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#### RESEARCH PAPER

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# Effect of cocoa butter content and degree of the fineness on the dispersibility and chromaticity values of cocoa powder

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#### Abstract

The present study was conducted to investigate the effect of cocoa butter contents (10 to 25%), degree of fineness of particle (40 to 200 mesh sieve) on the dispersibility and colour of cocoa powder. The obtained results indicated that the higher the cocoa butter content in cocoa powder, the higher the water separated with time. The dispersibility of cocoa powder also depends on the degree of fineness is the best in the samples where the particles were smaller (200 mesh sieve), and much worse in cocoa powder with particles 40 mesh sieve (in the experimental ranging). Cocoa powder of 15% cocoa butter gave the better dispersibility than cocoa powder with 20 and 25% cocoa butter. A very high correlation between the degree of fineness, cocoa butter and time on dispersibility (percentage of water separated) of cocoa powder drink was observed. Besides, there were significant differences among the chromaticity value of cocoa powder for the different degree of fineness and cocoa butter content, the more cocoa butter content, the darker in cocoa colour. The most important effects on L and b values are cocoa butter content and degree of fineness, while a value is most affected by cocoa powder degree of fineness.

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#### Introduction

The cocoa beans of commerce are the seeds of tree-species *Theobroma cacao* L. and the genus *Theobroma* (family *Sterculiaceae*) consists of some twenty-two species of small bushed and tree, is commercially the most important, due to the value of its seeds (Bartley, 2005). Cocoa powder is the product from roasted ground seeds (cocoa beans). For processing into cocoa powder, about half of the fat (cocoa butter) is pressed out, or extracted from the cocoa mass.

The main problem in cocoa powder processing is the production of standard quality acceptable for marketing. The primary requirements for both marketing and manufacturing stages of cocoa powder flavour and colour with dispersibility. Dispersibility defined as a uniform distribution of particles in solution, these mechanisms are closely related to the particle size and surface area of the particles in the final product in powder form (Kwapińska and Zbiciński, 2005). The relationship between colour and particle size was studied in several fields including food (McKee et al., 2012) and sediments (Storlazzi et al., 2015). The influences of the configuration of a spraying system on particle size and dispersibility of soy powders were investigated by Syll et al. (2013). The effect of particle size distribution on properties of powder mixtures was studied by Peterson and Small (1993). Powder coatings and the effects of particle size has been previously reported by Claase et al. (2014). Additionally, particle size also influenced the colour of the powder (Haas et al., 2019), as particle size decreases, the increase in the particle surface area causes higher affinity with moisture and higher ability to agglomerate during the drying process. Kurozawa et al. (2009) indicated that the lower the particle size, the lower the solubility and flowability of the powder.

So far, studies on the effects of cocoa butter content and degree of fineness on quality of cocoa powder are very limited in Vietnam and are qualitative. So, indepth studies on this subject are still needed. To obtain the best quality of the cocoa powder, the parameters which would likely affect cocoa powder quality not only the degree of fineness of particle size but also the cocoa butter remaining. The formation on the quality of cocoa powder as affected by these process are needed. This study attempts to develop a process methodology to improve the quality of cocoa powder product, especially for the dispersibility/suspension and chromaticity value of cocoa powder.

#### Materials and methods

Preparation of cocoa sample

Shelled raw cocoa beans (Forastero variety), used for this study were obtained from the Lab of Food Technology, Can Tho University, Vietnam. The moisture content of cocoa beans was around 5-6% wet basis. The cocoa beans were fermented and dried in an oven at a temperature of 70-75°C until the moisture content reduced to 3%. The dried bean (Fig. 1) was packed in bags, sealed and stored at room temperature. Before using, the dried beans were roasted in batches of 10kg in a rotary roaster. At the roasting time of 40 min and temperature of 120°C, full chocolate flavour developed.



