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# Source Rock Analysis Related with the Change of Organic Substances using SEM and TGA Method

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**Abstract.** Several preliminary studies on oil shale have motivated researchers to find out several methods and parameters that underlie the process of converting oil shale into fuel. This process requires several appropriate parameters to react to physical and chemical changes as desired. One related to organic maturity in oil and shale is Tmax, the maximum temperature to release hydrocarbon from the cracking process of oil shale during pyrolysis. Tmax value is used to determine the maturity level of oil shale turning into oil fluid. The time needed to obtain the Tmax value and the character of this converting is influenced by lithology. This study aims to determine the effect of lithology on Tmax in oil shale. This study begins by selecting the type of oil shale according to the purpose, clay, and nature carbonate at a certain depth. After obtaining several oil shale samples, characterization and several tests were carried out using SEM and TGA tools. By knowing the value of Tmax, type of carbonate oil shale, and clay in oil shale, the conversion of oil shale into oil in terms of temperature control can be determined as needed, so there are no errors during the heating process. Something that greatly affects the maximum temperature of oil shale (Tmax) is lithology. Clay is more influential than carbonate and will speed up the change from oil shale to oil and natural gas.

**Keywords:** oil shale, Tmax, SEM, and TGA.

## INTRODUCTION

In the context of new energy exploration, oil shale is a renewable energy that is expected to be used as a future alternative energy [1]. Oil shale is a clay or carbonate material containing immature organics; when heated to a certain temperature, it can produce energy substances such as oil and gas [2,3]. Currently, oil shale is an energy source that is being developed to be used as alternative energy in the future. Kogerman [4] mentions that oil shale research became a research center in the Soviet Union. The establishment of this research institute saw the development of research in oil shale quite rapidly.

The discovery of oil shale by the United States is considered the cause of the shock in world oil prices. The United States, which was originally only a consumer of crude oil, is now a producer. The latest analysis also shows that shale gas could provide up to half of the US gas supply by 2020. Oil shale reserves in Indonesia have begun to be mapped at 53 locations in Indonesia.

In response to this, Indonesian researchers began to research these new energy sources to help overcome the oil and gas crisis. In this study, researchers are trying to find new theories and methods in processing oil shale to be used as the basis for converting oil shale into fuel oil or gas. The theoretical basis used is the level of maturation of hydrocarbons. The temperature required for converting oil shale into oil and gas is called Tmax. This value is influenced by oil shale lithology; therefore, it is necessary to analyze these parameters. The specific objectives of this study were: To analyze the effect of lithology on Tmax in oil shale.

Oil shale is clay or carbonate material that contains organic matter, an energy source that produces oil and gas [3, 5]. Oil shale processing residue is also valuable for agriculture and industry [6-8]. Kogerman [4] mentions that oil shale research became a research center in the Soviet Union. Berraja et al. [9] initiated a study on thermal analysis studies on oil shale combustion at Tarfaya. Although the method used is not efficient, the research resulted in a heating theory of the pyrolysis method.

Research by Bartis et al. [10] stated that exploitation of oil shale that has been collected is sent to a processing site by burning the shale directly to be used as a source of electrical energy. Bartis also performs underground oil shale mining using the chamber and pillar method. Then Burnham et al. [11] extracted the results of oil shale processing, which was carried out above ground, even though it was underground *in-situ*.

Determination of temperature on pyrolysis to determine the maturity level and reaction rate analysis [12]. Determination of temperature at the maturation stage and reaction speed from TGA analysis refers to several research results: Katarzyna et al. [13], Himawanto et al. [14], Himawanto [15], Riyanto [16], Emam [17], Marnoto, and Sulistyowati [18], Yan et al. [19], Sugondo [20], Sukma [21], Malika et al. [22], Martono et al. [23], Suyitno [24], Kholisoh [25], Minarsih [26], Jiang et al. [27].

The urgency of this research is that oil shale gets attention as a new energy source because the price of a conventional oil is increasing, and its quantity is limited. Indonesia has many new energy sources, oil shale, and has begun to be mapped. The development of research for alternative energy is expected to be used commercially as a fuel and as a producer of electrical energy and is relatively cheap for industry and human needs.

This research is a theoretical technology developed to be applied experimentally analytically. This research will produce new theories and methods to convert oil shale to fuel oil or gas, which is expected to help overcome the oil and gas crisis.

# METHOD

## Sites and Equipment

The equipment used is glassware for preparation purposes and rock cutting equipment available at the Chemistry Lab, FMIPA Unila, and Lemigas Jakarta. TGA analysis was carried out at the Biomass Chemistry Laboratory, FMIPA Unila. SEM analysis to determine the concentration of oxide species/characteristics was carried out at the Chemical Biomass Laboratory, FMIPA Unila, and Lemigas Jakarta.

