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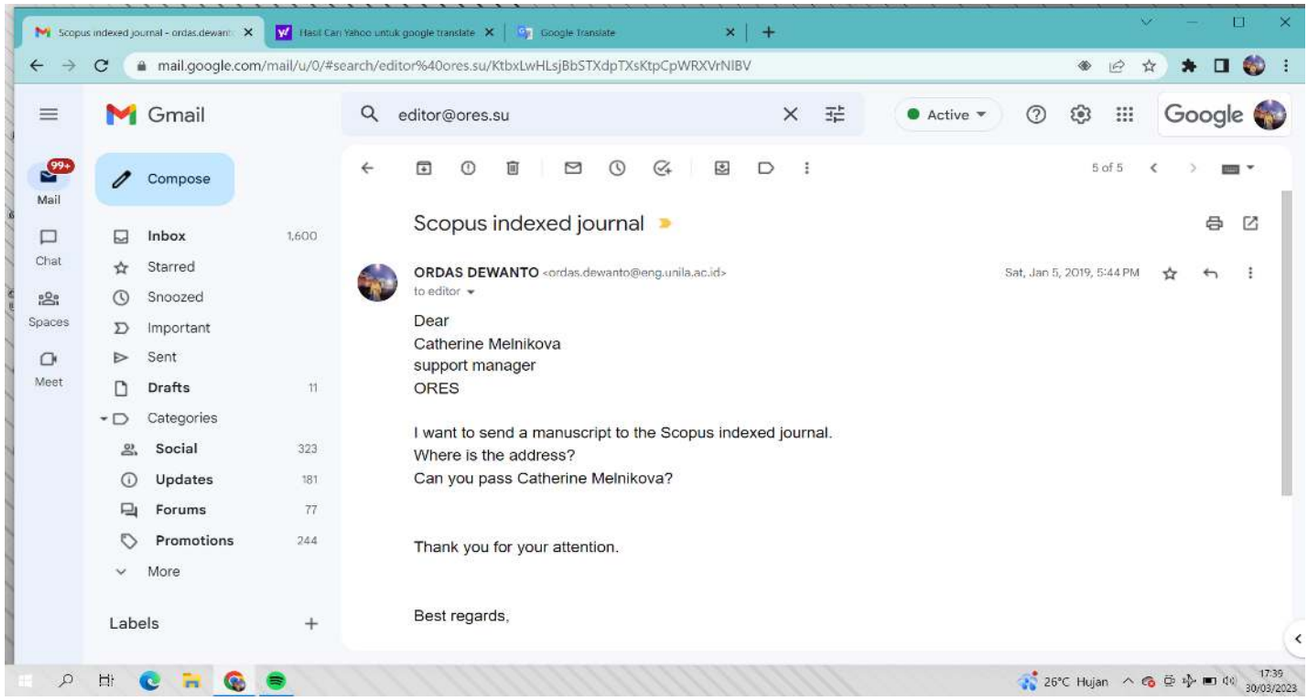

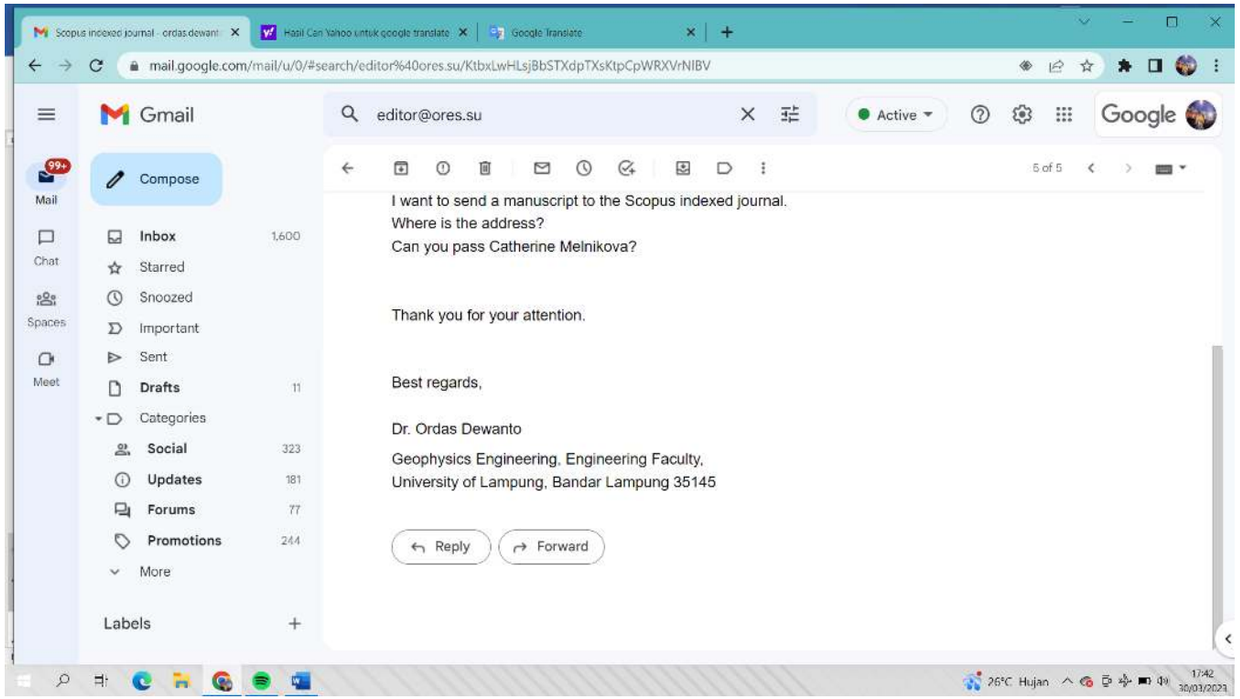
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The Maturity Estimation of Material Organic in $CaCO_3$ with Determining Tmax and Energy Activation Using Pyrolysis Method

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ABSTRACT

Shale material is shale oil that is clay or carbonate material contain excessively immature organic. When heated to a certain temperature, the organic content changed to mature and changed in physics and chemistry, so it can produce energy materials such as oil and gas. The testing of TOC that produced carbonates-organic showed excellent quality as shale oil (TOC≥12.0%). The results of thermogravimetric analysis showed activation energy of carbonates was 749-1339 kJ/mol and the temperature of the reaction process was 75-740^{O}C. Organic composition that was larger than carbonate can caused a smaller activation energy. The carbonate content of OD7-Asl2 had Ea=1083.7 kJ/mol smaller than OD7-Asl1 with Ea=1338.1 kJ/mol. A very large TOC value affected the activation energy to be smaller, as the carbonate of OD7-Asl2 was smaller than OD7-Asl1. The maturity of the OD7-Asl2 shale occurred at T=(380-445)^{O}C, Ea=1083.7 kJ/mol and Tmax=415^{O}C, better than OD7-Asl1. The Rock Eval Pyrolisis test results showed shale carbonate had a high potential to produce oil and gas. Shale material heating result reinforced by FTIR testing that the compounds with specific functional groups apart and a new peak appeared at wavenumber 2900 cm^{-1} which indicate the presence of hydrocarbons single bonds.

Keywords: oil shale, TOC, activation energy, pyrolysis.

1. INTRODUCTION

Oil shale is a kind of clay or carbonate shale material that contains a lot of organic materials, and an energy source that can produce oil and gas (Kantsler and Cook, 1980; Dewanto et. al, 2008). The result of oil shale processing is very useful in the agricultural sector and property industry (Barkia et. al, 2004; AL-Hasan, 2006; Al-Hamaiedh et. al, 2010). A research on the oil shale becomes a main research in Soviet Union (Kogerman, 2001). Berraja, Barkia, Belkbir, and Jayaweera (1988) started the research on thermal analysis study at the combustion of oil shale in Tafaya.

The *Rock-Eval Pyrolysis* method has been initiated by Katz (1983) to analyze the organic material. Bartis et al (2005) did the oil shale exploitation that was gathered and sent to the processing place by burning the oil shale to be utilized as a source of electrical energy. Then Burnham et al (2006) did the extraction on the result of shale material processing which was done on the ground (*ex-situ* processing), and there were some new technologies which carried out the extraction on the shale material underground (*insitu* processing).

The processing of carbonate or clay shale material has not been done in Indonesia, but the reserve of shale material in Indonesia has been mapped. Geological Resource Centre has conducted the research on the oil shale material in 53 locations in Indonesia (Hadiyanto, 2009). The processing of shale material by heating requires some appropriate parameters, so that the changing reaction (maturation) in physics, chemistry and biology can occur in accordance with the desire. Some of the parameters associated with the variation or organic maturity level is the temperature, the energy activation (inversely proportional to the velocity of the reaction) and the material type. In this case, the maturation is defined as maturation of the organic material in the carbonate material, or often referred to shale material or oil shale.

In this research, the production of carbonate shale material is by compounding the organic material and $CaCO_3$. The compounding is done using the way of weight percentage ratio variation, mixing, mixing time

and the last is the result of TOC testing (pyrolysis), where the value of TOC is ≥ 12.0% (Waples, 1985) as a requirement of good oil shale material. The material mixture is modified by the ratio of: organic=calcite and organic>calcite. The TOC testing that produces carbonate shale material shows an excellent quality of oil shale (TOC≥12.0%), which is confirmed by the result of SEM analysis (morphology and composition) and XRD (the interaction of two materials). By determining the level of organic maturation in $CaCO_3$, then the task will be more structured and accurate. Reaction stage-1 is *immature,* which is the immature organic material; reaction stage-2 is *mature-1*, which is the mature organic material or starting to crack material; reaction stage-3 is mature-2 and *over mature-1*, which is the organic material turning into hydrocarbon and some generating the gas; reaction stage-4 the release of all gases.

The reaction stages are closely related to the energy activation (including temperature and reaction velocity), and the type of shale material (carbonate-organic). By knowing the parameter value (from the result of the energy activation) and the type of material, then the shale material processing in term of temperature setting can be determined, so that no error occurs in the heating process.

The *Rock-Eval Pyrolisis* testing is not only used to determine the total organic carbon (TOC), but also to determine the value of Tmax (maximum temperature). Then to determine the maturity of organic material, detect oil and gas reserve and to reidentify the type of some material mixture.

The TGA testing is conducted to determine the value of the energy activation. By conducting a series of tests to obtain a pair of *dY/dt* and T*solid*, so the chart of *ln* (*dY/dt*) with 1/T*solid* can be made. Then the straight-line equation of the chart is searched by using linear regression, so the value of the energy activation can be determined from: $E=-aR$, the value of pre-exponential factor (A) is found when the chart of $y=ax+c$ intersects the y axis or 1/T*solid*=0 (Suyitno, 2009; Indrati et.al, 2000; Rufiati, 2011; Cahyadi et. al, 2011).

The formulation and analysis on the energy activation is based on some previous researchers, namely: Ravindra Pogaku et.al (2012) conducted a research on the energy activation and the velocity pf enzyme-catalyzed reaction. Balloni et. al (1995) conducted a research on the energy activation of SiO_2 which depending on the input power. Tundjung Indrati et.al (2000) conducted a research on the energy activation of pellet (Th,U)Oz at the stage of pellet growth using a dilatometer and Scanning Electrone Microscope (SEM). Cahyadi et. al (2011) conducted a research on the behavior of Indonesia's coal particle ignition using *Thermogravimetric Analysis* at the condition of O_2/N_2 and O_2/CO_2. Sato et. al (2010) conducted a research on the activation free energy that has a dependence on the temperature. The temperature dependence is found greater for the calculation. The determination of the energy activation, pre-eksponensial factor and reaction velocity from TGA analysis refers to several researchers, they are: Katarzyna et. al (2011), Farzuhana and Zakaria (2013), Dwi Aries Himawanto et. al (2011), Nugroho Dewayantoa (2014), Dwi Aries Himawanto (2013), Nukman (2001), Dwi Aries Himawanto et. al (2013), Ahmad Syafiq (2009), Sugeng Riyanto (2009), Any Kurniawati (2012), Eman A. Emam (2013), Tjukup Marnoto and Endang Sulistyowati (2012), Yan and Zhang (2014), Sugondo (2012), Harit Sukma (2012), Malika et. al (2014), Yohanes Martono et. al (2012), Cantrell et. al (2010), Suyitno (2009), Siti Diyar Kholisoh (2011), Tri Minarsih (2011), Longbo Jiang et. al (2014).

Through this research, it is expected that the shale material (Carbonate-Organic) which has been characterized by a variety of methods, can be obtained its physics and chemistry property information to understand the detail of mechanism and the active site on both shale materials, then compare and determine the best one, so that the parameter of laboratorium test can be determined for making the convertion model on the reaction of shale material into crude oil. Further, it is hoped that it can be used as the raw material of the oil shale processing, as the raw material which is excessively available in Indonesia and ready to be used as one of the alternative energy resources.

2. METHOD OF RESEARCH

The Selection of Carbonate and Organic

The stage of determining material type is the most important stage in the material selection. Firstly, choose the material from the

result of coring drilling, then the material is grouped, namely the carbonate material group type calcite ($CaCO_3$). The carbonate material must be completely clean from fluid and natural hydrocarbon, therefore prior to the measurement of porosity and permeability, the material should be in dry condition (dry sample). Secondly, determine the material organic cyclic group, namely salicylic acid ($C_7H_6O_3$).

The Characterization of Carbonate and Organic

The tools used for the characterization of clay and carbonate are SEM and XRD, in order to know the identity of the material. SEM is used to know the type of elements, distribution, topography and the surface shape. The XRD technique can show the type and compound percentage as well as the characteristic of the crystallography.

The Making of Shale Material (Carbonate-Organic) and TOC Testing

The forming method of carbonate shale material is by stirring for a long time, then pressing slowly and leaving to stand for a moment (72 hours) and stirring again and also pressing slowly again, the purpose is to make the organic material fills all pores of the carbonate material. Furthermore, the shale material is left to stand at least 48 hours, the purpose is to make the trapped organic material (fills) the pores and be more binding and cohesive. Then the TOC is tested and made like an oil shale, with the same characteristic, which is TOC≥12%.

The Characterization of Carbonate Shale Material

The carbonate shale material that has been made is characterized by using SEM. The purpose is to know the morphology, the particle size, the content of the material, the pores of the material and the elements. Whereas the characterization using XRD has a purpose to know the compound type, the compound percentage and the crystallography, in addition to know the distance of basal area (d_{001}) from natural $CaCO_3$ that has been mixed with the organic material (salicylic acid).

The TGA Testing on the Carbonate Shale Material

Thermogravimetry is a technique to measure the weight change of a compound as a function of temperature or time. The result is a continuous diagram recording; the schematic single stage decomposition reaction. The two types of main thermal analysis are thermogravimetry analysis, which automatically records the weight change of the sample as a function of temperature or time, and differential thermal analysis (DTA), which measures the difference of T temperature between the sample and the referen inert material as a function of temperature.

The Determination of Temperature and Energy activation

The calculation of the energy activation in this research uses the formula of kinetics calculation of order one reaction or commonly called as *global kinetic*. The determination of energy activation quantity is using graphical method with the formula that is based on Arrhenius equotation. The reason in choosing *global kinetic* method is since this research does not consider the elementery reaction that occurs, but only considers the velocity of shale material in reacting, so it can turn into hydrocarbon. The formulation used in global kinetic is:

$$\frac{dx}{dt} = Ae^{-\frac{Ea}{RT}}(1-x) \quad (1)$$

where;

dx : the loss in mass fraction
dt : the change of time (dt)
A : the pre-eksponential factor
e : the natural number (2,72)
E : the energy activation (J/mol) or Ea
R : the gas constant (8,31 J/mol ^{O}K)
T : the material temperature (^{O}K)

x is the mass fraction, which is calculated by the formula of,

$$x = \frac{m_0 - m}{m_0 - m_f} \quad (2)$$

where m is the mass of the sample when the time is ke-t, m_0 is the initial mass of the sample and m_f is the final mass of the sample. The heating rate is defined,

$$\beta - \frac{dT}{dt} \quad (3)$$

By combining the equotations (1), (2) and (3), so the equotation (1) becomes,

$$\frac{dx}{dT} = \frac{A}{\beta} e^{-\frac{Ea}{RT}} (1-x) \quad (4)$$

$$\frac{dx}{(1-x)} = \frac{A}{\beta} e^{-\frac{Ea}{RT}} dT \quad (5)$$

If both sides are integrated, so the equotation (5) will be,

$$-ln(1-x) = \frac{A}{\beta} \int e^{-\frac{Ea}{RT}} dT \quad (6)$$

In the equaotation (6), the term $\int e^{-\frac{Ea}{RT}} dT$, is an inexact integral but it can be expressed in the *asymptotic series,* so that the equotation (6) can be integrated to be,

$$-ln(1-x) = \frac{ART^2}{\beta E_a} \left(1 - \frac{2RT}{E_a}\right) e^{-\frac{E_a}{RT}} \quad (7)$$

$$-ln\frac{(1-x)}{T^2} = \frac{AR}{\beta E_a} \left(1 - \frac{2RT}{Ea}\right) e^{-\frac{E_a}{RT}} \quad (8)$$

$$ln\left[-\frac{ln(1-x)}{T^2}\right] = ln\left[\frac{AR}{\beta E_a}\left(1 - \frac{2RT}{E_a}\right)\right] - \frac{E_a}{RT} \quad (9)$$

In the fact, the term of:

$$\frac{2RT}{E_a} \ll 1$$

So it can be ignored and the equotation becomes,

$$ln\left[-\frac{ln(1-x)}{T^2}\right] - ln\left(\frac{AR}{\beta E_a}\right) - \frac{E_a}{RT} \quad (10)$$

By making the connection chart between $\ln\left[-\frac{\ln(1-x)}{T^2}\right]$ and $\frac{1}{T}$, the straight line can be obtained where the slope of the line is –Ea/R, so that the value obtained is Ea. The delineation chart of the relationship between $\ln\left[-\frac{\ln(1-x)}{T^2}\right]$ and 1/T heating process as a basis for calculating the energy activation on the shale material heating.

Determining the Maximum Temperature (Tmax) by Pyrolysis Testing

Tmax is the maximum temperature to release the hydrocarbon from the cracking process of the material mixture that occurs during the pyrolysis. Tmax is an indication of organic maturation stage in the shale-carbonate. The Tmax value is one of the geochemical parameters used to determine the maturity level of shale-carbonate. The value Tmax that recorded is influenced by several types of material mixtures mentioned above. Some of the material mixtures will form the different hydrocarbons at the same temperature condition. The Tmax value as an indicator of maturity also has some limitations such as it can not be used for some types of materials that have low TOC.

3. RESULT AND DISCUSSION

Figure 1 shows the description of determination of material type to the processing step in the laboratorium, starting from the coring process, the material selection, the production of carbonate-organic material sample, and also the tests.

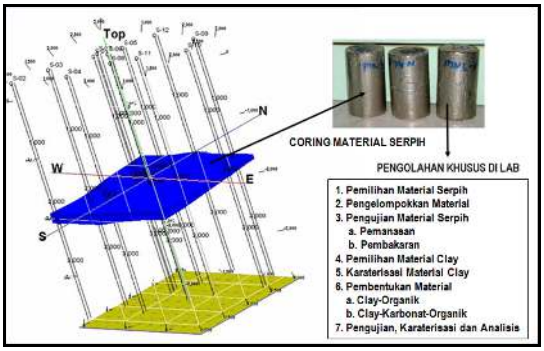


Figure 1.
The coring process, the determination of material type to the processing step in the laboratorium

The Selection of Carbonate and Organic

The carbonate material that has been formed as pellet with a variety of diameter sizes is shown at Figure 2, where the material is already in dry condition.

Figure 2. The carbonate material is formed as pellet

Some of carbonate materials used is the material with the sample number OD7, while the organic material choosen is group of cyclic compound such as salicylic acid.

The Determination of TOC (Total Organic Carbon) and Tmax on Carbonate Shale Material

The result of shale material manufacture ($CaCO_3$- $C_7H_6O_3$) shows a very good result as

oil shale, because it has a value of TOC≥12% and the characteristic is the same as oil shale. The TOC testing is successfully performed on the sample of carbonate-organic material (OD7-Asl). This TOC value is used as one of the parameters for initial selection stage on the material selection in order that can be used as shale material (oil shale), so that the bad and good material can be separated to be the raw material of further processing.

The carbonate-organic shale material has shown the excellent quality, which has a value of TOC≥12.0%. Table 1 shows the value of TOC dan Tmax from the result of pyrolysis that is used as an initial indicator of the thermal maturity level of carbonate-organic shale material. The material maturity shows a varied value, and the carbonate shale of OD7-Asl1 requires a greater temperature than the carbonate shale of OD7-Asl2. The combination between TOC and Tmax indicates that the carbonate shale material that acts as oil shale is likely more potential as oil and gas.

Table 1. The Result of TOC (Total Organic Carbon) Testing

No	Sample Name	TOC (%)	Tmax (^{O}C)
1.	OD7-Asl1 (50% $CaCO_3$ + 50% $C_7H_6O_3$)	12.01	432
2.	OD7-Asl2 (33% $CaCO_3$ + 67% $C_7H_6O_3$)	12.89	415
3.	OD7-Asl3 (67% $CaCO_3$ + 33% $C_7H_6O_3$)	9.14	493

The SEM Analysis on the Organic Material

Figure 3 shows the SEM image and Edax on the salicylic acid ($C_7H_6O_3$) organic material.

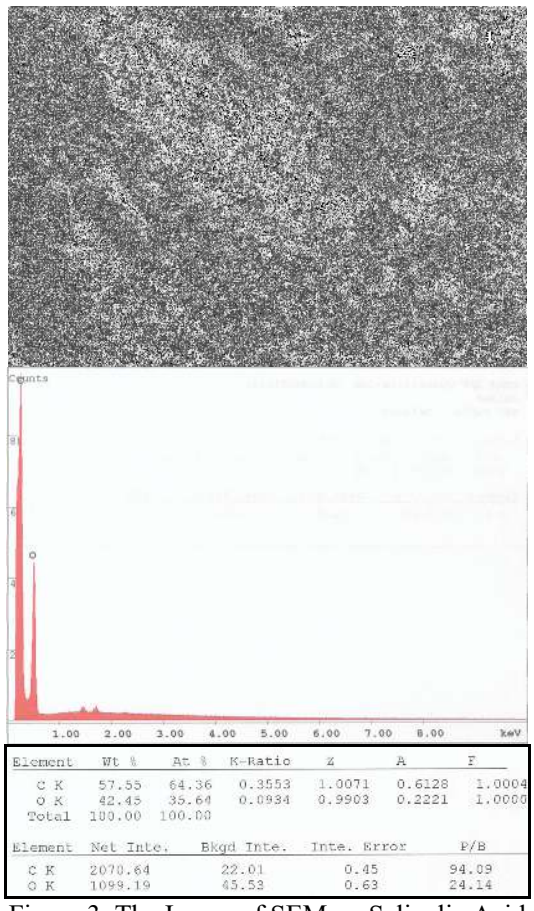


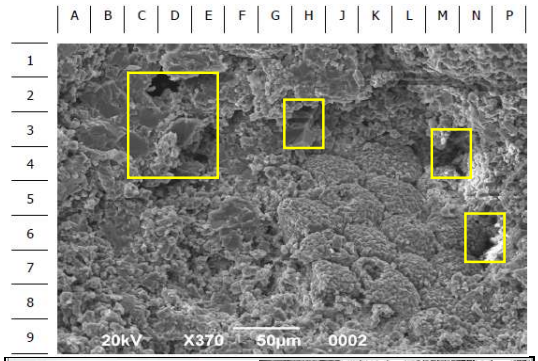
Figure 3. The Image of SEM on Salicylic Acid

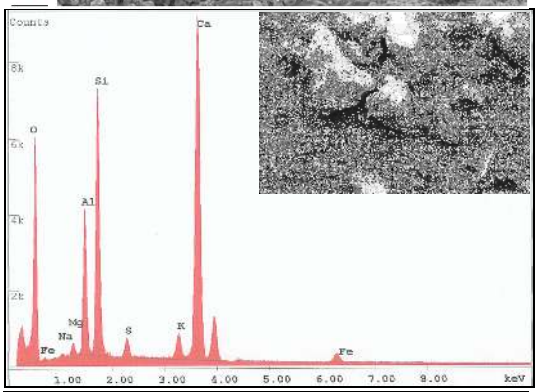
From the result of Edax, the organic material has a dominant content of C and O. The organic material infiltrates into the Kerogen type II which can be formed from some different sources, namely the marine algae, pollen and spore, wax layer of plant, resin fossil, beside that it also derives from the plan fat. This occurs due to the mixing of organic material *autochton* with *allochton* material that dominated by material from plants such as pollen and spore. Its SEM image looks like the bonded white blobs.

The SEM Analysis on the Carbonate Material

Figure 4 shows the result of SEM analysis on the carbonate material (OD7). The carbonate material (OD7) has many pores. Some areas of the pore cavity can be filled with other material (C-E, 2-4). The carbonate material is dominated by *calcite* (D-E, 2-3; B-D, 2-4) and a little dolomite (C-D, 5-6).

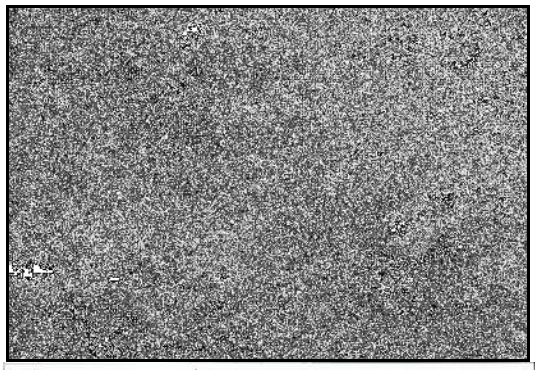
Besides the carbonate material is dominated by the carbonate, it also has little clay material (illite dan kaolinite) that is situated around the pores. The type of the pore is secondary pore, where the distribution of the secondary pore is caused by the dissolution of planktonic (C-D, 2). From the result of Edax SEM on the carbonate $CaCO_3$, it has a great content of Ca and O (dominant).

