Comparative Study of Kraft Paper Aged in Natural Ester with XRD and TG/DTG Analysis

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Abstract: High voltage transformer is an essential electrical equipment that determine the reliability and continuity of power system. Basically, it consists of solid and liquid insulating materials. Kraft paper as solid insulation should have an ability to insulate the winding connection, while oil transformer, aside of its main function as an insulation, it is essential to provide cooling mechanism. This paper analyzes the accelerated thermal aging performance of kraft paper immersed in the natural ester from RBD palm oil by using X-Ray Diffraction (XRD) characterization and thermogravimetric analysis. XRD characterization was used to determine the existence of crystalline structure in kraft paper. Crystalline structure in kraft paper is composed by cellulose. The toughness of kraft paper was determined by its amorphous region, lignin, that bind the cellulose and make them hard. The influence of thermal expansion was observed to discover the effect of thermal stress to the crystallinity of kraft paper. Besides, thermal degradation characteristics of kraft paper were examined by using thermogravimetric analysis. The sample was aged in 120^0C and 150^0C for two weeks. The results also show that crystalline region of the sample that heated in higher aging temperature tends to drop faster than the sample with lower aging temperature.

Keywords: thermal aging, kraft paper, natural ester, palm oil, XRD, thermogravimetric analysis.

1. Introduction

Transformer is the one of the most important high voltage apparatus in power transmission and distribution system, which converting voltage level from one to another. Transformer provide an essential role in determining the reliability and efficiency of power transmission. Over a hundred-year, transformer employee mineral oil as its insulation material, owing to preferable its stability of chemical and thermal performance during operation [1]. Mineral oil is not only playing a role as insulation material, but it is also used to dissipate heat to the environment [2]. However, the usage of mineral oil creates a new problem. Firstly, mineral oil is a hazardous material and it can be a contaminant when it spills on soil and water-ways, disturbing the ecosystem of water and soil organism [3]. Secondly, it is going to run out in the future since its raw material, petroleum, which is nonrenewable energy [4]. Third, its limited fire resistance creates an issue when it should be operated in high operation temperature [1,2]. Natural ester was projected to be a new environmentally friendly insulation material that replacing mineral oil. In Germany, natural ester is considered as nontoxic material due to aquatic biodegradation tests [5]. In addition, its fire point as insulation liquid is more than 300^0C, which is good for high temperature operation.

Kraft paper is one of the first insulation material that is commonly used in high voltage equipment. In transformer operation, the main function of kraft paper is to separate the winding connection. Due to thermal and electrical aging, kraft paper and liquid based insulation are degrade. The degradation characteristic of kraft paper is irreversible. Therefore, lifetime of transformer is then determined by its solid insulation condition. Paper degradation is mainly determined by using degree of polymerization, furan analysis, and tensile strength measurement. Degree of polymerization determine average length of cellulose chains. Besides, the reaction between oil and paper during operation yields a stable molecule which accumulated in oil namely furan. Tensile strength measurement generally used to determine the mechanical strength of kraft paper which also getting down by aging duration [6]. Based on our latest research before, there is

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a deep correlation between the decrease of degree polymerization and its mechanical strength, that is mainly influenced by initial moisture, aging duration and aging temperature.

Several measurement analyses have been done to determine the characteristic of paper degradation immersed in natural ester oil. In this paper, X-Ray diffraction (XRD) characterization and thermogravimetric analysis (TG/DTG) measurement was used to analyze the transformation of crystalline structure and thermal degradation characteristics in initial paper condition (T0), sample aged in 120^0C for 336 hours (T120), and sample aged in 150^0C for 336 hours (T150). The relative crystalline of kraft paper is decreasing during the aging due to high aging temperature, water molecule and oxygen dissolved lead the degradation processes in both paper and oil. Besides, it can be shown that cellulose, hemicellulose and lignin take an essential part in the thermal degradation of kraft paper.

