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Development of a Compact Vacuum Freeze Drying for Jelly Fish (Schypomedusae)

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Graphical abstract



Abstract

A new design of a vacuum freeze drying with internal cooling and heater from condenser's heat loss was built and tested. The dryer was used to dry jelly fish (*scyphomedusae*) to study the effect of drying parameter such as temperature within the drying chamber on mass losses (evaporation) during freezing stage and moisture ratio at the end of drying process and also the drying rate of vacuum drying process. The cold trap temperature rise in when activated the heating from condenser's heat loss. The midili thin layer mathematical drying model was used to estimate and predict the moisture ratio curve base on different drying chamber temperature. The result of this experiment show that mass loss during freezing stage decreased with a decrease in drying chamber temperature with constant pressure. Drying time reduced with an increase in drying temperature. Drying chamber temperature decreasing has a result pressure saturation of material lower than drying chamber pressure have an effect mass transfer should not occurs.

Keywords: Vacuum freeze drying, mass losses, moisture ratio, jellyfish (Scyphomedusae), thin layer models, internal cooling, condenser's heat loss

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1.0 INTRODUCTION

Vacuum Freeze drying (VFD) is an optimal drying technology method because it maintains the structure, nutrients, and color of the original substance [1, 2, and 3]. Vacuum freeze drying has also been described as a method of drying that achieves a high drying rate because the boiling point of water in the product, held under the vacuum, is lower than atmospheric pressure and water vapor is removed by a vacuum pump [4]. Characteristic of this drying technique such as high drying rate, low drying temperature and oxygen deficient drying environment may help to maintain the qualities such as shape, color, aroma and flavor and nutritive value of the dried product [5]. The development of vacuum freeze drying there are two serious problems, maintain the quality of product and then reduce the energy use for the vacuum freeze drying operation.

Vacuum freeze drying process consists of three processes that are freezing process, primary drying and secondary drying [6]. In freezing process, actually use vacuum freezing method. It is a freezing process based on the rapid evaporation of moisture from the surface and within the products due to the low surrounding pressure below triple point (pressure below 6.1mbar). Temperatures of product begin to decrease when the water starts to evaporate which is latent heat of evaporation remove sensible heat of product due to pressure reducing [7]. Vacuum freezing caused high evaporation and mass loss, this phenomenon for any

product make a serious damage [8]. Many experiments have been investigated by many researchers to reduce mass loss during vacuum freezing. The innovation in vacuum freeze drying to reduce mass loss and product damage by combine between vacuum freezing with air blast freezing and immersion freezing [8, 9, 10]. The next process of vacuum freeze drying is primary drying. Primary drying is a process of mass transfer or change phase of product from ice (solid) directly at low pressure below triple point condition. In this process need a lot of energy to sublime and remove unbound water in the product. Many experiments have been investigated by many researches to reduce energy consumption and drying time of product. In general, prolonged drying time can be reduced by techniques such as increasing temperature or decreasing pressure in the chamber. An experiment with VFD using a microwave heat source was developed for drying potato slices [11], and the total drying time was reduced by 37% as a result of microwave freeze drying and not conventional freeze drying. Results of another experiment using microwave freeze drying for drying sea cucumbers indicated that the technique can effectively replace conventional freeze drying because it greatly reduces drying time and energy consumption while producing the same product quality. In order to avoid corona discharge during microwave freeze drying, the cavity pressure should be applied within the range of 50-100 Pa [12, 13]. An experiment using an electrical heat source in the top position of a freeze vacuum dryer was conducted for drying

foodstuff; the surface temperature of the sample initially increased quickly due to the higher initial temperature difference between the sample surface and electrical heater, resulting in an increase in the drying rate and a reduction in drying time [2]. Other experiments using electrical heat sources have been performed to compare differences attributable to placement of Aloe Vera in either top or bottom positions, resulting in a faster drying rate at the bottom position [14]. This phenomenon occurred because the thermal conductivity of ice or a frozen layer, if using a bottom position heater, is 45 times higher than the thermal conductivity of a dry layer if using a top position heater. An innovative experiment using infrared radiation as a heat source when drying peaches was compared to microwave freeze drying, and results showed that less energy was consumed using infrared radiation [15]. Recently, chili peppers were freeze dried using a heat pump inside a product container and a dehumidifier and blower to reduce humidity at the drying chamber, resulting in reduced drying time accomplished from either reducing drying pressure or from increasing drying temperature. The moisture content of 20 kg of fresh chilies was reduced from 34% to 15% when dried for 7 h [16].

