

## Air Blast Freezing of Lime Juice: Effect of Processing Parameters

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**Abstract:** This research characterized the effects of air velocity and lime juice layer thickness on freezing time. In the experiment, the air velocity of the freezer and the thickness of the lime juice layer were set to be 4-12 m sec<sup>-1</sup> and 4-10 mm, respectively. The temperatures of the lime juice were measured from 15°C until it reached -20°C and were continuously recorded during each test. The experimental freezing time curves showed a decreased freezing rate period. In addition, the mathematical model of freezing time was fit to a set of the experimental sample data, which was characterized by 6 different regression models. The results showed that the freezing times increased when decreasing air velocity. Moreover, increasing the lime juice layer thickness would also increase the freezing time of lime juice in which occurred mostly in S-2 stage. With the air velocity exceeding 8 m/s and the lime juice layer thickness less than 8 mm, the experiment gave the best operating condition for the freezing time of lime juice in this freezing process. The Model (6) was found to satisfactorily describe the curves freezing time of lime juice with R<sup>2</sup> of 0.9656.

**Key words:** Air blast freezer, freezing time, lime juice

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### INTRODUCTION

Lime or *Citrus aurantifolia* is an important agricultural product in Thailand. It is the smallest member of the true citrus family and native to Southeast Asia and India. Lime contains unique flavonoid compounds that have antioxidant and anti-cancer properties. Lime has been used to prevent scurvy, a disease caused by a deficiency of vitamin C. Traditionally, lime has been used as a remedy for indigestion, heartburn and nausea. It also has cooling effects on fevers and can help ease coughs and various respiratory disorders<sup>[13]</sup>.

Freezing and ultimately freeze drying, is one of the important processes which widely applied in food preservation. Air blast freezer is a common type of freezer, which can be used for a variety of irregular shapes including small sized products<sup>[1-2]</sup>. There are two major considerations in an air freezer system: an energy input which required moving the air past the food product and the air velocity distribution in the freezer chamber<sup>[7]</sup>. In the freezing Process, the temperature of the product falls in a manner of consisting of three stages; the first stage involves sensible pre-cooling. The second stage involves the extraction of latent heat and

in the last stage, the remaining water in the frozen lime juice involves sensible sub-cooling<sup>[1]</sup>. The total freezing time ( $t = t_1 + t_2 + t_3$ ) is the sum of the pre-cooling time ( $t_1$ ), the latent heat of time ( $t_2$ ) and sub-cooling time ( $t_3$ )<sup>[2]</sup>.

Several researchers have studied the effect of processing parameters in air blast freezing of foods. Muftugil<sup>[14]</sup> studied the freezing time of strawberries at different air velocities in an air freezer at -30°C. Leblanc *et al.*<sup>[9]</sup> investigated the freezing time that required to decrease the temperature of a french fry in air blast freezer. Chevalier *et al.*<sup>[4]</sup> studied that the cylindrical gelatin gels that were frozen at atmospheric pressure with different operating conditions (air-blast freezing at different air temperatures and brine freezing). Boonsumrej *et al.*<sup>[3]</sup> studied the changes of quality of tiger shrimp which frozen by air blast freezing on different air velocity. Martins *et al.*<sup>[10]</sup> investigated the quality of frozen strawberries which were influenced by the super-cooling capacity during air blast freezing on operational variables: initial temperature, air temperature, air velocity and strawberry maximum diameter.

In this study, an air blast freezer, which is a part of the freeze dryer<sup>[17]</sup>, was designed to freeze lime juice. The aim of the study was to investigate the effects of

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the freezer's air velocity and the layer thickness of the lime juice on freezing time of lime juice. In addition, the freezing time mathematical model was fit to a set of the experimental sample data, which was characterized by a regression model.

