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Evaluation of hydrocarbon generation potential of source rocks by Thermogravimetric and Differential Scanning Calorimetry

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Abstract

Understanding the association of organic matter and minerals in petroleum source rocks is a vital component of the hydrocarbon-generation process, especially in unconventional reservoirs, which are the targets of hydrocarbon exploration today. In this study, five source rock samples selected from three wells namely, Chalbi-3, Sirius-1, and Ndovu-1wells from the Anza basin, were investigated to characterise the thermal decomposition process using differential scanning calorimetry (DSC) and thermogravimetry (TG/DTG) with a heating rate of 10 °C min^{-1} in an oxidizing atmosphere. The Anza basin is a sedimentary basin located in the north-eastern part of Kenya. The source rocks displayed variable degrees of organic richness, with TOC contents ranging from 0.54 to 1.99 wt. %, at subsurface depths ranging from 1067–3100 m. The Ndovu-1 (H) sample had the highest TOC value, while Sirius-1 (F) had the lowest. In all the analyses, the TG/DSC curves allowed three distinct decomposition regions of organic matter and inorganic matter decomposition identified as i) low-temperature oxidation of bitumen (temperature ranging from 257 °C to 350 ° C), ii) thermo-oxidation of the kerogen (between 430 and 560 °C), and iii) high-temperature decomposition of calcite mineral (between 723 and 747 °C), and clay minerals in the temperature range 907 and 923 °C. There was also a strong correlation between the mass loss and combustion of organic matter in the source rocks.

Keywords: source rocks, TG/DTG/DSC analysis, Thermo-oxidative decomposition, Anza basin

1. Introduction

Since the beginning of the industrial revolution, global energy consumption has been growing steadily, and it is expected to grow by 41 % from 2012 until 2035, according to BP Outlook 2035 presented in January 2014 (Rivera-González *et al.*, 2020). Advancements in the petroleum industry and continuously rising energy demand have extensively attracted the attention of researchers toward the exploration of both conventional and unconventional hydrocarbon resources (Khan *et al.*, 2022). Even though energy can be generated from various resources, the most significant resource remains fossil fuels: including coals and hydrocarbons that can be derived from source rocks and used as alternative energy (Askari & Krichene, 2010). Other Alternative energy sources include oil shale, tar sand, extra heavy oil, shale gas, synthetic fuels from coal, and nuclear energy.

Source rock is a sedimentary rock containing significant amounts of kerogen and smaller amounts of bitumen, and is a potential reservoir for most of the conventional oils or hydrocarbons that can release petroleum-like liquid and provide secure access to transportation fuels (Shoieb *et al.*, 2019) (Al-Alla & Nassef, 2015). It consists of kerogen and bitumen (Organic matter), and inorganic matrix like quartz; feldspars; clays-mainly illite and chlorite; carbonates, calcite and dolomite, alumina-silicates, and pyrite. The kerogen in the source rock can be converted to petroleum oil through the chemical processes of pyrolysis or can be utilized by direct combustion in an oxidising environment. However, (Labus & Matyasik, 2019) noted that the main problem encountered during the analysis of organics is that organic matter is dispersed within the rock matrix, which makes it difficult to examine them without taking into consideration the significant influence mineral components of the rock have on kerogen decomposition. Thermogravimetric (TG) and differential scanning calorimetry (DSC) methods are a powerful group of analytical techniques and can provide information on organic matter content and the minerals present in fossil fuels: coal and oil shales (Labus, 2017). Also, the corresponding shapes and peaks of curves obtained during thermal analysis are

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indicators of the composition and structure of the organic matter in the source rock (Labus & Matyasik, 2019). The greatest advantage of thermal methods is in allowing not only concentrated organic matter (OM) but also finely dispersed OM, even as low as 0.25%, to be detected and examined (Yusupova *et al.*, 1999).