## Materials

1. Nature clay (kaolinite/illite) and carbonate ($CaCO_3$), two have characteristics as the vessel according to the purpose, obtained from the coring at a certain depth in area X.
2. Carbonate and clay oil shale (good quality).

## Design

This research activity will be carried out entirely by conducting experiments in the laboratory. The detailed and complete implementation of the research is shown in the Research Flowchart in Fig.1.

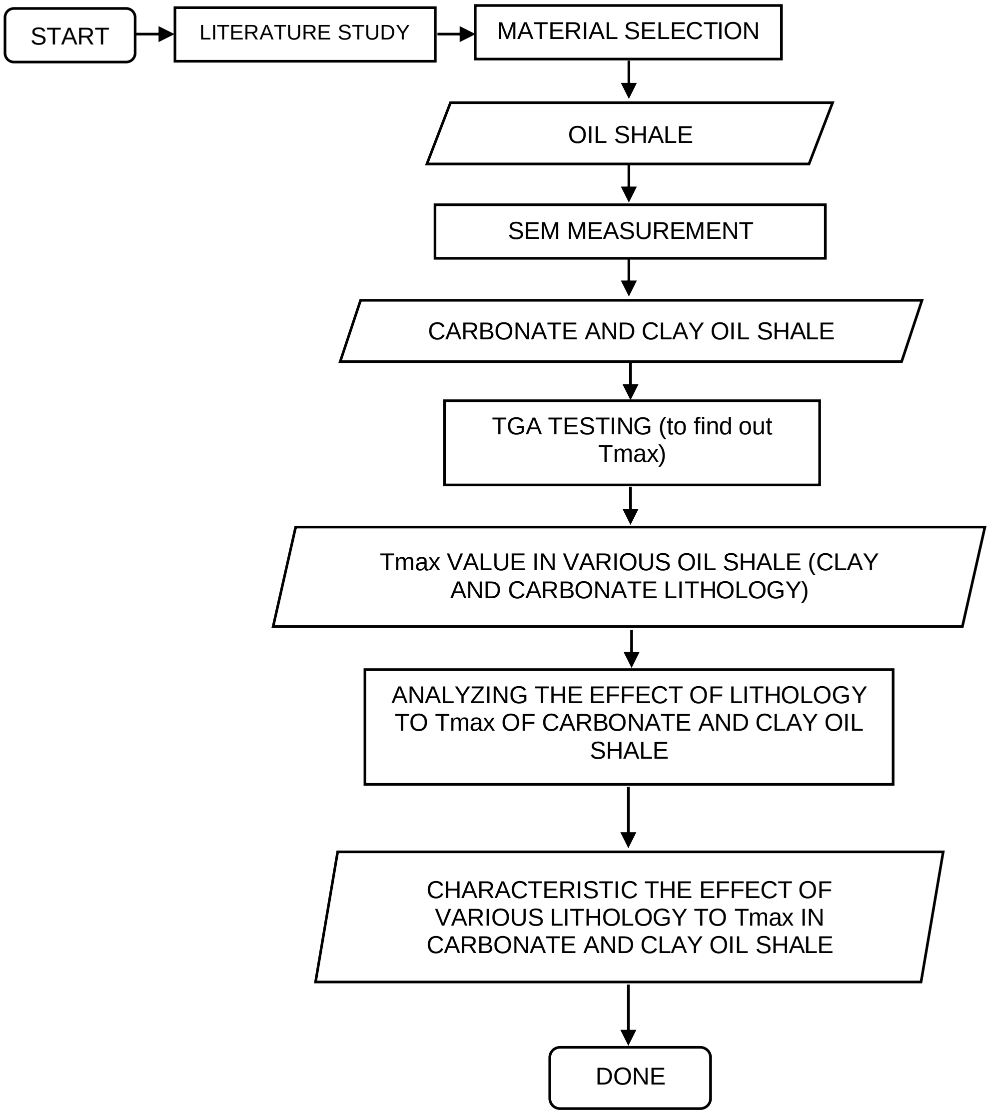
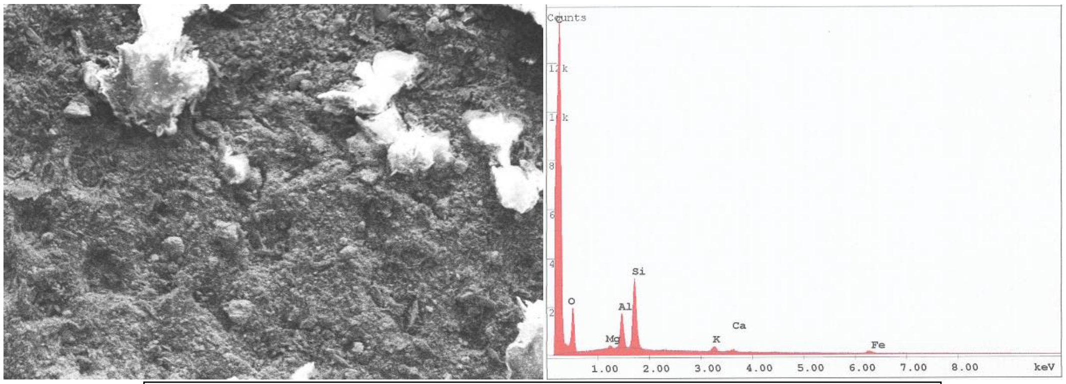