## MATERIALS AND METHODS

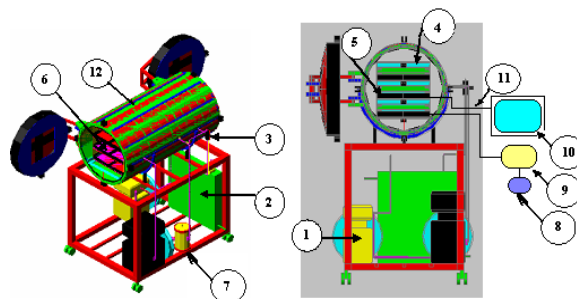
**Sample preparation:** Lime (*Citrus aurantifolia*) was purchased from a local market in Khon Kaen, Thailand. It was unsorted, washed and squeezed into lime juice (60 units/1 liters of juice) using a stainless steel juice squeezer. The average amount of lime juice per one kilogram of lime is 582.6 mL or 0.5532 kg. The lime juice was collected and examined the properties by the Laboratory Center for Food and Agricultural Products Co., Ltd, Thailand (LCFA), indicated in Table 1.

**Experimental apparatus preparation:** Figure 1 illustrates the schematic diagram of air blast freezer, which was designed by researcher<sup>[17]</sup>. Briefly, it consists of refrigeration system, cooling fan, freezing chamber and measurement instruments. The rectangular trays were made from 0.8 mm-stainless steel thick. The trays were divided into 8 blocks. Each tray had a small hole at the bottom of the block to allow lime juice to flow from one block to the other. The cooling unit was designed to be 3.75 kW of cooling capacity using R-22 refrigerant. The fin spacing of the evaporator is 10 mm and area surface is 10 m<sup>2</sup>. The air velocity ranged between 0-12 m sec<sup>-1</sup> in order to determine the suitable conditions for freezing process in each tray. The mechanical devices in the freezing chamber were used to control the direction of air flow into each tray in order to equalize the velocity. The wall was covered with 0.005 m polystyrene sheets to improve thermal insulation. Two thermocouple (type-T) wires were immersed in the lime juice in each tray. They were placed 133.33 mm. apart at the middle of the tray. The speed of the cooling fan was controlled via an inverter (T-VERTER 2N-Series-220\*1.5kW; Model N2-202-M). The electrical signals of the samples were collected by a data logger (YOKOGAWA; Model DAQSTATION DX200) and the data were stored on a floppy disk.

**Experimental method:** The fresh lime was used in the experiment. Before freezing, lime was washed, cut into two pieces and squeezed to get lime juice (60 units/1 L of juice) using a stainless steel juice squeezer. The juice was poured into six trays and placed on three shelves in the freezing chamber. Lime juice was frozen as layer

Table 1: Properties of lime juice treated under air blast freezing

Description	Result	Unit
Vitamin C	330.20	mg 100 g <sup>-1</sup> (by dry weight)
Citric acid	28.14	g 100 g <sup>-1</sup> (by dry weight)
pH	2.45	pH-range
Moisture content	92.22	g 100 g <sup>-1</sup>
Water activity	0.99	-



Description	Particular
1. Compressor	3.5 kW of cooling capacity on -40°C evaporator unit and 40°C condenser unit, motor 2 hp, 3 phase (Open reciprocating)
2. Condenser	5.25 kW of heat rejection (air cooled)
3. Expansion valve	3.5 kW (Sporlan, Model CG-032) Thermostatic 3.5 kW, thermostatic charges available -18°C to -40°C
4. Evaporator	Cooling capacity 3.75 kW, Ø 15 mm of tubing (50×50 mm aligned parallel), 10 mm of the fin spacing, 10 m <sup>2</sup> (heat transfer surface)
5. Cooling fan	1/3 hp, single phase, 1450 rpm
6. Tray	250×400×20 mm (SS-304)
7. Receiver tank	3.5 kW, Ø 10 mm of tubing
8. Flow meter	Testo GmbH and Co, Model testo 645
9. Inverter	T-VERTER 2N-Series-220*1.5 kW, Model N2-202-M
10. Data logger	Yokogawa, Model: DAQSTATION X200
11. Thermocouple	Type-S
12. Insulation	Polystyrene 50 mm of thickness

Fig. 1: Experimental freezing apparatus and sample tray

with the thickness of 4, 6, 8 and 10 mm at the velocity of freezing air of 4, 6, 8, 10 and 12 m sec<sup>-1</sup>. Freezing process started with an initial temperature of 15±1°C and continued until final temperature of -20±1°C.