Fig. 1. Drying of fermented cocoa beans

Effect of the cocoa butter content, degree of fineness and time on dispersibility of the cocoa powder Sample preparation

Cocoa powder is obtained by hydraulic pressing (HYP305HP, Japan) of finely ground cocoa liquor, which must have been made from winnowed cocoa beans. Hydraulic presses can accurately obtain the required fat content in the cocoa cake. Representative samples of 1kg each were obtained with different

cocoa butter content (10, 15, 20, 25%) and degree of fineness (40, 100, 140, 200 mesh sieve or 400, 149, 105, 74 $\mu$ m, respectively). O3g cocoa powder was weighed and stirred in hot water until free from lumps and stirred vigorously for 1 minute by a magnetic stirrer, then transferred to the measurement cylinder. After that, the volume of water separated from the mixture was recorded at 5 min interval for 50 min.

# Sample analysis

Testing of the dispersibility of the cocoa powder

The response was measured: per cent of water separated (Y) defined as water separate from the mixture (cocoa powder in hot water). It depends on butter content in cocoa butter, degree of fineness of cocoa powder and the time. The experiment was randomized to get the optimum variability in the observed responses due to extraneous factor.

# Determination of cocoa powder colour indicators

The colour of cocoa powder is determined by three factors, A frequently used representation is the one established by the Commission Internationale de l'Eclairage (CIE), where the colour is a three-dimensional point: L\*, a\* and b\*. L\* shows the difference between light and dark (100=white and 0=black), a\* shows the difference between red and green (+ a\*=red and - a\*=green) and b\* shows the difference between yellow and blue (+ b\* = yellow and - b\* = blue).

Cocoa powder colour indicators were determined using a CR-410 Chroma meter (U.S.A.). The purpose of this test was to select the optimum degree of fineness of cocoa bean sample which gives light colour and gave the smallest coefficient of variation in output readings.

The formula for the sample standard deviation (equation 1) is:

Standard deviation: 
$$S = \sqrt{\frac{1}{N-1} \sum_{i=1}^{N} (x_i - \overline{x})^2}$$
 (1)

Where  $x_1$ ,  $x_2$ , .....,  $x_n$  are the observed values of the sample items,  $\bar{x}$  is the mean value of these

observations, and N is the number of observations in the sample.

Statistical analysis

A statistical analysis (STATGRAPHIC) was used to fit the model to the observed data. The proposed model (equation 2) for each response (Y) was:

$$Y = b_o + \sum_{n=1}^{3} b_n X_n + \sum_{n=1}^{3} b_{nn} X_n^2 + \sum_{n \neq m=1}^{3} b_{nm} X_n X_m(2)$$

Where: b<sub>0</sub>: Y intercept (constant)

 $b_n\colon regression$  coefficient for the linear effect of  $X_n$  on Y  $b_{nn}$  and  $b_{nm}\colon regression$  coefficient for the quadratic effect on Y

X<sub>n</sub>, X<sub>m</sub>: independent values

To select the acceptable condition, the multiple regression analysis was used for the data from each reference cocoa butter content, degree of fineness and the time taken. In the multiple regression analysis, the dispersibility in terms of water separated was defined as the dependent variable and the cocoa butter content, degree of fineness and the time recorded which were presented in  $X_1$ ,  $X_2$  and  $X_3$  values were defined as independent variables. The reference equation was selected to fit the data, based on the  $R^2$  value obtained from the multiple regression. The selected reference equation should have a higher  $R^2$  value compare to  $R^2$  value for the other reference interaction.

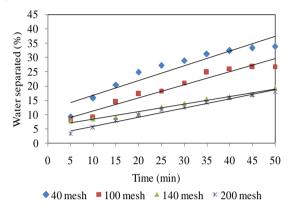
#### Results and discussion

Effect of degree of fineness and cocoa butter on Dispersibility of cocoa powder

 $Degree\ of\ fineness$ 

One of the most important physical parameters of food powders is particle size. The results in Fig. 2 showed the relationship of dispersibility varied with time as determined by water separated (cocoa powder with 20% cocoa butter and different degree of fineness). The amount of water separated increased linearly with time. At 50 min, the water separated was highest with cocoa powder of 40 mesh (33.82%) and less with cocoa powder of 200 mesh (18%). The results for the other cocoa butter content (10, 15, 25%) with different degree of fineness of cocoa powder showed a similar trend (data not shown).