Thermogravimetric (TG) analysis has been extensively used to accurately measure the weight loss of oil shale subjected to a temperature raised at a uniform rate (Liu *et al.*, 2015); and has advantages of short experimental time, small error and consistency of conditions in large-scale oil shale retorting processes. TG technique can analyse mass loss or gain of samples due to decomposition, oxidation, or loss of volatiles such as moisture. On the other hand, Differential Scanning Calorimetry (DSC) is a thermal technique in which the heat capacity of a material is measured as a function of temperature and time. The DSC instrument records the temperature and heat flow associated with material transitions, giving quantitative and qualitative data on endothermic and exothermic processes observed during chemical or physical changes. DSC technique has been used for organic matter recognition in oil shales and/or kerogen evaluation in source rocks (Abu El-Rub et al., 2019); in most cases, the kerogen was separated from the rock.

The Anza Basin, located in the extensional arm of the central African rift system in the North-Eastern part of Kenya has attracted much attention due to the richness in hydrocarbon potential of its source rocks (Rop, 2013); (Gilbert *et al.*,2021); (Tiercelin *et al.*, 2012). Therefore, it was necessary to evaluate these petroleum resources and their characteristics in order to address the desired gap in knowledge. Also, a combination of TG/DTG/DSC in a single instrument is fast, though only qualitative information is reported since no calculations were done.

This study aimed to assess the organic matter content (%TOC); thermal maturity levels and hydrocarbon generation potential of five source rock samples from the Anza Basin using combined thermal methods (TG/DSC), under oxidising atmosphere (in air/O_2). Based on the results of this study, it will be possible to have better insight into the quality of the source rocks for hydrocarbon generation potential. Above and beyond economic contributions, the generated information will certainly improve the energy supply conditions in Kenya.

2. Experimental

2.1. Sampling

The examined seven rock samples were provided by the National Oil Corporation of Kenya (NOCK) laboratories in Nairobi. They were collected from three wells namely, Chalbi-3, Sirius-1, and Ndovu-1 at different depth intervals with the potential of production of hydrocarbons listed in Table 1. The source rock samples were crushed and sieved to a particle size less than 0.2 mm and stored in air-tight brown paper bags at room temperature conditions until use. Total Organic Carbon (TOC) as % C, which is generally regarded as an indicator of kerogen and bitumen amounts in the rock, was analysed on a Thermo scientific™ Flash 2000 CHNS/O, at the University of KwaZulu-Natal, South Africa.

2.2. Thermal analyses

The TG/DTG/DSC analyses were performed at the University of KwaZulu-Natal and Durban University of Technology in South Africa. Approximately 10 mg of each powdered source rock sample was heated under atmospheric air/oxygen conditions in the temperature range of 30 to 1000 °C, at a controlled flow rate of 50 ml/min (in the oxidising atmosphere) and a heating rate of 10 °C/min, for TG/DTG/DSC analysis. They were heated up to 900 °C, at heating rates of 10 °C/min, and held at the final treatment temperature for 10 min to complete the oxidation process. The device used in this work provided the possibility to implement all measurements simultaneously under the same experimental conditions. Thus, TG and DSC were able to complement one another to characterise the material studied. After 15 minutes, the TG/DTG/DSC curves were generated. All the source rock samples were treated with 0.1 M HCl before analysis to minimise interferences by the carbonate and sulphate ions.

It should be noted that the TG/DSC apparatus offers an opportunity to switch the gases from N_2 (inert gas) to air or O_2, without altering the sample. If an inert atmosphere is settled, pyrolysis takes place, on the contrary, if the air is settled, oxidation or combustion process occurs. The two processes can, thus, be distinguished.

3. Results and Discussion

3.1. Total Organic Carbon (Organic Richness)

The representative %TOC values for the analysed samples are given in Table 1. Inspection of the data shows that the source rocks displayed variable degrees of organic richness, with TOC contents ranging from 0.54 wt. % to 1.99 wt. %, at subsurface depths ranging from 1067–3100 m. The Ndovu-1 (H) sample has the highest TOC value, while Sirius-1 (F) has the lowest. A majority of the source rock samples contain organic matter with TOC (wt. %) values above the accepted threshold of 0.5 wt. % for potential source rocks. TOC content is a strong indicator of organic richness, which is the ability of a source rock to generate hydrocarbons (Rivera-González *et al.*, 2020). Based on this characteristic alone, these source rocks can very well be able to generate hydrocarbons provided that the internal thickness and the organic matter concentration are high. In the final analysis, the variation of TOC content can be attributed to the combined effects of the environment on deposition, and burial depth of the drilled wells. Based on TOC results, it seems that little or no petroleum has been generated from the source rocks due to their moderate to high TOC values. Alternatively, they might have had higher TOC values and some of it has already been converted to hydrocarbons. This is suggestive of low maturity or early mature source rocks in agreement with the report by (Gilbert *et al*., 2021).