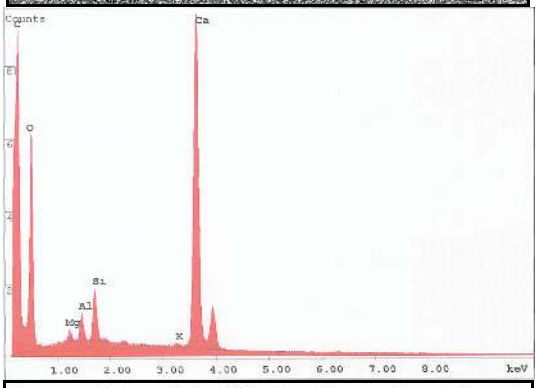




Element	Wt %	At %	K-Ratio	Z	A	F
O K	50.74	67.17	0.1207	1.0291	0.2312	1.0003
NaK	0.31	0.29	0.0011	0.9631	0.3741	1.0032
MgK	0.98	0.85	0.0051	0.9872	0.5257	1.0063
AlK	9.97	7.83	0.0638	0.9582	0.6629	1.0073
SiK	16.29	12.29	0.1114	0.9861	0.6913	1.0029
S K	1.27	0.84	0.0096	0.9716	0.7664	1.0075
K K	1.83	0.99	0.0163	0.9356	0.9277	1.0310
CaK	18.00	9.51	0.1634	0.9577	0.9477	1.0003
FeK	0.61	0.23	0.0052	0.8701	0.9857	1.0000
Total	100.00	100.00				

Figure 4. The result of SEM and Edax OD7 (calcite)





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 5.
The Image of SEM on the material of OD7-Asl2

The SEM Analysis on the Carbonate Shale Material (Carbonate-Organic)

The surface area of the shale material that has been filled by the organic is smaller, so it can be indicated that the place of the carbonate has been filled by the organic, because the pores becomes smaller. The SEM image of the carbonate shale (calcite) has the dominant secondary pores. At a certain time, the porosity will change and cause the organic material exits and enters the pores. The calcite material is success as a place of perfect maturation of organic material.

Figure 5 shows the carbonate shale material OD7-Asl2 (33% $CaCO_3$ and 67% salicylic acid). From the result of Edax SEM on the carbonate shale OD7-Asl2, it contains some elements with (wt, %) dominated by Ca, O and C, as follows Carbon (43.65%), Oxygen (40.81%) and Calcium (11.78%).

The Measurement of X-Ray Diffraction (XRD)

The result of the carbonate OD7 characterization is in the form of diffraction patterns which are the peaks characteristic of crystal $CaCO_3$ structure.

Picture 6 shows the chart of the XRD result on the shale material (carbonate-organic) OD7, OD7-Asl1, OD7-Asl2, and OD7-Asl3. The diffraction patterns of $CaCO_3$ are identified at the angle of 2θ. If the peaks of $CaCO_3$ characteristic appear, so the compound phase can be identified. The X-ray diffraction pattern that formed is the result of atoms scattering that is located on the hkl plane in the crystal. The carbonate OD7 has five high peaks and the intensity will change when there is an organic addition. The angle 2θ position is at the angle of 26.66^O, 29.48^O, 36.05^O, 39.50^O and 43.27^O with the intensity values captured by the X-ray detector are 779 cts, 20383 cts, 3477 cts, 4172

and 3252 cts. The peaks that formed are the peak of crystal Graphite C_1H_2 and Calcite $CaCO_3$ and Caron. The crystal planes or Miller indices hkl are (011), (104), (110), (161) and (202).

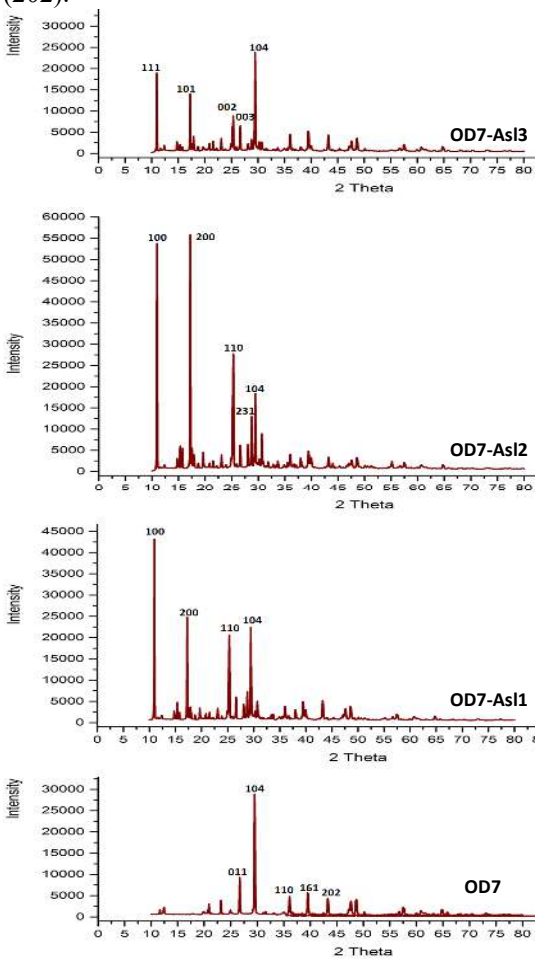


Figure 6.
The XRD characterization on the carbonate and shale material (carbonate-organic)

The carbonate shale material OD7-Asl1 has five highest peaks at the same angle position of 2θ as shale material OD7-Asl2 , which are at the angle of 10.94^O, 17.21^O, 25.23^O, 28.70^O and 29.41^O, with the intensity values captured by the X-ray detector are 47282, 44513, 18466, 8361 and 12199 cts. The peaks that formed are the peak of crystal Carbon Dioxide, Fichtelite $C_{19}H_{34}$, Calcium, Carbon and Calcite $CaCO_3$, and it can be identified that the crystal planes or Miller indices hkl are (100), (200), (110), (231) and (104). The shale material (carbonate-organic) OD7-Asl3 has 5 highest peaks at the position of 2θ, which are 10.96^O, 17.23^O, 25.24^O, 26.61^O and 29.43^O, with the intensity values captured by the X-ray detector are 12771, 13314, 4687 and 18095. From the result, it can be concluded that the peaks that formed are the peak of crystal Fullerite, Nitrammite $N_2H_4O_3$ and Calcite $CaCO_3$, and it can be identified that the crystal planes or Miller indices hkl are (111), (101), (002), (003) and (104).

The addition of organic material ($C_7H_6O_3$) is perfomed on the natural carbonate (OD7) with a particular composition. At the angle of 2theta=29^O with the same hkl plane, the calcite (104) condition experienced on the shale material is the intensity of OD7 ≥ OD7-Asl3 > OD7-Asl1 > OD7-Asl2. While at the angel of 2theta=25^O and the angle of 2theta=17^O, the condition experienced on the carbonate material is the intensity of OD7 ≤ OD7-Asl3 < OD7-Asl1 < OD7-Asl2. The result of XRD characterization on the carbonate shale material which has the highest intensity is shown on the Figure 6 and Table 2.

Table 2. The XRD characterization on the carbonate shale material which has the highest intensity

No.	Material Name	Pos. [°2Th.]	Height [cts]	d-spacing [Å]	hkl	Compound
1.	OD7	29.48	20383	3.03	104	Calcite, $CaCO_3$
2.	OD7-Asl1	10.94	46314	8.08	100	Carbon Dioxide
3.	OD7-Asl2	10.94	47282	8.08	100	Carbon Dioxide
4.	OD7-Asl3	29.43	18095	3.03	104	Calcite, $CaCO_3$

The organic addition causes the *preferred orientation* on the specific crystal planes, it leads the crystal planes having the higher intensity than before. While the angle of 2theta and its hkl plane do not change. Based on the image, it can be seen that the intensity of X-ray that absorbed by the detector on every sample, has different value. High or low theX-ray intensity that captured by the X-ray detector is influenced by the level of the regularity of atom formation in the crystal that is diffracted by X-ray. The more atoms are structured regularly,

the higher the intensity that captured by the detector.

When the intensity is greater means the sample has the greater crystal regularity or more well arranged atoms in the layers. The X-ray diffraction patterns that formed are the result of atoms scattering that located at the hkl plane in the crystal. Any difference or change in the compound and hkl plane at the same angle of 2theta at every organic addition, it means the reaction occurs between $CaCO_3$ material and organic.

The Result of TGA Testing on the Shale Material (Carbonate-Organic)

The test is carried out on the carbonate shale material OD7-Asl1, with the composition of 50% $CaCO_3$ and 50% $C_7H_6O_3$. The result of TGA testing on the shale material OD7-Asl1 is shown on Figure 7. The first change occurs at the temperature of 75^OC-170^OC, this is where the loss of water molecules happens in the crystal strcuture. At the temperature of 225^OC-275^OC, the second weight change that significant occurs, and it is indicated as the change in the structure on the carbonate shale material and the loss of water molecules chemically.

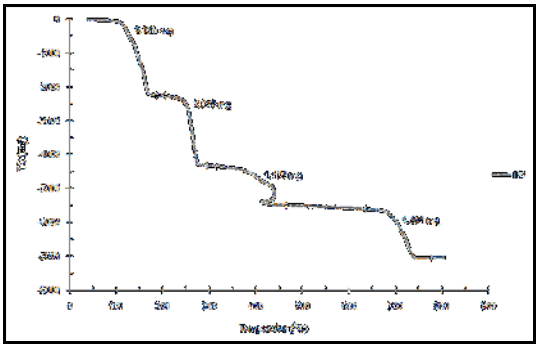


Figure 7.
The chart of Thermogravimetry (TGA) analysis result on the shale material (OD7-Asl1)

When the calcination is reperformed, the weight loss occurs at the temperature of 325^OC-450^OC, then the molecules in the carbonate shale are released, so that it will affect the change on the pore size. The interval temperature of 325^OC-450^OC indicates the maximum temperature that required for carbonate shale to turn into oil. The last change phase occurs at the temperature about 650^OC-740^OC. This condition can be called as over mature, where the chart line has shown a tendency of straight horizontal. Physically, the material that has been calcined at the temperature above 740^OC, the color becomes blackish. So the temperature that required at the process of the change (reaction) of oil shale material on the carbonate shale to be oil is about ± (325^OC-450^OC).

Then the TGA testing is performed on the material OD7-Asl2 (calcite-salicylate), namely the test on the carbonate shale material where the composition is the mixture of 33% $CaCO_3$ and 67% $C_7H_6O_3$. This test is performed to know the structural damage when heating at the high temperature is carried out (over 400^OC), because it can provide the description of the change process in the subtance mass. The result of TGA testing on the shale material OD7-Asl2 is shown on the Figure 8.

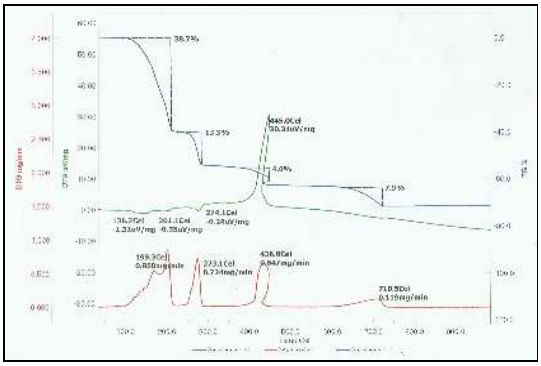


Figure 8.
The chart of Thermogravimetry (TGA) analysis result on the shale material (OD7-Asl2)

The first weight loss occurs at the temperature of ± 95^OC-205^OC, it indicates the water molecules apart from the crystal structure of OD7-Asl2. Then the second weight loss occurs at the temperature around 225^OC-285^OC. The second significant weight change indicates the change in the structure of the material OD7-Asl2 and the loss of water molecules chemically. Then the third weight loss occurs at the temperature of 380^OC-445^OC, where the molecules in OD7-Asl2 is apart. The interval of temperature indicates the maximum temperature that required by OD7-Asl2 (carbonate shale) to begin turning into oil.

When the calcination is always performed to the last loss until becoming constant, which is at the temperature of 610^OC-720^OC, then many molecules in OD7-Asl2 (the carbonate shale) are apart, so that there is part of the shale in the pores is also apart. This

condition can be called as *over mature*, where the line of the chart shows a tendency of straight horizontal along with the increasing of the temperature up to 720^{O}C. Physically, the material that has been calcined at the temperature above 720^{O}C, the color becomes blackish. So the temperature required for the maturation process of the oil shale OD7-Asl2 into crude oil is about ± (380^{O}C-445^{O}C).

The Duration of Heating the Material

The carbonate shale material OD7-Asl2 undergoes the heating process faster than the material of OD7-Asl1.

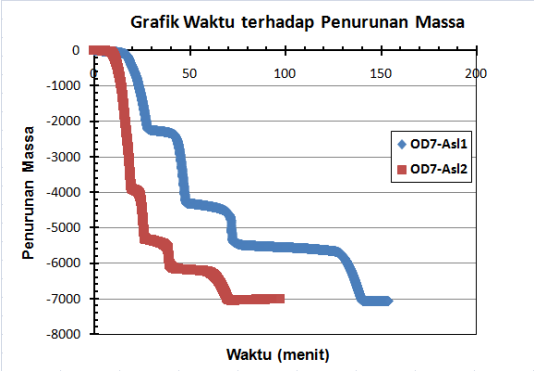


Figure 9.
The chart of mass loss versus time of reaction on the shale material (carbonate-organic)

Figure 9 shows the relationship between the mass loss versus the time on the carbonate shale material. The greater ratio of organic compotition than calcite causes maturation reaction in the heating process becoming faster.

The Energy Activation and Pre Exponential Factor of Shale Material OD7-Asl1

The TGA gradual decomposition process that is used alone or combined with DTA can separate and determine each stage. On this shale material sample (carbonate-organic), the four-level decomposition occurs, it can be seen on Figure 7.

The result of TGA shows that the decomposition level of shale material is divided into four levels of temperature, which are level I is between the temperature of 75^{O}C-170^{O}C, level II occurs between the temperature of 225^{O}C-275^{O}C, level III is between the temperature of 325^{O}C-450^{O}C and level IV occurs between the temperature of 670^{O}C-740^{O}C. Figure 10 shows the method of determining the energy activation (Ea) and the pre exponential factor of OD7-Asl1.

In the process of heating the material OD7-Asl1, the decomposition goes through four stages. The TGA and DTA can be used in a variety of kinetic studies. The fast and accurate TGA method is used to study the decomposition reactions isothermally. This process can be repeated at other temperature and the result is analyzed to determine its energy acivation.

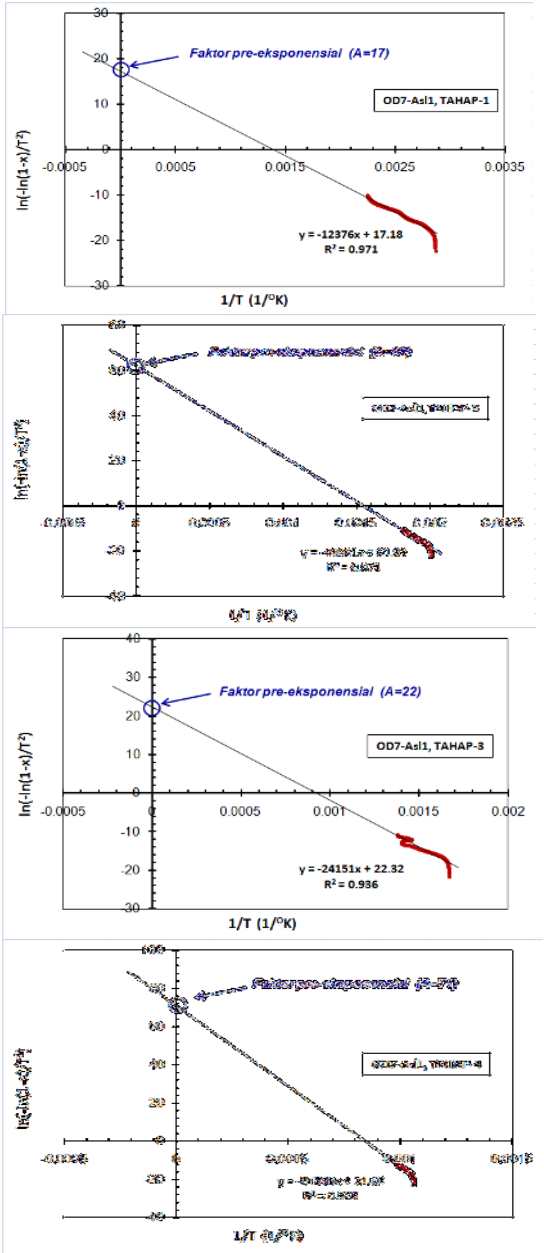


Figure 10.
The chart of ln[-ln(1-x)/T^{2}] vs 1/T, for Ea and A determination on the OD7-Asl1 using TGA

The energy activation (Ea) values and pre exponential factor of each reaction stage at 4 levels reaction are shown in Table 3.

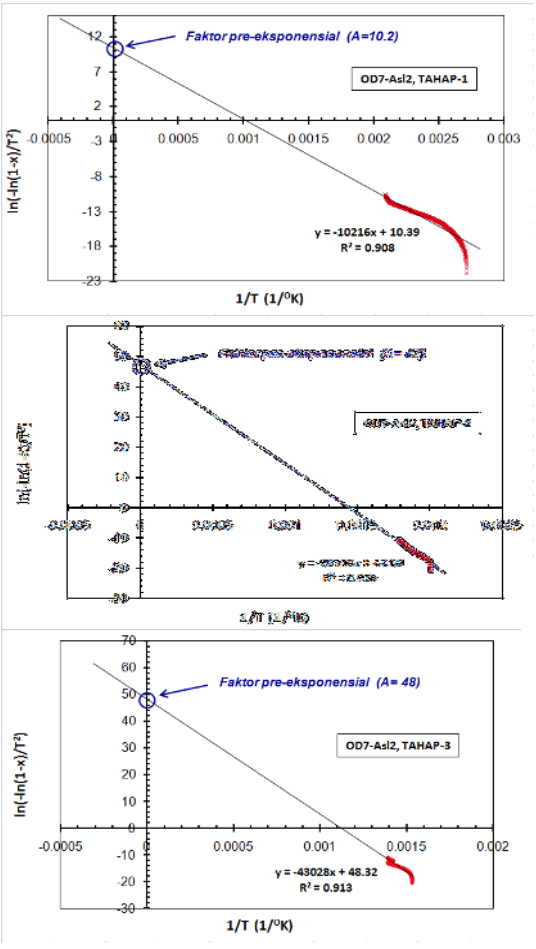
Table 3. The Ea and A Values of OD7-Asl1 Material

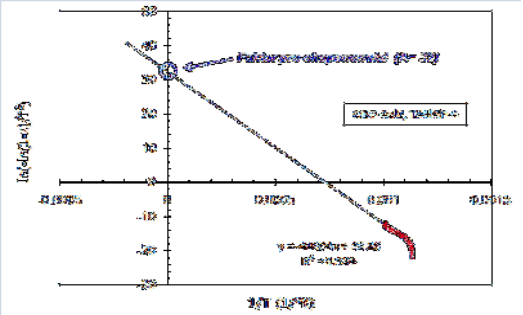
Reaction Level	Ea (kJ/mol)	A (%/s)
75^{O}C-170^{O}C	102.89	17
225^{O}C-275^{O}C	333.32	62
325^{O}C-450^{O}C	200.79	22
670^{O}C-740^{O}C	701.14	71

The total value of the energy activation on the heating of the material OD7-Asl1 is Ea=1338.1 kJ/mol and the pre exponential factor is A=172.

The Energy Activation and Pre Exponensial Factor of Shale Material OD7-Asl2

The gradual decomposition process on the shale material OD7-Asl2 (carbonate-organic) occurs on four levels decomposition, it is shown on the result of TGA testing on Picture 8.





Picture 11.
The chart ln[-ln(1-x)/T^2] vs 1/T, for Ea and A determination on OD7-Asl2 using TGA

The decomposition level of shale material OD7-Asl2, from the result of TGA is divided into four levels of temperature, which are level I is between the temperature of 95^{O}C-205^{O}C, level II occurs between the temperature of 225^{O}C-285^{O}C, level III occurs between the temperature of 380^{O}C-445^{O}C and level IV occurs between the temperature of 610^{O}C-720^{O}C.

The chart of the energy activation (Ea) and pre exponential factor determination of OD7-Asl2 is shown on Picture 11. The total value of the energy activation on this shale material (the organic ratio is greater) has value of Ea=1083.7 kJ/mol, and the pre exponential factor of A=137.2, where the value of Ea and A is smaller than the OD7-Asl1 material, so that the velocity of the reaction is faster. The complete energy activation (Ea) values and pre exponential factors for each reaction stage on 4 levels temperature are shown in Table 4.

Table 4. The Ea and A of OD7-Asl2 Material

Reaction Level	Ea (kJ/mol)	A (%/s)
95^{O}C - 205^{O}C	84.936	10.2
225^{O}C - 285^{O}C	271.93	47
380^{O}C - 445^{O}C	357.73	48
610^{O}C - 720^{O}C	369.09	32

4. CONCLUSION

The energy activation of carbonate shale material is Ea=749-1339 kJ/mol and temperature for the process of carbonate shale material reaction is T=75-740^{O}C.

The composition ratio (wt.%) of organic that is greater than carbonate causes the carbonate shale material (with TOC≥12%) having a lower energy activation. The

carbonate shale material of OD7-Asl2 (33% carbonate + 67% organic) has Ea=1083.7 kJ/mol lower than OD7-Asl1 (50% carbonate + 50% organic) that has Ea=1338.1 kJ/mol.

The TOC value that is so high, influences the energy activation becoming smaller (see number 2), namely carbonate shale material OD7-Asl2 is smaller than OD7-Asl1.

The maturity of shale material OD7-Asl2 occurs at T=(380-445^{O}C, Ea=1083.7 kJ/mol and Tmax=41^{5O}C, it is better than OD7-Asl1 which occurs at T=(325-450^{O}C, Ea=1338.1 kJ/mol and Tmax=43^{2O}C

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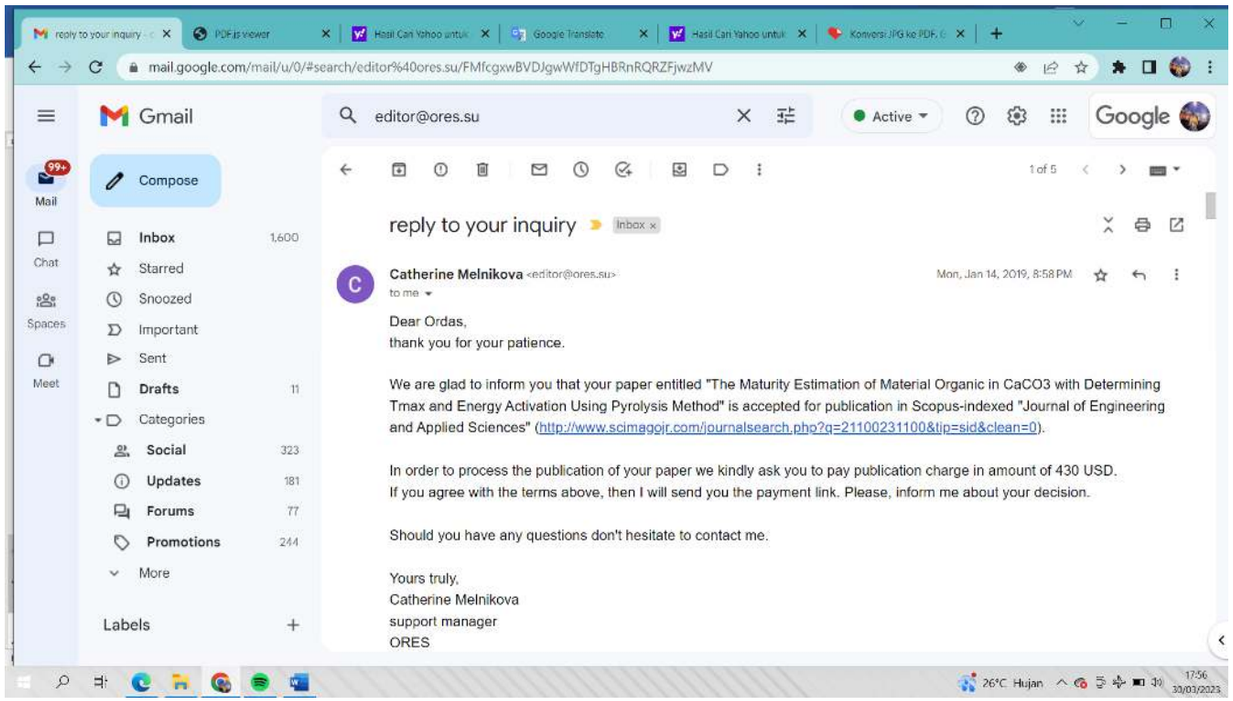