The results indicated that the most important property of cocoa powder is its fineness, for its solubility is low. In water or milk suspension, coarse particles will rapidly settle giving objectionable rough sediment. The large particle will also give a specky appearance in a milk drink or ice cream. A moderate increase in particle size (from 15 to 24  $\mu$ m) improved powder dispersibility in most cases (Syll *et al.*, 2013).



**Fig. 2.** The percentage of water separated as a function of the degree of fineness with time (cocoa butter content 20%).

# Cocoa butter content

Fig. 3 showed the dispersibility data of the product at the degree of fineness of 100 mesh with different cocoa butter contents. It can be seen that at 50 min the water separated was highest with 25% cocoa butter content in cocoa powder sample (37.33%) and less with 10% and 15% of cocoa butter content in cocoa powder (21.04% and 20.85%, respectively).

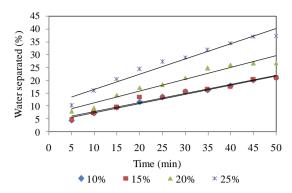
The results for the others degree of fineness of cocoa powder (40, 140, 200 mesh) with different cocoa butter content showed a similar trend (e.g. Table 1). The dispersibility of cocoa powder at a different percentage of cocoa butter content decreased with increased in cocoa butter content that remained in cocoa powder. The less hydrophilic property might be due to the formation of the surfactant with the CH and -CO groups more than -OH group, when the surface of individual particle coating by a high amount of cocoa butter. It was observed that increasing dispersibility of cocoa powders could be achieved by removal or reduction of their fat content. Cocoa butter might be the cause for the failure of cocoa powders to disperse rapidly in cold liquids. Therefore, cocoa butter can be separated from cocoa powder by many methods such as pressing and extracting with appropriate solvents (Acton, 2013). Moreover, from a nutrition standpoint, there has been an increase in the percentage of the population that is either overweight or obese over the last years, the growing concern with the relationship between health with reduced energy value in food was interested (Boff et al., 2013). Reducing fat content is currently one of the primary trends in food product innovation (Ma & Boye, 2013). The dispersibility that determined as water separated from the mixture increased significantly with decreased cocoa butter content in cocoa powder and also increased when the degree of fineness increased. Fig. 4 summarized three

**Table 1.** The percentage of water separated from the mixture cocoa powder (different degree of fineness) with 15% of cocoa butter.

main effects for water separated.

	1			
Time (min)	40 mesh	100 mesh	140 mesh	200 mesh
5	4.79**±0.17***	4.59±0.17	3.15±0.23	2.52±0.07
10	7.15±0.06	$7.29\pm0.17$	$5.22 \pm 0.30$	$4.78 \pm 0.11$
15	10.18±0.17	9.48±0.07	$7.26 \pm 0.06$	5.00±0.22
20	13.85±0.06	11.74±0.06	9.41±0.68	$7.00\pm0.11$
25	15.89±0.11	13.60±0.06	11.33±0.45	7.93±0.17
30	17.93±0.34	$15.74 \pm 0.26$	13.15±0.17	$8.33 \pm 0.11$
35	20.15±0.23	16.29±0.06	14.93±0.34	10.00±0.22
40	21.33±0.11	17.67±0.34	15.67±0.11	11.48±0.13
45	23.41±0.28	20.22±0.11	$16.74 \pm 0.23$	11.67±0.34
50	24.74±0.17	20.85±0.32	18.22±0.29	12.48±0.75

<sup>\*\*</sup>Data are means of triplicates; \*\*\*The standard deviation (STD) of the mean.