3.2. TG/DSC analysis

The results of the thermo-oxidative behaviour of different source rock samples, under air atmosphere, at 10 °C min^{-1} heating rate are given as TG, DTG, and DSC curves, as a function of temperature or time, and are displayed in Fig. 1

Table 1: Summary of the relationship between depth and %TOC of selected source rock samples from the drilled wells in the Anza basin.

Well/Sample		**Depth Interval of well (m)**	***TOC(wt. %) Mean± SE for n=3**
Chalbi-3			
	B	2819–3100	1.54± 0.053
Sirius-1			
	D	2316	1.46± 0.105
	E	1186–2344	n.a
	F	2082–2231	0.54± 0.056
Ndovu-1			
	H	1067	1.99± 0.150

*%TOC of four acid treated (in 0.1 M HCl) samples; n.a = not applicable

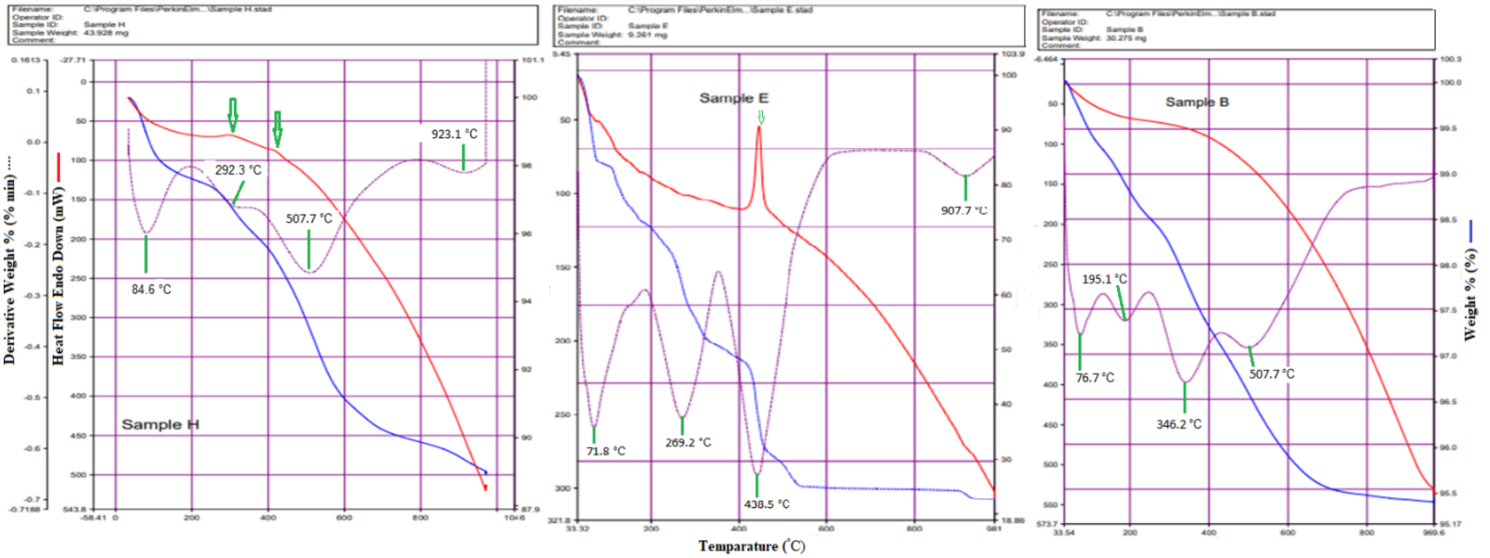

Fig. 1. Reaction regions in TG/DTG/DSC curves of source rock samples H, E, and B (ordered from left to right, respectively) at a heating rate of 10 °C/min. Weight loss in subsequent non-isothermal stages is determined from the TG curve (solid line); whereas, the DTG curve (dashed line) enables the maximum temperature change of the reaction to be determined. *Inset*: The arrow (↓) indicates an exothermic process in DSC.

