# The Optimization of Maltodextrin and Arabic Gum in the Microencapsulation of Aqueous Fraction of Clinacanthus nutans Using Simplex Lattice Design

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# The Optimization of Maltodextrin and Arabic Gum in the Microencapsulation of Aqueous Fraction of *Clinacanthus nutans* Using Simplex Lattice Design

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**ABSTRACT**

The aqueous fraction of *Clinacanthus nutans* leaf extracts contains flavonoids which known had antioxidative properties. To improve acceptability, this viscous and bitter aqueous fraction was microencapsulated using maltodextrin and Arabic gum. This research aims to discover the effectivity of maltodextrin and Arabic gum and the concentrations for optimum microencapsulation. Optimization design was done using Design Expert with simplex lattice design with ratios of 1:0; 0.75:0.25; 0.5:0.5; 0.25:0.75 and 0:1. The evaluations done to the results were microcapsule yield, moisture content, flow rate, and antioxidant activity. The optimum ratio of maltodextrin and Arabic gum was obtained at 0.806:0.194 with 1.49% moisture content, flow rate 4.375 g/s and antioxidant activity at the value of 842,499 ppm. The result of one-sample T-test showed that the prediction result of Design Expert did not differ significantly from the experiment result. From the data, it was concluded that the resulting equation was valid.

**Keywords**: aqueous fraction, *Clinacanthus nutans*, microencapsulation, maltodextrin, Arabic gum.

## INTRODUCTION

Currently, there are many research on plant antioxidants because the common synthetic ones such as BHA and BHT was suspected to be carcinogenic[10]. *Clinacanthus nutans* is one of the plants commonly found in Indonesia and has potential as antioxidants. *Clinacanthus nutans* is a shrub commonly used as hedgerows in Indonesia. In traditional medicines, this plant is used as antidiabetics, antioxidants, antiinflamation, analgetics, antiviral, and also for wound treatment.

The leaves of *Clinacanthus nutans* contain alkaloids, triterpenoids/steroids, glycosides, tannins, saponins, and flavonoids. The isolates of *Clinacanthus nutans* methanolic leaf extracts contains six C-glycosides flavons, namely shaftoside, isomollupentin7-O-β-glukopyranoside, orientin, isoorientin, vitexin, and isovitexin[2]. The flavonoids are known to acts as antioxidants by capturing free radicals. The aqueous fraction of *Clinacanthus nutans* leaf extracts had value of $EC_{50}$ 532,24 ppm, showing antioxidants properties[8].

The usage of *Clinacanthus nutans* leaf extracts fractions has several shortcomings, amongst them are its viscosity that gave difficulties in formulation and its astringent and bitter taste that made it hard to use orally. To overcome these shortcomings, microencapsulation can be used[6].

Microencapsulation is a process of miscroscopic encapsulation of drug particles with specific coat that result in better physical and chemical properties of those particles. Microencapsulation intends to protect sensitive components, reduce the loss of nutritions, and converts liquids to solids. The choice of microencapsulation methods depends on its applications and specific parameters, such as desired particle size, physicochemical properties of the core and the coating, release mechanism, process cost, etc. Freeze drying is one of drying method for encapsulation which had advantages in preserving the quality of the drying products, especially for heat-sensitive materials. Encapsulation can be attained as homogenous core in matrix solution which then was co-lyophilized, resulting in irregular shapes[4]. Freeze drying is a suitable method for encapsulation of antioxidants to preserve their properties because antioxidants are easily damaged by heat and light.

The choice of coating materials for microencapsulation of aqueous fraction of *Clinacanthus nutans* leaf extracts was based on the usage of combination of Arabic gum and maltodextrin for microencapsulation of grape anthocyanins[3] and on encapsulation of *Berberis vulgaris* extracts using Arabic gum and maltodextrin in 1:3 ratio[5]. The combination of maltodextrin and Arabic gum as encapsulant is expected to form stable microcapsule and protect the aqueous fraction of *Clinacanthus nutans* because of the film-forming properties of Arabic gum and the ability of maltodextrin to protect microcapsules from oxidation. This research intended to find the optimum

concentrations of microencapsulants of the aqueous fraction of *Clinacanthus nutans*.

## MATERIALS AND METHODS

*Tools and Materials*

Materials that were used on this research were *Clinacanthus nutans* leaves, analytical grade ethanol 96% (Merck), analytical grade methanol (Merck), analytical grade n-hexane (Merck), analytical grade ethyl acetate (Merck), DPPH, maltodextrin (*Dextrose Equivalent* 10,6), Arabic gum (*Tic Gums*), and distilled water. Tools used were separation funnels, *r*otary evaporator (*Buchi Rotavapor R-200*), freezer, freeze dryer (*Thermo Scientific Powerdry LL 1500*), moisture analyzer *(Mettler Toledo HE53)*, flow funnels, ovens, UV-visible spectrophotometer (*Shimadzu*), SEM (*Scanning Electron Microscopy*).