2. Methodology

A. Sample Preparation

The sample used in the experiment were kraft insulating paper and natural ester from palm oil. The oil has made in the laboratory through transesterification process. 4 liter of methanol is reacted with 20 liters of RBD palm oil in the reaction chamber with 216 grams of KOH as catalyst. The transesterification process results are alkyl ester which is then be used as insulation liquid and byproduct glycerol. The ester oil has passed new ester oil standard based on IEEE C 57.147/IEC 62770:2013. Oil characteristic is shown in Table 1.

Table 1. Oil Sample Characteristics

Oil Characteristic	Initial condition	New ester oil standard (IEEE C 57.147/IEC 62770:2013)
Visual	Bright and Clear	Bright and Clear
Resistivity	2.2×10^{10}	-
Breakdown voltage gap 2 mm (kV)	70.3	≥ 35
Oil moisture (mg/kg)	262.33	≤ 300
Acidity (mg KOH/g_{oil})	0.0315	≤ 0.06
Color	L 0.8	≤ 1.0
Dielectric losses (%)	0.15	≤ 0.5

(a) (b)

Figure 1. (a) Copper wrapped by kraft paper; (b) Sample under accelerated thermal aging

In order to obtain the real condition of transformer, the paper was wrapped into copper coil and then dissolved into the bottle of natural ester oil, shown in Figure 1. Natural ester oil, paper, and coil in a bottle was each 600 grams, 60 grams and 26.25 grams [7,8]. A pre-treatment process was carried out on the ester oil by putting in the oven at 100^0C for 24^0C. The sample were then subjected to accelerated thermal aging at 120^0C and 150^0C. 150^0C was chosen in accordance to the hot-spot temperature for bushing insulation, while 120^0C was chosen in accordance with the maximum hotspot of power transformer according to IEEE [5].

Sampling were taken regularly for every two weeks. Meanwhile, the aging was conducted for 1008 hours. In this experiment, a list of samples and its treatment is shown in Table 2.

Table 2. Samples and their treatments

Sample	Treatment
T0	Initial condition
T1.120	120^0C for 336 hours
T2.120	120^0C for 672 hours
T3.120	120^0C for 1008 hours
T1.150	150^0C for 336 hours
T2.150	150^0C for 672 hours
T3.150	150^0C for 1008 hours

B. Scanning Electron Microscopy (SEM)

Scanning electron microscopy is a method in imaging the morphology of the material over large range of magnification. An electron beam with low energy was radiated to the surface of material observed, interaction between both of them, lead the emission of photons and electrons from or near the sample surface. The detector will receive signals that is produced from electron and sample interaction and consequently transform it into an image. Figure 2 is the Hitachi SU 3500 that is used for SEM analysis. In this experiment, degradation of kraft paper due to thermal aging is observed by analyzing the its morphological structure. In order to be analyzed, sample under observation should be in a dry condition. Therefore, kraft paper was dissolved in methylene chloride for several minutes to remove the oil. The sample was then cut down into 1x1 cm and measured with 200, 500, and 1000 times of magnification.

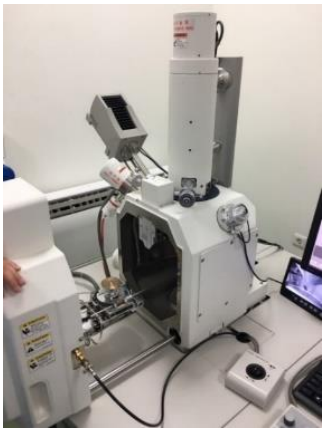


Figure 2. SEM analyzer SU3500 Hitachi

C. X-Ray Diffraction

Figure 3 represent the schematic work of XRD analyzer and the measurement tools, Bruker D8 Advance. X-Ray diffraction phenomenon was reported by W. H. Bragg in 1913 when the beamed X-rays into crystals of a specimen [9]. Part of X-ray will be diffracted and transmitted. The interaction between X-ray diffraction and specimen crystal is then used to identify the crystallographic structure. Braggs law is then used to measure distance between the crystal (d-spacing) of the specimen atoms [10].

$$n\lambda = 2d \sin\theta \quad (1)$$

Where n is integer (order of diffracted beam), λ represent the wavelength of X-ray, d is the spacing of the specimen, and θ is the angle between specimen plane and incident ray, which

known as Bragg angle. The sequential d-spacing is obtained at different incident angles, creating a fine pattern of diffraction peaks that represent the crystallographic structure of the sample called diffractograph [11].