From Fig.1, it is seen that combustion of the source rock(s) is a three or four-step process: i) evaporation of moisture and release of interlayer water (free and bound water) associated with quartz and clay minerals that are dispersed in the rock; and light fractions of organic matter (30–250 °C); ii) combustion of bitumen (the temperature ranging from 250 to 425 °C); iii) the thermo-oxidation of the kerogen (between 365 and 600 °C). The fourth and final step, between 600 and 1000 °C, is the decomposition of calcite and other minerals/residues and conforms with studies by other researchers; (Moine *et al.*, 2018); (Labus, 2017) and (Foltin *et al.*, 2017).

The observed differences between the reaction regions could be ascribed to differences in carbon and volatile matter contents of source rocks. Also, the combustion or thermo-oxidation of a source rock is a complex process and might involve a series of parallel reactions (Ots, 2009). All of the determined reactions in the examined samples are summarised in Table 2. The observed differences between reaction regions are due to the different carbon and volatile matter contents of the source rocks. Most of these reactions are connected to mass losses which were determined from the TG curves as a function of temperature and are computed in Table 3.

Table 2. Reaction regions of source rock samples (at heating rate of 10 °C/min)

		Reaction Intervals, °C			
Heating Rate	**Source Rock Sample**	**Vaporization of moisture and devolatilization of volatile matter**	**Combustion of light compounds**	**Combustion of heavy compound**	**Decomposition of minerals/ Residues**
10 °C/min	**Chalbi-3**				
	B	30–250	250–410	410–600	
	Sirius-1				
	D	30-200	200-400	400–600	600–850
	F	30–296		296–600	600–800
	E	30–200	200–380	380–600	600–970
	Ndovu-1				
	H	30–250	250–400	400–600	800–990

Table 3. Representative % weight loss of source rock samples from the Anza basin

Sample	**Depth, m**	**†Weight loss (%)**				
		1	**2**	**3**	**4**	**5**
Chalbi-3						
B	2819–3100	0.8	1.0	0.6	nd	2.4
Sirius-1						
D	2317	0.7	nd	nd	5.0	5.7
E	1186–2344	39.8	10.8	nd	0.2	50.8
F	2082–2231	2.3	nd	2.0	2.2	6.5
Ndovu-1						
H	1067	0.7	nd	3.7	0.4	4.8

† **1**—weight loss during TG analysis from 30 to 250 °C (%); **2**—weight loss during TG analysis from 250 to 410 °C (%), **3**—weight loss during TG analysis from 30 to 600 °C (%), **4**—weight loss during TG analysis from 600 to 1000 °C (%); **5**—total weight loss during TG analysis from 30 to 1000 °C (%). nd = not detected.

These results take into account the weight losses in individual temperature ranges or intervals recorded in Table 2 and Fig.1, respectively.

The first peaks observed at temperatures below 100 °C for samples B, D, E F, and H, when subjected to a heating rate of 10.0 °C/min, under air/O_2 atmosphere, as indicated in Fig. 1, resulted in a total mass loss of 0.2–19.8%. The mass loss is attributed to evaporation of moisture and light fractions of organic matter. The second stage took place at a higher temperature in samples H (292.3 °C), E (269.2 °C), B (195.1 °C), and D (257.1 °C). The high temperature values reflect a large amount of energy required to release interlayer water (bound water) associated with quartz and clay minerals that are dispersed in the rock, volatilization of free hydrocarbons and lighter materials in the organic matter as well as CO_2 emissions (Labus & Matyasik, 2019).

Dehydration of clay minerals has been demonstrated to exhibit visible peak maxima in the range of 70 to 250 °C on the DTG curves (Labus, 2017). Likewise, the first segment on the DSC curves, up to about 300 °C displays small endothermic peaks for all the examined samples, confirming dehydration of clay minerals and combustion of light volatile matters dispersed in the source rocks. Accordingly, the H_2O components in the crystalline structure and light organic matter are both decomposed by DSC (Moine *et al.*, 2018); (Labus, 2017) and (Foltin *et al.*, 2017). However, the size of the endothermic DSC peak at the onset appears much smaller than expected because organic matter present in the source rock(s) is replaced by the adsorbed water on the clay minerals (Labus, 2017). The presence of organic molecules on the surface of clay, due to their sorptive properties, makes the surface hydrophobic, leading to the characteristic peak maxima to shift to lower temperatures as was observed here on the TG/ΔTG thermograms (first segment at the onset of DSC curves) on Fig.1.