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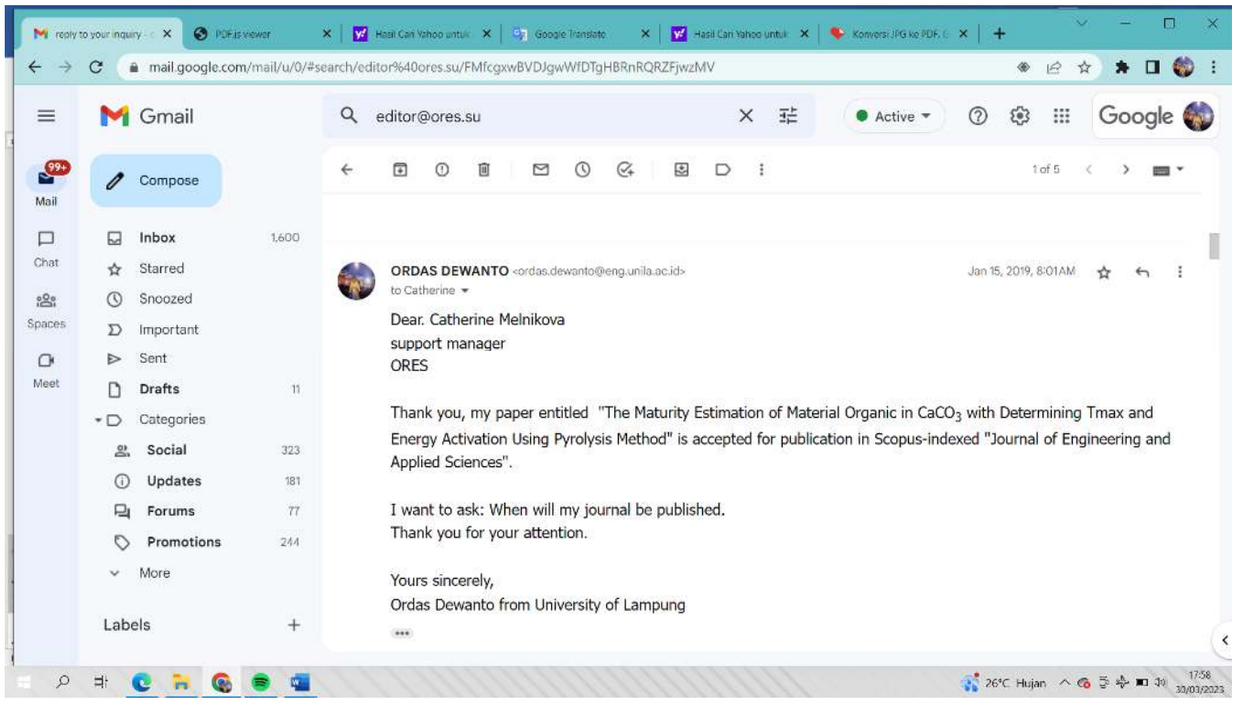
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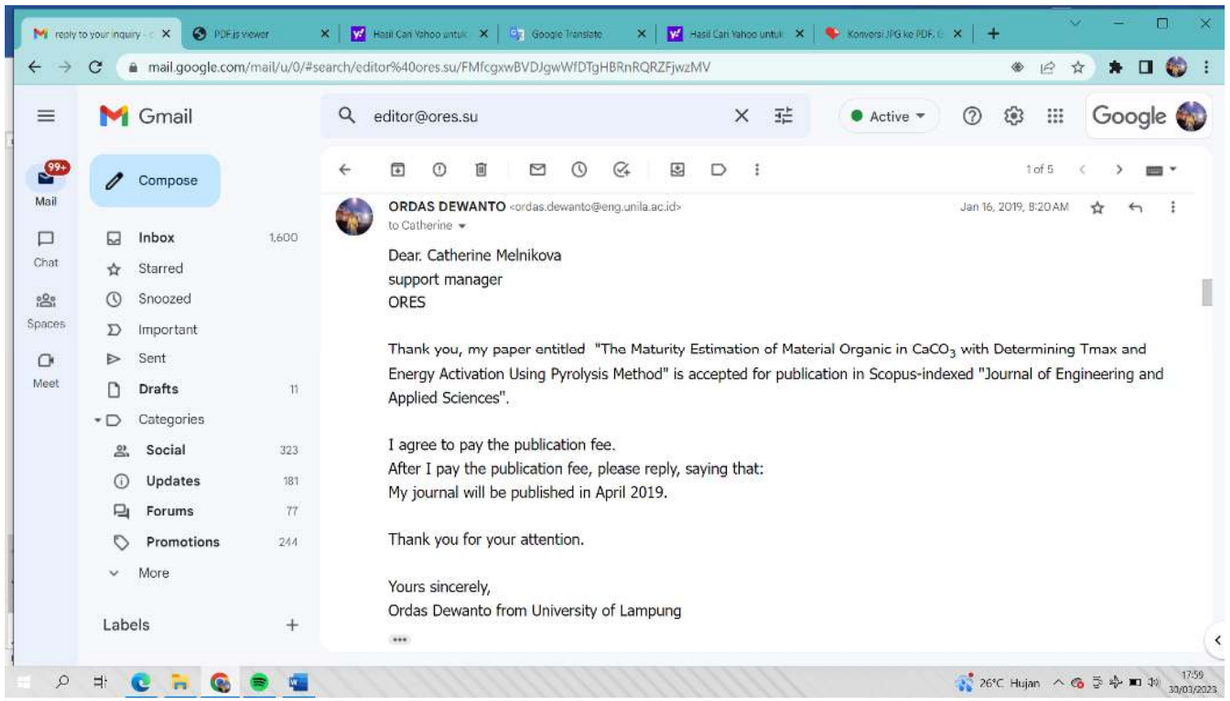

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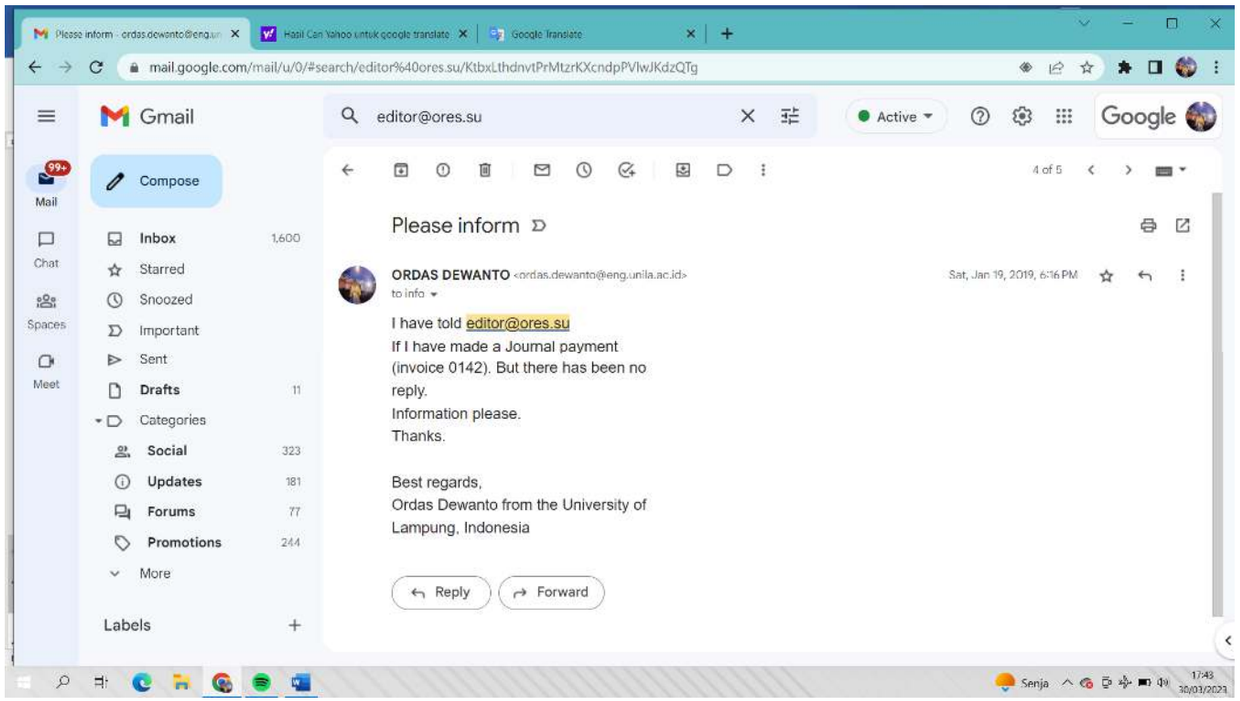
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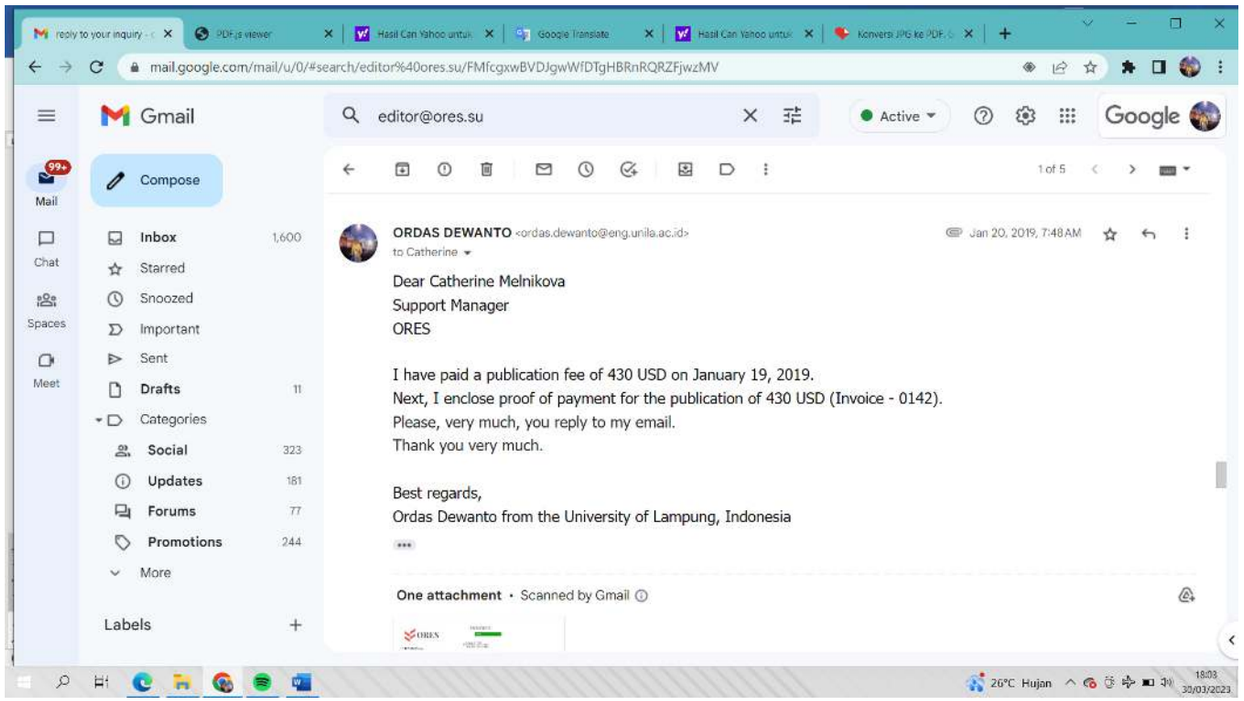
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Categories
Social 323
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Forums 77
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to Catherine
Jan 20, 2019, 7:48 AM
Dear Catherine Melnikova
Support Manager
ORES
I have paid a publication fee of 430 USD on January 19, 2019.
Next, I enclose proof of payment for the publication of 430 USD (Invoice - 0142).
Please, very much, you reply to my email.
Thank you very much.
Best regards,
Ordas Dewanto from the University of Lampung, Indonesia
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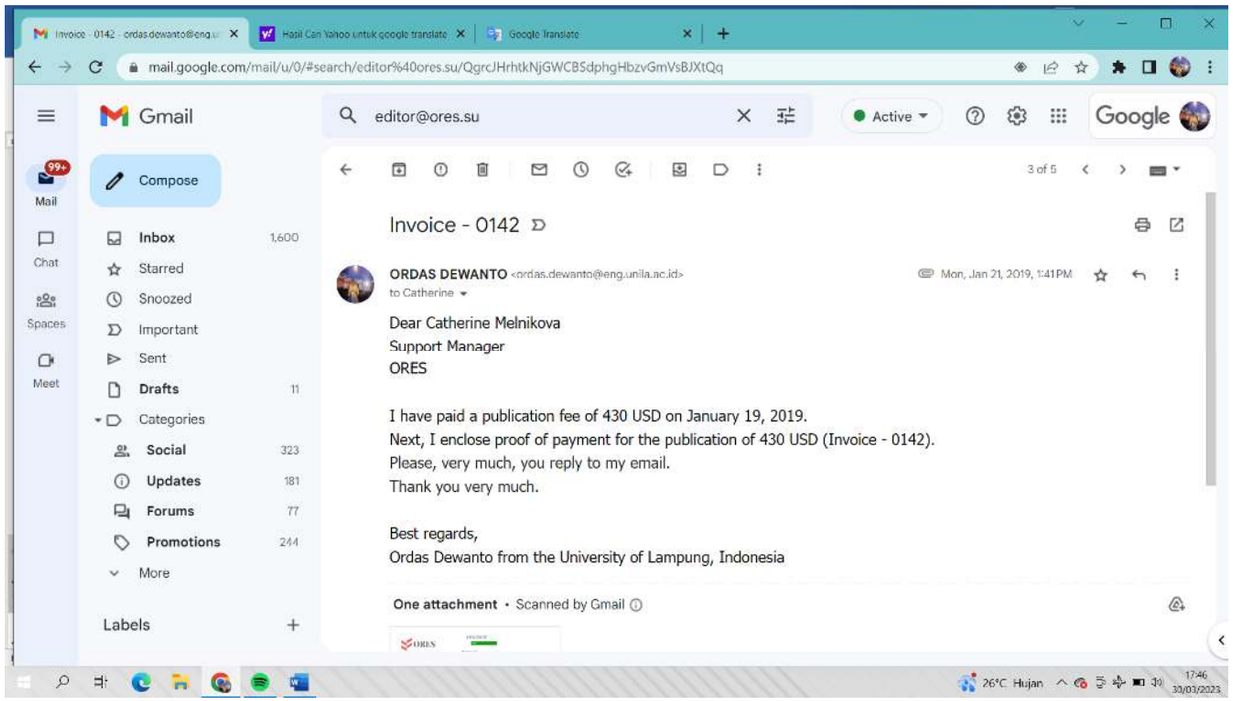
21-1-2019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Forums 77
Promotions 244
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Invoice - 0142
ORDAS DEWANTO <ordas.dewanto@eng.unila.ac.id>
to Catherine
Mon, Jan 21, 2019, 1:41 PM
Dear Catherine Melnikova
Support Manager
ORES
I have paid a publication fee of 430 USD on January 19, 2019.
Next, I enclose proof of payment for the publication of 430 USD (Invoice - 0142).
Please, very much, you reply to my email.
Thank you very much.
Best regards,
Ordas Dewanto from the University of Lampung, Indonesia
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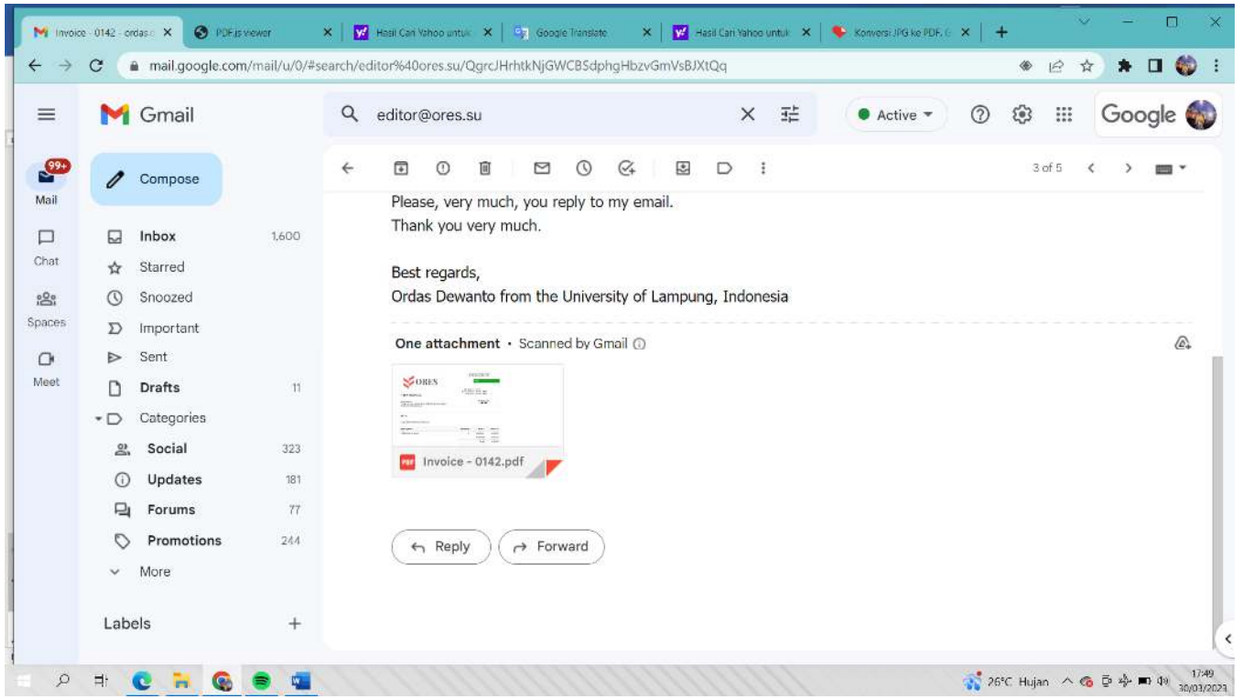
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Thank you very much.
Best regards,
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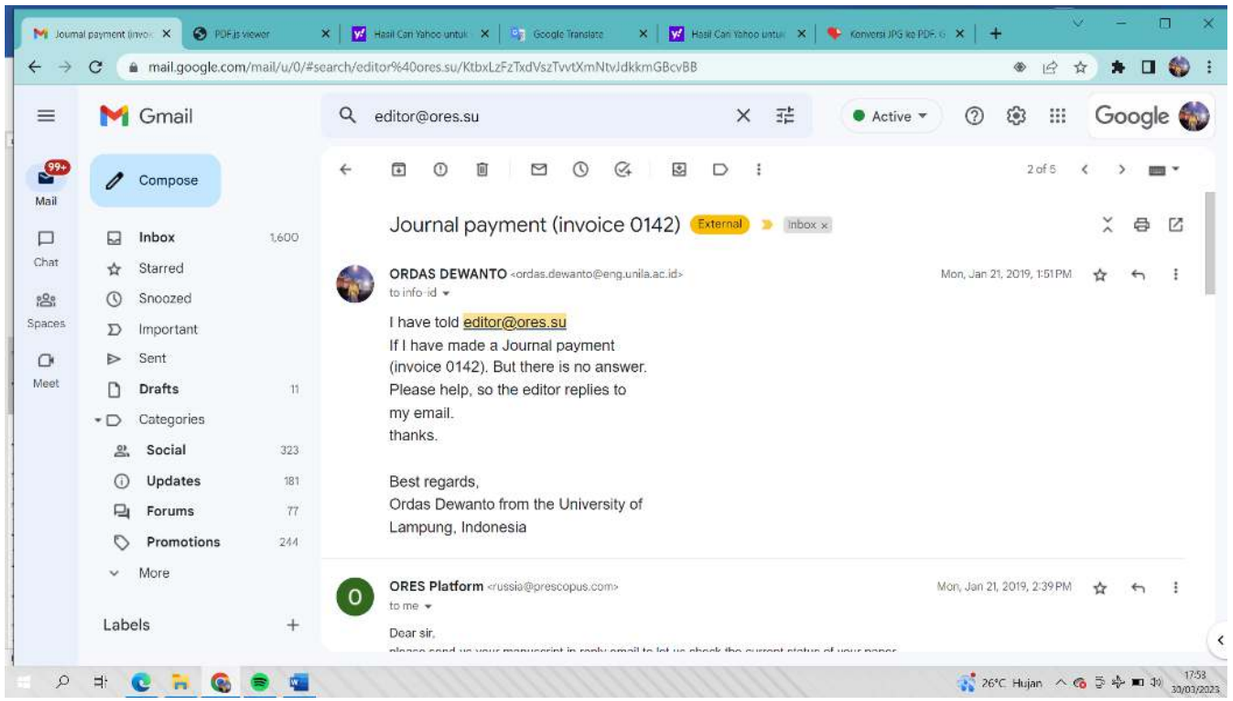