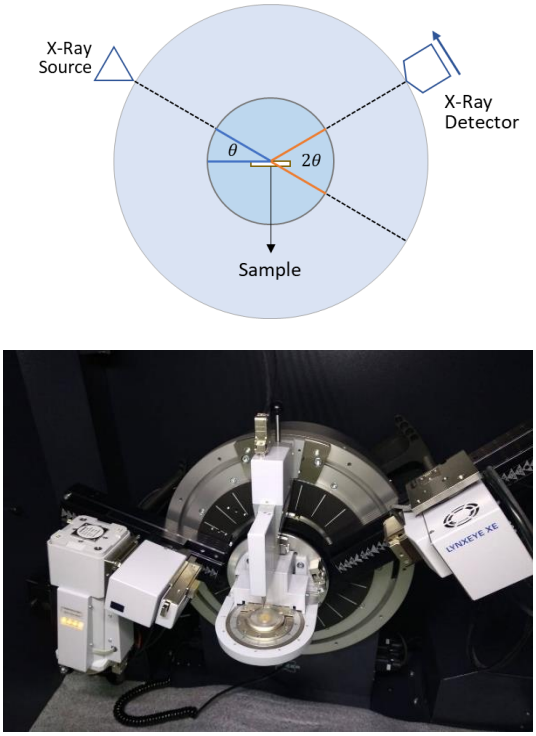


Figure 3. Schematic XRD Characterization and Bruker D8 Advance

D. Thermogravimetric (TG) Analyzer

Thermogravimetric analysis was done in order to analyze the pyrolysis characteristics of kraft paper. This measurement was done by using a thermogravimetric analyzer Hitachi STA7300 shown by Figure 4 below. During TG measurement, the weight of the paper was constantly measured. Specifically, thermogravimetric measurement will measure and record the dynamics of sample weight loss while increasing the temperature. 5 mg of kraft paper was cut into small pieces and then placed into aluminum crucible. The pyrolysis characteristics was carried out with heating temperature range of 30-550 ^{0}C and heating rate was controlled at 10^{0}C per minute. TGA was conducted by using nitrogen as the carrier gas so that the pyrolysis can take place in an inert environment without oxygen [12]. As the measurement take place, the other measurement such as derivative thermogravimetric (DTG) and differential thermal analysis (DTA) was carried out. DTG curve is the first derivative of the TG curve that determine the inflection points and represent the rate of mass loss. While, DTA is a technique to monitor the temperature difference between reference and sample as the temperature is increased.

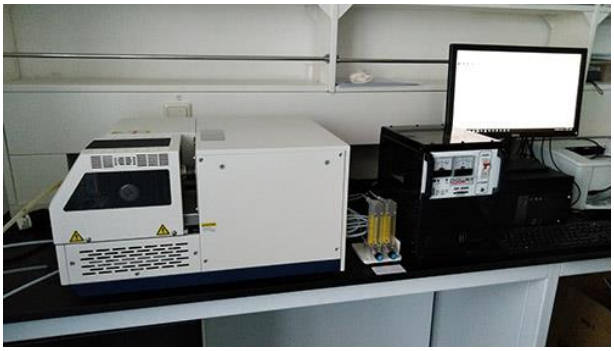


Figure 4. TG Analyzer Hitachi STA7300

3. Result and Analysis

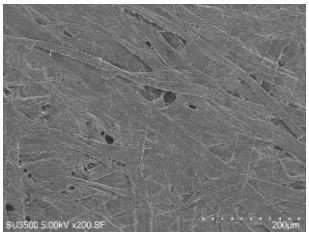
A. Visual and SEM Observation Result

The visual appearance of new kraft paper, sample aged at 120^0C and sample aged at 150^0C is shown in Table 3. The significant color transition can be seen clearly from initial paper condition to sample which aged at 1008 hours. It changes from the brighter one to the dark brown color, indicated that significant degradation had occurred.