The third region lies between 300 and 650 °C temperature ranges (Table 2.). Between 400 and 650°C, which is the main temperature range of oxidation of organic matter, most of the weight loss occurred as a result of decomposition of organic structures to CO_2 and H_2O coupled with thermo-oxidation of the heavier fraction of kerogen and fixed carbon in the source rocks (Labus, 2017). Thus, two reaction steps: low-temperature (first peak) and high-temperature (second peak) intervals were observed in samples B (at 346.2 and 507.7 °C), D (at 257.1 and a507.7 °C), E (at 269.2 and 438.5 °C), and H (at 22.3 and 507.7 °C).

The first reaction lying in the low-temperature interval (between 257.1 and 346.1 °C) corresponds to the thermo-oxidation of volatile organic compounds, mainly bitumen. While the second reaction at (438.5–507.7 °C interval) shows the thermo-oxidation of heavier macro–molecules of kerogen plus fixed carbon in the source rocks. The differences observed in temperature for the second reaction phase are brought about by variations in the complex structure of macro-molecules of kerogen, and from the activation energies of the reactions involved brought about by mineral matter (inorganic minerals) catalysis (Shekarifard et al., 2021).

Kerogen is a macro-molecular material (usually insoluble in common organic solvents) that is the main source of TOC in source rocks (Labus & Matyasik, 2019). The inorganic minerals are known to significantly influence the decomposition of kerogen due to ‘in-situ mineral matter catalysis’ (Ariskina *et al.*, 2021). Additionally, in the range 492.3 to 507.7 °C for samples A, B, and C oxidation of pyrite, FeS, (if any), could accompany the decomposition of organic matter; because in XRF analysis sulphur was only detected in sample C (results not reported here). It is, therefore, sensible enough to suggest that the overall reaction taking place in the studied samples is a single-stage thermo-oxidation of the heavier molecular-weight kerogen and fixed carbons in the organic matter.

The fourth and final region lies between 600 and 1000 °C. Most of the weight loss was in the range 0.2–5.0 wt. % loss (mean = 1.56 wt. %) for respective samples A, D, E, F, and H. This was caused by the decomposition of minerals and/or residual materials and was accompanied by episodes of gas evolutions during the oxidation/decomposition of the source rocks. Samples C, D, and F gave a reaction in the temperature range of 723.1–746.7 °C, which was credited to the decomposition of calcite ($CaCO_3$) and or dolomite ($CaMg(CO_3)_2$. This agrees with the work of (Labus & Matyasik, 2019), where calcite decomposition occurred between 541 and 845 °C. But, for samples A, E, and H, the reaction occurred in the temperature range 907.7 and 923.1 °C, and could be explained by decomposition/dehydroxylation of clay minerals such as chlorites($(Mg,Fe,Al)_3(Si,Al)_4O_{10}(OH)_2$), muscovite ($(KF)_2(Al_2O_3)_3(SiO_2)_6(H_2O)$), carbonates (calcite) and residual organic matter present in the source rocks (Labus & Matyasik, 2019); residual organic matter also burnt up to a temperature of almost 956 °C (Dziadzio & Matyasik, 2021).

However, a single-step decomposition process was observed in samples D (at 507.7 °C), F (at 538.5 °C), and H (at 507.7 °C). The rock is a porous medium where oxidation reactions occur, and as such, it has a significant influence on the chemical reactions taking place in the in-situ combustion (Ariskina *et al.*, 2021). The logical explanation, then is that in the temperature range 400 to 600 °C, both medium and high-temperature oxidation reactions merged into a single peak; an effect caused by the minerals in the source rocks. Minerals such as calcite and dolomite tend to bring about the merging of combustion-oxidation reactions into a single-step process (Ariskina *et al.*, 2021). The major reason is that inorganic minerals act as catalysts that lower the activation energies, and because a catalyst accelerates both medium and high-temperature oxidation processes, this results in a single–step reaction to generate coke, CO and CO_2 simultaneously.