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		Total	$430.00

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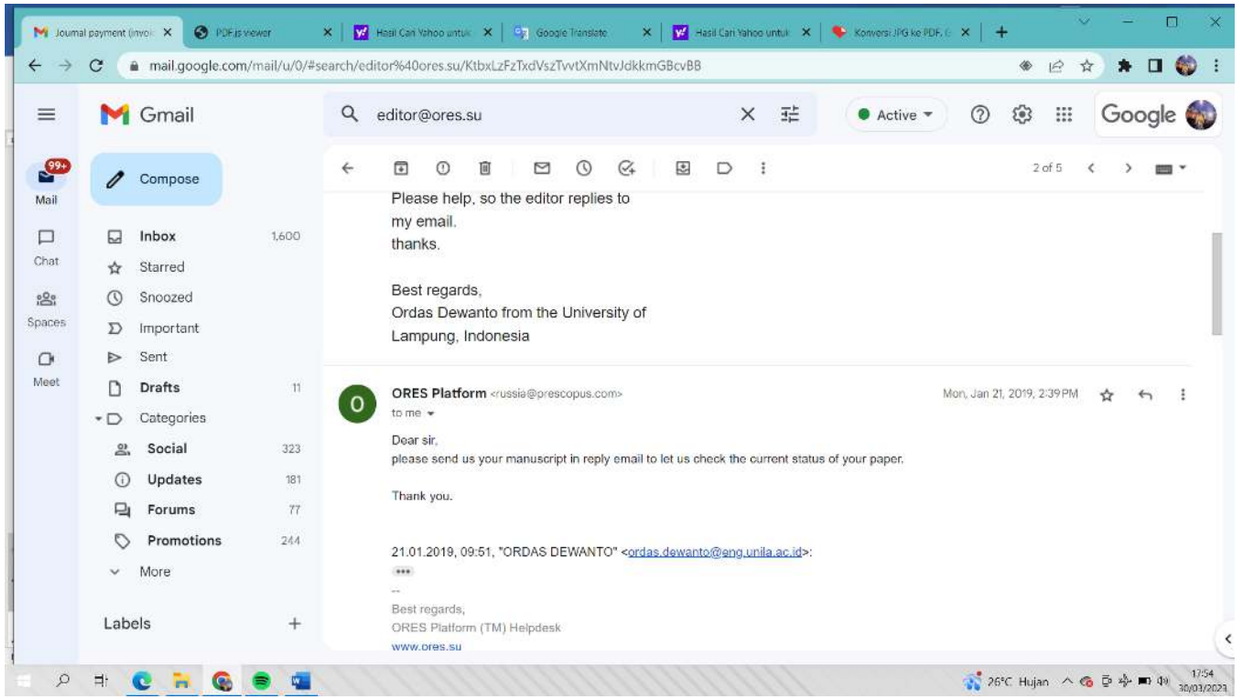
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thanks.
Best regards,
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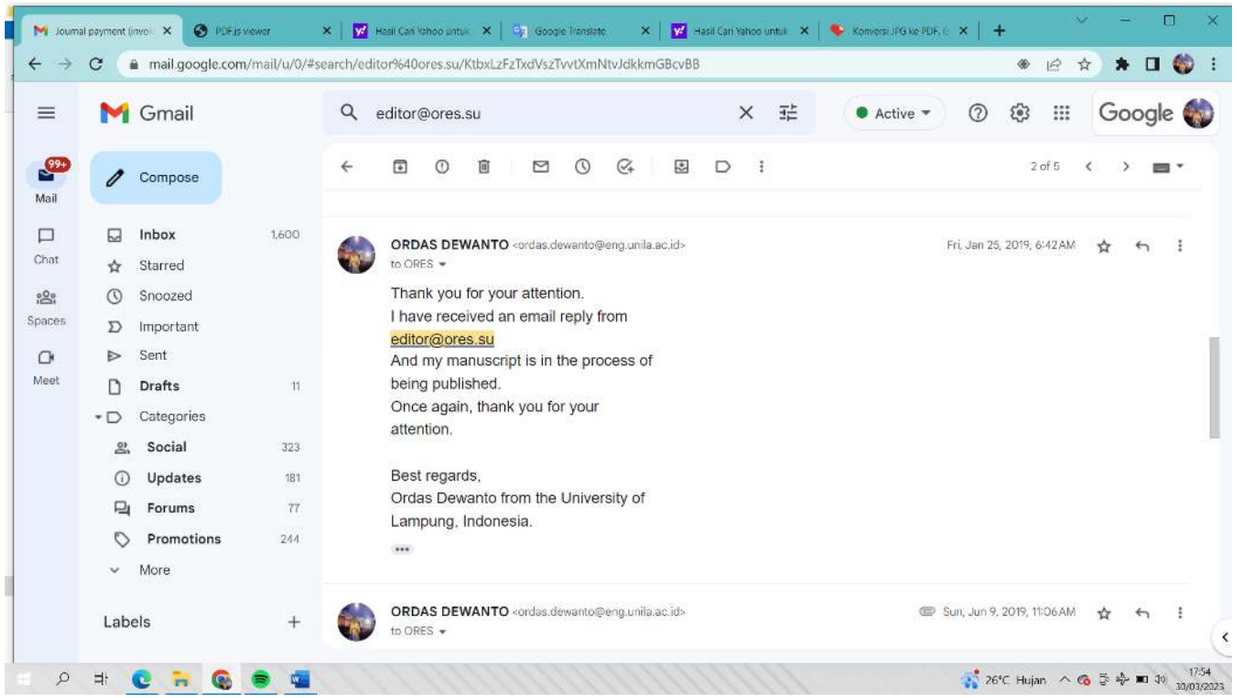
25-1-2019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2 of 5
ORDAS DEWANTO <ordas.dewanto@eng.unila.ac.id>
to ORES
Fri, Jan 25, 2019, 6:42 AM
Thank you for your attention.
I have received an email reply from
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And my manuscript is in the process of
being published.
Once again, thank you for your
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Best regards,
Ordas Dewanto from the University of
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to ORES
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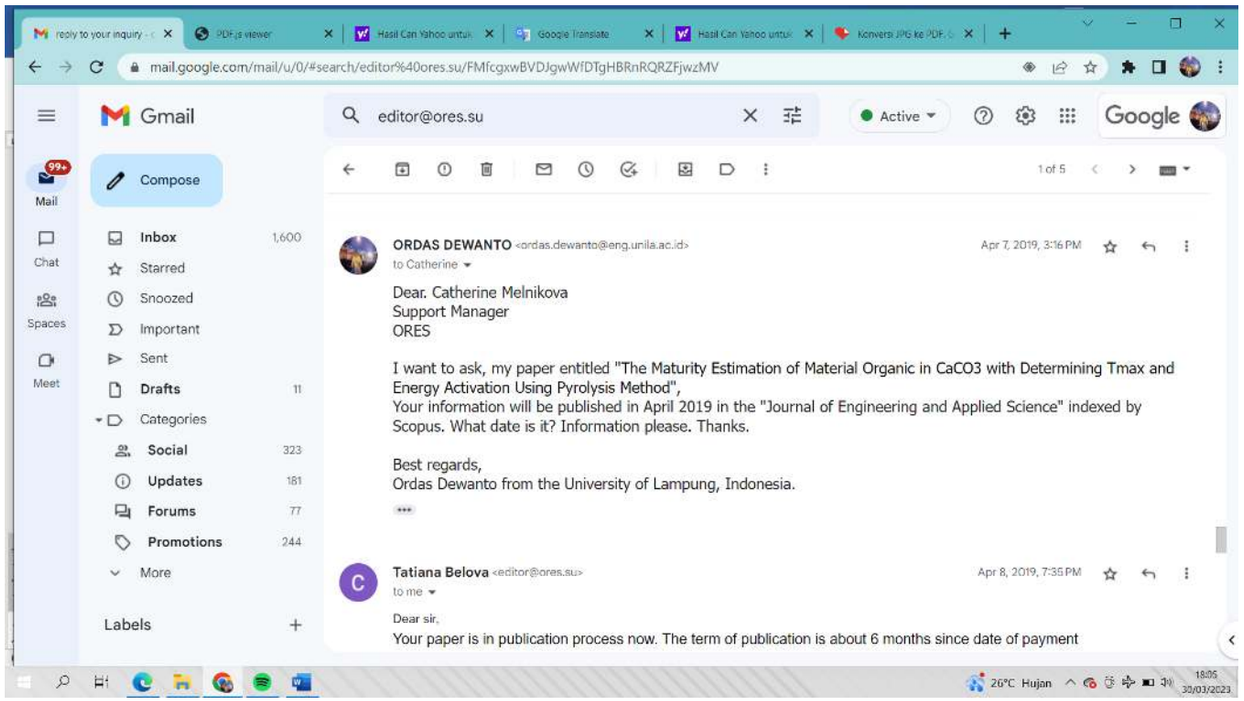
7-4-2019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Updates 181
Forums 77
Promotions 244
More
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ORDAS DEWANTO <ordas.dewanto@eng.unila.ac.id>
to Catherine
Apr 7, 2019, 3:16 PM
Dear. Catherine Melnikova
Support Manager
ORES
I want to ask, my paper entitled "The Maturity Estimation of Material Organic in CaCO3 with Determining Tmax and Energy Activation Using Pyrolysis Method",
Your information will be published in April 2019 in the "Journal of Engineering and Applied Science" indexed by Scopus. What date is it? Information please. Thanks.
Best regards,
Ordas Dewanto from the University of Lampung, Indonesia.
Tatiana Belova <editor@ores.su>
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Your paper is in publication process now. The term of publication is about 6 months since date of payment
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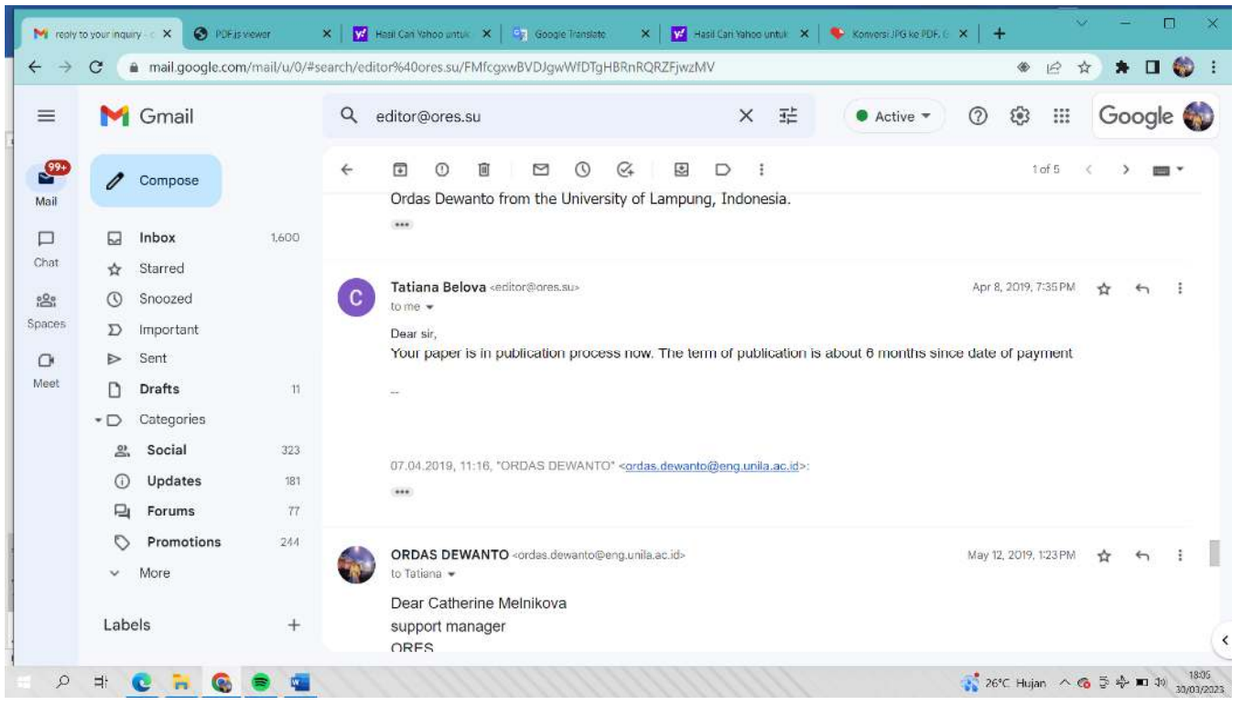
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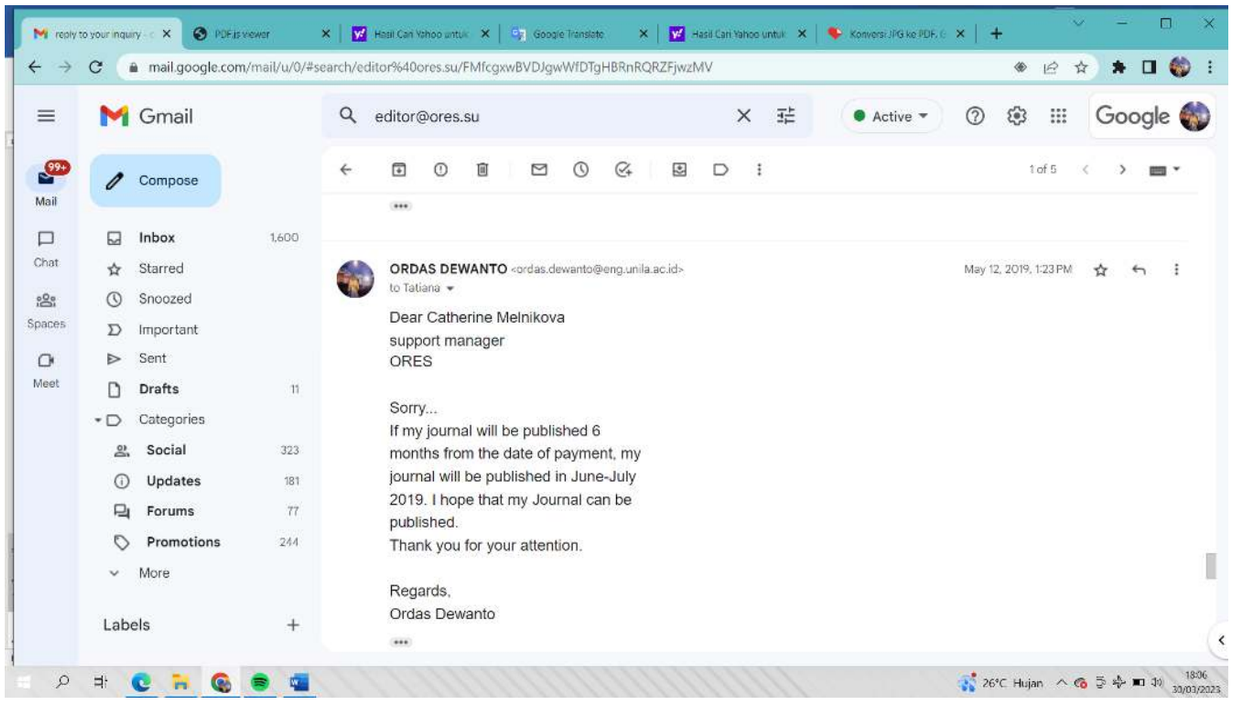
Ordas Dewanto from the University of Lampung, Indonesia.
Tatiana Belova <editor@ores.su>
Apr 8, 2019, 7:35 PM
to me
Dear sir,
Your paper is in publication process now. The term of publication is about 6 months since date of payment
07.04.2019, 11:16, "ORDAS DEWANTO" <ordas.dewanto@eng.unila.ac.id>:
ORDAS DEWANTO <ordas.dewanto@eng.unila.ac.id>
May 12, 2019, 1:23 PM
to Tatiana
Dear Catherine Melnikova
support manager
ORES

12-5-2019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Forums 77
Promotions 244
More
Labels
Mail
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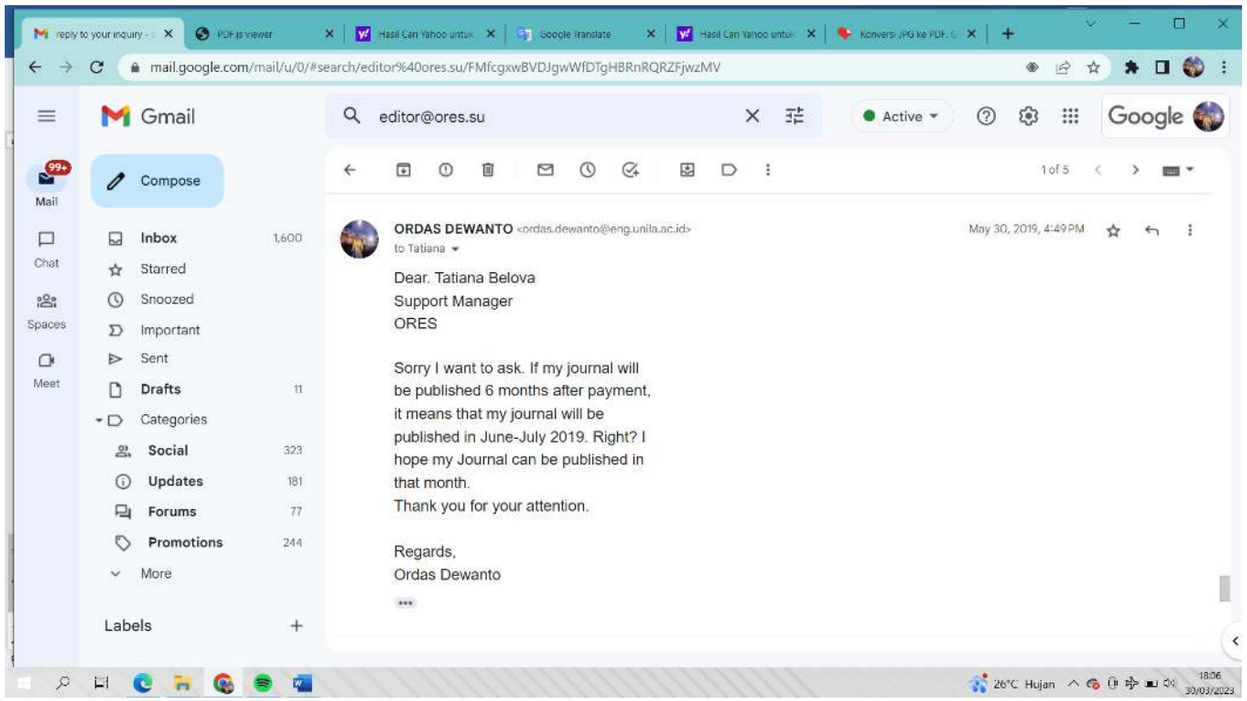
Spaces
Meet
1 of 5
ORDAS DEWANTO <ordas.dewanto@eng.unila.ac.id>
to Tatiana
May 12, 2019, 1:23 PM
Dear Catherine Melnikova
support manager
ORES
Sorry...
If my journal will be published 6 months from the date of payment, my journal will be published in June-July 2019. I hope that my Journal can be published.
Thank you for your attention.
Regards,
Ordas Dewanto