During the experiments, temperature of lime juice, inlet and outlet temperature of compressor, inlet and outlet temperature of evaporator, inlet and outlet temperature of condenser, inlet and outlet temperature of expansion valve and inlet and outlet pressure of compressor were recorded. The operating conditions of the freezer are shown in Table 2.

**Empirical model:** In this experiment, the temperature of the lime juice was recorded by the data logger every 4 min, which was started with an initial

Table 2: Operating conditions of air blast freezer of the experiments

Description	Operating condition		
	Mean	SD	S.E.M.
Inlet temperature of compressor (°C)	-9.9	4.53	1.43
Outlet temperature of compressor (°C)	100.0	3.80	1.20
Inlet temperature of evaporator unit (°C)	-30.0	3.65	1.15
Outlet temperature of evaporator unit (°C)	-20.0	4.11	1.30
Inlet temperature of condenser unit (°C)	98.0	3.37	1.06
Outlet temperature of condenser unit (°C)	30.0	3.37	1.06
Inlet temperature of expansion valve (°C)	28.0	3.37	1.06
Outlet temperature of expansion valve (°C)	-32.0	3.62	1.15
Inlet Pressure of compressor (psi)	5.0	3.23	1.02
Outlet Pressure of compressor (psi)	175.0	3.09	0.98

temperature of 15±1°C until it reached -20±1°C. The boundary conditions were; u = air velocity = 4, 6, 8, 10 and 12 m sec<sup>-1</sup> and Δx = lime juice layer thickness = 4, 6, 8 and 10 mm. The freezing process experiments have 3 repetitions.

The freezing time model was fit to a set of experimental sample data, which was characterized by a regression model. The freezing time, τ is a dependent variable, which was assumed to be function of air velocity (u) and limejuice layer thickness (Δx) as:

$$\tau = f(u, \Delta x) \quad (1)$$

According to Plank's model (Nagaoka *at et.*, 1955; Muftugil, 1986; Mannapperuma *at et.*, 1994), the fitting models were considered as the second-order in two variables which is the basic method for estimating the freezing times of the foods. Therefore, the models were selected and experimentally obtained:

$$\text{Model 1: } \tau = \beta_1(\Delta x)(u) + \beta_2(\Delta x)(u^2) + \beta_3(\Delta x^2)(u) + \beta_4(\Delta x^2)(u^2) + \beta_5 + e_{ij} \quad (2)$$

$$\text{Model 2: } \tau = \beta_1(\Delta x)(u^{1/2}) + \beta_2(\Delta x^2)(u^{1/2}) + \beta_3 + e_{ijk} \quad (4)$$

$$\text{Model 3: } \tau = \beta_1(\Delta x^{1/2})(u) + \beta_2(\Delta x^{1/2})(u^2) + \beta_3 + e_{ijk} \quad (5)$$

$$\text{Model 4: } \tau = \beta_1(\Delta x^{1/2})(u^{1/2}) + \beta_2(\Delta x^{1/2})(u^2) + \beta_3(\Delta x^2)(u^{1/2}) + \beta_4(\Delta x^2)(u^2) + \beta_5 + e_{ijk} \quad (6)$$

$$\text{Model 5: } \tau = \beta_1(\Delta x)(u) + \beta_2(\Delta x)(u^2) + \beta_3(\Delta x^{1/2})(u) + \beta_4(\Delta x^{1/2})(u^2) + \beta_5 + e_{ijk} \quad (7)$$

$$\text{Model 6: } \tau = \beta_1(\Delta x^{1/2})(u^{1/2}) + \beta_2(\Delta x^{1/2})(u) + \beta_3(\Delta x^{1/2})(u^2) + \beta_4(\Delta x)(u^{1/2}) + \beta_5(\Delta x)(u) + \beta_6(\Delta x)(u^2) + \beta_7(\Delta x^2)(u^{1/2}) + \beta_8(\Delta x^2)(u) + \beta_9(\Delta x^2)(u^2) + \beta_{10} + e_{ijk} \quad (8)$$