Table 3. Visual Appearances of Kraft Paper samples aged at different temperatures and aging times

Temperature	Aging Duration (h)			
	0	336	672	1008
120^0C				
150^0C				

Kraft paper comprises by a long microfibril structure with 90% of cellulose, 6-7% of hemicellulose and 3-4% of lignin [13]. The formation of cellulose fibers is well-organized, increasing the incompressibility, rigidity, deformation resistance, plasticity, and toughness of kraft paper [14]. Whereas, hemicellulose and lignin form an irregular formation known as amorphous region. Lignin are gums and resins which bond the cellulose fiber, making them hard and compact. Therefore, toughness level of cellulose is mainly determined by its amorphous structure. The sensitivity of amorphous region to the temperature is higher than crystalline region. The crystalline structure can perform relatively stable under high temperatures [15]. The morphological surface of initial kraft paper was then observed with Scanning Electron Microscope (SEM) analysis in 200, 500 and 1000 magnifications showed in Figure 5.



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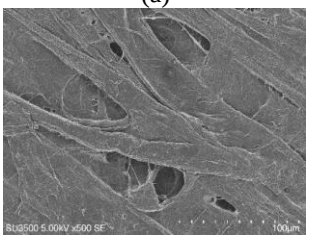

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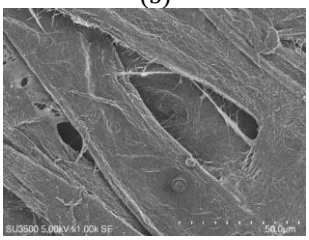
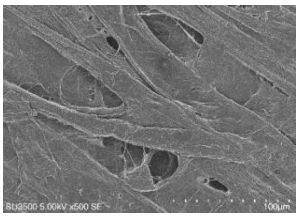


(c)

Figure 5. SEM results for new sample with (a) 200; (b) 500; (c) 1000 times magnifications



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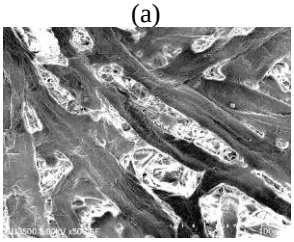

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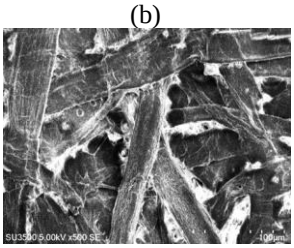


(c)

Figure 6. SEM results of (a) new sample ; (b) T3.120; (c) T3.150

The comparation of morphological structure of kraft paper before and after aging period is shown in Figure 6. It is clearly shown that in the initial condition the microfibril structure is well elongated. However, thermal aging usually starts from the amorphous region since it is sensitive to the high temperature and water molecule are more concentrated in this area. Thermal aging leads the development of microcrack on the microfibril side. Microcrack is then gradually ruining the deeper layer of cellulose. As a result, the average width of microfibril is getting thinner and it will affect the mechanical strength of the paper. Irregular structure on the side of cellulose after aging as shown by Figure 6 (b) and (c) is predicted to be the amorphous region which is formed from crystalline degradation.

B. X-Ray Diffraction Analysis

Crystalline structure and crystallinity are the key properties of crystalline polymer such as cellulose for deciding its electrical and mechanical performance. The flexibility of cellulose fiber decreases and their rigidity increases with increasing ratio of crystalline to amorphous regions [16]. XRD analysis determine the crystallinity of a cellulose and its chemical phase by identifying its length, width, height, and diffraction angle. The XRD diffraction spectrum of new kraft paper is described in Figure 7. From Figure 7, sharp peak correspondents to cellulose fibers and broad peak correspondents to amorphous regions of insulation paper [17]. Diffraction pattern for new kraft paper has two different peaks at $2\theta = 15.57^0$ and $2\theta = 22.29^0$, which indicated the diffraction of the crystalline structure.

