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THE EFFECT OF INFRARED DRYING TO THE MICROSTRUCTURAL STRUCTURE AND TEXTURE OF WHOLE DUKU INTACT SKIN BY MEANS OF SCANNING ELECTRON MICROSCOPY (SEM) TECHNIQUE

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ABSTRACT

The Infrared method has the potential to extend the shelf life of duku fruit by drying the duku's skin into "shell likeness". Duku's skin drying using infrared method could change the shape and characteristics of duku's skin which would significantly affect the length of fruit shelf life. The texture of duku's skin for the treatment of infrared emitter distance of 6 cm, temperature of 400 °C and exposure time of 80 seconds was increasing with the storage time which made the fruit inside the skin to experience a passive modified atmosphere and increase the shelf life of duku. The 3D visual depiction of the optimization result on drying process using infrared had the largest porosity and cavity value in the treatment of infrared emitter distance of 10 cm, temperature of 300 °C, and exposure time of 80 seconds. At the magnification of 2500 times, with a resolution of 10 µm, it was found that the porosity and thickness of the duku's void were greater than duku fruit without treatment. The result of the porosity also found that drying process with the infrared emitter distance of 6 cm at temperature of 400 °C, and exposure time of 80 seconds has more stable porosity (without collapsing) which confirmed the result found on the texture of the skin. The results of scanning electron microscopy analysis and 3D visual analysis confirmed the results of optimization that had previously performed in the drying process of duku fruit using infrared method.

Keywords: Infrared; drying; SEM Image; duku

INTRODUCTION

Electron microscopy is a method of using a beam of electron instead of light that interact with the atom in the sample. This interaction is in the form of sample reflectance which contain the information of the sample. One type of electron microscopy is called Scanning Electron Microscopy (SEM). The image produced by SEM is a result of a reflectance of heat, emission of low energy and high energy backscattered electrons, light emission which due to the beam of electron will contain the detail information about the properties of the surface of the sample. These signals were then converted into a very detailed image. SEM has been used to analyse the conditions of processing materials and food structures. Besides being used to analyse the material and structure of the food, SEM also provides more complete information about the structure of food materials by displaying three-dimensional imaging and could facilitate to obtain quantitative and qualitative information. SEM is used to visualize the structure of food materials because it combines several features of both light microscopy and transmission electron microscopy **(Aguilera et al., 2000; Falcone et al., 2006)**.

The study of the microstructure of food has become important part of food research because the structure of food could have an influence on nutritional value, rheology and some texture attributes **(Lyu et al., 2017)**. Food processing such as thermal and non-thermal processes could affect the structure and composition of the food **(Mercier et al., 2011; Lewicki and Pawlak, 2003)**. Thermal food processing could reduce the nutrition or the nutritional bioavailability, and cause damage to the chemical and the organoleptic properties. Some examples of thermal food processing technology is drying. Drying is one of the food processing methods that could prolong the shelf life or preserve grains, fruits, vegetables and food in all varieties. The quality of dried fruits depends on the conditions of the drying process. Infrared radiation has been used in drying food product. Infrared radiation has been widely implemented in the food process because it has several advantages including reducing water content in food, having low energy consumption and relatively short processing time, and could maintain and ensure the condition of product quality **(Pan et al., 2009)**. Infrared radiation could also inactivate pathogens in the material. Infrared radiation could preserve volatiles **(An et al., 2015)** inactivate bacteria, spore, yeast, and mold by controlling some of the influential parameters such as power of infrared heater **(Hamanaka et al., 2000)**, sample temperature **(Sawai et al., 2003)**, wavelength and the target wavelength **(Krishnamurthy et al., 2008)**, sample thickness **(Sawai et al., 2000)**, and sample water content **(Hamanaka et al., 2006)**. Some drying processes with infrared have been widely used including for fruits and foods. The unique characteristics of infrared radiation is the heat from emitter energy only hit the surface of food ingredients in a short time without raising the temperature of the material **(Li and Pan, 2014a)**.

