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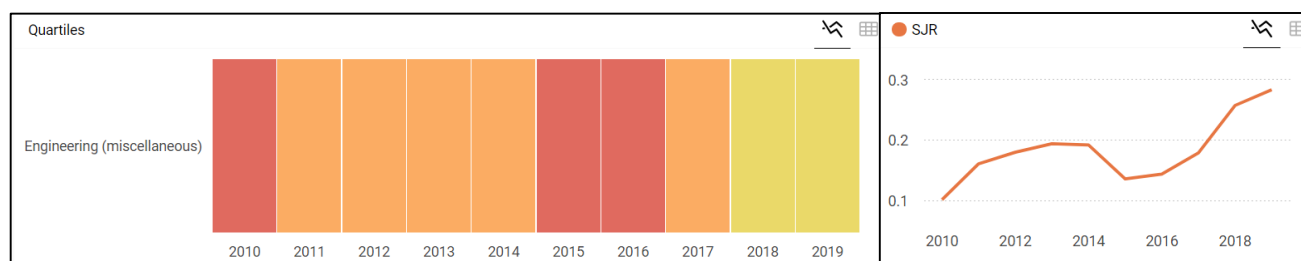
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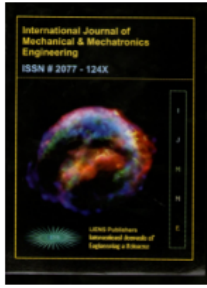
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# Determining the Temperature of Shale Material Conversion Into Crude Oil Based on Organic Clay and Organic Carbonate Test Outside Reservoir

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**Abstract--** The amount of shale material in Indonesia is so plentiful, so it is expected in the next few years there will be available many sources of oil and gas. If we wait the process of shale material changes into oil and gas, it obviously will require a long time. Therefore in this reseach, the process of converting shale material into oil and gas will be carried out in the laboratory. The result of its exploration and exploitation, which is in the form of shale material, will be heated to a certain temperature , the content of its organic material reacts and produces energy which can be utilized as fuel as a substitute of oil and gas. The waste of the process is also very useful for human life. Four shale materials used has a TOC value of  $\geq 12\%$ , which are 12.02%, 12.42%, 12.57% and 12.58%. These values are already qualified as oil shale. The time to change the shale material (organic-clay and organic-carbonate) into oil and gas, is determined by the characteristics of the shale material, which are: immature hydrocarbon substance that has API gravity and boiling point which is close to the boiling point of petroleum. The heating process at temperature of 200°C to 400°C changes the substance into shale material with low boiling point, this is due to its high API degree so it contains alot of light fractions such as gasoline, hence its boiling point is low. The heating process affects the characteristics of flash point of the shale material. The process of change requires a short time around  $\pm 15$  hours, so it is very efficient processing method outside reservoir, although further research need to be done to make it more perfect.

**Index Term--** shale material, TOC, organic clay, organic carbonate, oil shale

## 1. INTRODUCTION

Effort to build laboratory acoustics data to detect the change of organic material properties in the rocks from the result of physics and chemistry properties analysis on reservoir rocks in Indonesia has been conducted by Siswoyo (1995), Subono (1995) and Dewanto et al (2002-2004). According to them, the result of measurement and analysis the change of rock chemical properties in the laboratory arises some methods which support this research. The method generated in the research is to determine the maturity of hydrocarbon and its parameter which are used as indicator to predict the level of change in organic material in the rocks (shale material).

In this research, pyrolysis is used to determine organic content (TOC), maturity of organic material, detecting the amount of oil and gas produced and also used to re-identify

type of some material mixtures. The heating process is carried out with reference to the method of pyrolysis of previous researchers, which are Katz (1983), Berraja et al (1988), Kamtono, Praptisih and Siregar (2005), Heryanto and Hermiyanto (2006), Hidayat and Fatimah (2007), Praptisih, Kamtono, Putra and Hendrizan (2009), Hermiyanto and Ningrum (2009).