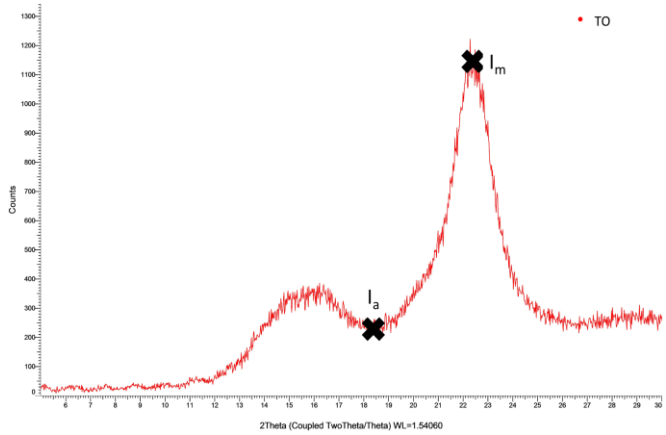


Figure 7. XRD spectrum for new paper insulation

By interpreting the diffractograph, some of parameters such as crystal size (D) and relative crystallinity (C_{rel}) can be measured [18]. Relative crystallinity of the kraft paper is calculated by using equation (2) [17]:

$$\%C_{rel} = \frac{I_m - I_a}{I_m} \times 100 \quad (2)$$

Where I_m and I_a show the intensity of crystalline and amorphous structure shown in Figure 7. Figure 8 shows the XRD spectrum of the sample which was aged in 120^0C and 150^0C of temperature for 1008 hours. Relative crystalline peak for new and aged paper remain the same. The peak positions are around $2\theta = 15.57^0$ and $2\theta = 22.29^0$. It indicates that during accelerated thermal aging, the crystal type has not changed [18]. By using equation (2), the relative crystallinity of kraft paper can be calculated. Relative crystallinity of kraft paper is tabulated in Table 2. It can be observed that relative crystallinity of kraft paper is decreasing. The crystallinity of new paper is 62.9%. Aging temperature have a big influence on the loose of crystalline formation in the kraft paper. Basically, the aging processes is begun with the deterioration of amorphous structure in the outer side of kraft paper. Amorphous structure is known as a region

which bind water molecules. Therefore, hydrolysis reaction mostly took place in this region, ruining the cellulose formation and devastating the deeper layer.

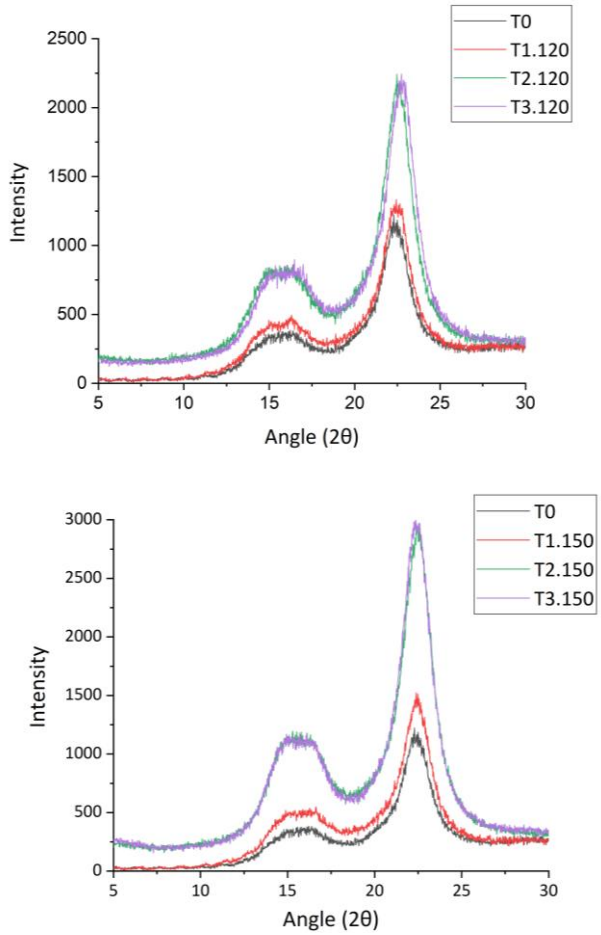


Figure 8. XRD spectrums of samples aged at (a) 120^0C and (b) 150^0C at different aging times