30-5-2019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Promotions 244
More
Labels
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1 of 5
ORDAS DEWANTO <ordas.dewanto@eng.unila.ac.id>
to Tatiana
May 30, 2019, 4:49 PM
Dear. Tatiana Belova
Support Manager
ORES
Sorry I want to ask. If my journal will be published 6 months after payment, it means that my journal will be published in June-July 2019. Right? I hope my Journal can be published in that month.
Thank you for your attention.
Regards,
Ordas Dewanto
26°C Hujan
18:06
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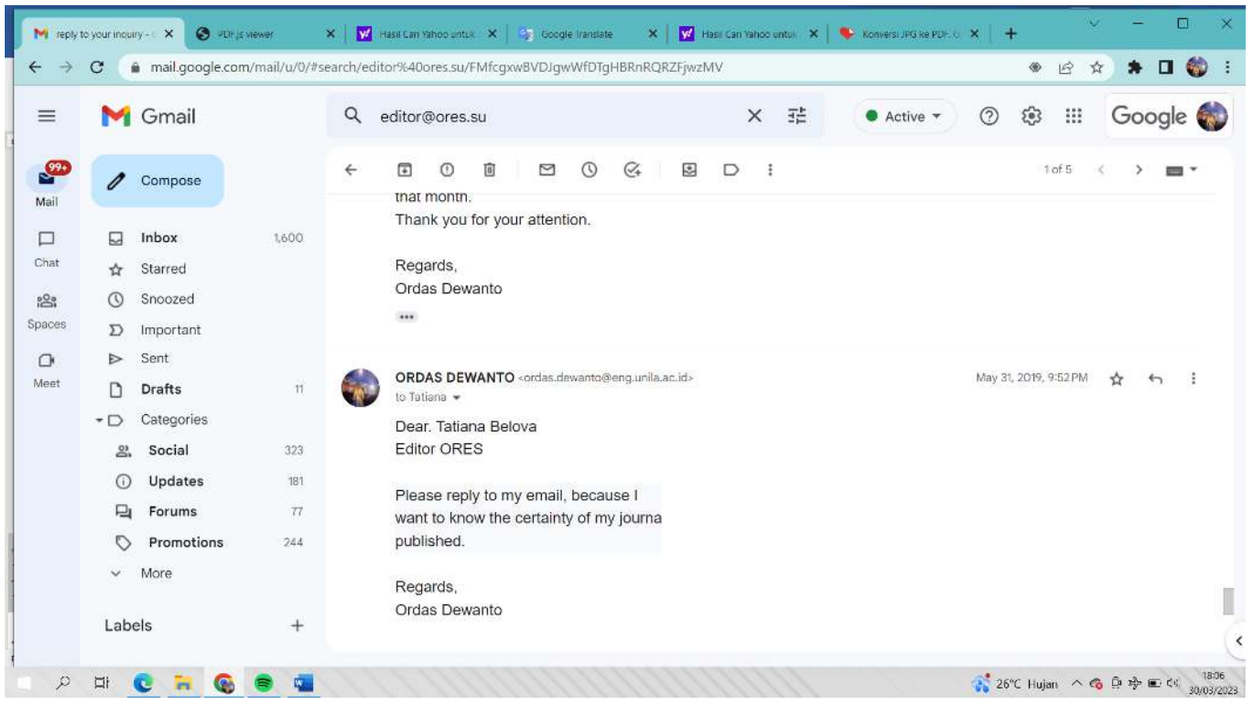
31-5-2019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that month.
Thank you for your attention.
Regards,
Ordas Dewanto
ORDAS DEWANTO <ordas.dewanto@eng.unila.ac.id>
to Tatiana
May 31, 2019, 9:52 PM
Dear. Tatiana Belova
Editor ORES
Please reply to my email, because I
want to know the certainty of my journa
published.
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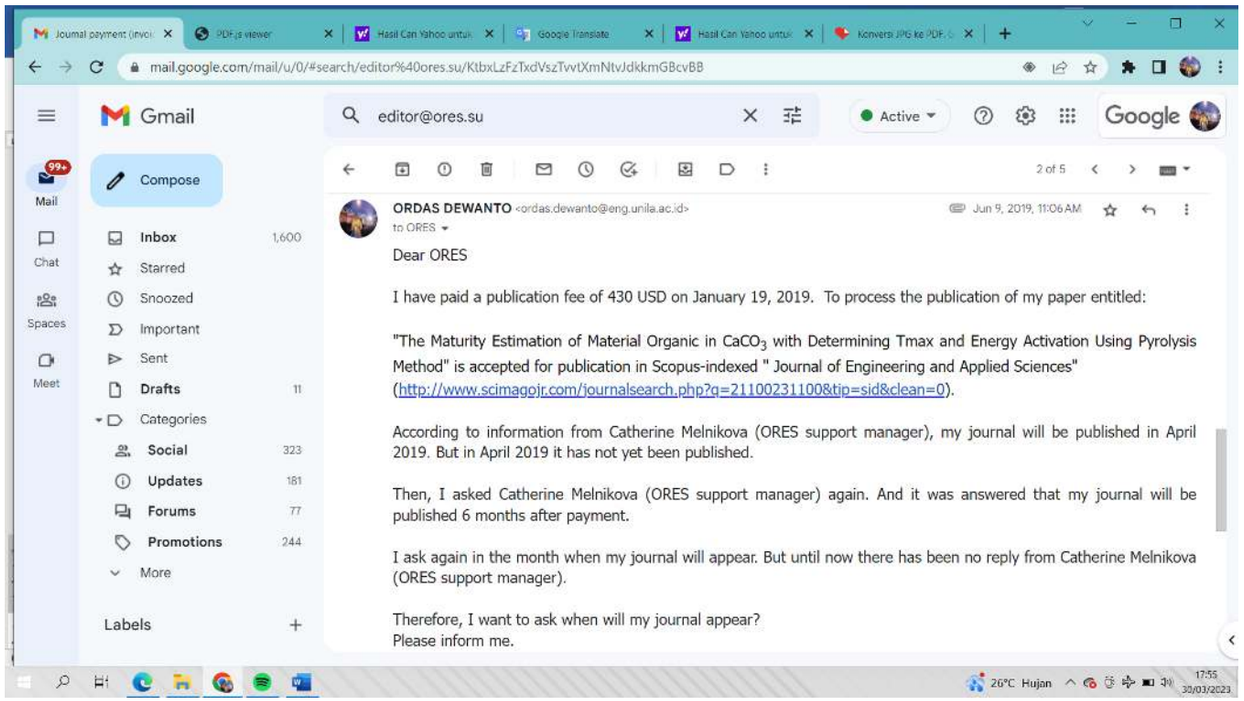
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ORDAS DEWANTO <ordas.dewanto@eng.unila.ac.id>
to ORES
Jun 9, 2019, 11:06 AM
Dear ORES
I have paid a publication fee of 430 USD on January 19, 2019. To process the publication of my paper entitled:
"The Maturity Estimation of Material Organic in $CaCO_3$ with Determining Tmax and Energy Activation Using Pyrolysis Method" is accepted for publication in Scopus-indexed " Journal of Engineering and Applied Sciences" (http://www.scimagojr.com/journalsearch.php?q=21100231100&tip=sid&clean=0).
According to information from Catherine Melnikova (ORES support manager), my journal will be published in April 2019. But in April 2019 it has not yet been published.
Then, I asked Catherine Melnikova (ORES support manager) again. And it was answered that my journal will be published 6 months after payment.
I ask again in the month when my journal will appear. But until now there has been no reply from Catherine Melnikova (ORES support manager).
Therefore, I want to ask when will my journal appear?
Please inform me.
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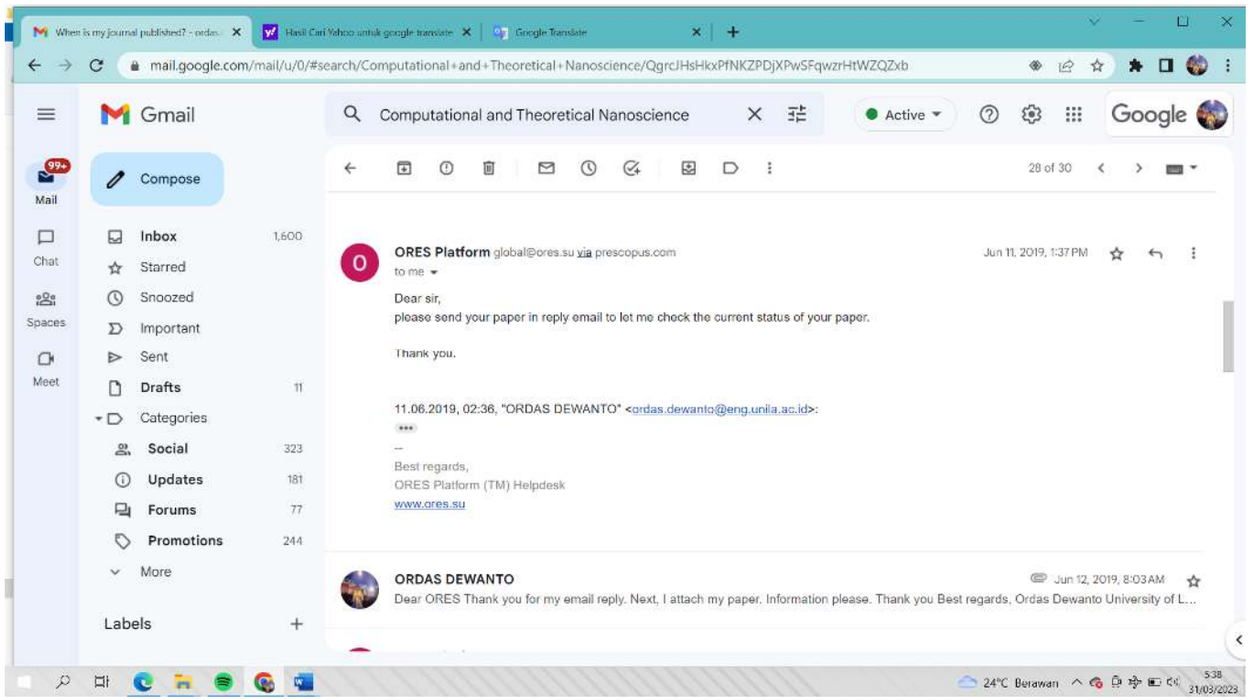
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Jun 11, 2019, 1:37 PM
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please send your paper in reply email to let me check the current status of your paper.
Thank you.
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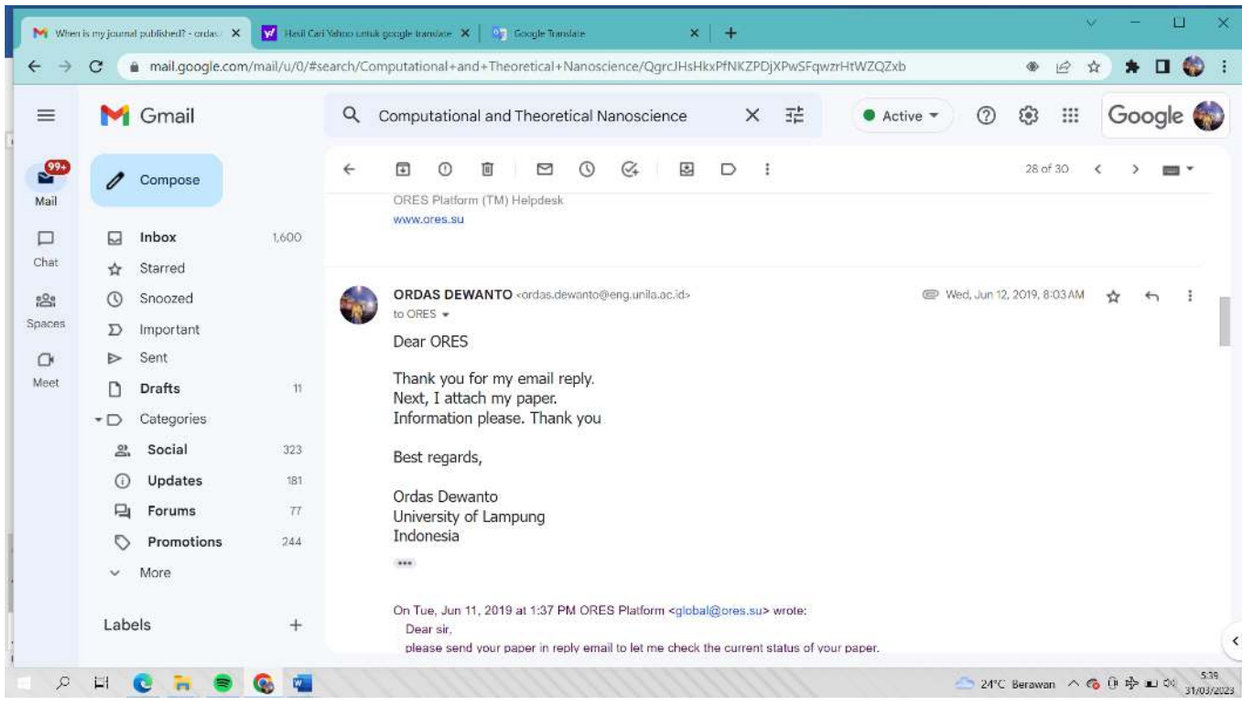
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Best regards,
Ordas Dewanto
University of Lampung
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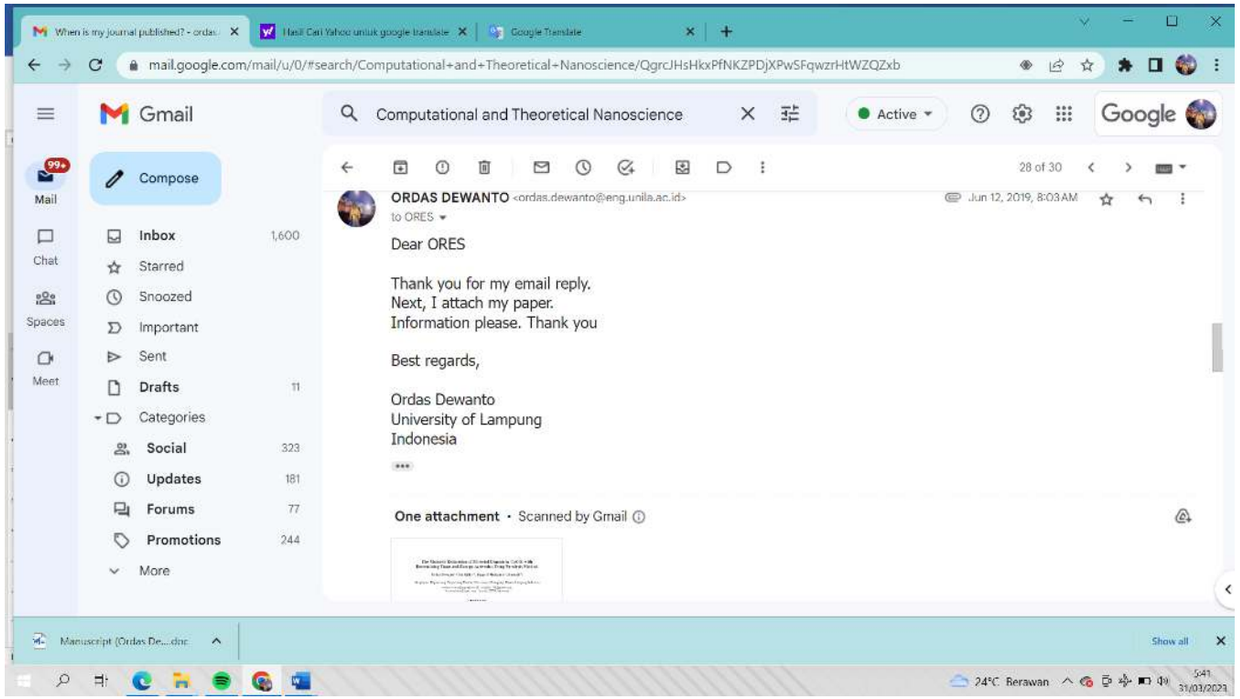
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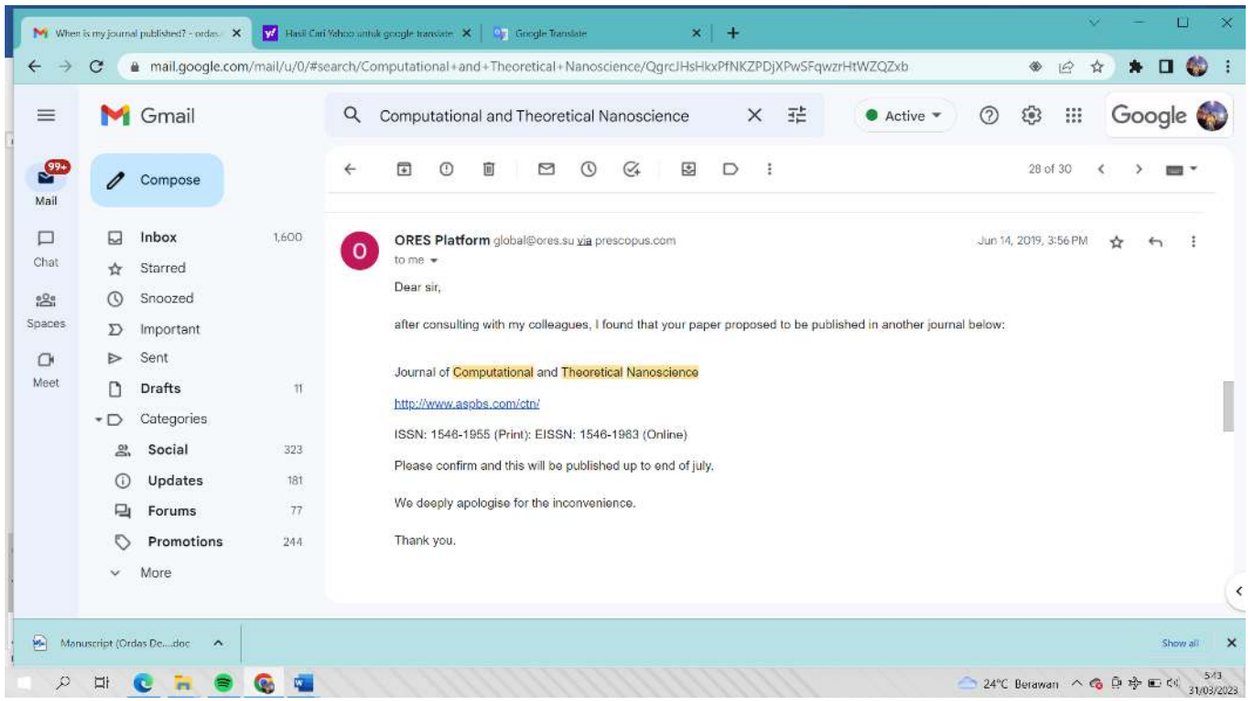
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Jun 14, 2019, 3:56 PM
to me
Dear sir,
after consulting with my colleagues, I found that your paper proposed to be published in another journal below:
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ISSN: 1546-1955 (Print); EISSN: 1546-1963 (Online)
Please confirm and this will be published up to end of july.
We deeply apologise for the inconvenience.
Thank you.
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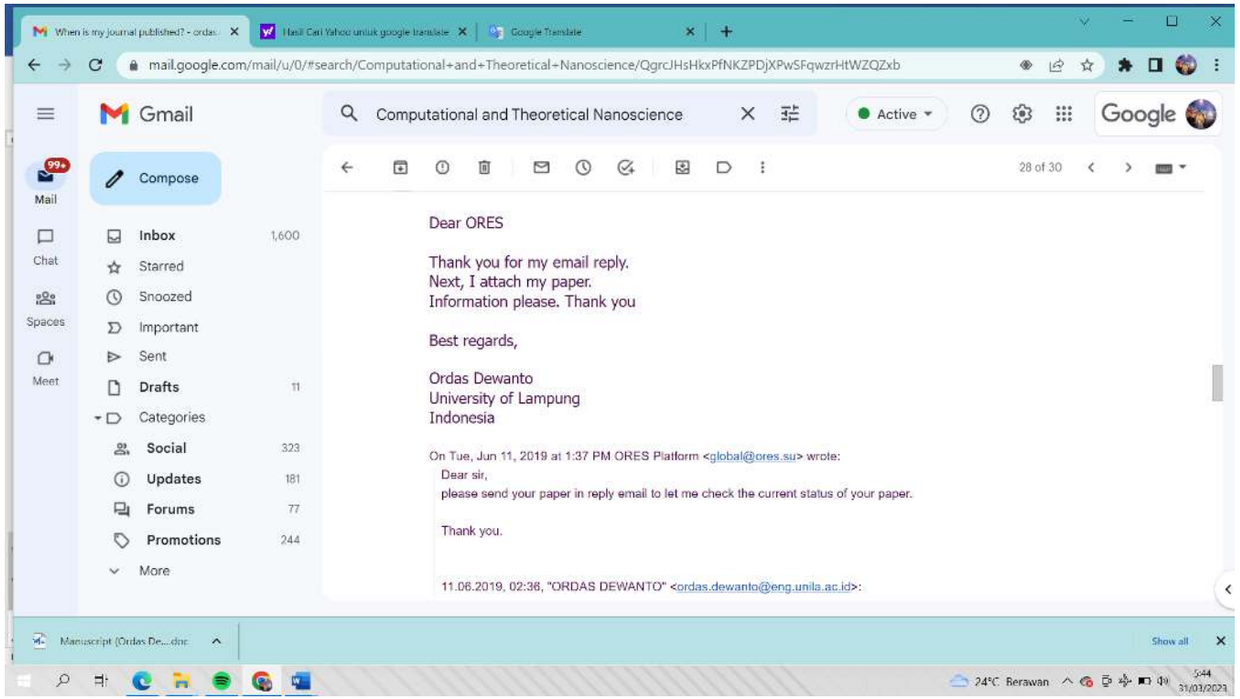
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Information please. Thank you
Best regards,
Ordas Dewanto
University of Lampung
Indonesia
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please send your paper in reply email to let me check the current status of your paper.
Thank you.
11.06.2019, 02:36, "ORDAS DEWANTO" <ordas.dewanto@eng.unila.ac.id>:
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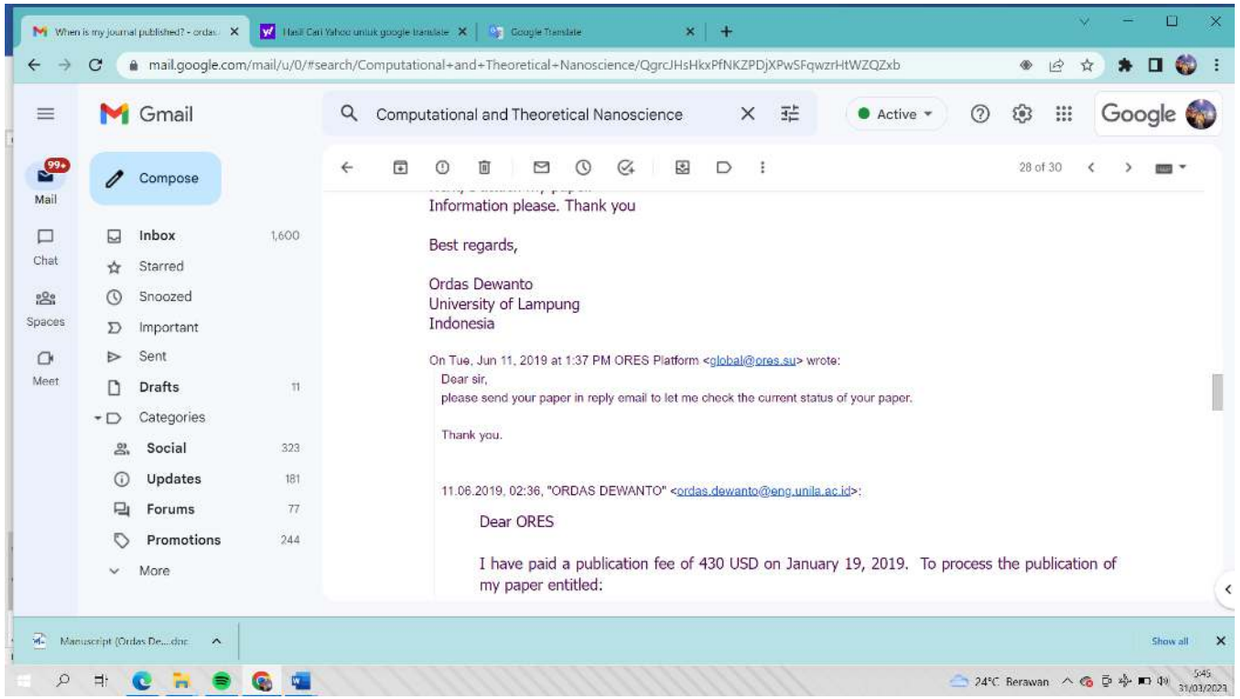
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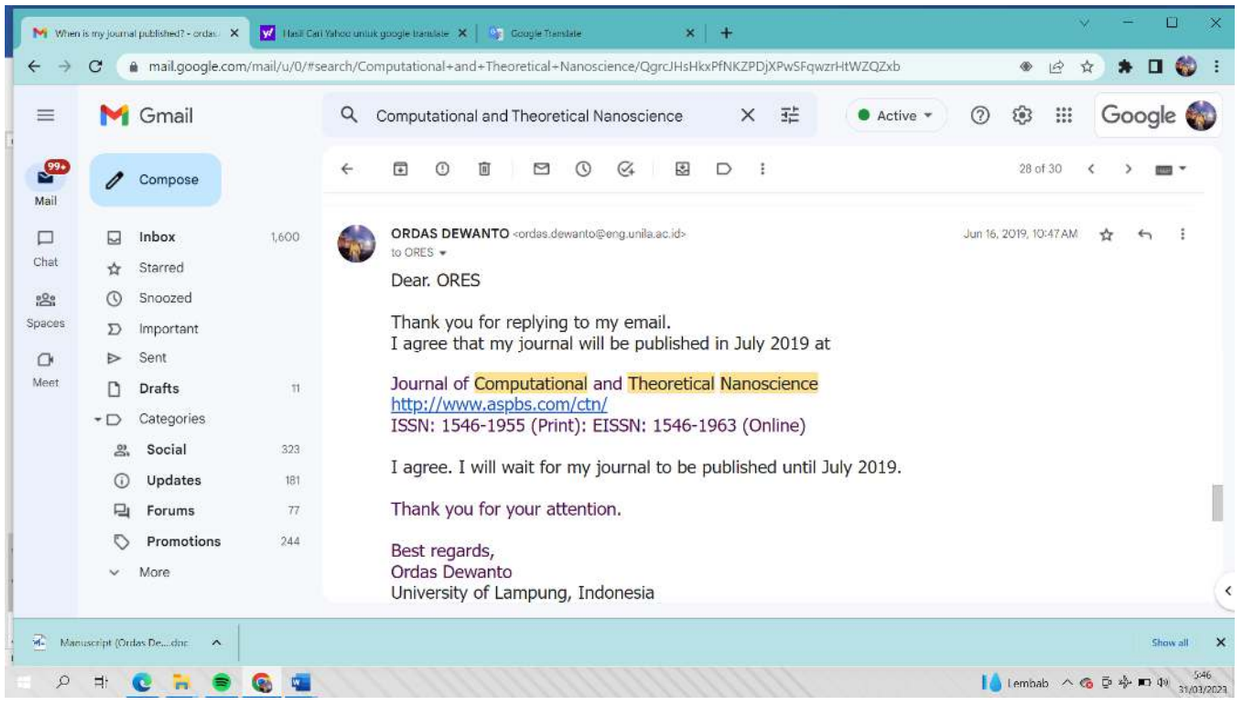
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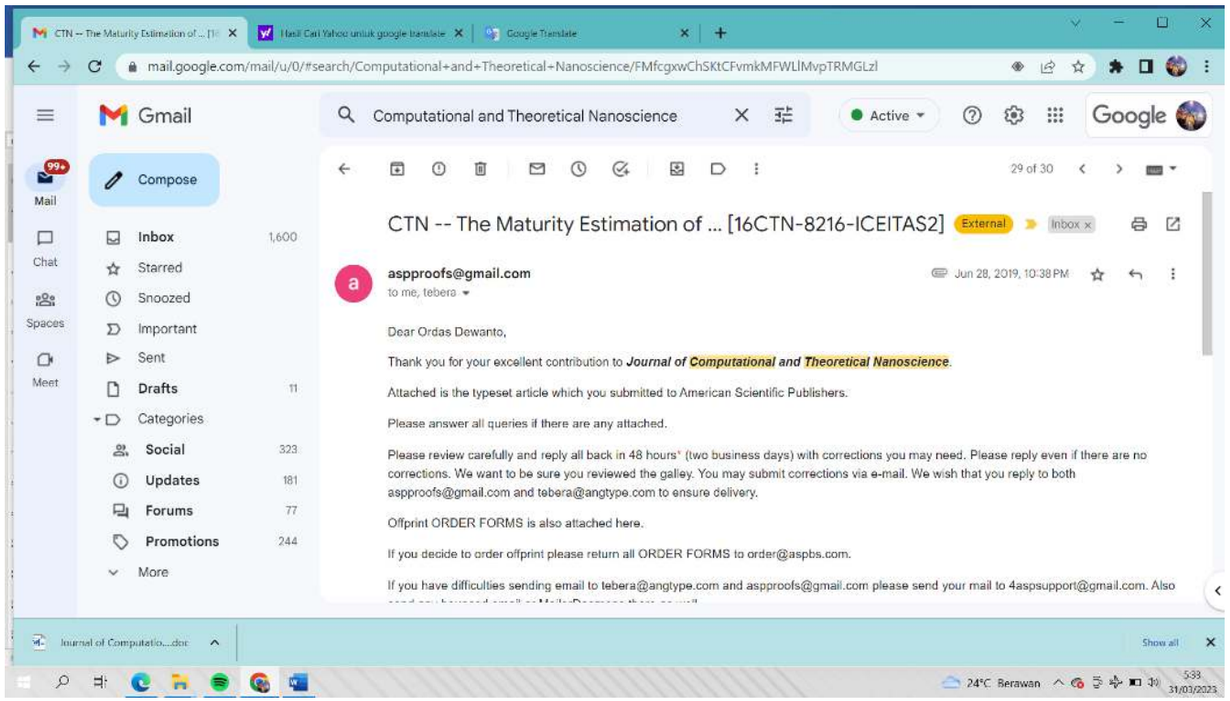
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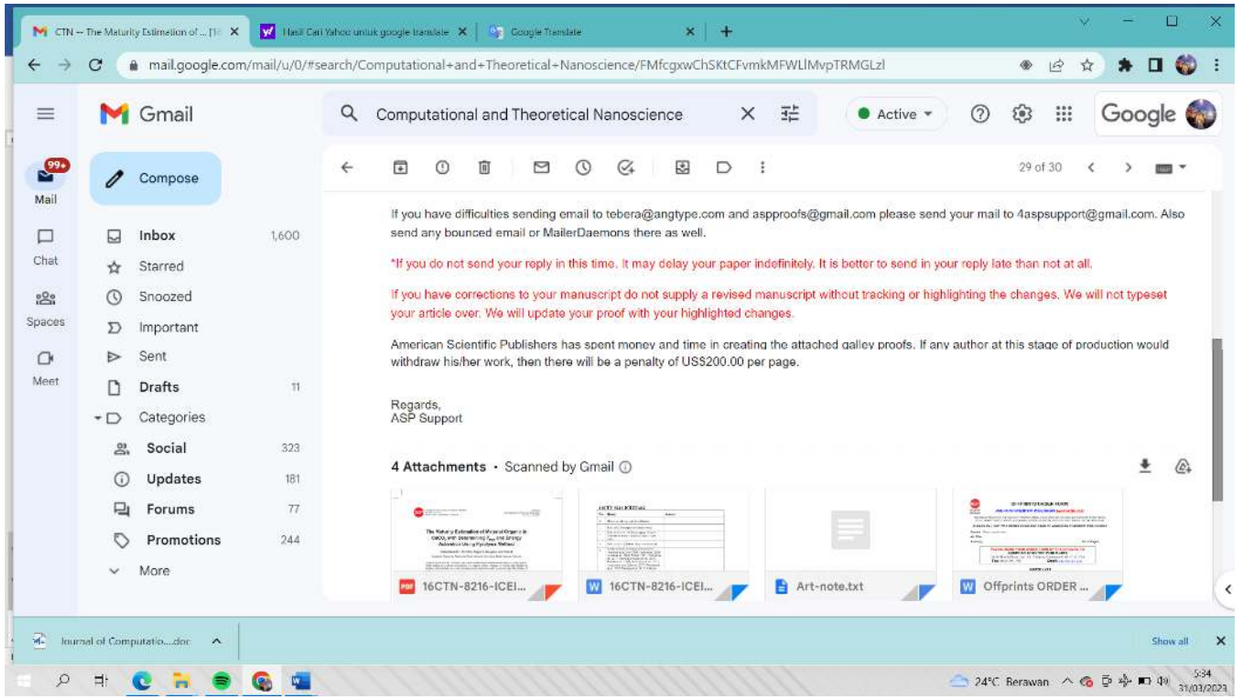
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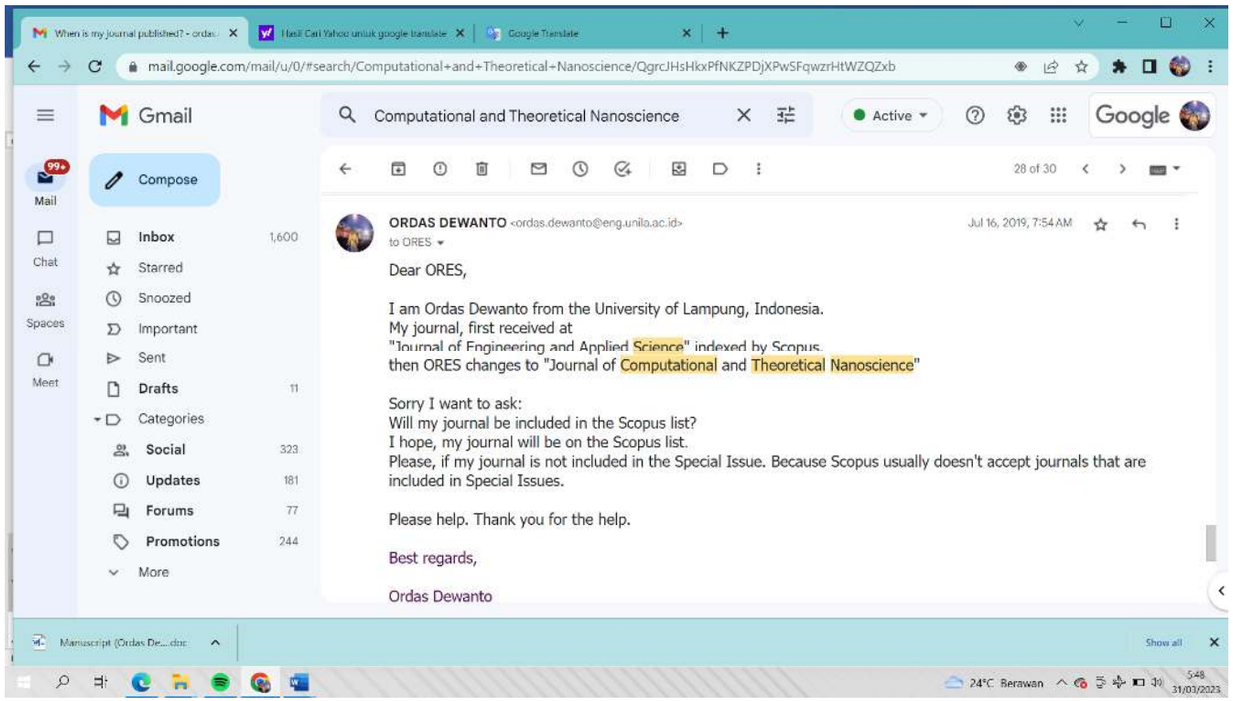
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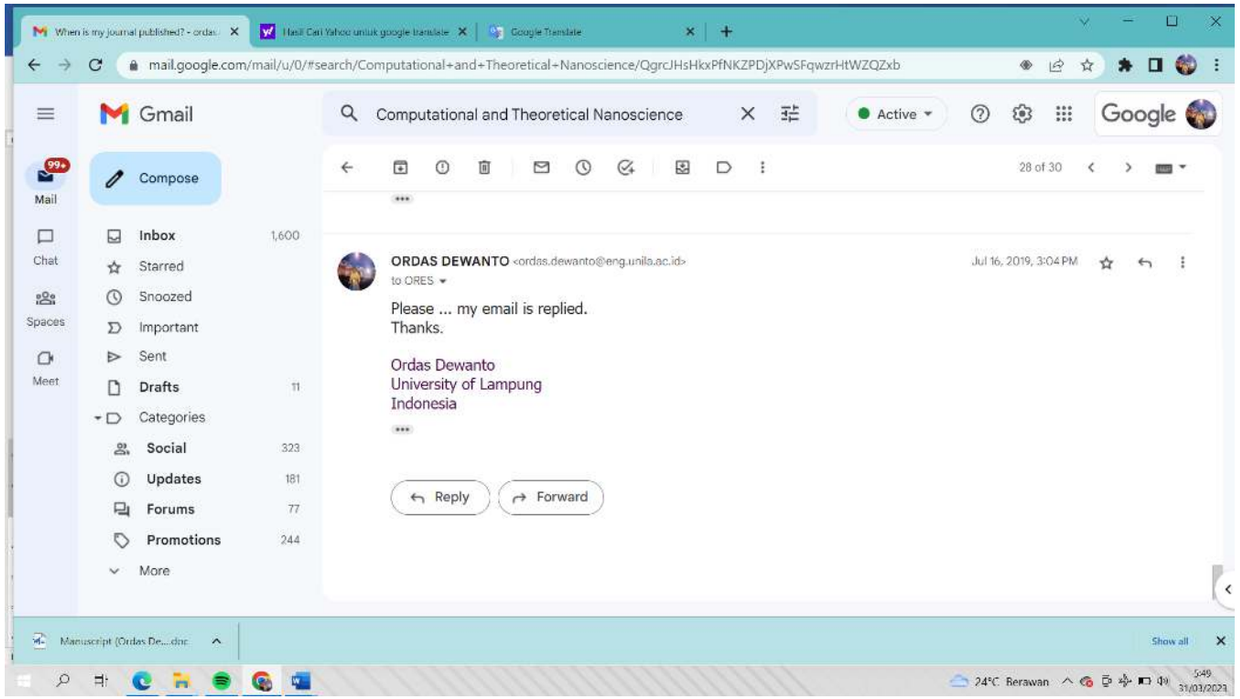
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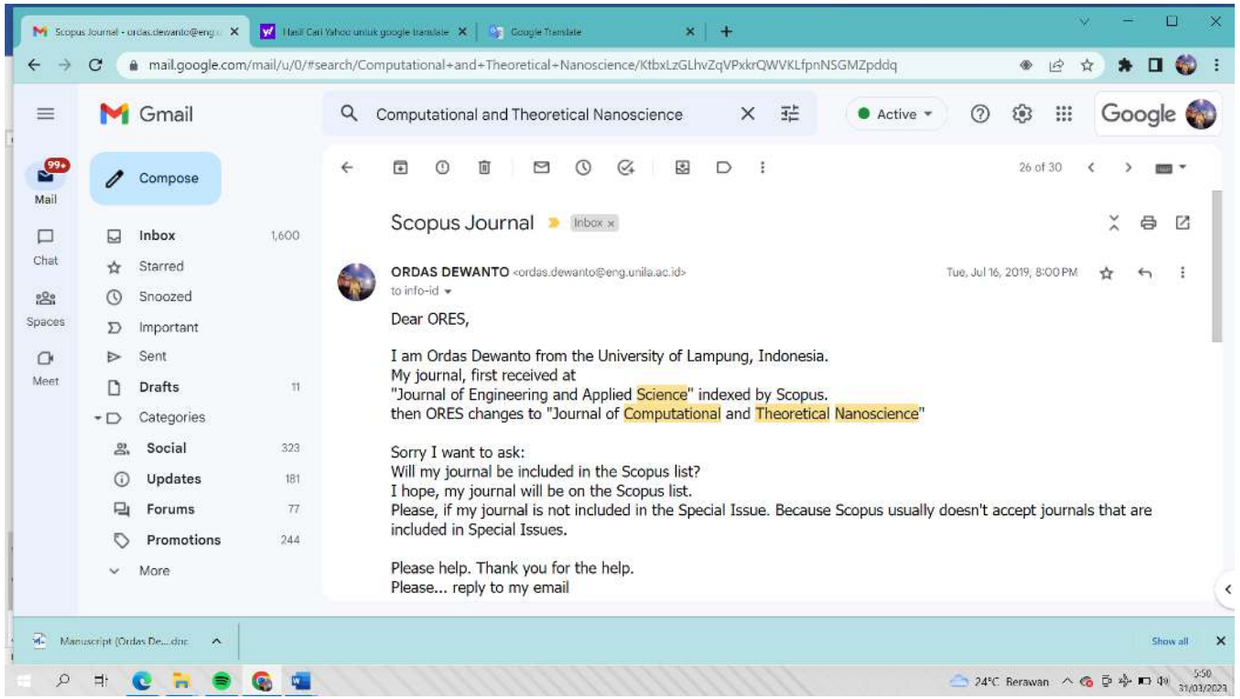
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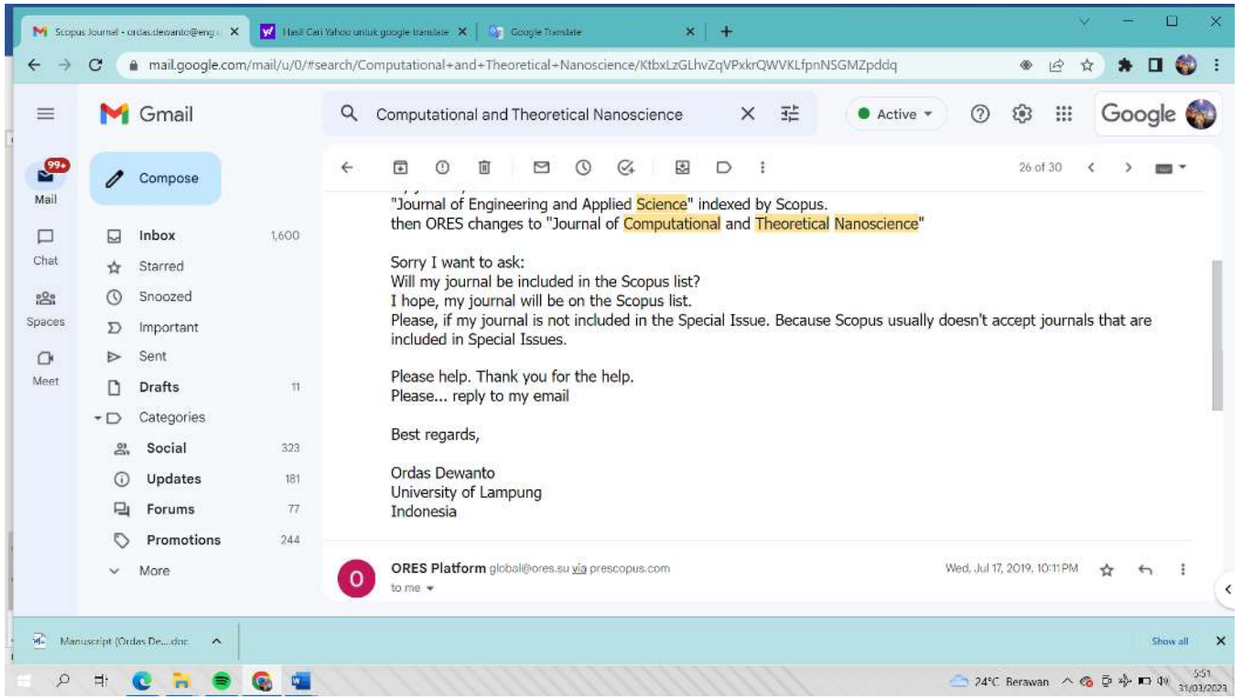
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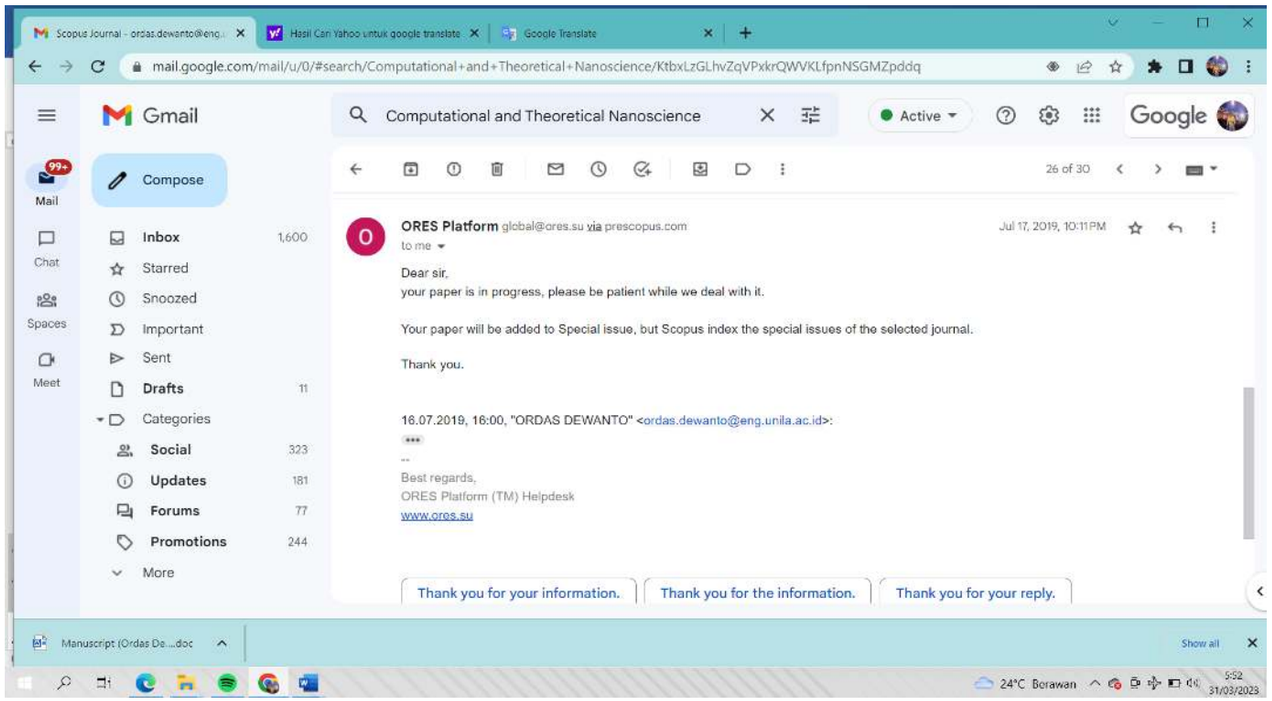
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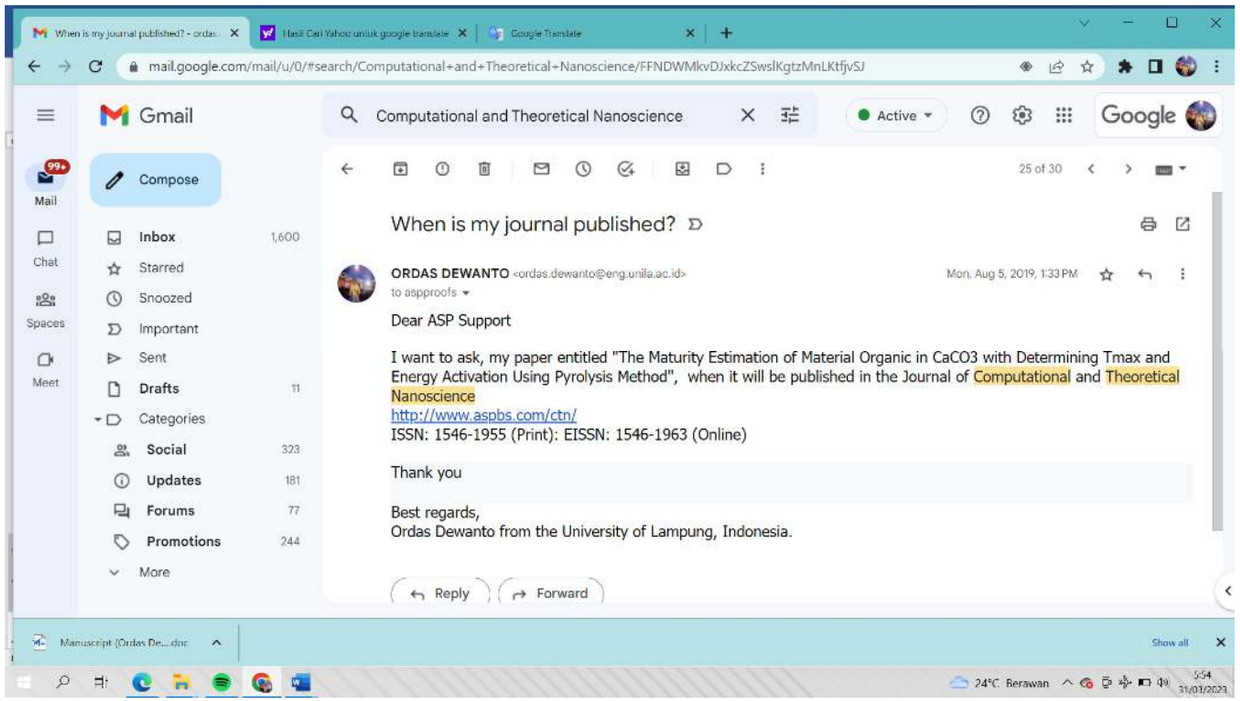
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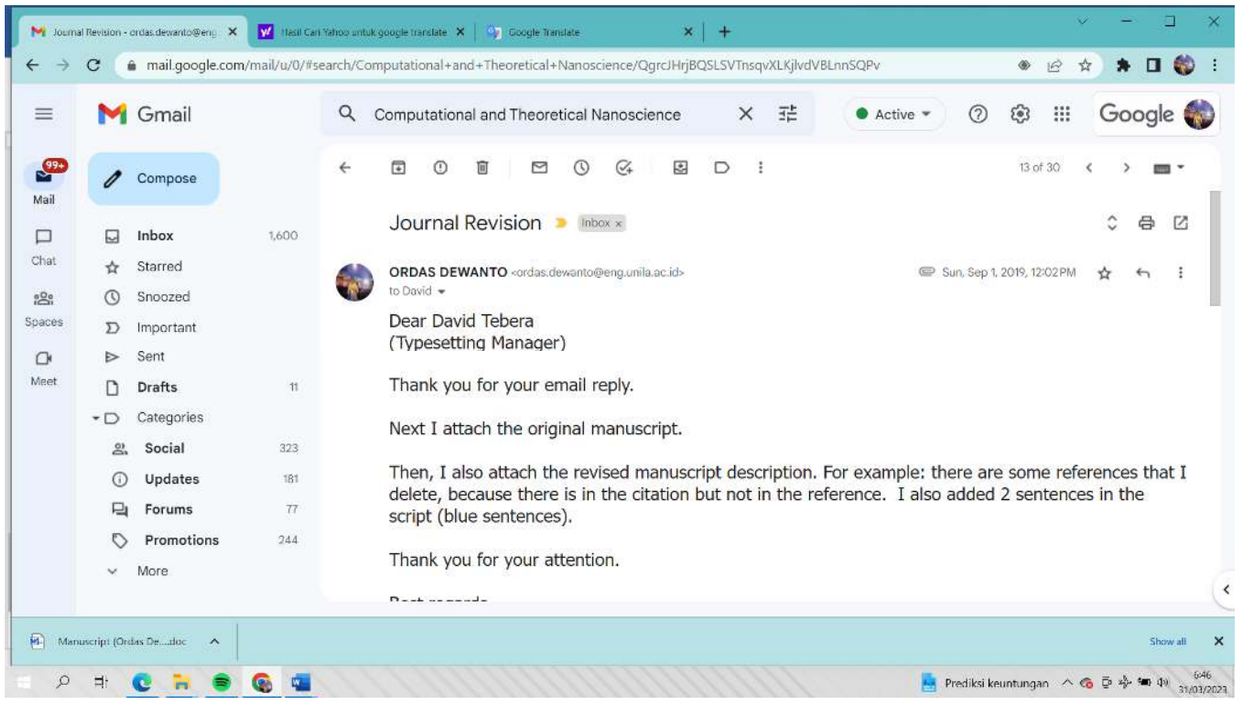
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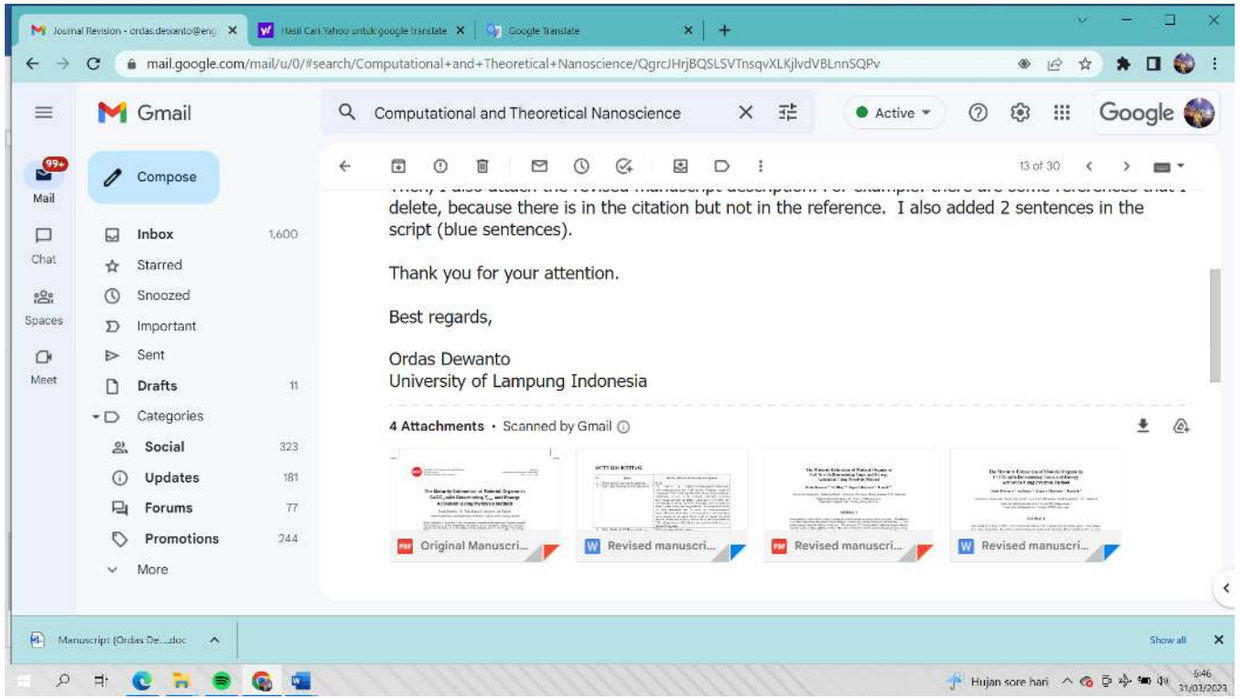
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3.	Refs. [14-16, 31, 39]:Please check, we have abbreviated author's name and initial as per style.	Ok No problem. Already correct
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		Himawanto et al. 2011; sorry to write wrong, the truth is: Himawanto, D.A., Indarto, Saptoadi, H. and Rohmat, T.A., 2013. Thermogravimetric analysis of single-particle RDF combustion. *Modern Applied Science, 7*(11), p.34. **already in the Reference** Dewayantoa, 2014; not used Nukman, 2001; not used Syafiq, A. 2009. *Uji Kualitas Fisik Dan Kinetika Reaksi Briket Kayu Kalimantan Dengan Dan Tanpa Pengikat*. Skripsi. Jurusan Teknik Mesin Fakultas Teknik Universitas Sebelas Maret Surakarta. Riyanto, S. 2009. Uji Kualitas Fisik Dan Uji Kinetika Pembakaran Briket Jerami Padi Dengan Dan Tanpa Bahan Pengikat. Skripsi. Jurusan Teknik Mesin Fakultas Teknik Universitas Sebelas Maret Surakarta. Any Kurniawati, 2012; was not used Marnoto, T. dan Sulistyowati, E. 2012. *Tinjauan Kinetika Pyrolysis Limbah Polystiren*. Prosiding Seminar Nasional Teknik Kimia. Pengembangan Teknologi Kimia untuk Pengolahan Sumber Daya Alam Indonesia. Teknik Kimia, Fak Teknologi Industri, UPN Veteran Yogyakarta. ISSN: 1693-4393. Yan Y.F., Zhang Z.E., Zhang L. and Zhang L. 2014. Influence of coal properties on the co-combustion characteristics of low-grade coal and city mud. *Global NEST Journal*. Vol. 16, No 2. pp 329-338. Printed in Greece. All rights reserved. Sugondo. 2012. Kinetika Pertumbuhan Butir Paduan Zry-4 Sn Rendah. *Urania*. Vol. 18 No. 3. Hal: 120-181. ISSN 0852-4777 Sukma, H.L. 2012. *Analisis Thermogravimetry Dan Pembuatan Briket Tandan Kosong Dengan Proses Pirolisis Lambat*. Tugas Akhir Konversi Energi. Fakultas Teknologi Industri. Institut Teknologi Sepuluh Nopember Surabaya. Martono, Y., Sari, Y.E.P., Hidarto, J. 2012. Penggunaan Model Arrhenius Untuk Pendugaan Masa Simpan Produk Minuman Kemasan Berdasarkan Kandungan Vit C. Paper. Program Studi Kimia, Fakultas Sains dan Matematika. Universitas Kristen Satya Wacana. Kholisoh, S.D. 2011. *Dasar-Dasar Kinetika Reaksi Kimia*. Slide Kinetika Dan Katalisis. Jurusan Teknik Kimia. FTI UPN "VETERAN" Yogyakarta Minarsih, T. 2011. Penentuan Energi Aktivasi Amlodipin Besilat Pada pH 1, 6 Dan 10 Dengan Metode Kromatografi Cair Kinerja Tinggi. *PHARMACY*. Vol.06 No.01 Agustus 2011. ISSN 1693-3591.
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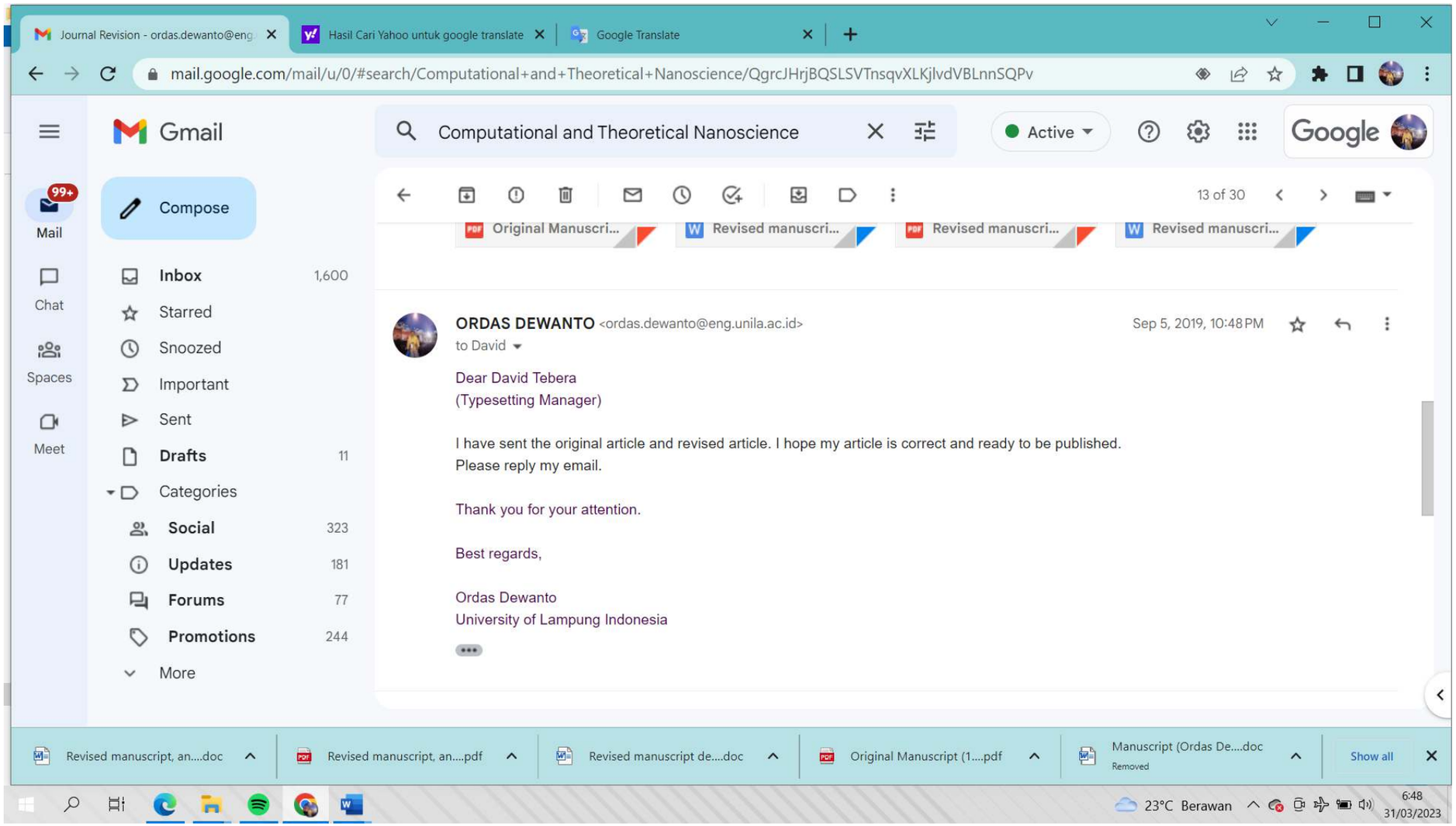
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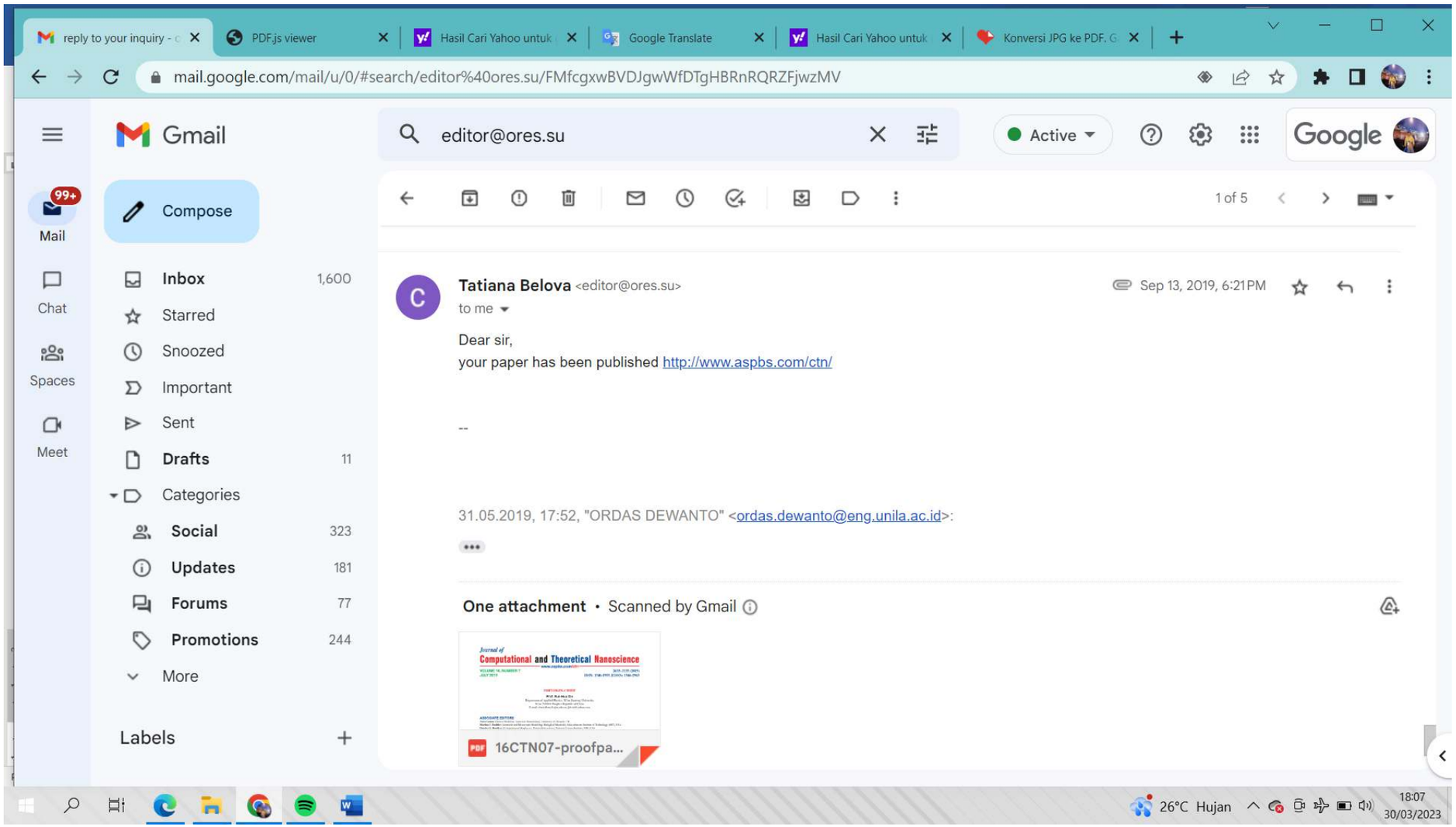
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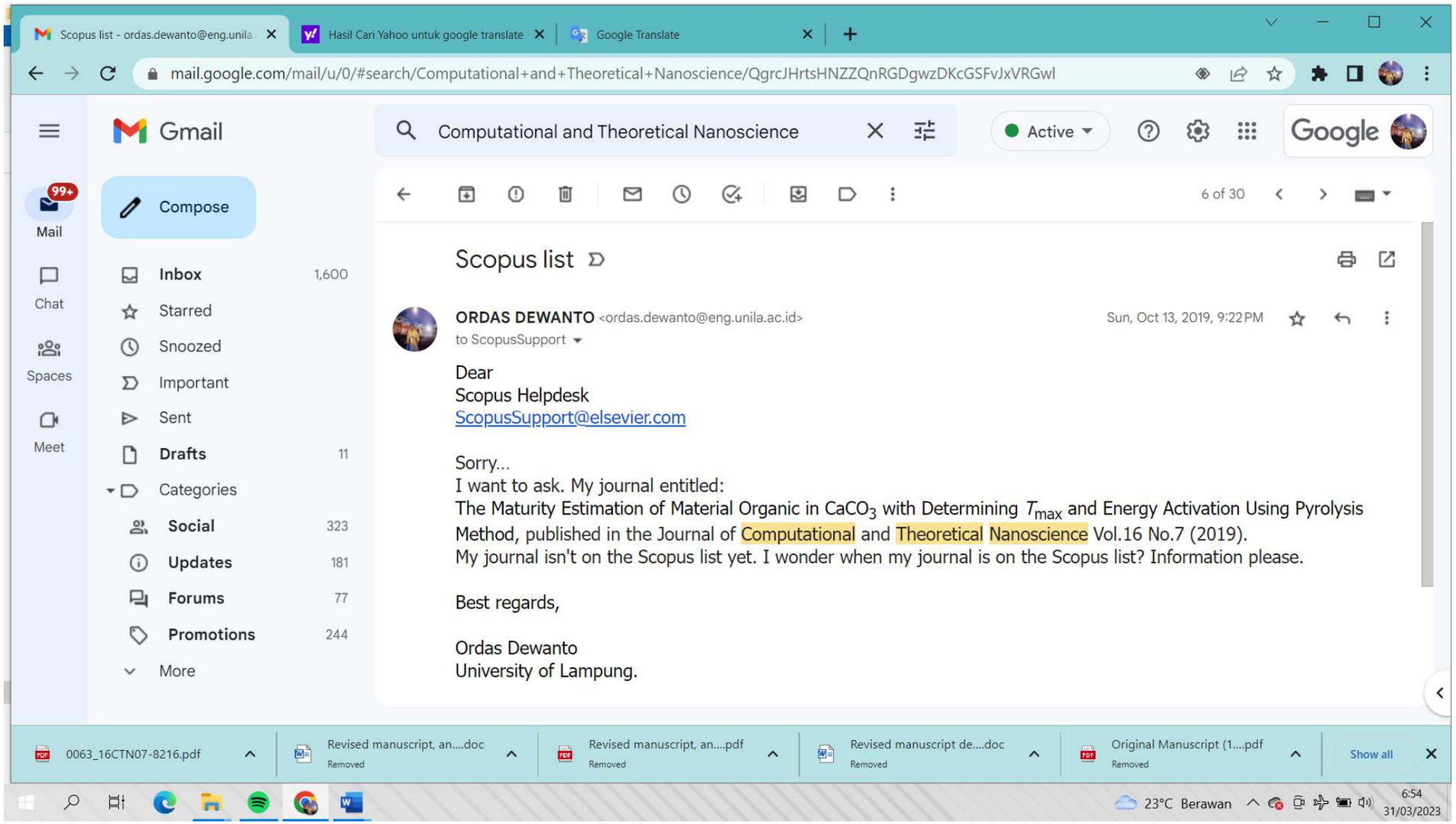
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The Maturity Estimation of Material Organic in $CaCO_3$ with Determining T_{max} and Energy Activation Using Pyrolysis Method