Due to the problems above, the aim of this research is to investigate the characteristics of vacuum freeze drying of jelly fish (scyphomedusae) and the effect combination internal cooling on mass loss and condenser's heat loss on final moisture ratio, drying rate and also the influence on the cold trap temperature.

2.0 MATERIALS AND METHOD

2.1 Sample Preparation

Jelly fish (scyphomedusae) a sea animal commonly grown in Indonesia as maritime country were used as the test material in this research. The tentacles of jelly fish used as basic medical ingredient to reduce the growth cell of cancer. Due to that use need extraction by vacuum freeze drying to retain nutrition content, color, taste, and biological structure. Initial moisture contents of jelly fish is 95.91% (w.b), respectively. Before tested using vacuum freeze drying, jelly fish is first stirred as seen in Figure 1. Initial and final mass measured using digital balance with range 0–600 g and accuracy of 0.4%. For each experiment, 50 g of samples was used on vacuum freeze drying process



Figure 1 Stirred of jelly fish tentacle

2.2 Experimental Set-up

A compact vacuum freeze drying with internal cooling and heat from condenser heat loss was designed, built and installed at Department of Mechanical Engineering, University of Indonesia (Fig 2 and 3). It consist of 2 Insulated cylindrical, drying and cold trap chamber with a diameter and a length of 20 cm. Cold trap is a cascade refrigeration with refrigerant at High stage R22 and at low stage HCR22. Cold coil installed at drying and cold trap chamber. Heat from condenser's heat loss also installed at drving chamber with same coil. To shut on and shut off cold and heat coil using shut off valve. A pressure transmitter PTX 1400 with an accuracy of 0.4% and measure range 0-1600mbar was used to measure pressure in the drying chamber. A tray of material used from Teflon and insulated to keep the heat transfer during process. The temperature at drying chamber, cold trap, material and also cascade refrigeration system were monitored by thermocouple type K with accuracy 0.4%. Dial pressure used to monitor pressure in cascade refrigeration system. The thermocouple and pressure transmitter connected to Data Acquisition which is have number of slot 4, total power 15 w and operating temperature -20°C until 55°C.



Figure 2 Compact vacuum freeze drying



Figure 3 A Schematic diagram of compact vacuum freeze drying. Blue line is cold refrigerant and red line is hot refrigerant. At High stage: (1) Compressor, (2) condenser, (3) needle valve as expansion valve, (4) PHE as evaporator. At low stage: (5) Compressor, (6) condenser, (7) Check Valve, (8) PHE as condenser, (9) Needle valve as expansion valve, (10) Cold/hot coil, (11) Cold trap coil, (12) Drying chamber, (13) Cold trap chamber, (14) Tray of material, (15-21) Shut off valve. Measurement equipment: (22-26) dial pressure gauge, (27) pressure transmitter, (28-40) Thermocouple type K, (41) Vacuum Pump

2.3 Methods

To start the process, the cascade refrigeration system in high stage was turned on until the Plate Heat Exchanger (PHE) as evaporator reached a temperature of -35° C. And then the low stage was turned on until cold trap reached a temperature of -35° C. For the using of internal cooling for freezing stage and condenser's heat loss as heater for drying stage, the shut off valve varied into 2 position (Partially and Fully). The vacuum pump was then turned on until the substance reached a freezing temperature and appeared stable. The variants of experiment show at Table 1.

Table 1 Experiment variation

Experiment	Freezing Stage		Drying Stage	
_	Internal Cooling	Condenser's Heat loss	Internal Cooling Valve	Condenser's Heat loss
Run no. 1	Off	Off	Off	Off
Run no. 2	Fully	Off	Off	Fully Open
Run no. 3	Open Partially Open	Off	Off	Partially Open
Run no. 4	Ôff	Fully Open	Off	Fully Open
Run no. 5	Fully	Off	Fully	Off
	Open		Open	

2.3.1 Characteristics of Vacuum Freeze Drying Temperature

Characteristics of vacuum freeze drying on jelly fish based on the reading of thermocouple type K for measure temperature which is installed at product. Temperature should describe three areas that are freezing, primary and secondary drying. The phenomena of change phase will be describes in Pressure and temperature graphic to know amount of water evaporate at freezing stage.