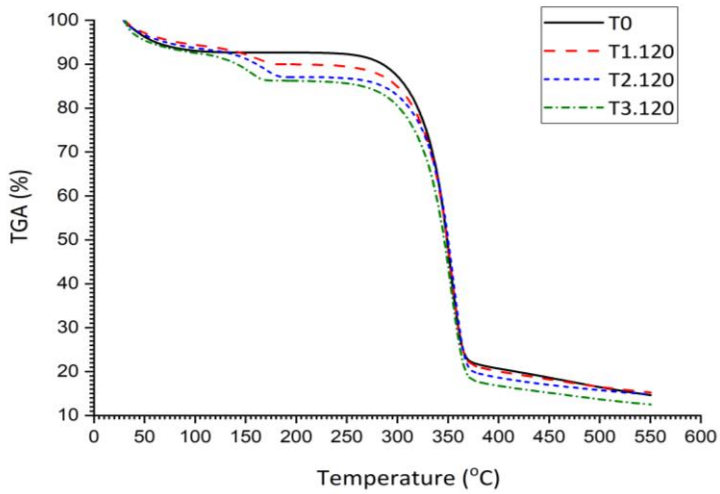
Table 2. Relative crystallinity of kraft paper samples before and after aging

Sample	*Crystallinity*	*Amorphous*
T0	62.90%	37.10%
T1.120	60.30%	39.70%
T2.120	50.40%	49.60%
T3.120	44.20%	55.80%
T1.150	57.80%	42.20%
T2.150	50.60%	49.40%
T3.150	49.80%	50.20%

C. Thermogravimatric Analysis

Thermogravimetric analysis (TG) is often used to measure the residual solvents and moisture. Moreover, it is also used to analyze the thermal stability, oxidation, and vaporization of a polymer material [19]. Thermogravimetric curve gives the percentage of weight loss as a function of temperature or time. Figure 9 shows the results of the thermogravimetric analysis performed on the different aged paper. Based on Figure 9, the decomposition of cellulose due to thermal heating can be divided into three stages.

Initial weight loss is taken place at around 30-100 ^{0}C. The weight of kraft paper has reduced for about 7% during this stage. Weight loss in the lower temperature may correspond to the water vaporization or breaking of water linkage [20–22]. The next stage of weight loss may correspond to the degradation of the whole polymer. The rate of weight loss and total weight loss is important to be observed during these stages.



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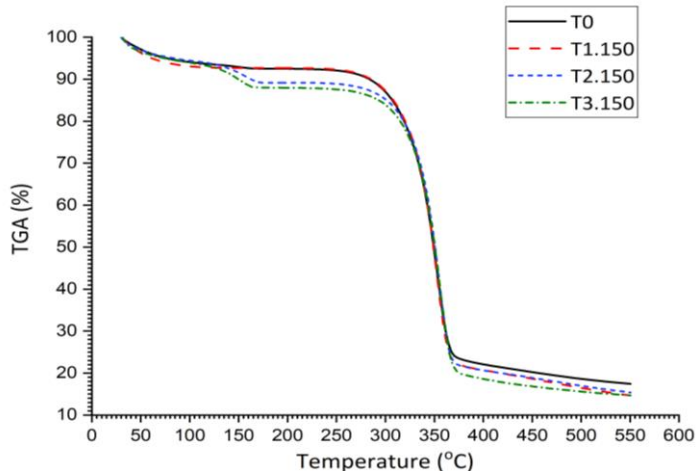
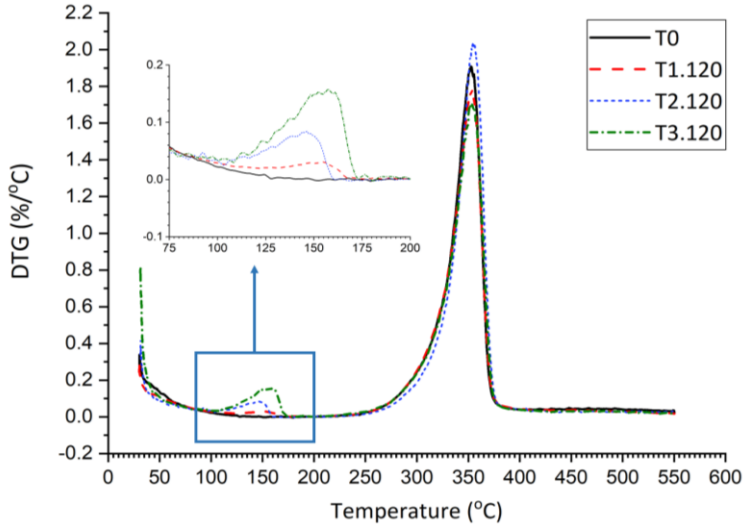