Ordas Dewanto*, Sri Rizky, Bagus S. Mulyanto, and Rustadi

Geophysics Engineering, Engineering Faculty, University of Lampung, Bandar Lampung, 35145, Indonesia

Shale material is shale oil that is clay or carbonate material contain excessively immature organic. When heated to a certain temperature, the organic content changed to mature and changed in physics and chemistry, so it can produce energy materials such as oil and gas. The testing of TOC that produced carbonates-organic showed excellent quality as shale oil (TOC $\geq$ 12.0%). The results of thermogravimetric analysis showed activation energy of carbonates was 749–1339 kJ/mol and the temperature of the reaction process was 75–740 °C. Organic composition that was larger than carbonate can caused a smaller activation energy. The carbonate content of OD7-Asl2 had $E_a = 1083.7$ kJ/mol smaller than OD7-Asl1 with $E_a = 1338.1$ kJ/mol. A very large TOC value affected the activation energy to be smaller, as the carbonate of OD7-Asl2 was smaller than OD7-Asl1. The maturity of the OD7-Asl2 shale occurred at $T = (380–445)$ °C, $E_a = 1083.7$ kJ/mol and $T_{max} = 415$ °C, better than OD7-Asl1. The Rock Eval Pyrolisis test results showed shale carbonate had a high potential to produce oil and gas. Shale material heating result reinforced by FTIR testing that the compounds with specific functional groups apart and a new peak appeared at wavenumber 2900 cm^{-1} which indicate the presence of hydrocarbons single bonds.