Where regression coefficients are β<sub>i</sub> = 1, 2, ..., 10 and e<sub>ijk</sub> is the errors or residuals (Montgomery *et al.*, 2001; Myers, 1990).

The regression analysis was performed via SPSS computer program. The coefficient of determination (R<sup>2</sup>) was primary criterion for selecting the best equation to describe the curve equation. In addition to R<sup>2</sup>, the mean square of the deviations between the experimental and calculated values for the models and the root mean square error analysis were used to determine the goodness of the fit<sup>[6,8]</sup>.

## RESULTS AND DISCUSSIONS

Figure 2 and Table 3 show the relationship between temperature of lime juice and freezing time. On the first stage (S-1), the initial temperature of the lime juice (15°C) was decreased rapidly until it reached the freezing point of water (0°C). The freezing rate on S-1 at different lime juice layer thickness and air velocity varied in range of 0.27-1.22 min/°C. In the second stage S-2, the temperature slightly changed from 0°C to -2°C. The freezing rate on S-2 at different lime juice layer thickness and air velocity varied in range of 0.96-15.67 min/°C. In the last stage (S-3) the temperature decreased again and reached the set point temperature of -20°C. The freezing rate on S-3 at different lime juice layer thickness and air velocity varied in range of 0.76-2.76 min/°C. The interesting point was in S-2, it was noticed that the increased freezing rate was changed proportional to the increase of lime juice layer thickness. In S-1 and S-3, the freezing rate were also increased when increasing the lime juice layer thickness but had no significance. In S-2, at every air velocities, the freezing rate was increased radically when increased the lime juice layer thickness from 4-6 mm., from 6-8 mm., from 8-10 mm. and from 8-10 mm. In S-1 and S-3, at the same condition, the freezing rate was slightly increased.

The relationship between ratio of freezing time and lime juice layer thickness and air velocity at different lime juice layer thickness was shown in Fig. 3. The ratio of freezing time and lime juice layer thickness at different lime juice layer thickness of 4, 6, 8, 10 and 12 mm. varied in range of 10.0-11.28(u = 4 m sec<sup>-1</sup>), 7.58-8.63 (u = 6 m sec<sup>-1</sup>), 6.0-7.93 (u = 8 m sec<sup>-1</sup>), 5.78-7.47 (u = 10 m sec<sup>-1</sup>) and 5.75-7.37 (u = 12 m sec<sup>-1</sup>) min/mm, respectively. In addition, when comparing the ratio of freezing time and layer thickness of lime juice at air velocity of 8 with 10 m/s and 8 with 12 m sec<sup>-1</sup>, the decreasing percentage in ratio of freezing times and layer thickness of lime juice was varied in range of