**Fig. 3.** The percentage of water separated as a function of cocoa butter content with time (degree of fineness of cocoa powder 100 mesh).

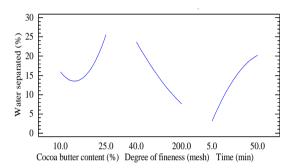


Fig. 4. Main effects plot for water separated.

The relationship between dispersibility of cocoa powder with the degree of fineness, cocoa butter content with time was presented in the multiple regression equation. The equation of the fitted model (Equation 3) is as follows:

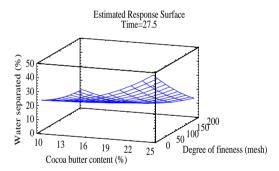
$$Y = 22.403 - 2.603 X_1 + 0.023 X_2 + 0.625 X_3 + 0.112 X_1^2 - 0.008 X_1 X_2 + 0.01 X_1 X_3 + 0.0002 X_2^2 - 0.001 X_2 X_3 - 0.005 X_3^2 (3)$$

Where Y is water separated (%),  $X_1$  is cocoa butter content (%),  $X_2$  is the degree of fineness (mesh) and  $X_3$  is time (min)

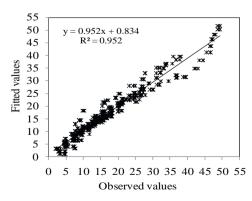
R-squared = 95.219 percent, P=0.0000

The effect of cocoa butter content and degree of fineness of cocoa powder on water separated at the time (27.5 min) is presented in Fig. 5. The ANOVA showed that in this case, 09 effects have P-values less than 0.05, indicating that they are significantly different from zero at the 95.0% confidence level. The R-Squared statistic indicates that the model as fitted explains 95.22% of the variability in water separate. The adjusted R-squared statistic, which is more suitable for comparing models with different numbers

of independent variables, is 95.13%. The standard error of the estimate shows the standard deviation of the residuals to be 2.386. The mean absolute error (MAE) of 1.68361 is the average value of the residuals. Since the P-value is less than 5.0%, there is an indication of possible serial correlation at the 5.0% significance level. The correlation between the fitted and the observed values of water separated was satisfactory (Fig. 6). The combination of factor levels which maximizes water separated over the indicated region is shown. The dispersibility that determined as water separated from the mixture increased significantly with decreased cocoa butter content in cocoa powder and also increased when the degree of fineness and time increased. Fig. 7 estimated in decreasing order of importance of nine effects on water separated. The most important effects are time and degree of fineness. Based on sensory evaluation of mango juice (from mango pulp powder), Sharma et al. (2013) found that the most acceptable mango pulp powder with the mean particle size of 260.09 µm compared to the particle size of 296.19 and 191.26µm.



**Fig. 5.** Influence of cocoa butter content and degree of fineness of cocoa powder on water separated.



**Fig. 6.** Correlation between the fitted values and the observed values estimated for water separated using the model described in equation 3.

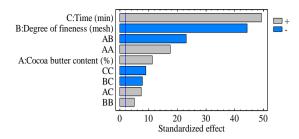


Fig. 7. Standardized Pareto chart for water separated.

The relationship between cocoa powder colour and degree of fineness and cocoa butter

The particle size of cocoa powder and the remaining cocoa butter content in powder had importance both organoleptically, in manufacturing processes and economically. Cocoa powder is used for its two major properties as flavour and colour of a product. The user also influences the colour by the processing method and the choice of ingredients in the recipe.

The colour of the cocoa powder also affects the colour of the user's end product. Thus colour is such an important aspect of cocoa powder. The cocoa powder properties as colour, degree of fineness and cocoa butter are important in all applications, as this ensures consistency in the appearance of the final product. This is desired by both the user of the powder and the consumer of this product. Cocoa powder also applies in the ice-cream industry, where the fineness of the powder is important for influencing the crystallization process and color of the ice cream. Coarse particles may produce unwanted dark specks.