Keywords: Oil Shale, TOC, Activation Energy, Pyrolysis.

RESEARCH ARTICLE

1. INTRODUCTION

Oil shale is a kind of clay or carbonate shale material that contains a lot of organic materials, and an energy source that can produce oil and gas [10] (Kantsler and Cook, 1980). The result of oil shale processing is very useful in the agricultural sector and property industry [1–3]. A research on the oil shale becomes a main research in Soviet Union [21]. Berraja et al. [5] started the research on thermal analysis study at the combustion of oil shale in Tafaya.

From the results of research by Mulyanto et al. [26]: Organic substances that are in sedimentary rocks or carbonate will undergo chemical and physical changes, caused by temperature, heat, pressure and age. Such changes can lead to the formation of oil or gas. To predict the properties of rock containing organic matter, inquiry can be done by looking the chemical and physical properties. To determine the chemical and physical properties, can be used petrophysical and geochemical technology, supported by geological data, seismic technology and technology geothermal (heat).

The *Rock-Eval Pyrolysis* method has been initiated by Katz [19] to analyze the organic material. Bartis et al. [4] did the oil shale exploitation that was gathered and sent to the processing place by burning the oil shale to be utilized as a source of electrical energy. Then Burnham et al. [6] did the extraction on the result of shale material processing which was done on the ground (*ex-situ* processing), and there were some new technologies which carried out the extraction on the shale material underground (*in situ* processing).

The processing of carbonate or clay shale material has not been done in Indonesia, but the reserve of shale material in Indonesia has been mapped. Geological Resource Centre has conducted the research on the oil shale material in 53 locations in Indonesia. The processing of shale material by heating requires some appropriate parameters, so that the changing reaction (maturation) in physics, chemistry and biology can occur in accordance with the desire. Some of the parameters associated with the variation or organic maturity level is the temperature, the energy activation (inversely proportional to the velocity of the reaction) and the material type. In this case, the maturation is defined as maturation of the organic material in the

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carbonate material, or often referred to shale material or oil shale.

From the results of Dewanto et al. [11] research: 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 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.

In this research, the production of carbonate shale material is by compounding the organic material and $CaCO_3$. The compounding is done using the way of weight percentage ratio variation, mixing, mixing time and the last is the result of TOC testing (pyrolysis), where the value of TOC is $\geq$12.0% [32] as a requirement of good oil shale material. The material mixture is modified by the ratio of: organic = calcite and organic > calcite. The TOC testing that produces carbonate shale material shows an excellent quality of oil shale (TOC $\geq$ 12.0%), which is confirmed by the result of SEM analysis (morphology and composition) and XRD (the interaction of two materials). By determining the level of organic maturation in $CaCO_3$, then the task will be more structured and accurate. Reaction stage-1 is *immature*, which is the immature organic material; reaction stage-2 is *mature-1*, which is the mature organic material or starting to crack material; reaction stage-3 is mature-2 and *over mature-1*, which is the organic material turning into hydrocarbon and some generating the gas; reaction stage-4 the release of all gases.

The reaction stages are closely related to the energy activation (including temperature and reaction velocity), and the type of shale material (carbonate-organic). By knowing the parameter value (from the result of the energy activation) and the type of material, then the shale material processing in term of temperature setting can be determined, so that no error occurs in the heating process.

The *Rock-Eval Pyrolisis* testing is not only used to determine the total organic carbon (TOC), but also to determine the value of T_{max} (maximum temperature). Then to determine the maturity of organic material, detect oil and gas reserve and to reidentify the type of some material mixture.

The TGA testing is conducted to determine the value of the energy activation. By conducting a series of tests to obtain a pair of dY/dt and T_{solid}, so the chart of $\ln(dY/dt)$ with $1/T_{solid}$ can be made. Then the straight-line equation of the chart is searched by using linear regression, so the value of the energy activation can be determined from: $E = -aR$, the value of pre-exponential factor (A) is found when the chart of $y = ax + c$ intersects the y axis or $1/T_{solid} = 0$ [7, 15, 31].

The formulation and analysis on the energy activation is based on some previous researchers, namely: Pogaku et al. [27] conducted a research on the energy activation and the velocity enzyme-catalyzed reaction. There are several researchers conducting research on the activation of SiO_2 energy which depends on the input power. Indrati et al. [15] conducted a research on the energy activation of pellet (Th, U)Oz at the stage of pellet growth using a dilatometer and Scanning Electrone Microscope (SEM). Cahyadi et al. [7] conducted a research on the behavior of Indonesia's coal particle ignition using *Thermogravimetric Analysis* at the condition of O_2/N_2 and O_2/CO_2. Sato et al. [28] conducted a research on the activation free energy that has a dependence on the temperature. The temperature dependence is found greater for the calculation. The determination of the energy activation, pre-exponential factor and reaction velocity from TGA analysis refers to several researchers, they are: Katarzyna et al. [18], Himawanto [13], Himawanto et al. [14], Emam [12], Marnoto [23], Yan and Zhang [33], Sugondo [29], Sukma [30], Malika et al. [22], Martono et al. [24], Cantrell et al. [8], Suyitno [31], Kholisoh [20], Minarsih [25], Jiang et al. [16].

Through this research, it is expected that the shale material (Carbonate-Organic) which has been characterized by a variety of methods, can be obtained its physics and chemistry property information to understand the detail of mechanism and the active site on both shale materials, then compare and determine the best one, so that the parameter of laboratorium test can be determined for making the convertion model on the reaction of shale material into crude oil. Further, it is hoped that it can be used as the raw material of the oil shale processing, as the raw material which is excessively available in Indonesia and ready to be used as one of the alternative energy resources.

2. METHOD OF RESEARCH

The Selection of Carbonate and Organic

The stage of determining material type is the most important stage in the material selection. Firstly, choose the material from the result of coring drilling, then the material is grouped, namely the carbonate material group type calcite ($CaCO_3$). The carbonate material must be completely clean from fluid and natural hydrocarbon, therefore prior to the measurement of porosity and permeability, the material should be in dry condition (dry sample). Secondly, determine the material organic cyclic group, namely salicylic acid ($C_7H_6O_3$).

The Characterization of Carbonate and Organic

The tools used for the characterization of clay and carbonate are SEM and XRD, in order to know the identity of the material. SEM is used to know the type of elements, distribution, topography and the surface shape. The XRD

technique can show the type and compound percentage as well as the characteristic of the crystallography.

The Making of Shale Material (Carbonate-Organic) and TOC Testing

The forming method of carbonate shale material is by stirring for a long time, then pressing slowly and leaving to stand for a moment (72 hours) and stirring again and also pressing slowly again, the purpose is to make the organic material fills all pores of the carbonate material. Furthermore, the shale material is left to stand at least 48 hours, the purpose is to make the trapped organic material (fills) the pores and be more binding and cohessive. Then the TOC is tested and made like an oil shale, with the same characteristic, which is TOC $\geq$ 12%.

The Characterization of Carbonate Shale Material

The carbonate shale material that has been made is characterized by using SEM. The purpose is to know the morphology, the particle size, the content of the material, the pores of the material and the elements. Whereas the characterization using XRD has a purpose to know the compound type, the compound percentage and the crystallography, in addition to know the distance of basal area (d_{001}) from natural $CaCO_3$ that has been mixed with the organic material (salicylic acid).

The TGA Testing on the Carbonate Shale Material

Thermogravimetry is a technique to measure the weight change of a compound as a function of temperature or time. The result is a continuous diagram recording; the schematic single stage decomposition reaction. The two types of main thermal analysis are thermogravimetry analysis, which automatically records the weight change of the sample as a function of temperature or time, and differential thermal analysis (DTA), which measures the difference of T temperature between the sample and the referen inert material as a function of temperature.

The Determination of Temperature and Energy Activation

The calculation of the energy activation in this research uses the formula of kinetics calculation of order one reaction or commonly called as *global kinetic*. The determination of energy activation quantity is using graphical method with the formula that is based on Arrhenius equation. The reason in choosing *global kinetic* method is since this research does not consider the elementary reaction that occurs, but only considers the velocity of shale material in reacting, so it can turn into hydrocarbon. The formulation used in global kinetic is:

$$\frac{dx}{dt} = Ae^{-E_a/RT}(1-x) \tag{1}$$

where; dx: The loss in mass fraction; dt: The change of time (dt); A: the pre-exponential factor; e: The natural number (2, 72); E: The energy activation (J/mol) or E_a; R: The gas constant (8, 31 J/mol °K); T: The material temperature (°K).

x is the mass fraction, which is calculated by the formula of,

$$x = \frac{m_0 - m}{m_0 - m_f} \tag{2}$$

where m is the mass of the sample when the time is $ke-t$, m_0 is the initial mass of the sample and m_f is the final mass of the sample. The heating rate is defined,

$$\beta = \frac{dT}{dt} \tag{3}$$

By combining the Eqs. (1)–(3), so the Eq. (1) becomes,

$$\frac{dx}{dT} = \frac{A}{\beta}e^{-E_a/RT}(1-x) \tag{4}$$

$$\frac{dx}{(1-x)} = \frac{A}{\beta}e^{-E_a/RT}dT \tag{5}$$

If both sides are integrated, so the Eq. (5) will be,

$$-\ln(1-x) = \frac{A}{\beta}\int e^{-E_a/RT}\,dT \tag{6}$$

In the Eq. (6), the term $\int e^{-E_a/RT}\,dT$, is an inexact integral but it can be expressed in the *asymptotic series*, so that the Eq. (6) can be integrated to be,

$$-\ln(1-x) = \frac{ART^2}{\beta E_a}\left(1 - \frac{2RT}{E_a}\right)e^{-E_a/RT} \tag{7}$$

$$-\ln\frac{(1-x)}{T^2} = \frac{AR}{\beta E_a}\left(1 - \frac{2RT}{E_a}\right)e^{-E_a/RT} \tag{8}$$

$$\ln\left[-\frac{\ln(1-x)}{T^2}\right] = \ln\left[\frac{AR}{\beta E_a}\left(1 - \frac{2RT}{E_a}\right)\right] - \frac{E_a}{RT} \tag{9}$$

In the fact, the term of:

$$\frac{2RT}{E_a} \ll 1$$

So it can be ignored and the equation becomes,

$$\ln\left[-\frac{ln(1-x)}{T^2}\right] = \ln\left(\frac{AR}{\beta E_a}\right) - \frac{E_a}{RT} \tag{10}$$

By making the connection chart between $\ln[-\ln(1-x)/T^2]$ and $1/T$, the straight line can be obtained where the slope of the line is $-E_a/R$, so that the value obtained is E_a. The delineation chart of the relationship between $\ln[-\ln(1-x)/T^2]$ and $1/T$ heating process as a basis for calculating the energy activation on the shale material heating.

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Determining the Maximum Temperature (T_{max}) by Pyrolysis Testing

T_{max} is the maximum temperature to release the hydrocarbon from the cracking process of the material mixture that occurs during the pyrolysis. T_{max} is an indication of organic maturation stage in the shale-carbonate. The T_{max} value is one of the geochemical parameters used to determine the maturity level of shale-carbonate. The value T_{max} that recorded is influenced by several types of material mixtures mentioned above. Some of the material mixtures will form the different hydrocarbons at the same temperature condition. The T_{max} value as an indicator of maturity also has some limitations such as it can not be used for some types of materials that have low TOC.

3. RESULTS AND DISCUSSION

Figure 1 shows the description of determination of material type to the processing step in the laboratorium, starting from the coring process, the material selection, the production of carbonate-organic material sample, and also the tests.

The Selection of Carbonate and Organic

The carbonate material that has been formed as pellet with a variety of diameter sizes is shown at Figure 2, where the material is already in dry condition.

Some of carbonate materials used is the material with the sample number OD7, while the organic material choosen is group of cyclic compound such as salicylic acid.

The Determination of TOC (Total Organic Carbon) and T_{max} on Carbonate Shale Material

The result of shale material manufacture ($CaCO_3$–$C_7H_6O_3$) shows a very good result as oil shale, because it has a value of TOC $\geq$ 12% and the characteristic is the same as oil shale. The TOC testing is successfully performed on the sample of carbonate-organic material (OD7-Asl). This TOC value is used as one of the parameters for initial selection stage on the material selection in order that can be used as shale material (oil shale), so that the bad and good material can be separated to be the raw material of further processing.

The carbonate-organic shale material has shown the excellent quality, which has a value of TOC $\geq$ 12.0%. Table I shows the value of TOC and T_{max} from the result of pyrolysis that is used as an initial indicator of the thermal maturity level of carbonate-organic shale material. The material maturity shows a varied value, and the carbonate shale of OD7-Asl1 requires a greater temperature than the carbonate shale of OD7-Asl2. The combination between TOC and T_{max} indicates that the carbonate shale material that acts as oil shale is likely more potential as oil and gas.

The SEM Analysis on the Organic Material

Figure 3 shows the SEM image and Edax on the salicylic acid ($C_7H_6O_3$) organic material.

From the result of Edax, the organic material has a dominant content of C and O. The organic material infiltrates into the Kerogen type II which can be formed from some different sources, namely the marine algae, pollen and spore, wax layer of plant, resin fossil, beside that it also derives from the plan fat. This occurs due to the mixing

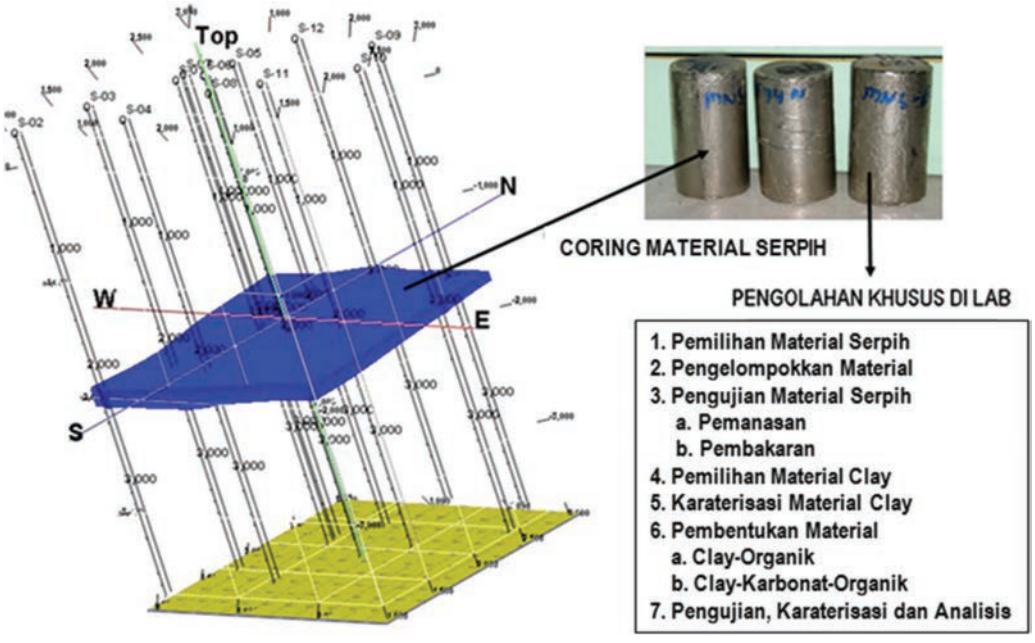


Fig. 1. The coring process, the determination of material type to the processing step in the laboratorium.

Fig. 2. The carbonate material is formed as pellet.

of organic material *autochton* with *allochton* material that dominated by material from plants such as pollen and spore. Its SEM image looks like the bonded white blobs.

The SEM Analysis on the Carbonate Material

Figure 4 shows the result of SEM analysis on the carbonate material (OD7). The carbonate material (OD7) has many pores. Some areas of the pore cavity can be filled with other material (C–E, 2–4). The carbonate material is dominated by *calcite* (D–E, 2–3; B–D, 2–4) and a little dolomite (C–D, 5–6). Besides the carbonate material is dominated by the carbonate, it also has little clay material (illite dan kaolinite) that is situated around the pores. The type of the pore is secondary pore, where the distribution of the secondary pore is caused by the dissolution of planktonic (C–D, 2). From the result of Edax SEM on the carbonate $CaCO_3$, it has a great content of Ca and O (dominant).

The SEM Analysis on the Carbonate Shale Material (Carbonate-Organic)

The surface area of the shale material that has been filled by the organic is smaller, so it can be indicated that the place of the carbonate has been filled by the organic, because the pores becomes smaller. The SEM image of the carbonate shale (calcite) has the dominant secondary pores. At a certain time, the porosity will change and cause the organic material exits and enters the pores. The calcite material is success as a place of perfect maturation of organic material.

Figure 5 shows the carbonate shale material OD7-Asl2 (33% $CaCO_3$ and 67% salicylic acid). From the result of Edax SEM on the carbonate shale OD7-Asl2, it contains

Table I. The result of TOC (total organic carbon) testing.

No.	Sample name	TOC (%)	T_{max} (°C)
1.	OD7-Asl1 (50% $CaCO_3 + 50\%$ $C_7H_6O_3$)	12.01	432
2.	OD7-Asl2 (33% $CaCO_3 + 67\%$ $C_7H_6O_3$)	12.89	415
3.	OD7-Asl3 (67% $CaCO_3 + 33\%$ $C_7H_6O_3$)	9.14	493

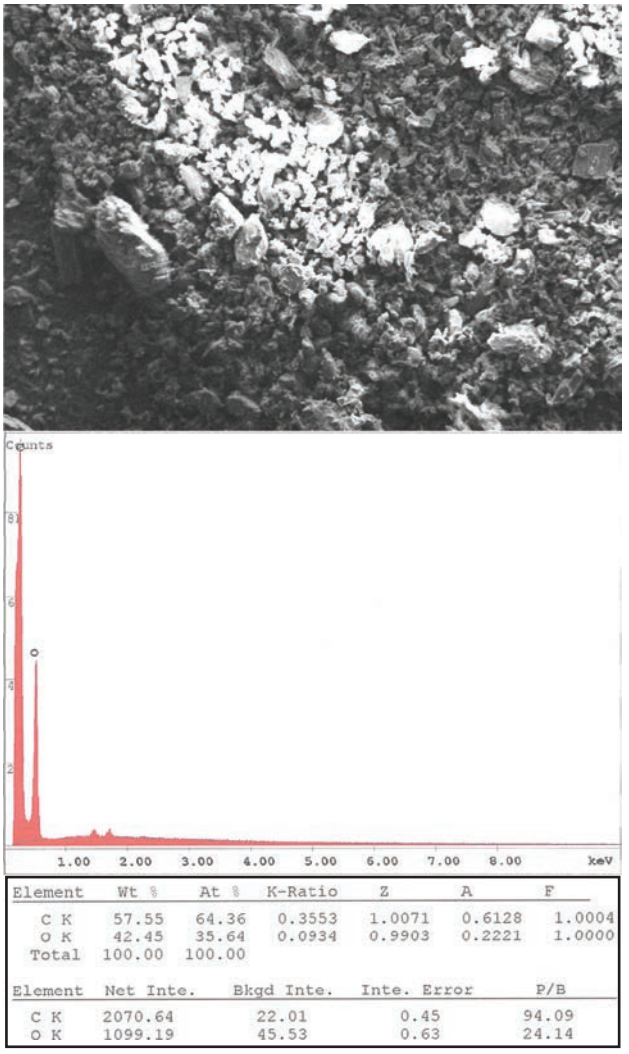
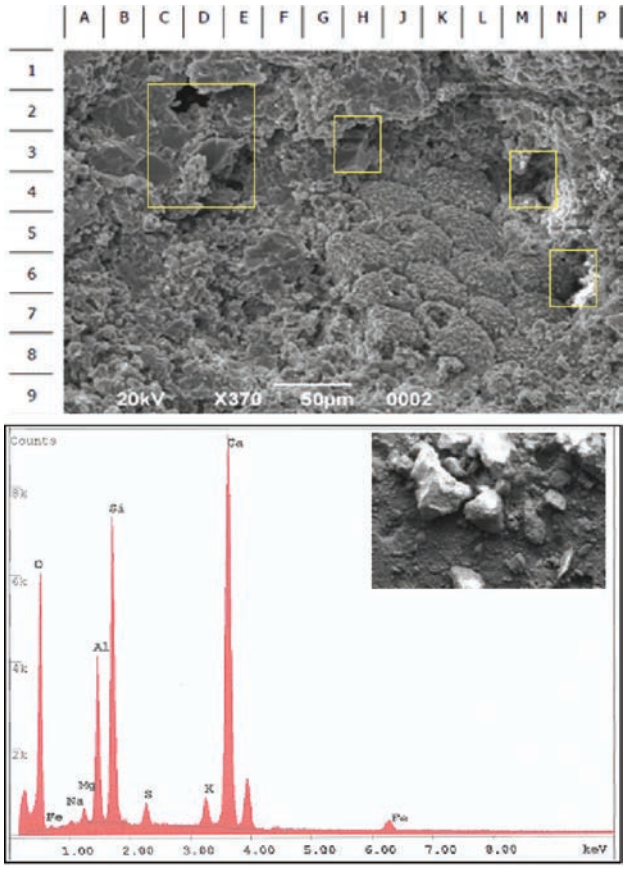


Fig. 3. The image of SEM on salicylic acid.

some elements with (wt, %) dominated by Ca, O and C, as follows Carbon (43.65%), Oxygen (40.81%) and Calcium (11.78%).