2.3.2 Mass Loss During Freezing Stage

In this section, a simple theoretical analysis of vacuum freezing process based on thermodynamic principle is presented. This analysis is limited to the mass loss based on temperature drop observed during vacuum freezing process. The heat required to lower the temperature of 1 kg jelly fish could be calculated by the following expression:

$$\Delta m.h_{fg} = m.C_{avg}.\Delta T \tag{1}$$

Where Δm is mass loss (kg), hfg is latent heat evaporation (J/kg), m is mass of jelly fish (kg), ΔT is = the change in product temperature (K). Average specific heat (C_{avg}) of product can be calculated by following expression [7]:

$$C_{avg} = 3349a + 837.36J / kg.K$$

Where a is the water content of jelly For instance, water content of jelly fish is 95.91% by mass.

2.3.3 Moisture Ratio

Moisture ratio or rehydration ratio was simplified to M_t/M_{in}

instead of $M_t - M_e / M_{in} - M_e$ because relative humidity of the drying air continuously fluctuated in vacuum freeze drying process [17]. The moisture ratio is of the sample was then calculating by [11, 16]:

$$MR = \frac{M_t}{M_{in}} \tag{3}$$

Where MR is the moisture ratio, M_{in} is the initial sample mass (kg), and M_t is the sample mass at time t (kg) Curve of moisture ratio was describe using midilli thin layer models as below [18]:

$$MR = a.\exp\left[-k t^{n}\right] + bt \qquad (4)$$

Where a, b is empirical constants in drying models, k is empirical coefficients in the drying models (h⁻¹), n is drying exponent and t is time (h). The empirical constants (a, b), empirical coefficients (k) and also drying exponent (n) have been described with variant of temperature by calculation below [16]:

$$k = -0.4680P^2 + 9.8691x10^{-2} + 0.2765$$
 (5)

$$a = 9.2616P^{3} - 6.2783P^{2} + 1.2200P + 0.9177$$

$$(6)$$

$$n = -18.0281P^{3} + 10.1802P^{2} - 1.6252P + 1.2608$$

$$(7)$$

$$b = -0.8140P^{3} + 0.5631P^{2} - 97221x10^{-2}P + 1.2166x10^{-3}$$

$$(8)$$

2.3.4 Drying Rate

Drying rate is one of important parameter to know energy consume of vacuum freeze drying. The higher drying rate will reduce drying time and finally reduce energy consume of vacuum freeze drying process. The equation to calculate drying time described as below [1, 2] with the schema at Figure 4 :



Figure 4 Scheme of heat and mass transfer in vacuum freeze drying

$$N_{w} = \frac{L}{M_{w} N_{s}} \left[\frac{x_{t+\Delta t} - x_{t}}{\Delta t} \right]$$
(9)

Substitution

$$m = N_w . M_w \tag{10}$$

$$\dot{m} = \frac{L}{V_s} \left(\frac{dx}{dt}\right) \tag{11}$$

$$\dot{m} = L.X_o.\rho_s \left(\frac{dx}{dt}\right) \tag{12}$$

And the heat flux that occurs to the material can be calculate based on conduction heat transfer since the condition at drying chamber is low vacuum below (Chakraborthy et al. 2006)

$$q = \frac{\lambda}{L - \Delta L} T_d - T_m \tag{13}$$

Substitution

Z

$$\Delta L = 1 - x \ L \tag{14}$$

$$q = \frac{\lambda}{xL} T_d - T_m \tag{15}$$

3.0 RESULTS AND DISCUSSION

3.1 Characteristics of Vacuum Freeze Drying Temperature

The characteristics of vacuum freeze drying temperature of jelly fish under different drying chamber temperature are given in fig 5 and 6, respectively. As expected, from Figure 5 can be seen that drying temperature consists of 3 area, freezing area, primary drying area and secondary drying area. As expected, from Figure 6 drying time decrease with an increase drying chamber temperature similar tendency is also observed in study of Apichart and Sonuk (2010) in vacuum drying of shiitake mushroom and jinda chilli and George and Data (2002) in vacuum drying of vegetable slices. Characteristics for adding combination internal cooling and condenser's heat loss the drying time longer than without internal cooling due to freezing process reach lower temperature.