The results from these experiments are presented in Table 2. Statistical analysis to compare the mean showed that there were significant differences among the chromaticity value for the different degree of fineness and cocoa butter content.

Table 2. The average L\*, a\*, b\* values of cocoa powder with different degree of fineness and cocoa butter content.

Cocoa butter content (%)	Chromaticity		
Degree of fineness	L*	a*	b*
10% Cocoa butter			
40 mesh	32.37**±0.12***	8.29±0.02	10.49±0.08
100 mesh	$32.84 \pm 0.05$	$10.10\pm0.01$	11.87±0.06
140 mesh	33.79±0.08	10.79±0.01	12.54±0.05
200 mesh	33.92±0.11	$10.78 \pm 0.03$	12.74±0.05
15% Cocoa butter			
40 mesh	30.96±0.08	9.14±0.05	10.71±0.07
100 mesh	$32.73\pm0.07$	$10.12 \pm 0.05$	11.74±0.08
140 mesh	33.31±0.11	$10.58 \pm 0.10$	12.17±0.05
200 mesh	33.26±0.12	$11.27 \pm 0.15$	$12.63 \pm 0.05$
20% Cocoa butter			
40 mesh	25.75±0.07	9.61±0.06	$8.79 \pm 0.04$
100 mesh	27.02±0.05	9.85±0.15	9.71±0.08
140 mesh	$28.11 \pm 0.05$	$10.50\pm0.20$	9.64±0.14
200 mesh	27.79±0.04	10.70±0.12	9.44±0.21
25% Cocoa butter			
40 mesh	27.25±0.12	$8.78 \pm 0.12$	$8.38 \pm 0.08$
100 mesh	26.6±0.05	10.22±0.10	9.39±0.07
140 mesh	28.39±0.10	11.09±0.08	10.15±0.10
200 mesh	25.77±0.10	11.43±0.06	10.03±0.05

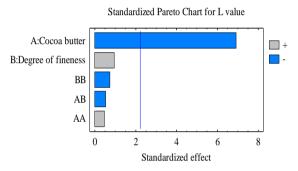
<sup>\*\*</sup>Data are means of triplicates; \*\*\*The standard deviation (STD) of the mean.

# L\* value

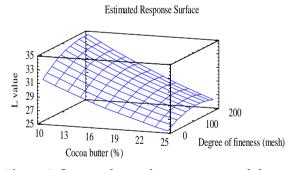
The results indicated that  $L^*$  values decreased with increasing cocoa butter content, which means that cocoa colour becomes darker with cocoa butter content. This may be due to the colour of cocoa butter content included. The ANOVA table partitions the variability in  $L^*$  value into separate pieces for each of the effects. It then tests the statistical significance of each effect by comparing the mean square against an

estimate of the experimental error. In this case, on effect (cocoa butter content) has P-value less than 0.05, indicating that they are significantly different from zero at the 95.0% confidence level. The R-Squared statistic indicated that the model as fitted explains 83.3091% of the variability in  $L^*$  value. The adjusted R-squared statistic, which is more suitable for comparing models with different numbers of independent variables, is 74.964%.

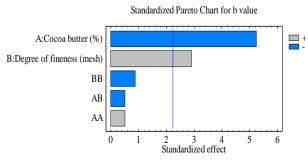
The standard error of the estimate shows the standard deviation of the residuals to be 1.601. The mean absolute error (MAE) of 1.143 is the average value of the residuals. 04 remained effects (degree of fineness. cocoa butter\*cocoa butter. cocoa butter\*degree of fineness, degree of fineness\*degree of fineness) have P-values higher than 0.05, stated that the treatment effects were not statistically significant. Fig. 8 estimated in decreasing order of importance of five effects on  $L^*$  value. The most important effect is the cocoa butter content. The effect of cocoa butter content and degree of fineness of cocoa powder on the L value of cocoa powder is presented in Fig. 9.