**FIGURE**.1. Research flow chart

## RESULT AND DISCUSSION

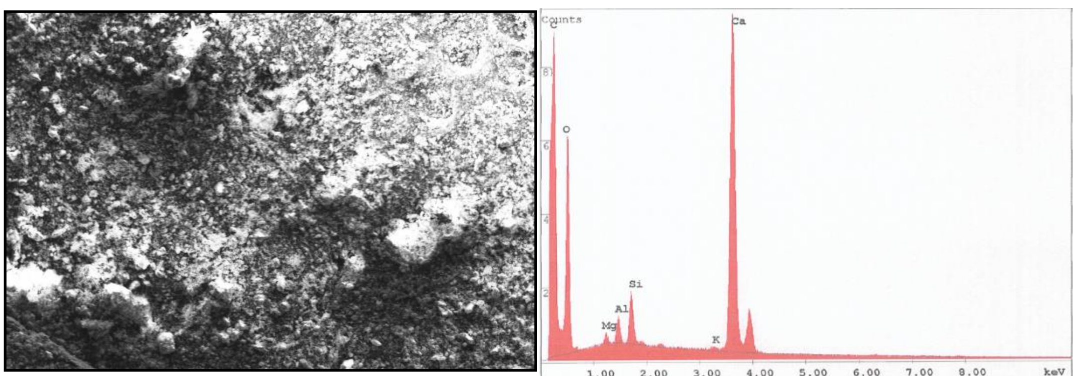
### Analysis Result Using SEM

The shale material that has been selected is then characterized using SEM. The aim is to determine the morphology, particle size, material content, material pores, and the elements contained. The most important characteristic is that the shale material exhibits excellent oil shale properties, according to the reference. Shale material with clay lithology (shown in Fig.2) and shale material with caronic lithology (shown in Fig. 3).




| Element | Wt % | At % | K-Ratio | Z | A | F |
|---|---|---|---|---|---|---|
| C K | 75.28 | 82.41 | 0.3681 | 1.0081 | 0.4849 | 1.0001 |
| O K | 17.24 | 14.17 | 0.0286 | 0.9913 | 0.1675 | 1.0001 |
| MgK | 0.22 | 0.12 | 0.0014 | 0.9516 | 0.6684 | 1.0020 |
| AlK | 2.34 | 1.14 | 0.0174 | 0.9238 | 0.8045 | 1.0023 |
| SiK | 4.08 | 1.91 | 0.0339 | 0.9509 | 0.8728 | 1.0002 |
| K K | 0.40 | 0.13 | 0.0036 | 0.8983 | 1.0176 | 1.0008 |
| CaK | 0.15 | 0.05 | 0.0014 | 0.9200 | 1.0220 | 1.0004 |
| FeK | 0.29 | 0.07 | 0.0025 | 0.8341 | 1.0218 | 1.0000 |
| Total | 100.00 | 100.00 | | | | |

**FIGURE. 2.** SEM photo on clay lithology oil shale




| Element | Wt % | At % | K-Ratio | Z | A | F |
|---|---|---|---|---|---|---|
| C K | 43.65 | 54.94 | 0.2018 | 1.0185 | 0.4538 | 1.0004 |
| O K | 40.81 | 38.57 | 0.0780 | 1.0015 | 0.1908 | 1.0001 |
| MgK | 0.50 | 0.31 | 0.0027 | 0.9612 | 0.5541 | 1.0013 |
| AlK | 1.13 | 0.63 | 0.0074 | 0.9330 | 0.6975 | 1.0020 |
| SiK | 1.84 | 0.99 | 0.0142 | 0.9604 | 0.8030 | 1.0024 |
| K K | 0.29 | 0.11 | 0.0027 | 0.9086 | 1.0100 | 1.0417 |
| CaK | 11.78 | 4.44 | 0.1114 | 0.9304 | 1.0169 | 1.0000 |
| Total | 100.00 | 100.00 | | | | |

**FIGURE. 3.** SEM photo on carbonate lithology oil shale

## Analysis Results Using TGA

Thermogravimetry is a technique for measuring the change in weight of a compound as a function of temperature or time. The result is usually a continuous diagram recording, a one-step decomposition reaction. The sample used, weighing several milligrams, is heated at a constant rate, ranging from 1–2 ºC/min, maintaining its initial weight, Wi, until it decomposes at temperature Ti.