Figure 5 Characteristic of material temperature consist of 3 area



Figure 6 Profile temperature versus drying time of material

3.2 Mass Loss During Freezing Stage

From Figure 7-11 can be seen that vacuum pressure in the drving chamber decreased rapidly from atmosphere to below triple point pressure, then decline slightly. When it reaches to set pressure it starts to fluctuate around it. When the pressure is lower or equal to the saturation pressure at local temperature, water evaporates (change phase from liquid to gas). After that the cooling effect comes from water evaporating from the sample and sample become freezing start from the surface. However, with decreasing the pressure, evaporation and freezing occur through the jelly fish and temperature decrease together. There are different temperatures decreasing of jelly fish due to different temperature drying chamber. For Run no. 1 temperature of jelly fish should decrease from 24°C to -5°C, Run no. 3 from 24°C to -23°C, Run no. 4 from 24°C to -2.5°C and Run no. 5 from 24°C to -20°C. But for Run no. 2 there is information that the vacuum freeze drying process occurs with pre freezing by internal cooling. This event does not involve the evaporation process due to different pressure between saturation pressure product and drying chamber pressure to reduce product temperature. The next process is drying or sublimation process which is change phase from ice to gas. For Run no. 4 there is mal function of vacuum freeze drying, the phase change at drying process occurs from liquid to gas (evaporation) due to the temperature of condenser's heat loss in activated. From Figure 12 there is information for Run no. 5 which is does not occurs sublimation or mass transfer so that the material not to dry. This event occurs due to drying chamber pressure higher than saturation pressure of material. When internal cooling activated until the on of drying stage



Figure 7 Diagram P-T on run no. 1





Figure 9 Diagram P-T on run no. 3



Figure 10 Diagram P-T on run no. 4







Figure 12 Grapich between saturation pressure of product with drying chamber pressure

Mass loss occurs during vacuum freezing since cooling effect directly comes from water evaporation from jelly fish and combination internal cooling. Mass losses of jelly fish with Run no.5 is given in table 2. The highest mass loss comes from Run no. 1, due to the process without internal cooling. An internal

cooling does not take high effect to the experiment due to between internal cooling and vacuum freezing run concurrently. Should be note that to reduce mass loss due to evaporation, temperature of drying room should be reduce first and then running vacuum pump. It is will give high effect to the process.

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Table 2	Mass	losses	tor	everv	experim	ient riin	no
I UDIC A	TTTCOD .	1000000	101	cicij	enperim	ione run	no.

Experiment	Initial Mass	Mass Loss	Percentages (%)
	(g)	(g)	
Run no. 1	50	1.68	3.4
Run no. 2	50	1.00	2.0
Run no. 3	50	1.80	3.6
Run no. 4	50	1.58	3.1
Run no. 5	50	1.59	3.1

3.3 Moisture Ratio

The moisture ratio curves of jelly fish under different drying temperature are given in Figure 13. As expected, moisture ratio reached maximum value at the beginning of the drying process due to a high moisture content in the substance during the first sublimation. After reaching EMC (Equalibrium Moisture Content), its moisture content remained in bound water condition; therefore, the moisture ratio decreased. In other words, the moisture ratio decreased with drying time in all drying processes. We observed that the processes which affect the moisture ratio internally are porosity and bulk density.



Figure 13 Moisture Ratio of Jelly Fish

In this research is using midili model to know the moisture ratio. Midili model is one of model for predicting the moisture ratio at a product, The predicting of this model is in a thin layer. Thin layer drying generally means to dry as one layer sample particles or slices. Because of its thin structure, the temperature distribution can be easily assumed as uniform and thin layer drying is very suitable for lumped parameter models. The advantages of using midili model is suitable for drying kinetics which is the condition of the product and the surrounding environment change over time.

The correlation between midilli thin layer model with Constanta (k, a, b, n) from Artnaseaw and experiment, 2010 can be seen at table 3 and Figure 14. The data from experiment compare with midilli model have differences with the value of R Square 0.686 and not linear. The error (not linear) occurs due to the Constanta (k, a, b, n) use from material which is have moisture content 80%, jelly fish have moisture content 95.91%. However, the midilli model was validate to estimate the moisture ratio but need to redefine the Constanta (k, a, b, n) especially for jelly fish used. For run no 5 the moisture ratio cannot be calculate due to in

run no. 5 mass transfer not occurs. This phenomena because of saturation pressure of product lower than drying chamber pressure. The reduction of mass at final mass run no. 5 due to evaporation on product at the freezing process

 Table 3
 Data comparison of Moisture content between midilli model and experiment