The uniqueness of infrared radiation could be used to dry the skin of whole intact duku fruit. The relatively short time infrared exposure in drying of the skin would make the water on the skin of the whole intact duku to evaporate and dry the skin. Skin drying would turn the skin of the whole intact duku to dry and in turn made it into the "shell likeness". This "shell likeness" of the duku skin would mimic the condition of the inner flesh of duku fruit into a state of passive modified atmosphere **(Rahmawati et al., 2018; Rahmawati et al., 2019** which was shown to increase the shelf life of duku **(Saputra and Pratama, 2013; Saputra and Pratama, 2018)**. During the process of forming "shell likeness" on duku's skin, the heat and mass transfer simultaneously were involved in this drying process. In these processes, the fruits would undergo some volume changes either by shrinkage due to moisture loss or by expansion due to gas generation or pore formation. This shrinkage or expansion process would result in the changes of porosity of the fruit **(Aydogdu et al., 2015)**. The measurement of porosity is needed to solve the transport process for these conditions **(Hayakawa and Futura, 1989; Witrowa-Rajchert and Rząca, 2009; Pongpichaiudom and Songsermpong, 2018)**. The changes in pore formation in food during the drying process could be used to predict the thermal conductivity, mass diffusivity, thermal diffusivity, and other transport properties of food **(Rahman, 2007; Mavroudis et al., 1998; Xiong et al., 2015; Vincent, 1989; Scanlon et al., 1998)**.

Textural and mechanical properties of food process had been shown to have a correlation to the porosity. Crispiness also was shown to have a high correlation to the food porosity. The higher the apparent density of the agglomerate of the lower porosity, the stronger the agglomerate. Besides of porosity, the binding forces or adhesion of particle (i.e., structure) also affect the mechanical strength **(Pietsch, 1999)**. The variation of porosity, size of pore and pore size distribution have significant effects on the textural characteristics of dried foods **(Huang and Clayton, 1990)**.

The objective of this study was to determine the effect of infrared drying on the microstructural structure (porosity) and texture of whole duku intact skin by means of Scanning Electron Microscopy (SEM) technique.

Scientific hypothesis

The hypothesis of this research was that the microstructural changes of intact duku's skin caused by the infrared drying could create a passive modified atmosphere within duku which was shown by the scanning electron microscopy

MATERIAL AND METHODOLOGY

Samples of Duku

Duku (*Lansium domesticum*) used in this study was bought from a local farm in the area of South Sumatra Province, Indonesia. Duku fruit selected based on the appearance of smooth yellowish colour of the skin without blemish, free from contamination of microorganism, and were selected in the diameter range of 2. 5 to 3. 5 cm. The fruits were then exposed to a pair of Infrared emitter (IRE) with the distance between the emitter at the top and bottom of 6 and 10 cm, the IRE temperature of 200, 300, and 400 °C, the exposure time of 50, 60, 70 and 80 seconds respectively according the treatment allocated by the experimental design. After exposing duku to the IRE, the fruit was stored in a showcase at 11 – 15 °C for 25 days. The physical and chemical properties of duku's were then measured periodically as described on previous study **(Rahmawati et al., 2018)**. The result of previous study on the effect of IRE distance, temperature, and time of exposure on the physical and chemical properties changes were then optimized to by means of using Response Surface Methodology **(Rahmawati et al., 2019)**. The skin of duku's from the result of the optimization were then peeled and its microstructure were determined by means of SEM to reconfirm the result of Response Surface Methodology.