*Methods*

*Extraction dan Fractionation of Clinacanthus nutans Leaves*

Two hundred grams of *Clinacanthus nutans* leaf powder was macerated with two liters of ethanol 96% for two days. Diluted extracts was separated from the solvent using rotary vacuum evaporator at 40°C. The extract then gradually fractionated using liquid-liquid partition in separation funnels with water, ethyl acetate, and n-hexane as solvents. The water fraction then was evaporated using rotary vacuum evaporator at $80^0$C.

*Microencapsulation of Aqueous Fraction of Clinacanthus nutans Leaf Extracts*

Maltodextrin and Arabic gum in various ratio (see Table 1) were suspended in distilled water and the to the mixture the aqueous fraction of *Clinacanthus nutans* leaf extracts was added. The resulting mixture was frozen for 24 hours and then dried using freeze-dried in the temperature -100°C. The dry samples were pulverized and then were sifted using 24 mesh sieve. The resulting microcapsules were kept in tigtly closed container and protected from light.

*The Evaluation of Microcapsule Characteristics*

*Microcapsule yield*

The yield was calculated by comparing the weight of microcapsule to the total active and coating materials[1]. The percentage of the microcapsule yield was calculated using Equation 1:

$$\text{Yield} = \frac{Wt - Wo}{Wo} \times 100\% \quad \text{.....(1)}$$

Note: Wt: initial weight (g)

Wo: end weight (g)

*Moisture content*

Moisture content was measured by weighing 0.5 grams of microcapsules and measuring them in the moisture meter. The start button was pressed and then the resulting numbers were noted when the notification sounds were heard.

*Solubility*

Solubility were evaluated by solving one gram of microcapsules in 25 ml distilled water. The resulting solutions were filtered using Whatman paper No. 42. The

Table 1: Microcapsule Formulation of Aqueous Fraction *Clinacanthus nutans* Leaf Extracts.

| Materials | F1 | F2 | F3 | F4 | F5 |
|---|---|---|---|---|---|
| Aqueous fraction (%) | 4 | 4 | 4 | 4 | 4 |
| Arabic gum | 1 | 0,75 | 0,50 | 0,25 | 0 |
| Maltodextrin | 0 | 0,25 | 0,50 | 0,75 | 1 |

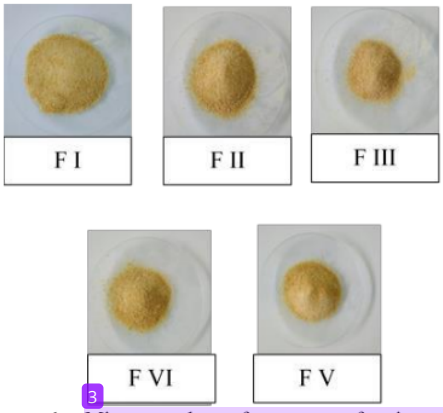



Figure 1: Microcapsules of aqueous fraction of *Clinacanthus nutans* leaf extract.

filter papers and residues were dried in oven at 105°C for three hours and then were cooled down and weighed. The solubility percentage then was calculated using equations 2 and 3.

Solubility percentage = 100% - residue percentage .....(2)

$$\text{Residue percentage} = \frac{\text{Weight of filter paper and residue} - \text{weight of filter paper}}{\text{weight of sample}} x\ 100\% \quad \text{..... (3)}$$

*Flow Rate*

Flow rate was measured by weighing 100 g microcapsules, and then inputting those microcapsules into a closed-end funnel. The cover at the end of the funnel then was opened ant then the microcapsules was let to flow until there weren't any microcapsules remaining in the funnel. The flow time was recorded as the time the microcapsules needed to flow from the time the cover was opened until all the granules flowed out.

*Antioxidant Activity Assay with DPPH Method*

Antioxidant activity was evaluated by solving 250 mg microcapsule in 50 ml of methanols and diluting the solution to 1000,1500, 2000, 2500 and 3000 ppm. From those solutions, 2 ml were taken and 4 ml DPPH solution was added. The mixtures then were incubated at room temperature in dark condition for 30 minutes. The lowering of absorbancy was measured with spectrophotometer at 517 nm. The negative controls were made without any samples. From the resulted absorbancy, percentage of inhibition and $EC_{50}$ value were calculated. Percentage of inhibition was calculated using Equation 4.