Experiment	Initial mass	Final mass	Moisture Ratio	
	(g)	(g)	Experiment	Midilli Model
Run no. 1	50	0.78	0.016	0.02
Run no. 2	50	1.73	0.035	0.019
Run no. 3	50	1.66	0.033	0.013
Run no. 4	50	0.55	0.011	0.026
Run no. 5	50	48	No mass transfer	No mass
			at sublimation	transfer at
			process	sublimation
			(Reduction	process
			because of	(Reduction
			evaporation at	because of
			freezing stage)	evaporation at
				freezing stage)



Figure 14 Graphic of linear correlation

3.4 Drying Rate

Heating have affect to the drying time and because the using of heating can increase drying rate during vacuum freeze drying as seen at Figure 15. Drying rate of vacuum drying process without heating can reach drying rate until 0.000232 g/m²s but with activated heating from condenser's heat loss 44°C drying rate reach 0.000505 g/m²s. Every raise in heating temperature also can increase the drying rate of vacuum drying process similar effect were describe by the others research with different heating source electrical heater [1, 2, 19) infrared heater [20, 21], but if using microwave every rise in power of microwave caused increase in drying rate of vacuum freeze drying [12, 13, 15]



Figure 15 Drying Rate result until the end of vacuum drying process

3.5 Cold Trap Temperature

At Figure 16 is profile of cold trap temperature. At the beginning process, there is a fluctuate of temperature because of flashing point of product o reach freezing temperature. Activated heating from condenser's heat loss make the temperature of cold trap rise in, this phenomena because of activated heating from condenser's heat loss is mean adding the cooling load of refrigeration system. The cold trap temperature also rise in at primary and secondary drying due to load from moisture content of material which is in this experiment is 97% of water at material



3.6 Final Product of Tentacle Jelly Fish

At Figure 17 can be seen the final product after vacuum freeze drying process with variation from run no 1 until 5. For variation run no until 4 can be seen that tentacle of jelly fish is dried with variant of final moisture content but for variation run no 5 the tentacle of jelly fish can't be dried but still in liquid condition because the pressure at drying chamber is higher than saturation product of jelly fish.

r

Х

t

n



Figure 17 Profile of cold trap temperature

4.0 CONCLUSION

The influences of combinations internal cooling and vacuum freezing have been tested. Result show that vacuum freezing have mass loss 1.4% higher than conventional freezing or internal freezing (Run no. 1 and 2), Combination between internal cooling and vacuum freezing have effect 0.3% better than vacuum freezing due to internal cooling and vacuum freezing run concurrently. The effect of condenser heat loss can be reducing drying time 30% (Run no. 1 and 2). Midilli thin layer model can use to predict moisture ratio curve but the value of Constanta (k, a, b, n) need to redefine for jelly fish use. moisture ratio decreased with drying time in all drying processes. When decrease drying chamber need to observe the pressure saturation of product, if pressure saturation product lower then pressure of drying chamber the mass transfer should not occurs. The activated of heating from condenser's heat loss can increased the drying rate of vacuum drying process from 0.000232g/m²s at without heating become 0.000505g/m²s with heating at 44°C. The activated heating from condenser's heat loss also have effect to cold trap temperature. Cooling load of cold trap at the beginning came from flash point of material before reach freezing temperatur and then at primary and secondary drying came from moisture content which is removed from material.

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Nomenclature

Notation

Pressure (bar) Ρ =

$$N = Molar Sublimation rate
\begin{pmatrix} kg.mol/\\m^2s \end{pmatrix}$$

$$L = Thickness of sample (m) = Specific volume of water in food
 $\binom{m^3}{kg}H_2O$

$$M = Molecular mass \begin{pmatrix} kg/\\kg.mol \end{pmatrix}$$

$$M = Fraction of initial moisture remaining (dimensionless) = Drying time (jam) = Sublimation rate $\binom{kg}{m^2.s}$

$$X = Water content
 $\binom{kg.H_2O}{kg.dry}$

$$q = Heat flux \frac{W/m^2}{m^2}$$$$$$$$

Greek letters

 ΔL

λ

р

Su

w

d

m

s

Distance of frozen surface from the top (m)

	=	Thermal conductivity of ice
	W	/ m.k
haaninta	=	Mass density $\begin{pmatrix} kg \\ m^3 \end{pmatrix}$
useripis	=	Water
	=	drying
	=	material
	=	solid

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