Scanning Electron Microscopy (SEM) Analysis

Analysis of SEM was carried out by selecting the optimum treatment of IRE with the RSM method (Design Expert Program® Version 11, 2018). Duku's were peeled and then the skin was freeze dried. The freeze dried of duku's skin was then cut longitudinally and the skin texture was observed using the Scanning Electron Microscope (SEM, JEOL® serial number 6510 LA) with the magnification of 100 times, 500 times, and 2500 times, and the resolution of 100 µm, 50 µm, 10 µm and with the depth of field 10 mm and 11 mm. The process of taking pictures and sample composition with a SEM instrument were performed by placing and pasting the sample on the SEM specimen holder with the longitudinal cross section leading upward of the objective lens. The space of the specimen holder was then vacuumed to 10^{-6} torr to ensure that the SEM column was free of air molecules. SEM was operated with the standard operating parameters including, High Voltage (HV) = 25 kV; Spot Size (SS) = 50; Work Distance (WD)= ±50 µm.

Data from SEM analysis were then analysed by using the Mountain Map Program® version 7. Mountain map measured the peak and valley of the duku's skin microstructure and measured the void within the surface of duku's skin.

Statistical analysis

The analysis of variance (ANOVA) and optimization for this research was performed with the help of Statistical Package (SAS® version 9. 4). The statistical design was a split-split plot. The details of the design could be seen on the previous paper **(Rahmawati et al., 2018; Rahmawati et al., 2019)**.

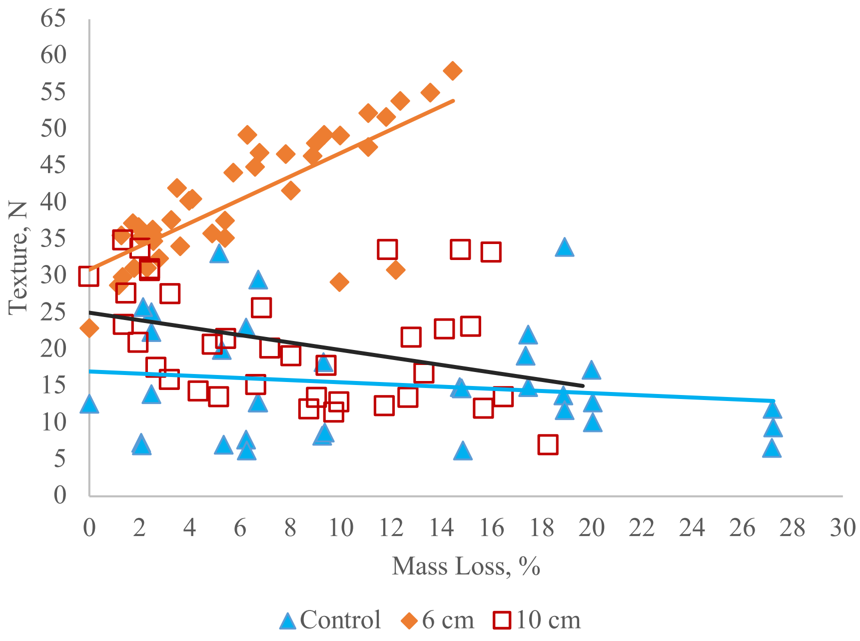


Figure 1 The correlation of duku's skin mass loss versus texture of duku.

RESULTS AND DISCUSSION

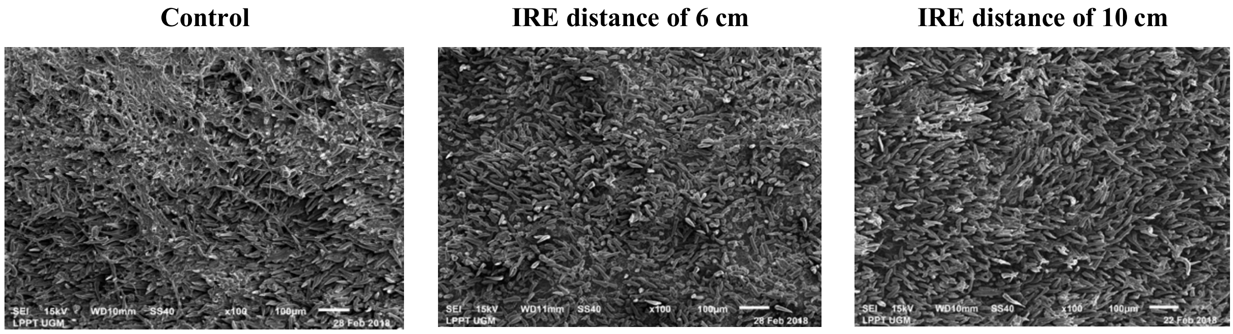
Mass loss on duku's skin and Fruit Firmness