(b)

Figure 9. TG curves of samples aged at (a) 120^{0}C and (b) and 150^{0}C at different aging times

Further thermal degradation is occurred in the second stage. The second stage degradation approximately take place at 300-375^{0}C. A prominent peak at around 352^{0}C that can be observed through DTG curves as shown in Figure 9 is corresponding to the degradation of cellulose and maximum decomposition rate [22]. As can be seen, compared to first stage degradation, a more significant weight loss has been observed. The mass depletion in this stage is around 70%, and only ±20% of sample weight is remained. However, from Figure 9 and 10, there is a significant difference of thermal degradation characteristic between new and aged paper. New kraft paper tends not to lose its weight at 150^{0}C, clarified by DTG curves (rate of weight loss almost 0 wt.%/^{0}C^{-1}). Weight loss between 150-350 ^{o}C is subjected by hemicellulose depolymerization [22,23]. The higher activity of hemicellulose degradation might be associated to the

transformation of its chemical structure after thermal aging [24,25]. Amorphous structure at the hemicellulose is in a random arrangement. Therefore, hydrolysis reaction can be taken place easily [24,25]. This event might be corresponded with the loss of crystalline structure as measured before in XRD characterization, accounted at 62.9% crystallinity for new paper and decrease after aging time. Cellulose is a long polymer of glucose unit in the paper, which can improve thermal stability of kraft paper.



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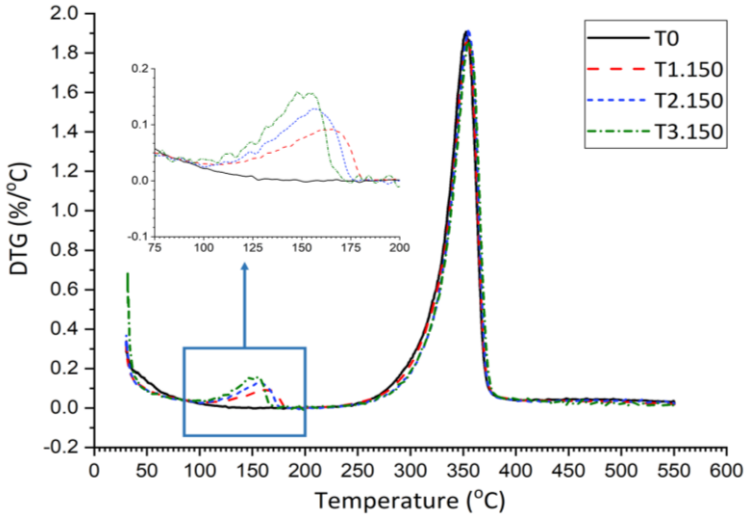
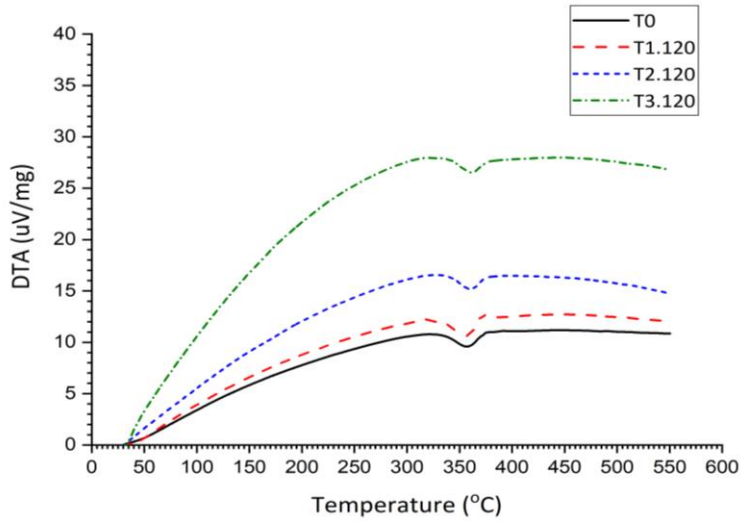