The uses of thermal analysis in solid-state science are many and varied. DTA is generally more valuable than TGA; TGA detects effects involving only mass changes. DTA can also detect this effect and other effects such as polymorphic transitions, which do not involve weight changes. For many problems, it is advantageous to use DTA and TGA because the thermal events detected in DTA can be classified into various processes involving heavy or not involving weight.

Figure 4 shows the weight loss before decreasing at the last temperature, indicating the maximum temperature required for clay shale to start turning into oil shale. So the temperature required for the conversion process of oil shale into crude oil requires a temperature of ± (285ºC-425ºC).

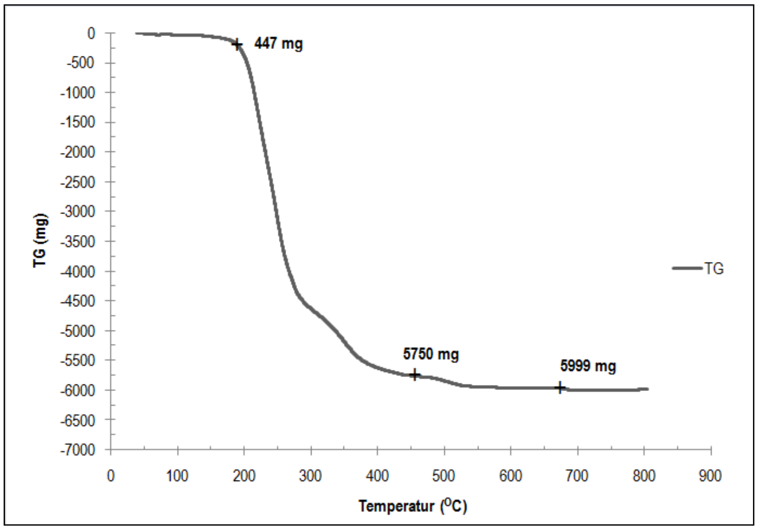



**FIGURE 4.** Graph of Thermogravimetry Analysis (TGA) for clay shale (clay lithology)

From the results of the TGA test on the carbonate shale material, as shown in Fig. 5, it was found that the last phase change occurred at a temperature of around 650OC. So it was slightly higher than the previous phase change in clay shale. Slightly different from clay flakes, the carbonate shale changes four times in weight. So the temperature required for the process of changing (reaction) oil shale material on carbonate shale into oil shale requires an ambient temperature of ± (320°C-455°C).

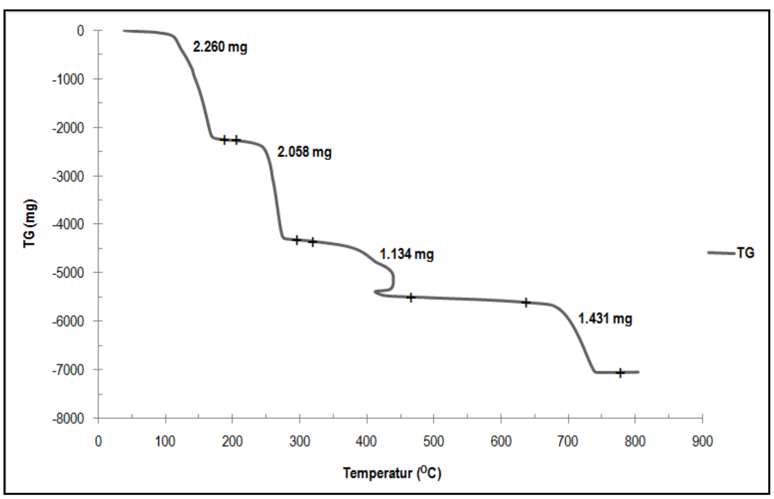



**FIGURE 5.** Graph of Thermogravimetry Analysis (TGA) for clay shale (carbonate lithology)

From the results of the TGA test, oil shale with clay lithology is faster than carbonate which is means lithology greatly affects the maximum temperature of a material (Tmax) and will accelerate the reaction to change from oil shale to oil and natural gas.

## CONCLUSION

Based on the SEM characteristics, there are two types of oil shale a clay and carbonate oil shale. Lithology greatly affects the maximum temperature, especially on clay and carbonate. Clay has a maximum temperature lower than carbonate and will accelerate the reaction from oil shale to oil and natural gas.

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