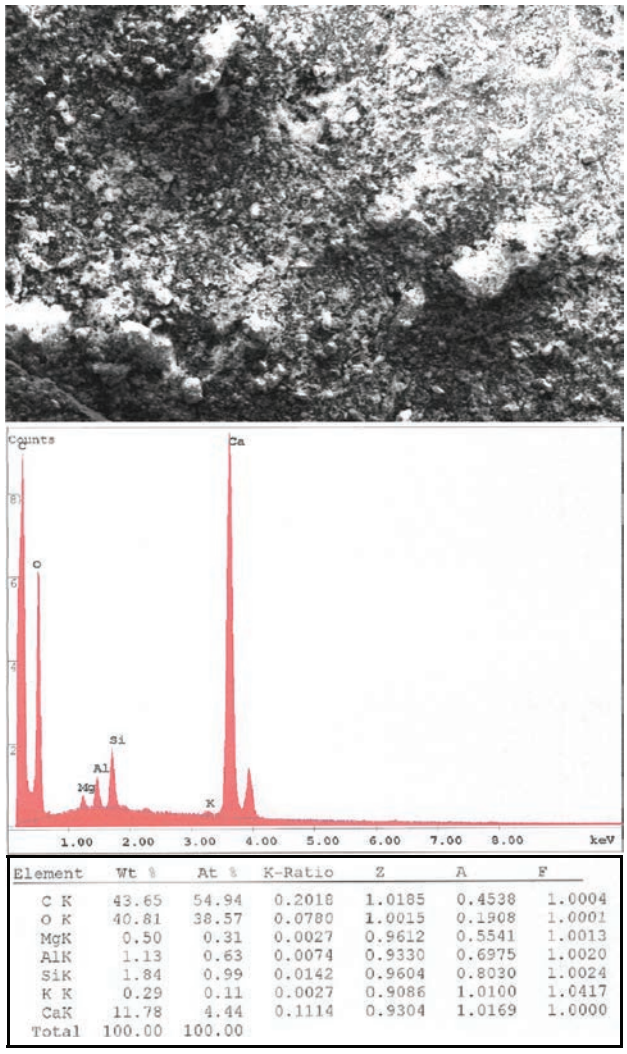
The Measurement of X-ray Diffraction (XRD)

The result of the carbonate OD7 characterization is in the form of diffraction patterns which are the peaks characteristic of crystal $CaCO_3$ structure. Figure 6 shows the chart of the XRD result on the shale material (carbonate-organic) OD7, OD7-Asl1, OD7-Asl2, and OD7-Asl3. The diffraction patterns of $CaCO_3$ are identified at the angle of 2θ. If the peaks of $CaCO_3$ characteristic appear, so the compound phase can be identified. The X-ray diffraction pattern that formed is the result of atoms scattering that is located on the *hkl* plane in the crystal. The carbonate OD7 has five high peaks and the intensity will change when there is an organic addition. The angle 2θ position is at the angle of 26.66°, 29.48°, 36.05°, 39.50° and 43.27° with the intensity values captured by the X-ray detector are 779 cts,



Element	Wt %	At %	K-Ratio	Z	A	F
O K	50.74	67.17	0.1207	1.0291	0.2312	1.0003
NaK	0.31	0.29	0.0011	0.9631	0.3741	1.0032
MgK	0.98	0.85	0.0051	0.9872	0.5257	1.0063
AlK	9.97	7.83	0.0638	0.9582	0.6629	1.0073
SiK	16.29	12.29	0.1114	0.9861	0.6913	1.0029
S K	1.27	0.84	0.0096	0.9716	0.7664	1.0075
K K	1.83	0.99	0.0163	0.9356	0.9277	1.0310
CaK	18.00	9.51	0.1634	0.9577	0.9477	1.0003
FeK	0.61	0.23	0.0052	0.8701	0.9857	1.0000
Total	100.00	100.00				

Fig. 4. The result of SEM and Edax OD7 (calcite).



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				

Fig. 5. The image of SEM on the material of OD7-Asl2.

20383 cts, 3477 cts, 4172 and 3252 cts. The peaks that formed are the peak of crystal Graphite C_1H_2 and Calcite $CaCO_3$ and Caron. The crystal planes or Miller indices *hkl* are (011), (104), (110), (161) and (202).

The carbonate shale material OD7-Asl1 has five highest peaks at the same angle position of 2θ as shale material OD7-Asl2, which are at the angle of 10.94°, 17.21°, 25.23°, 28.70° and 29.41°, with the intensity values captured by the X-ray detector are 47282, 44513, 18466, 8361 and 12199 cts. The peaks that formed are the peak of crystal Carbon Dioxide, Fichtelite $C_{19}H_{34}$, Calcium, Carbon and Calcite $CaCO_3$, and it can be identified that the crystal planes or Miller indices *hkl* are (100), (200), (110), (231) and (104). The shale material (carbonate-organic) OD7-Asl3 has 5 highest peaks at the position of 2θ, which are 10.96°, 17.23°, 25.24°, 26.61° and 29.43°, with the intensity values captured by the X-ray detector are 12771, 13314, 4687 and 18095. From the result, it can be concluded that the peaks that formed are the peak of crystal Fullerite, Nitrammite $N_2H_4O_3$ and Calcite $CaCO_3$, and it can be identified that the crystal planes or Miller indices *hkl* are (111), (101), (002), (003) and (104). The addition of organic material ($C_7H_6O_3$) is performed on the natural carbonate (OD7) with a particular composition. At the angle of 2 theta = 29° with the same *hkl* plane, the calcite (104) condition experienced on the shale material is the intensity of OD7 ≥ OD7-Asl3 > OD7-Asl1 > OD7-Asl2. While at the angel of 2 theta = 25° and the angle of 2 theta = 17°, the condition experienced on the carbonate material is the intensity of OD7 ≤ OD7-Asl3 < OD7-Asl1 < OD7-Asl2. The result of XRD characterization on the carbonate shale material which has the highest intensity is shown on the Figure 6 and Table II.

The organic addition causes the *preferred orientation* on the specific crystal planes, it leads the crystal planes having the higher intensity than before. While the angle of 2 theta and its *hkl* plane do not change. Based on the image, it can be seen that the intensity of X-ray that absorbed by

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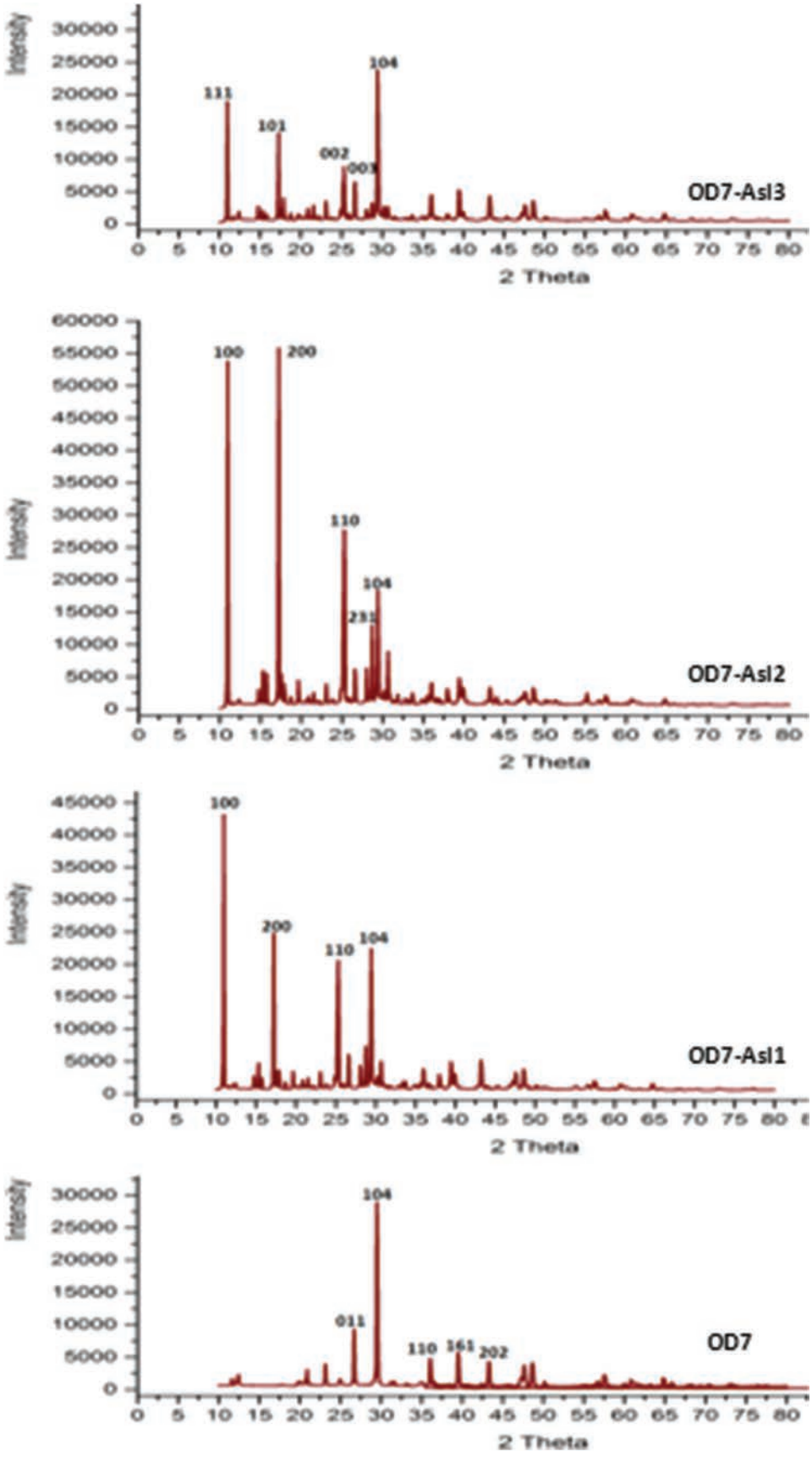

Fig. 6. The XRD characterization on the carbonate and shale material (carbonate-organic).

Table II. The XRD characterization on the carbonate shale material which has the highest intensity.

No.	Material name	Pos. [°2Th.]	Height [cts]	*d*-spacing [Å]	*hkl*	Compound
1.	OD7	29.48	20383	3.03	104	Calcite, $CaCO_3$
2.	OD7-Asl1	10.94	46314	8.08	100	Carbon dioxide
3.	OD7-Asl2	10.94	47282	8.08	100	Carbon dioxide
4.	OD7-Asl3	29.43	18095	3.03	104	Calcite, $CaCO_3$

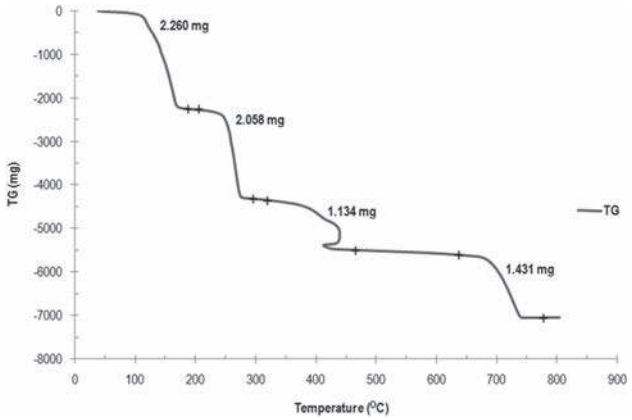

Fig. 7. The chart of thermogravimetry (TGA) analysis result on the shale material (OD7-Asl1).

the detector on every sample, has different value. High or low the X-ray intensity that captured by the X-ray detector is influenced by the level of the regularity of atom formation in the crystal that is diffracted by X-ray. The more atoms are structured regularly, the higher the intensity that captured by the detector. When the intensity is greater means the sample has the greater crystal regularity or more well arranged atoms in the layers. The X-ray diffraction patterns that formed are the result of atoms scattering that located at the *hkl* plane in the crystal. Any difference or change in the compound and *hkl* plane at the same angle of 2 theta at every organic addition, it means the reaction occurs between $CaCO_3$ material and organic.

The Result of TGA Testing on the Shale Material (Carbonate-Organic)

The test is carried out on the carbonate shale material OD7-Asl1, with the composition of 50% $CaCO_3$ and 50% $C_7H_6O_3$. The result of TGA testing on the shale material OD7-Asl1 is shown on Figure 7. The first change occurs at the temperature of 75 °C–170 °C, this is where the loss of water molecules happens in the crystal structure. At the temperature of 225 °C–275 °C, the second weight change that significant occurs, and it is indicated as the change in the structure on the carbonate shale material and the loss of water molecules chemically.

When the calcination is reperformed, the weight loss occurs at the temperature of 325 °C–450 °C, then the molecules in the carbonate shale are released, so that it will affect the change on the pore size. The interval temperature of 325 °C–450 °C indicates the maximum temperature that required for carbonate shale to turn into oil. The last change phase occurs at the temperature about 650 °C–740 °C. This condition can be called as over mature, where the chart line has shown a tendency of straight horizontal. Physically, the material that

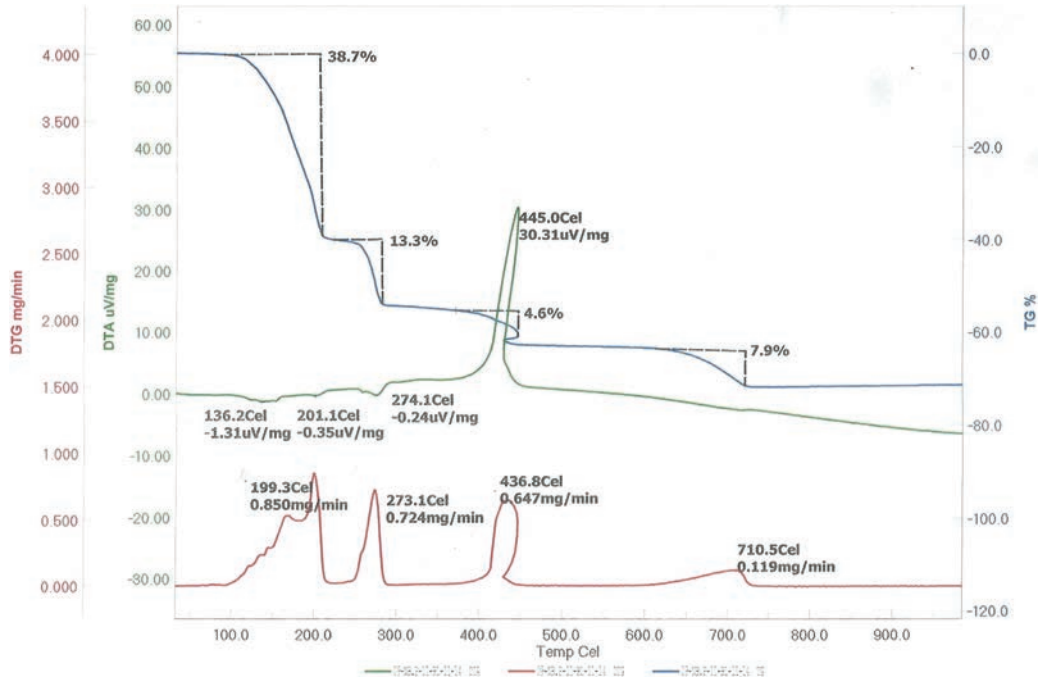

Fig. 8. The chart of thermogravimetry (TGA) analysis result on the shale material (OD7-Asl2).

has been calcined at the temperature above 740 °C, the color becomes blackish. So the temperature that required at the process of the change (reaction) of oil shale material on the carbonate shale to be oil is about ±(325 °C–450 °C).

Then the TGA testing is performed on the material OD7-Asl2 (calcite-salicylate), namely the test on the carbonate shale material where the composition is the mixture of 33% $CaCO_3$ and 67% $C_7H_6O_3$. This test is performed to know the structural damage when heating at the high temperature is carried out (over 400 °C), because it can provide the description of the change process in the substance mass. The result of TGA testing on the shale material OD7-Asl2 is shown on the Figure 8.

The first weight loss occurs at the temperature of ±95 °C–205 °C, it indicates the water molecules apart from the crystal structure of OD7-Asl2. Then the second weight loss occurs at the temperature around 225 °C–285 °C. The second significant weight change indicates the change in the structure of the material OD7-Asl2 and the loss of water molecules chemically. Then the third weight loss occurs at the temperature of 380 °C–445 °C, where the molecules in OD7-Asl2 is apart. The interval of temperature indicates the maximum temperature that required by OD7-Asl2 (carbonate shale) to begin turning into oil. When the calcination is always performed to the last loss until becoming constant, which is at the temperature of 610 °C–720 °C, then many molecules in OD7-Asl2 (the carbonate shale) are apart, so that there is part of the shale in the pores is also apart. This condition can be called as *over mature*, where the line of the chart shows a tendency of straight horizontal along with the increasing of the temperature up to 720 °C. Physically, the material that has been calcined at the temperature above 720 °C, the color becomes blackish. So the temperature required for the maturation process of the oil shale OD7-Asl2 into crude oil is about ±(380 °C–445 °C).

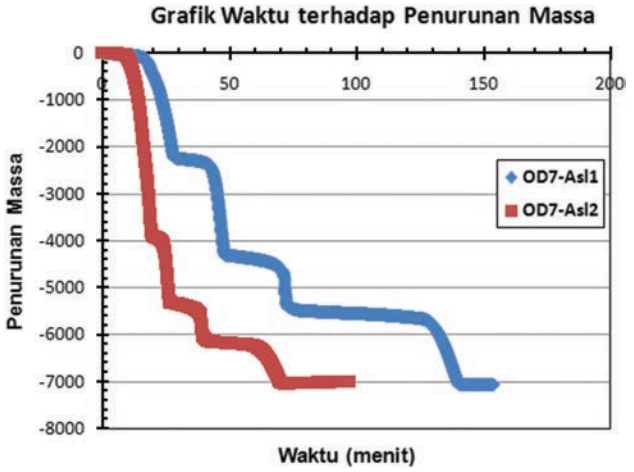

Fig. 9. The chart of mass loss versus time of reaction on the shale material (carbonate-organic).

The Duration of Heating the Material

The carbonate shale material OD7-Asl2 undergoes the heating process faster than the material of OD7-Asl1.

Figure 9 shows the relationship between the mass loss versus the time on the carbonate shale material. The

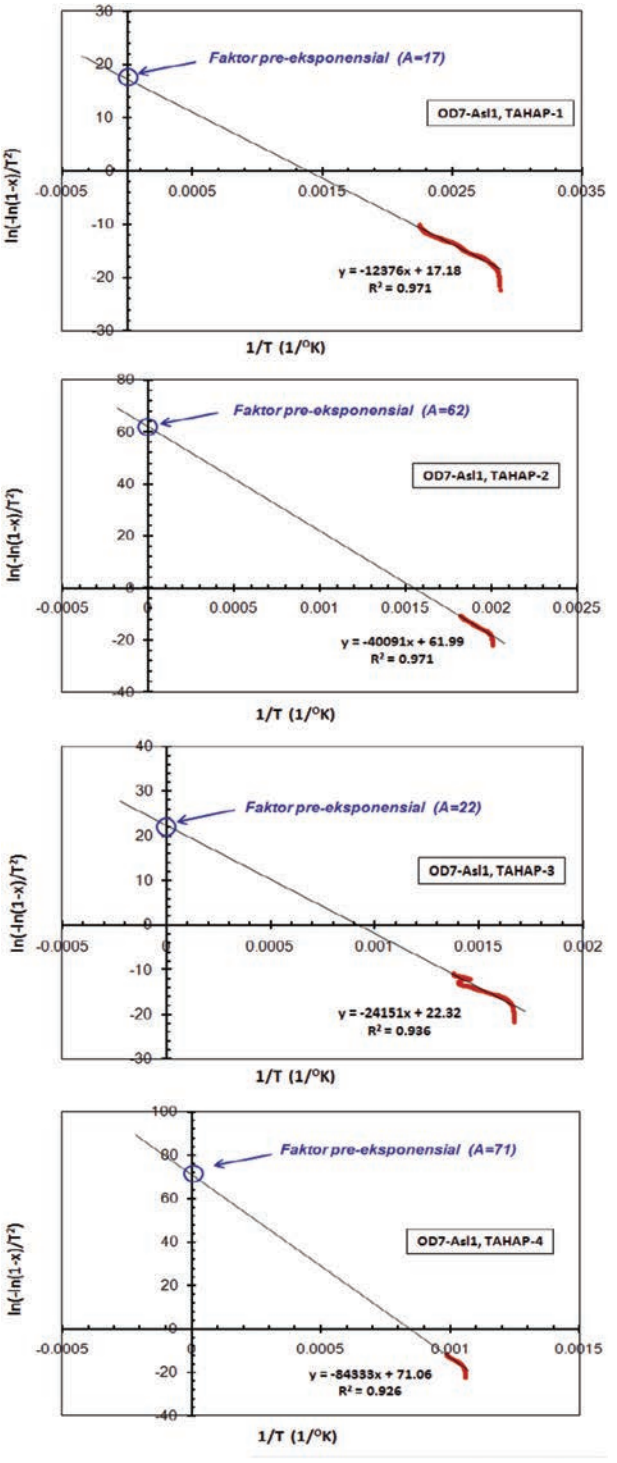

Fig. 10. The chart of $\ln[-ln(1-x)/T^2]$ versus $1/T$, for E_a and A determination on the OD7-Asl1 using TGA.

greater ratio of organic composition than calcite causes maturation reaction in the heating process becoming faster.

The Energy Activation and Pre Exponential Factor of Shale Material OD7-Asl1

The TGA gradual decomposition process that is used alone or combined with DTA can separate and determine each stage. On this shale material sample (carbonate-organic), the four-level decomposition occurs, it can be seen on Figure 7. The result of TGA shows that the decomposition level of shale material is divided into four levels of temperature, which are level I is between the temperature of 75 °C–170 °C, level II occurs between the temperature of 225 °C–275 °C, level III is between the temperature of 325 °C–450 °C and level IV occurs between the temperature of 670 °C–740 °C. Figure 10 shows the method of determining the energy activation (E_a) and the pre exponential factor of OD7-Asl1. In the process of heating the material OD7-Asl1, the decomposition goes through four stages. The TGA and DTA can be used in a variety of kinetic studies. The fast and accurate TGA method is used to study the decomposition reactions isothermally. This process can be repeated at other temperature and the result is analyzed to determine its energy activation.

The energy activation (E_a) values and pre exponential factor of each reaction stage at 4 levels reaction are shown in Table III.

The total value of the energy activation on the heating of the material OD7-Asl1 is $E_a = 1338.1$ kJ/mol and the pre exponential factor is $A = 172$.

The Energy Activation and Pre Exponensial Factor of Shale Material OD7-Asl2

The gradual decomposition process on the shale material OD7-Asl2 (carbonate-organic) occurs on four levels decomposition, it is shown on the result of TGA testing on Figure 8.

The decomposition level of shale material OD7-Asl2, from the result of TGA is divided into four levels of temperature, which are level I is between the temperature of 95 °C–205 °C, level II occurs between the temperature of 225 °C–285 °C, level III occurs between the temperature of 380 °C–445 °C and level IV occurs between the temperature of 610 °C–720 °C. The chart of the energy activation (E_a) and pre exponential factor determination of OD7-Asl2 is shown on Figure 11. The total value of the energy activation on this shale material (the organic ratio is greater) has value of $E_a = 1083.7$ kJ/mol, and the pre exponential factor of $A = 137.2$, where the value of E_a and A is smaller than the OD7-Asl1 material, so that the velocity of the reaction is faster. The complete energy activation (E_a) values and pre exponential factors for each reaction stage on 4 levels temperature are shown in Table IV.

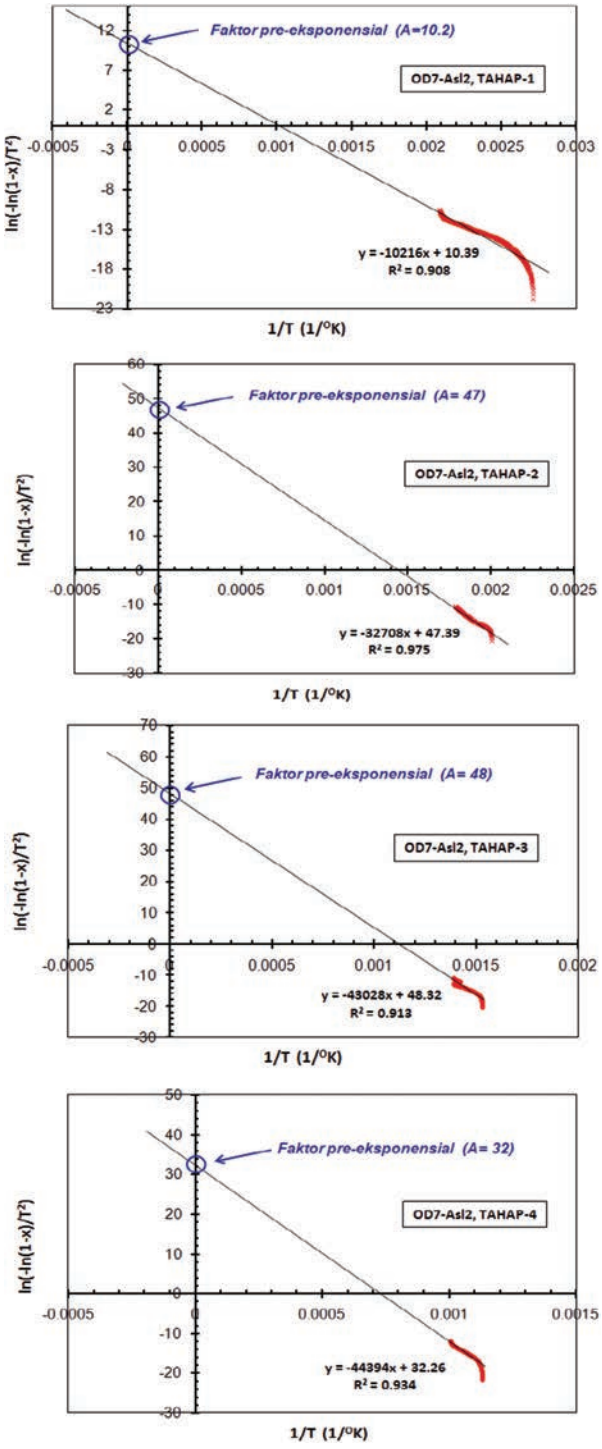

Fig. 11. The chart $\ln[-\ln(1-x)/T^2]$ versus $1/T$, for E_a and A determination on OD7-Asl2 using TGA.

Table III. The E_a and A values of OD7-Asl1 material.

Reaction level	E_a (kJ/mol)	A (%/s)
75 °C–170 °C	102.89	17
225 °C–275 °C	333.32	62
325 °C–450 °C	200.79	22
670 °C–740 °C	701.14	71

Table IV. The E_a and A of OD7-Asl2 material.

Reaction level	E_a (kJ/mol)	A (%/s)
95 °C–205 °C	84.936	10.2
225 °C–285 °C	271.93	47
380 °C–445 °C	357.73	48
610 °C–720 °C	369.09	32

4. CONCLUSION

The energy activation of carbonate shale material is E_a = 749–1339 kJ/mol and temperature for the process of carbonate shale material reaction is T = 75–740 °C.

The composition ratio (wt.%) of organic that is greater than carbonate causes the carbonate shale material (with TOC $\geq$ 12%) having a lower energy activation. The carbonate shale material of OD7-Asl2 (33% carbonate +67% organic) has E_a = 1083.7 kJ/mol lower than OD7-Asl1 (50% carbonate +50% organic) that has E_a = 1338.1 kJ/mol.

The TOC value that is so high, influences the energy activation becoming smaller (see number 2), namely carbonate shale material OD7-Asl2 is smaller than OD7-Asl1.

The maturity of shale material OD7-Asl2 occurs at T = (380–445 °C), E_a = 1083.7 kJ/mol and T_{max} = 415 °C, it is better than OD7-Asl1 which occurs at T = (325–450 °C), E_a = 1338.1 kJ/mol and T_{max} = 432 °C.

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