Although on the scale of commercial production it will be generated a large enough waste, but apparently according to the theory, the waste can be employed as growing media, road embankment construction materials, cement raw materials. AL-Hasan (2006) has succeeded in doing research on the behavior of concrete made by using oil shale ash and mixture of cement, and the result is incredible, the concrete has a strong and elastic behavior. Another potential of industrial products of oil shale are such as carbon fiber, carbon absorption, black carbon, brick, building decoration, soil fertility, fertilizer and raw material of glass industry. Barkia, Belkbir, and Jayaweera (2004) have conducted a research once again about thermal analysis which was used to determine the effect of heating on oil shale material in Morocco, at temperature of 500°C. A fairly rapid research development, which was originally only conducting combustion without looking at its effect. The result of the research is quite gladdening, encourages the researches to conduct a research development on the shale material, as what has done by Peters, Walters, and Moldowan (2006). Then Al-Hamaiedh, Maaitah, and Mahadin (2010) conducted a research on the result of shale material combined with producing a large amount of ash, then examined the replacement of cement mortar with oil shale ash with a ratio of 10%, 20%, and 30%, which each of them is compared to the strength of mortar cube with pure cement. In 2002, Yoshioka and Ishiwatari conducted a research on micropyrolysis infrared laser system for analyzing organic material in the rock, which was then developed and applied on Green River's shale sample. Peters, Walters, and Moldowan, conducted a research on oil shale on shale.

Due to the different condition and observation scale between condition in the laboratory and in the revoir, so conversion of the result of modeling in the laboratory to reservoir condition should be conducted (Nakayama, 1987), with the help of heat capacity at each depth observed to the

laboratory scale which is calibrated, so it can be considered valid in the process of changing shale material into oil and gas.

According to the result of research from Bartis et al (2005), exploitation of shale material that has been collected is sent to a processing place by burning the shale directly to be utilized as source of electrical energy. Bartis et al (2005) also conducted underground shale material mining by applying room and pillar method. Then Burnham et al (2006) extracted the result of shale material processing, which was done on the ground (ex-situ processing), although there were some new technologies to extract the result of shale material processing under ground at the location or at in-situ processing.

To know hydrocarbon potential, shale material type, as well as the level of thermal maturity, chemical-material analysis is performed, namely organic geochemical analysis which is done by conducting *total organic carbon* analysis and also *Rock-Eval pyrolysis* (Tjahjono, 2004 and Tobing, 2003).

The heating test is begun by furnace, then conducted combustion test. The main result of the first test of organic clay and organic carbonate is obtained within the physical changes (color and phase). The exact temperature value is indispensable to change shale material into a solid material, liquid or gas (carbon substance) with a low boiling point. The changing process is expected to require a short time ( $\pm 20$  hours).

## 2. RESEARCH METHOD

### 2.1 Material Selection

Started by coring activity at each material which has been known. Picture 1 shows the coring on the material type facies A. Some materials used in this research are clay material (illite), carbonate (dolomite) and organic (naftalen).

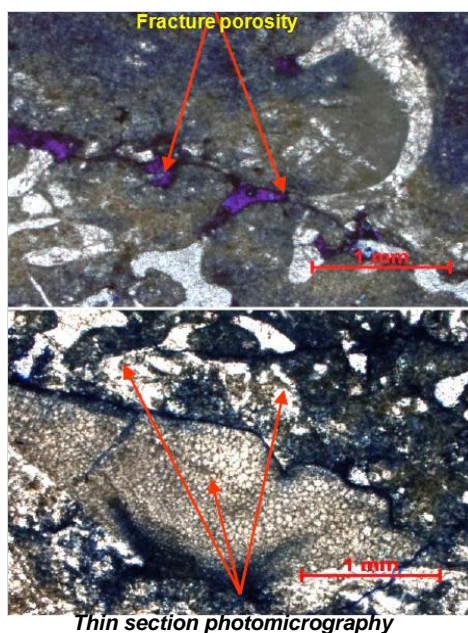


Fig. 1. Type of material taken from the Core Facies A

### 2.2 Shale Material Type Determination

Firstly, prepare the sample of clay and carbonate material which then are smashed using mechanical tool until it turns into rock powder. After that, do the unification process of the size of the rock powder particle radius by using the sieve which has been measured the size of its radius pores, then weigh.

Secondly, the organic material used is taken into a more specific, which is the organic material group of cyclic compound in the form of naphthalene.

Thirdly, material preparation with desired composition is gotten from composition of organic clay and organic carbonate material. Shale material is synthesized with a certain ratio combined with the same grain size (Widjaya, 2012), followed by formation process with pres pressure differently to get material that is ready to be analyzed.