Mass loss on the fruit skin, in general, had a correlation with the destruction of the skin due to deterioration. As expected for the plant product especially fruit and vegetables, the higher the mass loss the lower the texture. The lowering of the texture was due to the loss of liquid in the cell that would lower the turgor pressure of the cell which in turn would lower the texture. The lowering of texture for the control one was due to the deterioration or decaying of cells within the skin (Figure 1). However, in this study, the objective of the study was to dry the skin of duku which would make the skin turn into a cocoon likes and would make the fruit flesh inside the fruit to experience a passive modified atmosphere. So, the drying of the duku's skin would be expected to increase the texture. Unfortunately, for the treatment of IRE 10 cm, the correlation between the mass loss and the texture was a negative one. The texture of the skin did not increase with the increase of mass loss. Similar tendency to the treatment of IRE 10 cm was also shown by the control one. Initially, it was expected that the treatment of IRE at 10 cm would follow the hypotheses, however, even though duku's treated with infrared at IRE 10 cm showing a higher texture due to infrared drying but its texture had a similar tendency to the control. It seems the infrared drying treatment with IRE at 10 cm was not good enough in creating the passive modified atmosphere within the duku. It might be that the infrared radiation only hit the outermost of the skin but was not deep enough to penetrate the whole skin. This would make the surface of the skin dry but the inner part did not dried enough and the water from the inner part diffuse to the surface of the skin and this diffusion would make the surface of the skin became wet and lower the texture of the skin. It was observed that the wet skin on the IRE treatment of 10 cm start showing a decaying process.

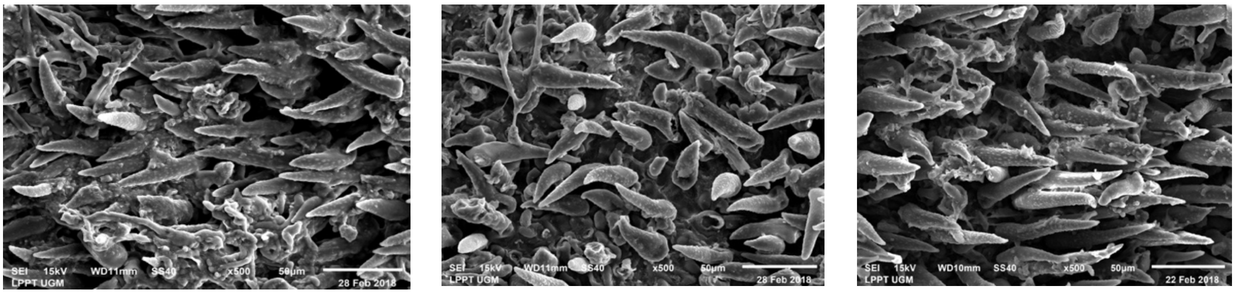
Contrary to the result showing by duku on the infrared treatment of 10 cm and the control one, it was found that on this study the treatment of IRE at distance of 6 cm show a result as expected on the hypotheses which was showing that the higher the mass loss of the skin, the higher the texture. This tendency indicates that the increasing of the mass loss was due to the infrared drying process. The treatment of IRE of 6 cm seems dried the skin of duku deep enough and did not resulted in the diffusing of water from the inner part. The complete drying of the skin from the inner part to the outer part of the skin would result in harder shell of the skin which covered the duku's flesh. The increase hardness of duku's skin would in turn increase the texture of the skin and creates a cocoon or shell like which enclose the duku's flesh. This enclosure would create or mimics the environment of a passive modified atmosphere to the flesh of duku which would slow down the gas transmission to and out of the inside part of the duku as expected on the hypotheses. This condition confirms the result showed on the previous study **(Rahmawati et al., 2018**; and **Rahmawati et al., 2019)** which show that the treatment of infrared at IRE 6 cm gave an optimum result for infrared treatment of duku. Another measurement was performed to confirm this finding by means of inspection of duku's skin with Scanning Electron Microscopy (SEM).