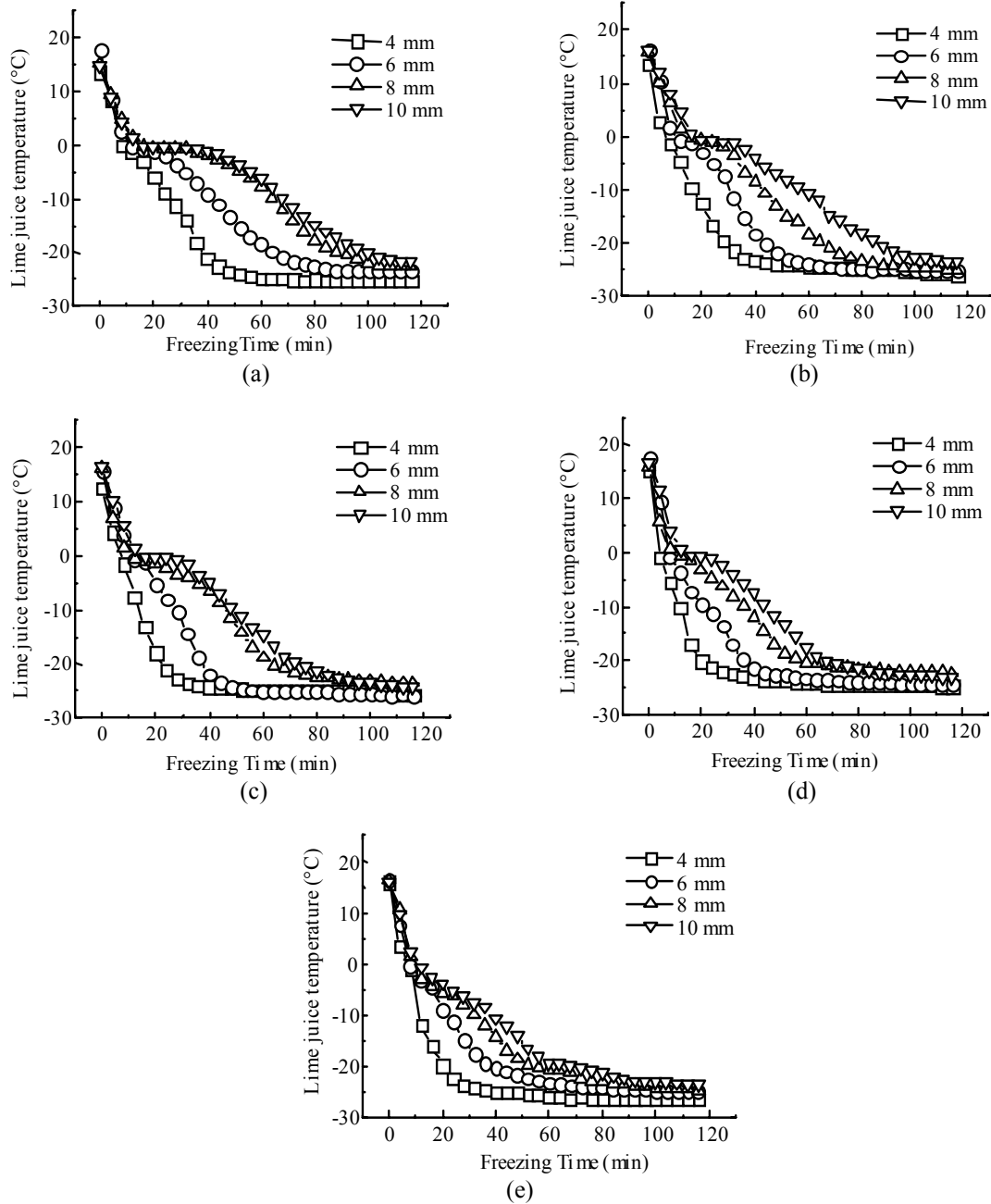


Fig. 2: The relationship between temperature of lime juice and time during freezing process at different lime juice layer thickness and air velocity of: a) 4 m sec<sup>-1</sup>, (b) 6 m sec<sup>-1</sup>, (c) 8 m sec<sup>-1</sup>, (d) 10m sec<sup>-1</sup> and (e) 12 m sec<sup>-1</sup>

1.95-8.0% at 4, 6, 8 and 10 mm of lime juice layer thickness. At the same lime juice layer thicknesses, the increasing percentage in ratio of freezing time and layer thickness of lime juice was varied in range of 17.0-46.81% when comparing the air velocity of 4 with 6 m sec<sup>-1</sup> and 4 with 8 m sec<sup>-1</sup>. Therefore, the ratio of

freezing time and layer thickness of lime juice was slightly changed at the air velocity of more than to 8 m sec<sup>-1</sup> at every lime juice layer thickness.