(b)

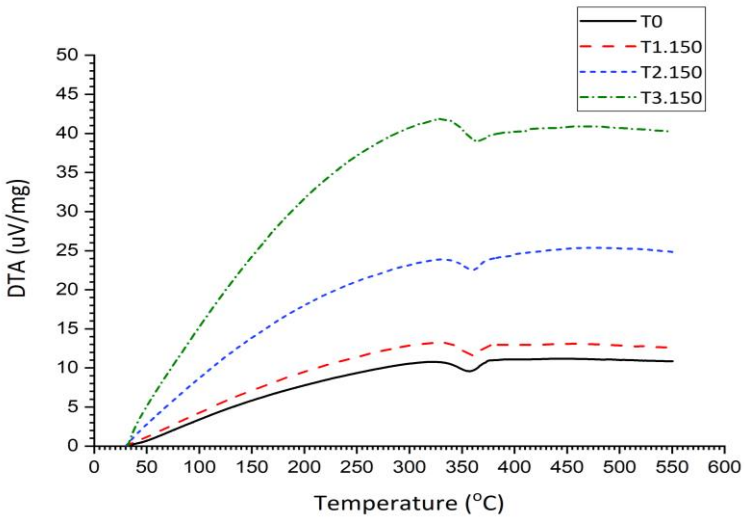
Figure 10. DTG curves of samples aged at (a) 120^0C and (b) 150^0C at different aging times

The third stage thermal degradation was indicated by lignin degradation. Lignin has different structure compared with hemicellulose and cellulose. It is cross-linked structure composed by benzene-propane units that has high molecular weight make it hard to degrade [25]. Lignin degradation starts at around 250 °C and end at around 500 °C [24,25]. As a result of this degradation characteristic, rate of degradation in the last stage is getting slower.

Figure 11 shows the DTA curves which represent the temperature difference between the sample and reference versus temperature. The DTA curves for all samples studied have similar patterns. There is an endothermic effect with maximum temperature at around 352^{0}C, corresponds to the decomposition of cellulose.



(a)



(b)

Figure 11. DTA curves of samples aged at (a) 120^{0}C and (b) 150^{0}C at different aging times

4. Conclusion

In this paper, the degradation of solid insulation, kraft paper, was observed by using XRD characterization. The main feature of XRD analysis is to observe the crystalline formation with measuring the length, width, and diffraction angle of the spectrum. The experimental analysis was done for initial paper condition, paper aged at 120^{0}C and 150^{0}C immersed in natural ester from palm oil. Kraft paper aged in different temperature are observed to identify the effect of thermal stress to the decrease of crystalline structure. The results show that from visual inspection, paper tend to be darker, especially for the sample that aged in high temperature, 150^{0}C. Besides, the relative crystalline of paper aged in higher temperature drops faster than the other. The amorphous

region tends to increase during the aging because of the degradation reaction. At the initial aging, hydrolysis reaction starts to degrade amorphous region which bind water molecule and ruined the deeper layer, which is crystalline structure.

On the other hand, thermal degradation was observed by using thermogravimetric analysis. Thermal degradation of kraft paper can be divided into three main processes. Water vaporization is contributed in the initial weight loss of kraft paper, which observed reducing 7% of its original weight. The second stage is a rapid drop of weight loss, caused by hemicellulose and cellulose decomposition. This phenomenon can be examined by using DTG measurement which indicate a light shoulder at around 150^0C showing hemicellulose degradation and a prominent peak at 352 ^{0}C which is subjected to cellulose decomposition. In the last stage of thermal degradation, a slow rate of weight loss is mainly caused by lignin decomposition. Three samples that is observed have different behavior of thermal degradation due to different percentage of crystalline and amorphous region, Therefore, it can be concluded that cellulose, hemicellulose and lignin take an essential part in the thermal degradation characteristic of kraft paper.

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