### 2.3 Material Characterization and Test

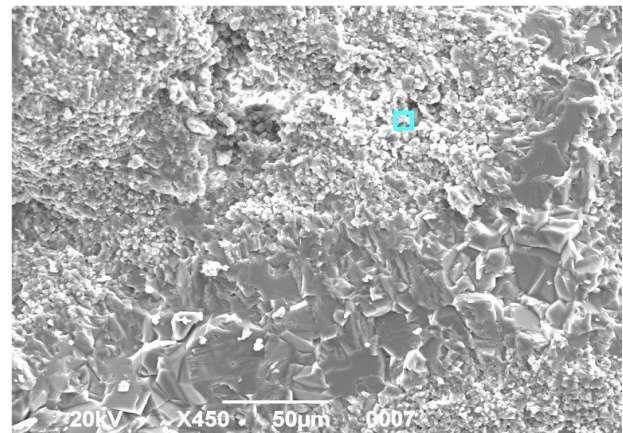
The most important characteristic is shale material that is made, has TOC value  $\geq 12$ , which shows the characteristics of good oil shale used as a reference for comparison. Then to know the distribution of oxide on the surface of the shale, will be analyzed by using X-ray diffraction (XRD). Whereas to know the morphology of the material, it will be analyzed by using SEM analysis. Further, shale material testing is conducted, which is by heating and combustion, so that the temperature of oil shale formation and the change of material into oil and gas can be determined.

## 3. DISCUSSION

### 3.1 Result of Material Characterization

Characterization result of SEM and EDAX shows that the clay material type is illite ( $\text{Si}_7\text{Al})\text{O}_{20}(\text{OH})$ , it can be seen at Figure 2. Clay material always accompanies the reservoir material containing organic material.

At the time of pyrolysis testing, this material will be added with material organic, so it can be known exactly whether this clay material acts as disturber or otherwise assists the maturation process of organic material in reservoir material.



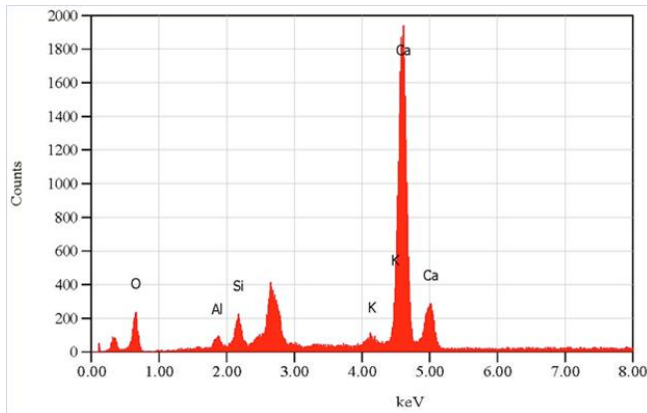
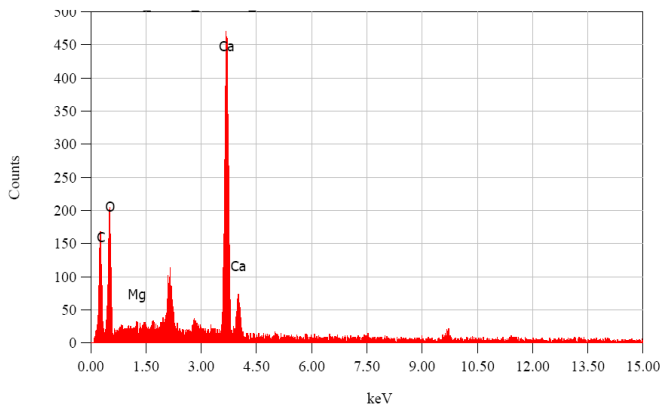
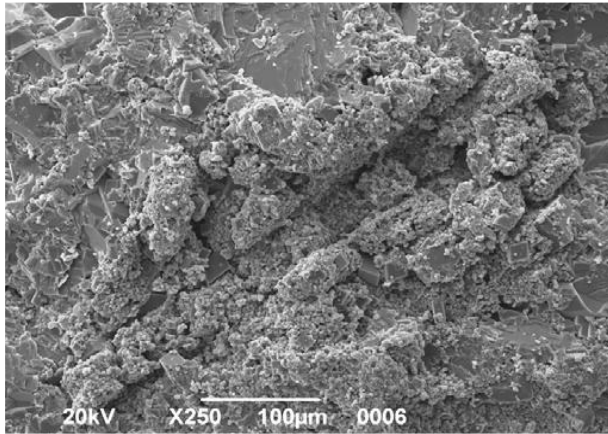


Fig. 2. Clay material type illite

Figure 3 is the result of material characterization which shows carbonate material with the type dolomite [ $\text{CaMg}(\text{CO}_3)_2$ ]. This is supported by the result of characterization using SEM and Edax analysis.