Figure 4 shows the relationship between ratio of freezing time and layer thickness of lime juice and lime juice layer thickness at different air velocity. The ratio

Table 3: Experimental freezing times at different air velocity and lime juice layer thickness

		Freezing times (min)			
Air velocity (m/s)	Layer thickness of lime juice (mm)	Freezing times (min)			
		4	6	8	10
4	S-1	8.62±0.18	13.25±0.16	16.56±0.19	18.36±0.26
	S-2	7.31±0.06	3.72±0.14*	27.33±0.58*	31.34±0.58*
	S-3	21.83±0.24	35.9±0.39	43.63±0.41	50.11±0.80
		17.70±0.5	24.14±0.19	34.07±0.16	45.37±0.33
6	S-1	8.43±0.31	10.20±0.33	19.59±0.17	16.75±0.28
	S-2	3.72±0.15	9.79±0.17*	19.59±0.17*	23.40±0.22*
	S-3	13.79±0.14	20.68±0.33	33.56±0.39	40.37±0.71
		13.6±0.24	20.67±0.42	33.53±0.36	37.64±0.42
8	S-1	7.66±0.26	10.37±0.29	10.41±0.26	15.67±0.35
	S-2	2.46±0.12	8.49±0.33*	15.88±0.14*	20.03±0.39*
	S-3	13.79±0.14	20.68±0.33	33.56±0.39	40.37±0.71
		13.6±0.24	20.67±0.42	33.53±0.36	37.64±0.42
10	S-1	4.34±0.17	9.22±0.23	9.48±0.26	13.13±0.26
	S-2	2.04±0.05	7.68±0.20*	14.84±0.29*	16.87±0.30*
	S-3	13.79±0.14	20.68±0.33	33.56±0.39	40.37±0.71
		13.6±0.24	20.67±0.42	33.53±0.36	37.64±0.42
12	S-1	3.99±0.13	8.37±0.16	9.39±0.34	12.95±0.29
	S-2	1.92±0.07	7.63±0.25*	13.55±0.25*	16.39±0.36*
	S-3	13.79±0.14	20.68±0.33	33.56±0.39	40.37±0.71
		13.6±0.24	20.67±0.42	33.53±0.36	37.64±0.42

S-1 = freezing time on the first stage, S-2 = freezing time on the second stage, S-3 = freezing time on the final stage. Values are mean ± S.E.M, The significance of the freezing time was evaluated by the analysis of variance (ANOVA). \*Significantly difference (p<0.05 as compare to the layer thickness of lime juice at 4 mm)

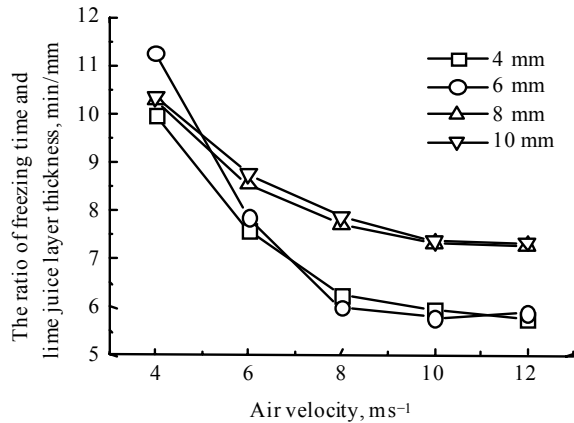


Fig. 3: Influence of air velocity on freezing times at different lime juice layer thickness