**Fig. 8.** Standardized Pareto chart for L\* value of cocoa powder.

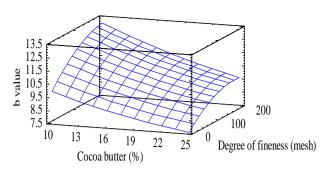


**Fig. 9.** Influence of cocoa butter content and degree of fineness of cocoa powder on  $L^*$  value.

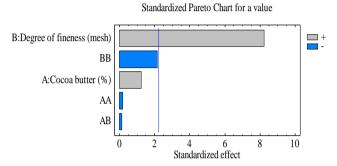


**Fig. 10.** Standardized Pareto chart for  $b^*$  value of cocoa powder

# Estimated Response Surface

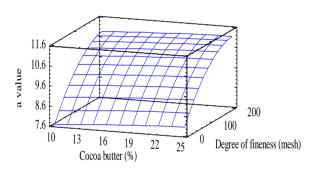


**Fig. 11.** Influence of cocoa butter content and degree of fineness of cocoa powder on b



**Fig. 12.** Standardized Pareto chart for  $a^*$  value of cocoa powder

Estimated Response Surface



**Fig. 13.** Influence of cocoa butter content and degree of fineness of cocoa powder on  $a^*$  value

#### b\* value

The ANOVA table partitions the variability in b value into separate pieces for each of the effects. In this case, 2 effects (cocoa butter content and degree of fineness) have P-values less than 0.05 (0.0004 and 0.0157, respectively), indicating that they are significantly different from zero at the 95.0% confidence level. The R-Squared statistic indicates that the model as fitted explains 78.87% of the variability in b value. The adjusted R-squared statistic, which is more suitable for comparing models with different numbers of independent variables, is 68.303%.

The standard error of the estimate shows the standard deviation of the residuals to be 0.846. Three remained effects (cocoa butter\*cocoa butter, cocoa butter\*degree of fineness, degree of fineness\*degree of fineness) have P-values higher than 0.05, stated that the treatment effects were not statistically significant. Fig. 10 showed the most important effects are cocoa butter content and degree of fineness. The effect of cocoa butter content and degree of fineness on  $b^*$  value of cocoa powder is presented in Fig. 11.

# a\* value

The ANOVA table partitions the variability in a into separate pieces for each of the effects was obtained. In this case, one effect (degree of fineness) has a P value less than 0.05, indicating that they are significantly different at the 95.0% confidence level. The remained 04 effects (cocoa butter, cocoa butter\* cocoa butter, cocoa butter\*degree of fineness, degree of fineness\* degree of fineness) have P values higher than 0.05, simply stated that there was no evidence for a treatment effect. The R-Squared statistic indicates that the model as fitted explains 88.112% of the variability in a value. The adjusted R-squared statistic, which is more suitable for comparing models with different numbers of independent variables, is 82.168%. The standard error of the estimate shows the standard deviation of the residuals to be 0.376. Different from L and b values, Fig. 12 represented the most important effect is the degree of fineness. The effect of cocoa butter content and degree of fineness of cocoa powder on a value of cocoa powder is presented in Fig. 13. These results are in agreement with published findings showing that the L\* a\* b\* values of the powders were measured to quantify the impact of particle size on the perceived colour intensity of carrot concentrate powder (Haas et al., 2019).

## Conclusion

The dispersibility and colour of cocoa powder drink prepared from different cocoa butter contents, degree of fitness of particle were investigated. As determined, the lowest particle size (200 mesh) of cocoa powder increased the dispersibility of the mixtures. Cocoa powder drink prepared with the cocoa powder containing 15% cocoa butter content gave the better dispersibility than cocoa powder with 20 and 25% cocoa butter.

These findings provide a potential approach to reduce the cocoa butter content in cocoa powder processing. Further study will be conducted to optimize the emulsion formulation is essential to improve the stability of these oil-in-fat dispersions. The displayed results also indicated the significant potential of using proper cocoa butter content for the processing of cocoa powder for a drink, which may provide good physical characteristics and high consumer preference.

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