Fig. 3. Material type Dolomite  $\text{CaMg}(\text{CO}_3)_2$ 

### 3.2 TOC Analysis

To know hydrocarbon potential, shale material type, as well as the level of thermal maturity, chemical-material analysis is conducted, that is by organic geochemical analysis in the form of *total organic carbon* (TOC). Material characterized in this research is natural clay that has been put

together with organic material, which is organic material in the form of naphthalene. Then the natural clay is replaced with natural carbonate which is also incorporated with the organic compound, a compound contained in the shale material (oil shale), so that two shale materials are formed (Organic Clay and Organic Carbonate) which have TOC value  $\geq 12\%$ . The comparison of organic material with clay or carbonate will affect the organic carbon content and the maximum temperature. In this research, the material properties of clay and carbonate mass are varied and tested. Before the material is selected, characterization is conducted first by using SEM and XRD analysis. Material in the nature contains a variety of substances, chemical compounds and different characteristics, therefore it is necessary to know the material identity. Table I shows the data of TOC testing result on 4 samples of Organic-Clay and Organic-Carbonate.

Table I  
TOC Data and Pyrolysis

| Sample Name                  | TOC (%) |
|------------------------------|---------|
| Sample-1 (Organic Clay)      | 12,02   |
| Sample-2 (Organic Clay)      | 12,42   |
| Sample-3 (Organic Carbonate) | 12,57   |
| Sample-4 (Organic Carbonate) | 12,86   |

Furthermore, both of shale materials (Organic-Clay and Organic-Carbonate), which have been characterized by using a variety of methods, will produce physical and chemical properties information. Beside that it can be obtained some laboratory test parameters and conceived in detail the mechanism of oil shale reaction model into crude oil.

### 3.3 Organic Clay Material Test

Figure 4 is organic clay material, that is illite and naphthalene mixture, which is ready to be heated.



Fig. 4. Organic clay material (illite+naphthalen)

The heating at temperature of  $300^\circ\text{C}$  produces a color change becomes brighter, cleaner and the form is smoother. Then it also produces fluid in two conditions, the muddy color and the clean one. The fluid is a form of oil shale material (immature hydrocarbon). It is shown at Figure 5.



Fig. 5. The heating of organic clay at  $T=300^{\circ}\text{C}$

Figure 6 shows the heating at  $T=400^{\circ}\text{C}$ , which results in a change of color becomes brighter, cleaner and the form is smoother than the heating at temperature of  $300^{\circ}\text{C}$ . Then it also produces fluid in two conditions, the muddy color and the clean one. The fluid is a form of shale oil material (immature hydrocarbon or crude oil).



Fig. 6. The heating of organic clay at  $T=400^{\circ}\text{C}$

Figure 7 shows the heating at  $T=1000^{\circ}\text{C}$ , which results in a change of color becomes brighter and the form is smoother than the heating at  $400^{\circ}\text{C}$ . Then it is also produced fluid in humid condition which adheres at the wall of glass tube, in a few hours, the liquid will disappear and leave crust (gas).



Fig. 7. The heating of organic clay at  $T=1000^{\circ}\text{C}$

### 3.4 Organic Carbonate Material Test

Figure 8 is a form of carbonate-organic, that is dolomite and naphthalene mixture, which is ready to be heated.



Fig. 8. Carbonate-Organic Material (dolomite+naphthalen)

Figure 9 is the initial heating process at temperature of  $400^{\circ}\text{C}$  which results in a change of color, becomes brighter and soft form. Then it is also produced fluid with muddy color and the clean one. The fluid is a form of oil shale material (immature hydrocarbon).



Fig. 9. The heating of organic carbonate material at temperature of  $400^{\circ}\text{C}$  and  $500^{\circ}\text{C}$

The result of heating at temperature of  $500^{\circ}\text{C}$  is the same as at temperature of  $400^{\circ}\text{C}$ , which results in a change of color, becomes brighter and smoother form. Then it is generated also fluid with muddy color and the clean one. The fluid is a form of oil shale material (immature hydrocarbon).