of freezing times and layer thickness of lime juice at different air velocity of 4, 6, 8, 10 and 12 m sec<sup>-1</sup> varied in range of 10.0-5.75 ( $\Delta x = 4$  mm), 11.28-5.78 ( $\Delta x = 6$ mm), 10.29-7.29 ( $\Delta x = 8$  mm) and 10.43-7.37 ( $\Delta x = 10$  mm) min/mm, respectively. When comparing the lime juice layer thickness of 6 with 8 mm and 6 with 10 mm., the increasing percentage in ratio of freezing time and limejuice layer thickness was varied in range of 23.87-32.22. at the air velocity of 8, 10 and 12 m sec<sup>-1</sup>. At the same air velocities of 8, 10 and 12 m sec<sup>-1</sup>, the increasing percentage in ratio of freezing time and limejuice layer thickness was varied in range of

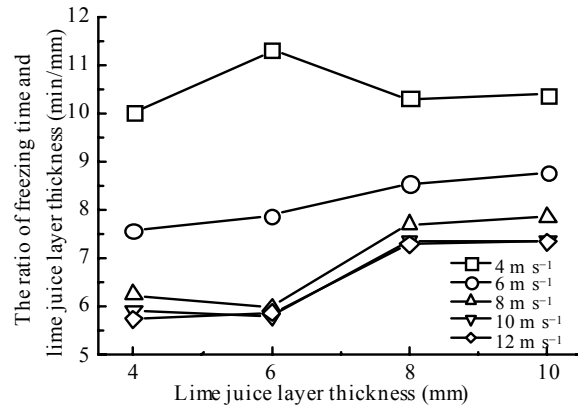


Fig. 4: Influence of lime juice layer thickness on freezing times at different lime air velocity

2.32-4.00. for the limejuice layer thickness of 4 and 6 mm. Therefore, the freezing time was extremely changed at the limejuice layer thickness of more than 6 mm and the air velocity of more than 8 m sec<sup>-1</sup>.

The results of statistical analysis undertaken on sum of squares and R<sup>2</sup>. These curve fitting criteria for these models were shown in Table 4. Generally, sum of squares (regression and residual) and R<sup>2</sup> values were varied between 196460.07-215510.35, 1096.65-20146.93 and 0.3685-0.9656, respectively. The Model (6) gave better prediction on the freezing time of lime juice than other models with R<sup>2</sup> of 0.9656.

The fitting curves procedure showed that the results of the Model (6) could be used to model the freezing time behavior of examined lime juice sample, but it could not indicate the effect of freezing air velocity and layer thickness. To account for the effect of the freezing variable on the models regression coefficient ( $\beta_1$ - $\beta_{10}$ ), the values of regression coefficient were shown in Table 4.

The accuracy of the established Model (6) was evaluated by comparing the computed freezing time with the experimental freezing time in sets of freezing condition. The performance of the Model (6) at the freezing air velocity and lime juice layer thickness has been illustrated in Fig. 5 (a) with plot of residuals ( $e_{ijk}$ ) versus predicted ( $\hat{y}_{ijk}$ ) ( $e_{ijk} = y_{ijk} - \hat{y}_{ijk}$ ,  $y_{ijk}$  is experimental data value), which indicates that a mild tendency for the variance of the residuals increased as the predicted freezing times increased. Figure 5 (b) is a plot of experimental freezing time versus predicted freezing time of lime juice. The predicted data generally banded around the straight line, which showed the suitability of the Model (6) in describing freezing time behavior of lime juice.

Table 4: Predicted model with nonlinear regression summary statistics and the values of the regression coefficients of the models determined through regression method for lime juice sample

