Optimization foam mat drying of roselle *(Hibiscus sabdariffa L.)* extract

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Abstract. In this study, foaming condition of roselle was optimized using response surface methodology (RSM) and the effect of drying characteristic was investigated. Roselle extract was foamed by addition of 1-5% w/w foaming agents (ovalbumin). The foaming stabilizer, glycerol mono stearate (0-1% w/w) was used to remain mechanic and thermodynamic stability of foam. As the response foam density and drainage volume was determined. The optimum foam variable was then dried at various drying temperatures (50-70˚C). The moisture content was observed by gravimetry every 10 minutes for 90 minutes. Result showed that optimum formulation was 3.31% egg albumin and 1% GMS. The constant rate of the foam mat drying (temperature 50ºC) was 3 times higher than non foam mat drying. Higher drying temperature can speed up the driving force but lead to color degradation.

1 Introduction

The roselle plant is cultivated in China, India, Sudan, Uganda, Indonesia, Malaysia, and Mexico. The total roselle plant in Indonesia around 6.23% of worldwide total production. The main bioactive compounds in Roselle calyces are phenols and anthocyanins that its concentration according to the roselle cultivation and extraction process [1]. Previous research observed that roselle extraction with roselle:water ratio of 1:10 resulted in 502.33±0.52 mg of anthocyanins. Roselle extract has the potential aspect as natural colorant and antioxidant [2].

Drying roselle extract can result stable powder and enhance the storage life. Foam mat drying can shorten the drying time and retain the quality of the product by transforming liquid and semisolid materials into stable foam, with addition of foaming agent and incorporation of air [3]. A foaming agent is a surfactant materials that can reduce the surface tension and improve the foam formation. Egg albumen (EA), whey protein, soy protein, guar foaming albumin are the most common foaming agent [4]. The egg albumin can produce lower foam diameter, enhanced the surface area, reduced drying time and increased the water diffusivity [3,5]. GMS as the foaming stabilizer are polysaccharides that were added to thicken the aqueous solution. Addition of GMS in foam mat drying can form the stable foam [4,6].

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The previous observation studied about the optimum foaming condition of shrimp using xanthan gum [7]. Result showed that 0.19% (w/w) xanthan gum and whipping time 5.89 minutes were optimum foaming condition. In foam drying of muskmelon, 11.59% w/w EA and 0.59% w/w CMC were optimum foaming condition [8]. There was no study about foam optimization using egg albumin and GMS. The aim of this study was to investigate the optimum parameter to foam mat drying using egg albumin and GMS, determine the constant rate of drying in various temperature, and determine the change of Total Color Ratio (TCR) in foam mat drying of roselle extract.

2 Materials and Methods

2.1 Roselle extract preparation

The red roselle *(Hibiscus sabdariffa Linn.)* calyces (dry) were obtained from a herbal medicine market in Solo, Indonesia. The roselle calyces were ground and sieved until the size 0.25 mm. The roselle extraction used water as the solvent and roselle calyces mass to water volume ratio was 1:10 [2]. The extraction was operated at temperature 50°C under continuous agitation. The extract was then filtered through a Buchner filter.

2.2 Foam formulation

Eggs were purchased from the local market in Semarang, Indonesia. The egg albumin was separated from the yolk. One hundred ml of roselle extract was mixed with foam agent (egg albumin 1-5% w/w) and foam stabilizer (GMS 0-1% w/w). Then the solution was whipped with a hand domestic mixer (360 W power) at maximum speed for 3 min. Before the drying process, the foam was analyzed for the foam characteristic. There was two characteristic of the foam: foam density and foam drainage volume.

Foam density was determined using previous method [9]. One hundred ml of the foam was transferred into 100 ml measuring cylinder and weighed. The foam density was calculated using the following equation:

$$foam\ density = weight\ of\ foam\ (g)/volume\ of\ foam\ (cm^3) \quad (1)$$

Drainage volume was determined using previous method with modification [7]. Fifteen grams of foam was poured into a Buchner filter (80 mm diameter) and was placed in a 100-mL graduated cylinder. The drainage volume was liquid volume of the collapsed foam during 30 min. Each experiment was carried out in triplicate and the average was reported.

2.3 Optimization

Design Expert 7.0.0 software was used to find optimized levels of independent factors, analysis of variance (ANOVA), and regression models. The goals of the optimization process were minimum value of foam density and foam drainage volume. The independent variables were placed in the experimental range and the levels can be seen in Table 1.

Table 1. Levels of the independent variables.

Independent variables	units	Coded levels		
		-1	0	1
(A):Egg albumin	% w/w	1	3	5
(B):GMS	% w/w	0	0.5	1

2.4 Drying process

The foams were then dried in tray dryer. The foams were placed in aluminium tray (length 20 cm, width 15 cm and thickness of 0.2 cm) under drying temperature (50ºC-70ºC). The water loss was determined by weighing the samples every 15 minutes. The drying process took place for 90 minutes with superficial velocity of 0.18 m s^{-1}. As a comparison, the control sample (without addition of foam agent and foam stabilizer) was dried.