Fig. 10. The heating of organic carbonate material at temperature of  $900^{\circ}\text{C}$  and  $1000^{\circ}\text{C}$

Figure 10 shows the heating at  $T=900^{\circ}\text{C}$ , which results in a change of color, becomes brighter and more refined form than heating at temperature of  $500^{\circ}\text{C}$ . Then the fluid is produced in humid condition which adheres to the wall of glass tube, in a few hours, the liquid will disappear and leave



the crust (gas). Then the heating at temperature of 1000°C is performed, and the result is the same as the heating at temperature of 900°C.

### 3.5 Combustion Test of Heating Result Organic Clay and Organic Carbonate

Through the heating tests of organic clay material and organic carbonate, all of the tests produce solid material that changes its color and material in a form of fluid. Then the combustion test is performed immediately on the fluid resulted from the heating process. Combustion test is performed by dripping the mixture of the fluid into charcoal (Figure 11). The result is the charcoal will flare up but it takes a long time.

The dry charcoal which has been dripped with the fluid is difficult to flare up. This is because the sample has low API degree, which means it contains a lot of heavy fractions (high density) and thus its boiling point is high.



Fig. 11. Combustion test on organic clay and organic carbonate

Furthermore the heating result of organic clay and organic carbonate material, which is in a form of fluid, is reheated using furnace (Figure 12).

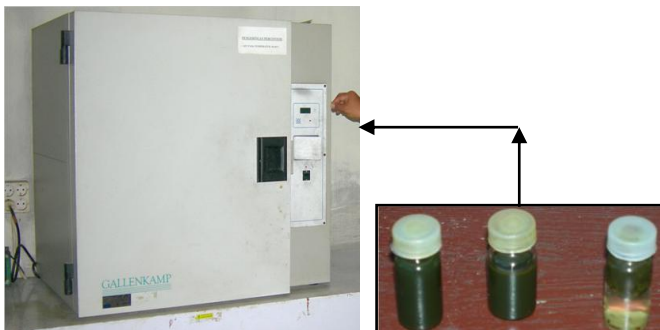


Fig. 12. The heating of organic clay and organic carbonate fluid at temperature of 200°C-400°C

The temperature will affect the fluid, due to the increasing of temperature will change the organic material into hydrocarbon. Figure 13 shows the combustion test on the fluid the result of organic clay and organic carbonate material heating. The result is easy to flare up (such as petroleum), and the flame flares in a quite long time.



Fig. 13. Combustion test on the fluid the result of organic clay and organic carbonate material

Clay or carbonate material also affects on the time and the value of maximum temperature required in the process of the phase change or the release of water molecule and the molecule contained in the shale material. Carbonate material requires greater time and temperature than clay material does (illite and kaolinite).

It can be seen here that the clay material (kaolinite or illite) has a dominant influence on the process of maturation of organic material (by looking at the value of T<sub>max</sub>) and the phase change (from the result of TGA test), compared to carbonate material (calcite or dolomite). Overall the test results can be employed as an indicator of this research aim.

The time to change the immature organic clay and organic carbonate material into oil and gas (energy source), is determined by the characteristics of the shale material, which are: the immature hydrocarbon substance has API gravity and boiling point which is close to petroleum's boiling point; the heating process at temperature of 200°C to 400°C changes the substance into shale material with low boiling point, this is due to the high degree of its API so it contains more light fractions such as gasoline, thus its boiling point is low. The heating process affects the flash point of the shale material. The process of change requires a short time, it is around ±20 hours, so it is an efficient processing method outside the reservoir, although further research need to be conducted to make it more perfect.

## 4. CONCLUSION

Based on the outcome of this research, it can be concluded that:

- 1) The selection of 4 shale materials have been in accordance with the characteristics of oil shale (oil shale) which has good quality with TOC value  $\geq 12\%$ , which are 12.02%, 12.42%, 12.57%, 12.86%.
- 2) The shale material derived from the heating test of organic clay material occurs at temperature of 300-400°C, and for organic carbonate occurs at temperature

of 400-500°C. Meanwhile at temperature of 900-1000°C both materials generate the gas.

- 3) The heating test on the shale oil at temperature of 200<sup>o</sup>-400°C changes the material into shale material with low boiling point, it is due to low degree of its API, so the heating process affects the flash point characteristic of the shale material.

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