SEM Analysis

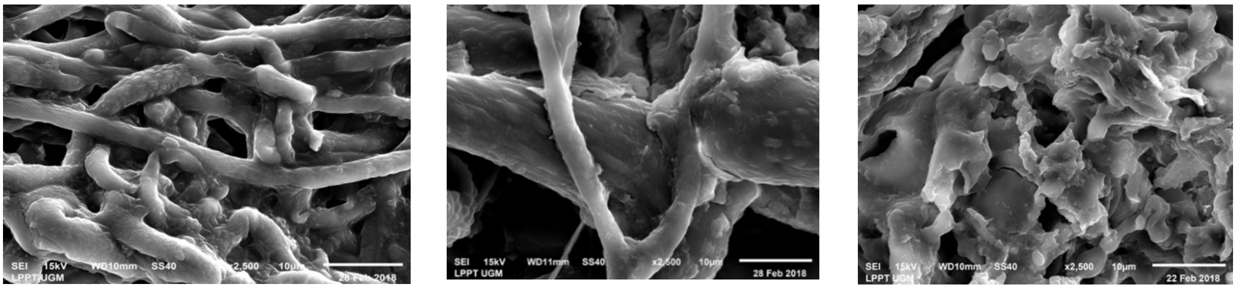
Drying of fruit using infrared has been widespread **(Li and Pan, 2014a; Li and Pan 2014b; Pan et al., 2009; Ding et al., 2015; Léonard et al., 2008)**, but there was a limited literature which explains infrared drying for fruit skin without damaging the flesh of the fruit. The infrared drying process was widely used because it has high heat transfer coefficient, short processing time, and relatively low costs. Two aspects that were important for designing infrared heaters are the distribution of the spectrum and the energy or power intensity.



(a) SEM magnification of 100 times



(b) SEM magnification of 500 times



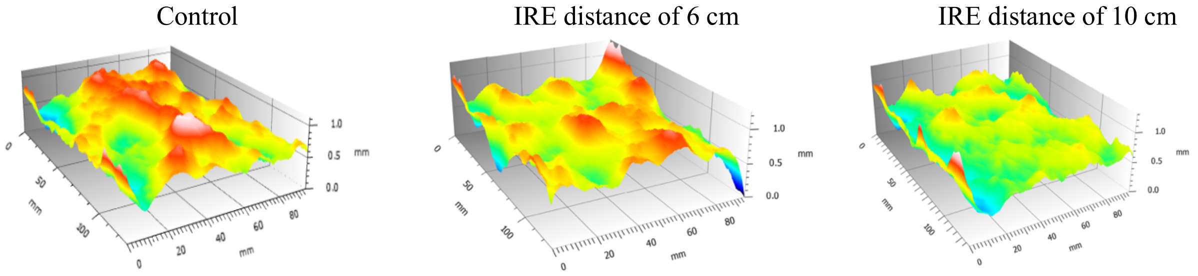
(c) SEM magnificaiton of 2500 times

Figure 2 The Scanning Electron Microscopy of duku's skin.