Description	Model No.					
	1	2	3	4	5	6
R <sup>2</sup> (Asymptotic 95%)	0.8346	0.7134	0.3685	0.8249	0.9080	0.9656
Sum of squares						
Regression	211328.9	207463.55	196460.07	211020.39	213672.07	215510.35
Residual	5278.10	9143.45	20146.93	5586.61	2934.93	1096.65
Regression coefficient						
$\beta_1$	-9.3298	0.9853	7.4032	-15.0081	7.1121	-463.6767
$\beta_2$	0.4893	0.0768	-0.4946	0.0913	-0.4125	133.8524
$\beta_3$	0.2951	80.9615	-6.1552	0.5228	-24.0419	-3.0819
$\beta_4$	-0.0170			-0.0051	1.3472	168.7904
$\beta_5$	97.0105			88.5459	113.9094	-52.8776
$\beta_6$						1.3316
$\beta_7$						-4.6321
$\beta_8$						1.5544
$\beta_9$						-0.0415
$\beta_{10}$						394.7945

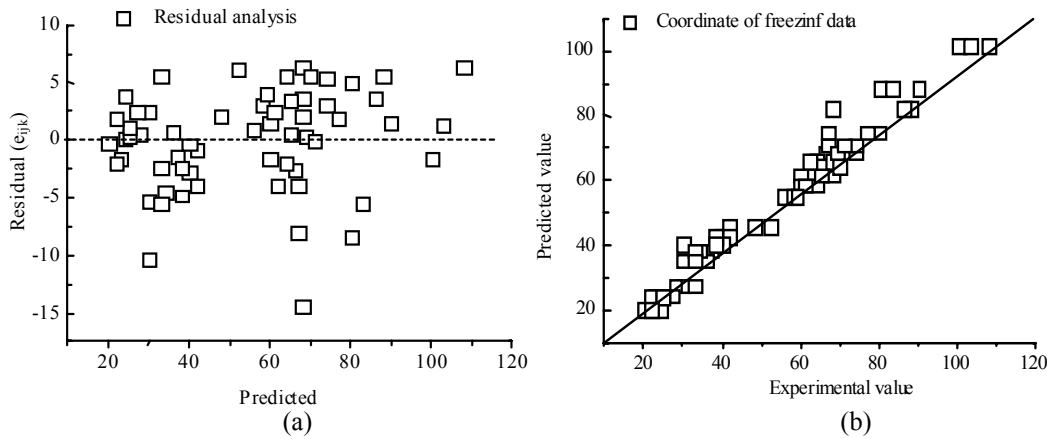


Fig. 5: Comparison of experimental and predicted freezing time by the Model (6) for different air velocity and lime juice layer thickness: (a) Plot of residuals ( $e_{ijk}$ ) versus predicted value ( $\hat{y}_{ijk}$ ), (b) Plot of predicted value versus Experiment value

### CONCLUSION

This study investigated the effect of air velocity of 4-12 m sec<sup>-1</sup> in range and lime juice layer thickness of 4-10 mm in range on freezing time of lime juice. The mathematical model was determined as the freezing time model. The freezing time model (Model (6)) was fit to a set of the experimental sample data, which was characterized by a regression model.

The freezing time of lime juice depended on air velocity and lime juice layer thickness. The freezing time was increased when decreased air velocity or increased lime juice layer thickness. Lime juice layer thicknesses of 8 mm caused non-linearity on the freezing time at the air velocity of 8, 10 and 12 m sec<sup>-1</sup>, so the practical condition of the freezing process

becomes uncertain. In addition, the freezing time at the second stage (S-2) was increased distinctly when the lime juice layer thickness was increased. Moreover, the freezing time was slightly decreased when the air velocity was increased more than 8 m/s at any given lime juice layer thickness.

In order to explain the freezing time behavior of lime juice, six regression models were compared to their coefficient of determination (R<sup>2</sup>). According to the results, the Model (6) could adequately describe the freezing time behavior of lime juice. The effects of the air velocity and lime juice layer thickness to the freezing time of lime juice were examined in the experiments at different conditions. The Model (6) gave the predicted results with an R<sup>2</sup> of 0.9656.

The future work of this study would be using the results to obtain the best operating condition for the air blast freezer in order to get the sensible freezing time of lime juice. It also could be used for designing the machine that could work both freezing and freeze-drying processes in order to get low cost production in producing lime juice powder for small scale manufacture.

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