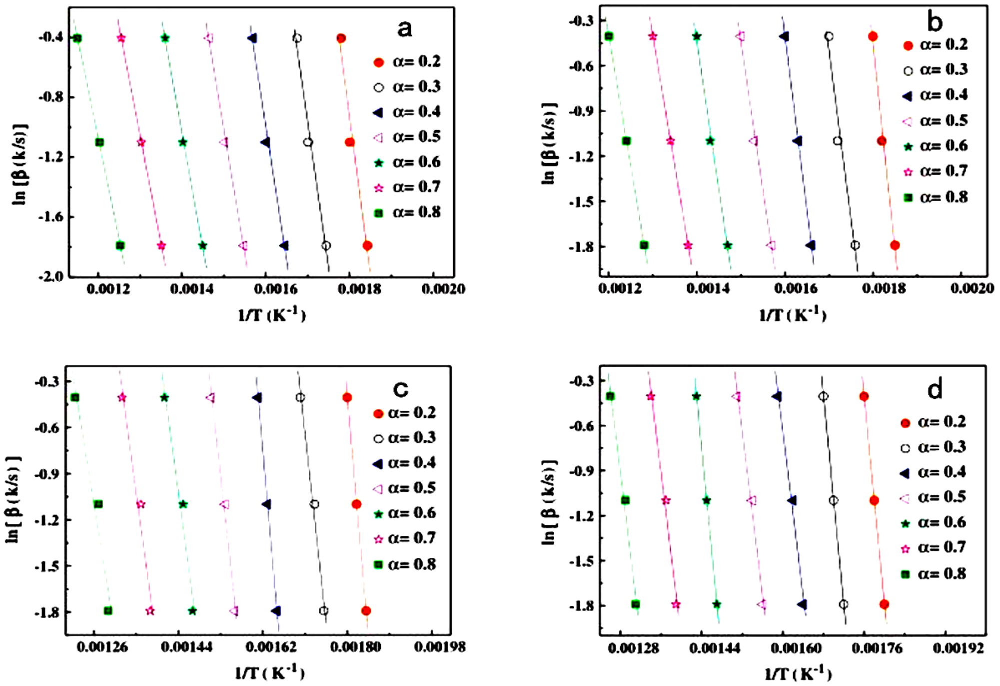

Figure 3: *Iso-conversional plot of FWO method at different α values of conversion under O_2/N_2 atmosphere(a) :11%O2/89%N_2 ,(b): 21%O_2/79%N_2 , (c) 42%O_2/58%N_2 and (d): 63%O_2/37%N_2*

4.2.2. Kissinger-Akahira–Sunose (KAS) methods:

According to KAS method, the activation energy of thermal decomposition process at several conversion degrees α, could be calculated from Eq.7 from the plot of ln(β /T^2) versus (1/T) the regression curves should be straight line.

The KAS method Curves for thermal decomposition of oil shale in different heating rates under different oxygen concentration atmosphere are shown in Fig.4 and the activation Energy (E) and regression coefficients (R) value at various conversions are reported on Table 5.

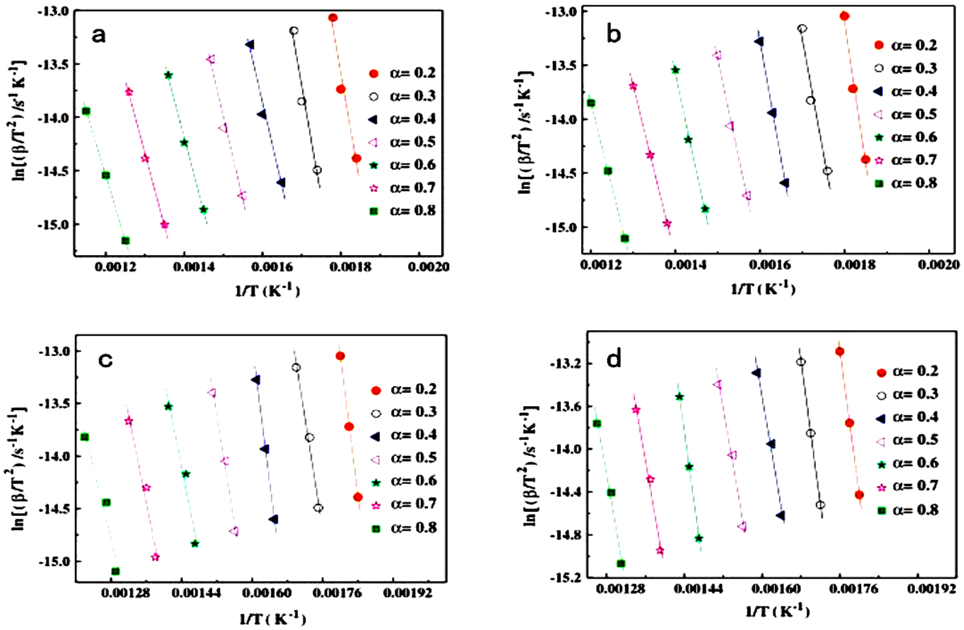

Figure 4*: Iso-conversional plot of KAS method at different α values of conversion under O_2/N_2 atmosphere(a) :11%O2/89%N_2 ,(b): 21%O_2/79%N_2 , (c) 42%O_2/58%N_2 and (d): 63%O_2/37%N_2*