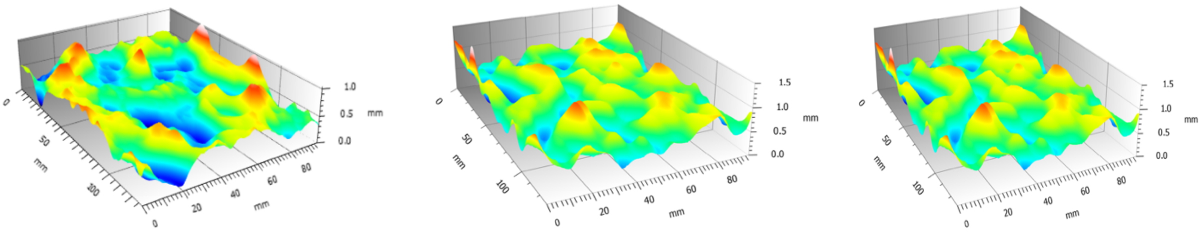
The infrared heating spectrum needs to be considered because the energy which coming out of the emitter consists of different wavelengths, and the radiation fraction depends on a number of factors such as the temperature of the emitter, emissivity and others that would affect the texture of the fruit surface/heated material. Increasing infrared power would result in an increase in the drying rate. However, the faster drying rate would increase the process of mass transfer and might damage the fruit skin texture. Based on the result of optimization on previous study **(Rahmawati et al., 2019)** and the finding on Figure 1 about the effect of infrared drying to duku's skin, an attempt was performed to confirmed this finding by performing a Scanning Electron Microscopy (SEM). The microstructural observations of duku's skin was performed using the SEM method with the magnifications of 100, 500, and 2500 times, and the resolution of 50 µm, 10 µm respectively. The SEM was performed for the treatment at IRE 6 cm and IRE 10 cm, temperature of 400 °C, and exposure time of 80 seconds on the 14th days storage as the best treatment (Figure 2).

Visually, it could be seen the effect of infrared radiation to the structure of duku's skin. It could be seen that on the magnification of 500 times, the cell structure of duku's skin experiencing a change to the skin microstructure. The epidermal cells of the duku's skin undergoing an exfoliation in the cuticle part. The exfoliation of the cuticle could also be indicated that the waxy layer of the duku's skin has been destroyed by the heating process. Further observation on the magnification of 2500 times and the resolution of 10µm show that the skin of duku with IRE distance of 10 cm had some damage to the microstructure of its epidermal which was similar to the finding of condition of duku without magnification that duku's skin exposed to infrared radiation with IRE distance of 10 cm had deteriorated. A different result was found on the duku exposed to infrared with IRE distance of 6 cm. The microstructure of the skin, relatively, more intact and firmer which was due to a better texture of the skin show on the Figure 1.

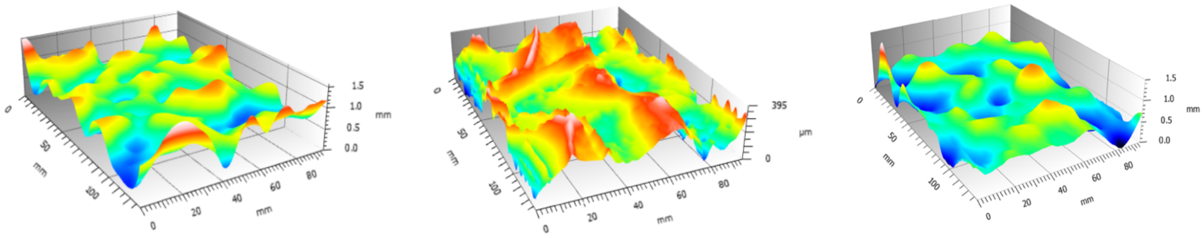
However, even though it could be seen from Figure 2 that the microstructure of duku for the control one show a similar pattern with the one exposed to infrared radiation with IRE distance of 6 cm but its size was smaller compare the the fiber on the skin of duku exposed to IRE distance of 6 cm and 10 cm.



(a) SEM magnification of 100 times



(b) SEM magnification of 500 times



(c) SEM magnification of 2500 times

Figure 3 The 3D SEM countour plot of duku's skin processed by the Mountain Map Program.

The smaller fiber on the skin of duku for the control one turns out to be a mycelium of mold or fungus which grew on the deteriorated skin of duku. The control duku used on this experiment had started to deteriorate not just due to the loss of turgor pressure but also due to the mold which grew on the surface of the skin.