Thin layer model was used to determine the constant rate of the drying. Based on the literature, the newton model (Eq. 2) has been proven to predict the constant rate of foam drying [10,11]. The moisture ratio in Eq. 3 was simplified [7,12,13].
Refer to thin layer drying model, the correlation between moisture ratios with time can be represented by Eqn (1) [9].

$$kt = -\ln(MR) \tag{2}$$

$$MR = M_t/M_0 \tag{3}$$

Where k was the drying rate constant ($minute^{-1}$), t was drying time (min), M_t was the moisture content at time t, M_0 was the initial moisture content. Arrhenius equation (Eq. 4) was used to correlate the dependency of k on temperature (T)

$$k = k_0 exp^{\frac{-E_a}{R(T+273)}} \tag{4}$$

with k_0 was the pre-exponential factor ($minute^{-1}$), Ea was the activation energy (J mol^{-1}), and R was the gas constant (8.32 J mol^{-1} K^{-1}).

2.5 Color measurement

Color measurement was done by using Chroma Meter (CR-300, KONICA MINOLTA SENSING INC, Japan) in terms of observing L (lightness), a (redness and greenness) and b (yellow and blueness) as previously used by Kumar *et al.* [10]. The changes of total color ratio (TCR) was used to evaluate the total color degradation during drying process (Eq. 5) [16]:

$$TCR=(L_s a_s b_s)/(L_o a_o b_o) \tag{5}$$

Where, L_s, a_s, and b_s were measured on a sample at a given time, while L_o, a_o, and b_o were measured on raw roselle extract (before the drying process).

3 Results and Discussion

3.1 Foam density and foam drainage volume

Foam density (FD) and foam drainage volume (FDV) are commonly used to evaluate the foam characteristic. The density of the foam varied from 0.118 g cm^{-3} to 0.419 g cm^{-3}. Based on ANOVA analysis, the P value less than 5% indicated that the addition of egg white and GMS give the significant effect on FD. The correlation was related in quadratic model (Eq. 6). The foam density decreased with increased concentration of egg albumin and GMS (Fig 1.a). A similar result was reported by previous observation, increasing egg albumin from 1 to 3% make the FD decreasing[17]. The decreased of FD indicated that addition of foam agent reduces the surface tension and interfacial tension to form the sufficiently interfacial film and reducing the droplet size [18,4]. The decreased FD lead to increased the surface area for drying and speed up the drying rate [15].

$$FD = 0.16 - 0.10A + 0.018B - 0.043AB + 0.11A^2 - 0.022B^2 \quad , R^2=0.943 \qquad (6)$$

$$FDV = 9.06 + 1.42A - 1.08B - 0.13AB - 0.58A^2 - 0.083B^2 \quad , R^2=0.997 \qquad (7)$$

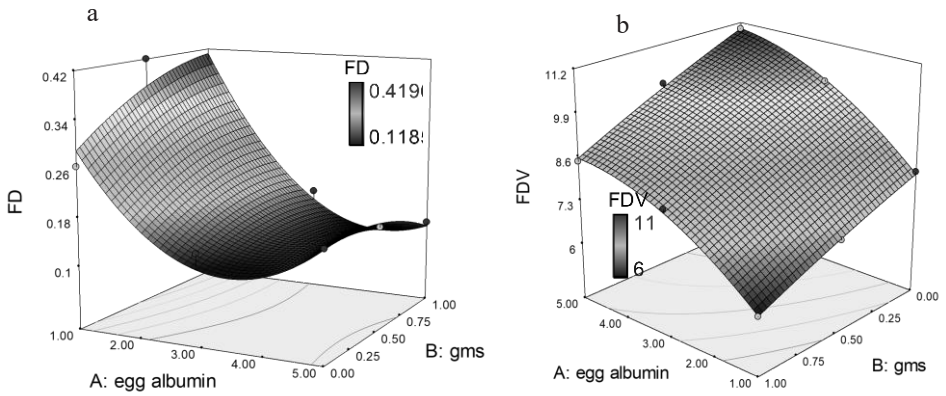

Fig. 1. a. Effect of addition egg albumin and GMS on Foam Density (FD) b. Effect of addition egg albumin and GMS on Foam Drainage Volume (FDV)

The FDV indicated the foam stability. The FDV varied from 6 to 11 ml. Based on ANOVA analysis, the P value less than 5% indicated that the addition of egg albumin and GMS give the significant effect on FDV. The correlation was related in quadratic model (Eq. 7). FDV decreased with increased concentration of and GMS (Fig 1.b). The GMS stabilization mechanism by form the lamellae and trapping the droplet in the microgel matrix [4].

3.2 Optimization

The goals of the optimization were to form the lower FD and lower FDV. Based on the optimization using software Design Expert, the formulation using 3.31% egg albumin and 1% GMS resulted in the lowest FD (0.135 g cm^{-3}) and the lowest FDV (8.07 ml) with desirability 86.2%. The optimum formulation was then validated and resulted FD 0.162 g cm^{-3} and FDV 9 ml. The results were more significant, compare with another foam formulation. The formulation resulted in the lower FD compare to foam formulation in muskmelon pulp [8]. In muskmelon foam formulation, using 11.59% egg albumin and 0.59% CMC resulted in FD 0.3390 g cm^{-3}. Bag et al. observed foam optimization in bael fruit using 3.1% w/w GMS and 0.32% w/w MC resulted higher FD (0.658) and lower FDV (1.75 ml) [18].