Looking at sample E, a more intense exothermic peak was observed at 438.5 °C on the DSC curve (Fig.1) and is characterised by the largest mass loss of 10.8%. This probably could indicate the combustion of aliphatic carbons that are characterised by long chains. This is supported by (Markova & Rustschev, 1991), who stated that mass loss during the combustion of organic matter (under an oxidative atmosphere) occurred up to the temperature of 500 °C. This was related to the presence of light hydrocarbons in bitumen. So, the major organic matter species present in the examined sedimentary source rock (sample E) are light hydrocarbons. What is more, a large weight loss in the temperature ranging from 350 to 450 ° C might indicate the presence of kerogen that is rich in oxygen. For that reason, sample E possibly contains kerogen type III.

However, the combustion of the source rock sample E is more exothermic (more energetic) compared to sample H combustion (Fig. 1). This fact can be semi-quantitatively verified based on the peak heat flow values in DSC curves: 60 *m*W/g for sample E and approximately 2 *m*W/g for sample H, respectively. And hence, is dependent on the quality of the source rock. In an oxidising atmosphere, the more intensive exothermic peak reflects organic matter combustion. Therefore, the release of the large quantity of energy observed in sample E is mainly due to the combustion of heavy volatiles of bitumen and kerogen, which is confirmed by the large area of the peak at 438.5 °C. Identification of the exothermic peaks fundamentally tells us at what temperature the oxidation reactions can be triggered, which in turn, helps one to determine the ignition condition of a fuel.

In this study, the 'quartz peak' at 573 °C signifying the presence of quartz in the source rocks was absent in the temperature 500–550 °C on the DSC curves. Because of the small quartz particle size, it (quartz) was strongly dispersed in the rock, and in turn, failed to yield the expected thermal polymorph inversion. Therefore, it can be concluded that, in this instance, the investigated rocks had a very fine grain structure with little or no detectable quartz (Labus, 2017).

It is worth noting that in all the analysed samples, the highest weight losses were recorded between 300 and 650 °C in TG/DTG analyses, thus, indicating that lower temperature values were necessary for complete decomposition of organic matter in the examined source rocks of the Anza basin.

4. Conclusion

Organic geochemical analyses (TOC wt. % coupled with TG/DTG/DSC measurements were performed on the organic-rich source rock samples in the Anza Basin of the North-Eastern part of Kenya to assess its source rock potential and resulted in the following conclusions:

1) The source rocks drilled from the three wells-Chalbi-3, Sirius-1, and Ndovu-1 had organic-richness ranging from 0.54 wt. % to 1.99 wt. %, at subsurface depths ranging from 1067–3100. These source rock samples were found to be rich in hydrocarbon content with a high potential to generate hydrocarbons.
2) Thermo-oxidative decomposition of the source rocks occurred in three steps: (1) the thermo-oxidation of bitumen (according to the heating rate ranging from 260 ° C to 401 ° C), (2) the thermo-oxidation of the kerogen (between 365 and 560 ° C), and (3) finally, the decomposition of calcite mineral (between 541° C and 815 ° C).
3) It can also be concluded that thermo-oxidative destruction of organic matter starts in the temperature region of 130-300 °C and ends at 400–600 °C.
4) The representative rock samples from the Anza Basin have shown good potential to produce liquid hydrocarbons since the Anza Basin has fair to excellent source rock intervals.
5) Due to their moderately high TOC values, it is suggestive of low maturity or early mature source rocks capable of generating gas and liquid hydrocarbons.
6) Overall, the Anza basin formation is thermally immature and can generate liquid hydrocarbons but largely can act as a source for gaseous hydrocarbons.
7) Some reactions represented not only oxidation of organic matter but also mineral matter catalysis that assisted thermo-oxidation of kerogen (hydrocarbons); especially in the temperature range 300–600 °C, which were assigned to thermos-reactive minerals such as carbonates (e.g. calcite), dolomite and sulphates (e.g. pyrite) in the sample.

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