Another way to show the microstructure of duku's skin was by showing the SEM on a 3D picture. By using Mountain Map Program® a 3D picture of duku's skin as shown on Figure 3 were generated. This program besides creating the 3D picture also measured the height of the peak and the depth of the valley of the skin. This valley were the result of the skin which deteriorated due to uncomplete dry of the skin which resulted in the deeper valley of the skin. It was expected that that the fruit which underwent drying would have changes of pressure that modified its shape and deformed its skin **(Mayor and Sereno, 2004)**. This pressure difference would create more valley than the hills on the skin of duku.

At a glance, it could be seen that the infrared radiation with IRE distance of 6 cm, temperature of 400°C, and exposure time of 80 seconds, for the magnification of 100 times and 500 times produces more visual representation of red colour (hill) which indicates that the duku's skin exposed to infrared at IRE distance of 6 cm had a relatively intact dry skin. The hill on the figures show that duku's skin, relatively, still retain the skin layer and a stronger skin which simulate the passive modified atmosphere condition for the flesh of duku and increase its shelf life. However, a closure look at the magnification of 2500 times and resolution of 10 µm, show that even though the skin of duku for IRE distance of 6 cm had more hills compare to the control and duku exposed for IRE 10 cm but the unit and scale of the hills and valley was completely different. The unit for the control and IRE 10 cm were mm but the unit for the IRE 6 cm was µm. This finding indicates that the thickness of duku's skin exposed to IRE 6 cm had a thinner and compact skin compare to the control and IRE 10 cm. This finding also confirm the result showing on Figure 1 which show that the skin of duku exposed to IRE 6 cm had a thinner, stronger, and compact skin which would simulate the packaging of a passive modified atmosphere.

The porosity is the most important part that affects the strength of the agglomerate from the dried material. Porosity, pore size and pore size distribution have a significant influence on the texture characteristics of dry matter **(Huang and Clayton, 1990)**. The pores of the material could be characterized by the fraction of its porosity **(Rahman, 2007)**, shape of the pore, size of pores, number of pores, pore distribution, pore wall thickness, and number wall or face pores. Porosity could be obtained by comparing the volume of void with the total volume of material. Porosity values basically influenced by the overall drawing of SEM results in each magnification, resolution and depth of field values.