3.3 Drying Characteristic

The roselle extract was dried with foam agent and foam stabilizer at 50-70ºC. As the comparison, the extract dried without foam agent and foam stabilizer. The drying curves in the form of moisture content versus time were depicted in Figure 2. The moisture content of roselle extract reduced drastically with foam mat drying than non foam mat drying. The moisture content in foam mat drying until 0.426 (dry basis) while the moisture content of non foam mat drying 6.085 (dry basis). The constant rate of the foam mat drying (temperature 50ºC) was 3 times higher than non foam mat drying (Table 2). The foam formulation forms the decreased droplet size and increased the total surface area. The larger surface area leads to increased significantly moisture evaporation rate [3,4].

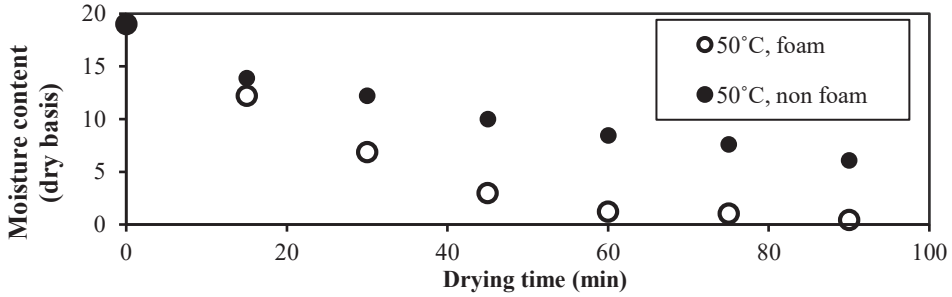

Fig. 2. Moisture ratio of foam mat roselle extract drying

The constant rate of the drying calculated using Eq. 2. The Newton model resulted the higher value of R^2 . The R^2 value varied from 0.853 to 0.981. The constant rate of the drying increased with increased drying temperature. The constant rate of foam mat drying in temperature 70ºC was 1.7 times higher than constant rate of foam mat drying in temperature 50ºC (Table 2). In higher drying temperature, the relative humidity decrease. The driving force for the drying higher and produced lower moisture content [19]. Arrhenius model was used to correlate the dependency of k on temperature (T) (Eq. 4). The value of Arrhenius constants were listed in Table 2. Using the value of Ea and K_0 , the value of constant rate of the drying in various temperature can be predicted.

Table 2. Constant rate of foam mat roselle extract drying

T (˚C)	Foam mat				Non foam mat			
	k (min^{-1})	R^2	$k_o \times 10^2$	Ea (kJ mol^{-1})	*k* (min^{-1})	R^2	$k_o \times 10^7$	Ea (kJ mol^{-1})
50	0.041	0.984	5.22	25.35	0.013	0.971	7.3	60.72
60	0.057	0.853			0.016	0.889		
70	0.071	0.941			0.049	0.947		

3.4 Color Quality

The total color ratio (TCR) between fresh roselle extract and dried roselle extract in different drying temperatures was depicted in Figure 3. After 90 minutes drying, the TCR decreased. The TCR reduction was higher at higher drying temperature. In drying temperature 70ºC, the TCR was the lowest (0.49). The color in roselle extract was from anthocyanins pigments. The constant rate of anthocyanin degradation depends on pH, temperature, copigmentation, ascorbic acid, oxygen, etc [2]. In this dying process, the degradation of anthocyanin because of the thermal degradation. The color change from red to brown indicated the thermal degradation in the drying process. [20].

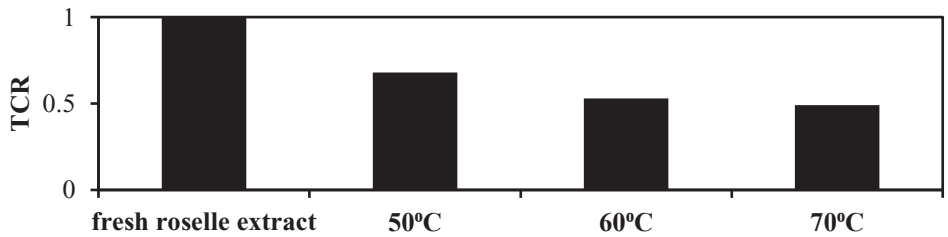

Fig. 3. Effect of drying temperatures on Total Color Ratio (TCR)

4 Conclusions

The roselle formulation using 3.31% egg albumin and 1% GMS resulted in the lowest FD (0.135 g cm^{-3}) and the lowest FDV (8.07 ml). Egg albumin contributed to form the sufficiently interfacial film and reducing the droplet size. GMS contributed to stabilizing the foam by during the drying process. The constant rate of the foam mat drying (temperature 50ºC) was 3 times higher than non foam mat drying. Higher drying temperature can speed up the driving force but lead to thermal degradation.

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