***Table 5**: Shows The Values of The Apparent Activation Energy (E) And Correlation Coefficients (R) Calculated By KAS And FWO Methods*

Atmosphere	α	KAS Method		FWO Method	
		E(KJ/mol)	R	E(KJ/mol)	R
$11\%O_2/89\%\ N_2$	0.2	175.35	0.9604	167.73	0.96546
	0.3	173.07	0.9608	154.89	0.99228
	0.4	132.58	0.9775	144.82	0.97863
	0.5	129.07	0.9783	134.35	0.98534
	0.6	116.80	0.9957	126.26	0.98902
	0.7	115.08	0.9963	118.37	0.97247
	0.8	102.00	0.9990	109.13	0.96990
	Average	**134.85**	**0.9811**	**136.50**	**0.97890**
$21\%\ O_2/79\%\ N_2$	0.2	218.22	0.9853	218.49	0.98316
	0.3	176.35	0.9624	179.52	0.98753
	0.4	181.58	0.9999	186.46	0.98984
	0.5	153.89	0.9921	158.26	0.99132
	0.6	151.12	0.9928	158.26	0.99137
	0.7	132.52	0.9996	136.09	0.99475
	0.8	130.19	0.9995	134.53	0.99953
	Average	**163.41**	**0.9902**	**167.37**	**0.99102**
$42\%\ O_2/58\%\ N_2$	0.2	279.26	0.9995	283.57	0.99835
	0.3	219.70	0.9867	219.85	0.98531
	0.4	275.33	0.9998	274.07	0.99146
	0.5	215.13	0.9926	206.00	0.98328
	0.6	174.49	0.9962	206.00	0.98325
	0.7	172.29	0.9927	177.14	0.98009
	0.8	142.14	0.9995	148.86	0.97894
	Average	**211.19**	**0.9952**	**216.49**	**0.98573**
$63\%\ O_2/37\%\ N_2$	0.2	278.25	0.9998	273.44	0.99999
	0.3	277.32	0.9998	242.26	0.99841
	0.4	218.85	0.9958	236.12	0.99294
	0.5	217.19	0.9921	216.85	0.99008
	0.6	274.44	0.9895	209.00	0.98846
	0.7	215.32	0.9979	201.57	0.98753
	0.8	214.91	0.9898	198.48	0.98687
	Average	**242.32**	**0.9949**	**225.38**	**0.99198**

The average values of activation Energy are 134.85; 163.41; 211.19 and 242.32kJ/mol at different oxygen concentration 11; 21; 42; 63 vol %, respectively. The average values of activation energy calculated bythe KAS methodwere:134.85; 163.41; 211.19 and 242.32 KJ/mol and the values determined by KWO methods were 136.50;167.37;216.49 and 225.38 KJ/mol for the oxygen concentration of;11, 21, 42, 63 vol.% respectively. The activation energy value determined by FWO and KAS methods at constant conversion α are quite similar (Table 5), however, these value are lower than those reported by Aboulkas[35]and by Bai [10].The activation energy of pyrolysis and combustion of oil shale was affected by conversion of organic matter. As oxygen concentration increases, heat released from the volatile oxidation increase then further increased the temperature. The oxygen concentration increased the organic matter oxidization as such, the temperature also increased. The model and parameters were credible [32] because the correlation coefficients were greater than 0.99 (Table 5) and the activation energy of oil shale is directly related to the heating rate and oxygen concentration.

5. Conclusions:

The thermal decomposition and kinetic behavior of oil shale has been studied under different N_2/O_2 mixture of gas of atmospheres (11, 21, 42, 63 vol % oxygen) and different heating rates (10, 30 and 50 °C/min) by non-isothermal thermogravimetric (TG) technique.

The TG curves showed three stages which could be distinguished during the combustion of oil shale: the first stage corresponding to evaporation and the second

stage is the decomposition of bitumen, and kerogenon this stage the rate of weight loss attained is maximum.
Increasing the oxygen concentration: decrease the final combustion temperature, increased the mass loss and the residue char mass decreased. Increasing the heating rate increased the mass loss rate at different atmosphere. The kinetic parameters of decomposition were estimated by iso-conversional methods Kissinger-Akahira–Sunose (KAS) and Flynn–Wall-Ozawa (FWO) methods. The results show that the average activation energy at different oxygen concentration (11, 21, 42, 63 vol%)was calculated as 123.28, 159.78 and 241.69 KJ/mol respectively from KAS method and 199 kJ/mol, 298.9 calculated from FWO method corresponding to the fraction conversion α ranging from 20% to 80%.It can noted that the increasing the oxygen concentration increased the value of activation energy for both method. The oxygen concentration in the atmosphere strongly influences the kinetic parameters. For the decomposition of kerogen and bitumen, the activation energy increases when the oxygen concentration increases. For evaporation and the char oxidation, the activation energy does not depend on oxygen concentration.

The present study may be helpful in understanding the thermal decomposition kinetics of oil shale under different atmospheres. Optimal oxygen concentration was 42vol % for the combustion of oil shale.

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