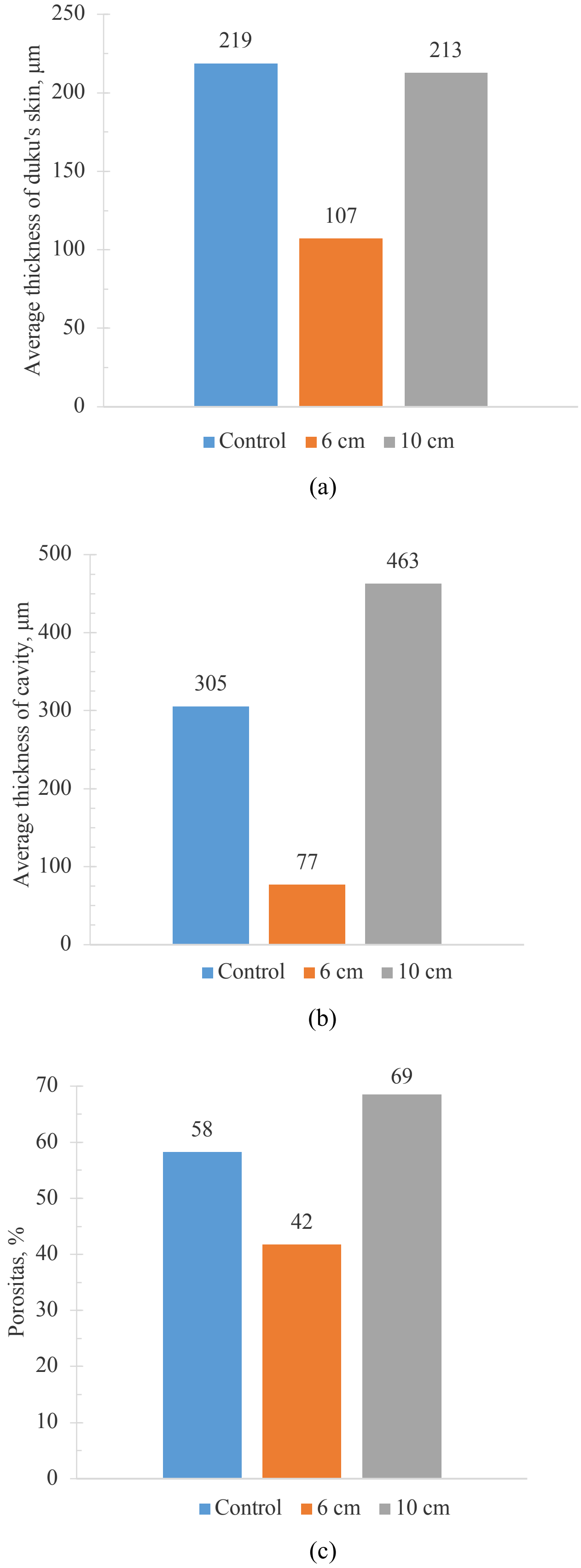


Figure 4 Average thickness (a), thickness of cavity (b), and porosity (c) of duku's skin for the control IRE 6 cm, and 10 cm.

The results of testing SEM with 2500 magnification, and 10 µm resolution show that the porosity and thickness of the duku's void without treatment was thicker than the exposure at 400 °C, 80 s at 6 cm distance of IRE. This was caused by the exposure process with IR has a solid and hard effect on the fruit skin, so the thickness of the void in the IR treatment was lower than the control treatment.

While the value of porosity and void thickness at the measurement of 2500 magnification, and 10 µm resolution at a distance of 10 cm has a higher value compared to the control, because during storage time the damage occurs on the texture besides that it could also be caused by the decay process.

A furthermore calculation of the peak and valley and the distribution of the area resulted on the average thickness of duku's skin, the average thickness of the void, and the porosity of the skin. The calculation shows that the thickness of duku's skin of 6 cm had the thinnest thickness (Figure 4 a). This result means duku's skin of IRE 6 cm is more compact than the duku's skin of IRE 10 cm and the control. The more compact skin would make duku's skin becoming a cocoon like and parallel to the result shown on Figure 1. A cocoon like skin would create an environment inside the skin like a passive modified atmosphere and prolong the shelf life duku.

The amount of porosity calculated for each SEM visual result would increase with the thickness of the voids **(Moon et al., 2014)**. The formation of pores could be divided into two types with inversion point and the other without inversion points. Factors that affecting the pore formation include internal and external factors. External factors include temperature, RH, pressure, gas atmosphere, and the radiation produced from dryers **(Rahman, 2007)**. From the statement it could be concluded that the biggest radiation produced at drying distance of 6 cm at 400 °C and exposure time 80 s has more stable porosity (without collapsing) when compared to drying with a distance of 10 cm at 300 °C and exposure time 80 s (Figure 4 b and 4 c).

CONCLUSION

The treatment of IRE 6 cm, temperature 400 °C and exposure time of 80 second increase the texture of duku's skin with the storage time. This increase made the fruit inside the skin to experience a passive modified atmosphere and increase the shelf life of duku. The 3D visual depiction of the optimization result on drying process using infrared had the largest porosity and cavity value in the treatment of 10 cm distance of IRE with 300 °C temperature of IR and 80s of exposure time. At the magnification of x2500, with a resolution of 10 µm, it was found that the porosity and thickness of the duku's void were greater than duku fruit without treatment. The result of the porosity also found that drying process with distance of 6 cm at 400 °C and exposure time 80 s has more stable porosity (without collapsing) which confirmed the result found on the texture of the skin. The results of SEM analysis and 3D visual analysis could confirm the results of optimization that had previously been done in the drying process of duku fruit using infrared method.

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