Table 2: Result of physical characteristics and antioxidant test.

| Test | Formula | | | | |
|---|---|---|---|---|---|
| | I | II | III | IV | V |
| Microcapsule yield (%) | 28,15 ± 0,49 | 34,20 ± 1,38 | 53,48 ± 1,66 | 59,07 ± 1,47 | 40,05 ± 1.92 |
| Moisture content (%) | 1,32 ± 0,12 | 1,34 ± 0,06 | 1,67 ± 0,13 | 1,87 ± 0,04 | 2,06 ± 0,13 |
| Solubility (%) | 97,97 ± 0,52 | 97,16 ± 0,47 | 95,90 ± 0,35 | 95,84 ± 0,43 | 95,17 ± 0,22 |
| Flow rate (g/s) | 4,46 ± 0,26 | 4,00 ± 0,13 | 3,89 ± 0,19 | 3,90 ± 0,25 | 3,89 ± 0,19 |
| Antioxidant activity (ppm) | 919,98 ± 80,48 | 886,87 ± 56,88 | 703,37 ± 23,70 | 981,78 ± 96,21 | 919,88 ± 12,54 |

Note: The result above were the mean of four replications

$$\text{Percentage of inhibition} = \frac{\text{Negative control absorbancy} - \text{Sample absorbancy}}{\text{Negative control absorbancy}} \times 100\% \quad \text{.....(4)}$$

*Scanning Electron Microscopy*

The optimized microcapsules was analyzed using SEM to examine particle size of the resulting microcapsules. The analysis was done by attaching the microcapsules to the holder using *dotile* then inputting the microcapsules into the vacuum evaporator. At certain degree of vacuum, the holder was set ablaze to make the gold vapor coat the tested material. The holder then was put into the instrument and the microcapsules were examined.

## RESULTS AND DISCUSSION

The concentration range of maltodextrin and Arabic gum which can be used as encapsulant are were v from the total mixture. The ratio of aqueous fraction to the encapsulants was 1:10. The microencapsulation results can be seen at Figure 1. The result of physical characteristics and antioxidant test can be seen at table 2.

*Microcapsule yield* was within the range of 28,15 ± 0,49 % to 59,07 ± 1,47%. Freeze-drying works by freezing the material and reducing the pressure around it and adding sufficient heat to enable the direct sublimation of ice[4]. therefore there are no other material other than water undergoes sublimation, even if the yield is less than 100%.

*Moisture content tests* were done to find the moisture content of the microcapsules. High moisture content result in damages of microcapsules and this damage influences the stabili of microcapsules themselves. The moisture content of the aqueous fraction of *Clinacanthus nutans* leaf extracts microcapsules were found to be relatively low, within the range of 1.32 ± 0.12% to. 2,06 ± 0,13%. This low moisture content also can improve the flow rate of the granules. According to *Design Expert version 10.0 Trial* analysis results, it was shown that maltodextrin (M) and Arabic gum (G) each contributes significantly to the moisture content. On the other hand, the M-G interaction affected significantly in lowering moisture content. It was proven from the prob>F value smaller than 0.05 (>0.0001) according to Equation 6. The addition of Arabic gum can increase moisture content because Arabic gum contains a larger amount of shorter hydrophilic groups, therefore it easily binds water molecules in the air. (Mahdavi, et.al., 2016).

Y = 1,28 (M) + 2,08 (G) – 0,22 (MG) .....(5)

*Solubility test* was done to find the capability of microcapsules to dissolve in water. The number of solubility value of the aqueous fraction of *Clinacanthus nutans* leaf extracts microcapsules was within the range of 95.17 ± 0.22 % to 97.97 ± 0,52%. The solubility was affected by the moisture content, in which the low moisture content enable the microcapsule to disperse easily in water. According to the analysis results of *Design Expert version 10.0 Trial*, maltodextrin (M), Arabic gum (G), and MG interaction give significant effect with the prob>F value smaller than 0.05 (<0.0001) according to equation 7.

Y = 98.00( M) + 95,24(G) – 1,69( MG) .....(6)

According to equation 7, it can be seen that the solubility will increase along the increase of the amount of maltodextrin dan Arabic gum. It was shown by the positive constants value. It can be seen from the equation that the addition of maltodextrin affected the increase of solubility since maltodextrin itself has high solubility[9].

*Flow Rate Test* was done to find the capability of certain amount of material to flow in certain time. High flow rate indicates that the material has good capability of flowing. Free-flowing microcapsules are highly desired because it makes measurement easier and ensures the homogeneity of composition and weight is all packages. The average flow rate of the resulting microcapsules was 4.03 g/s. A preparation can be said having good flow rate if it's flow rate is less than ten seconds[11]. According to analysis results of *Design Expert version 10.0 Trial,* maltodextrin (M), Arabic gum (G), and MG interaction give significant effects , with prob>F less than 0.05 (<0.0011) according to Equation 8.

Y = 4,42 (M) + 3,92 (G) – 1,15 (MG) .....(7)

According to Equation 8, the value of flow rate wilin increase along the increase the maltodextrin because maltodextrin is a free-flowing material[9].

*Antioxidant activity test*. The test was done quantitatively using DPPH method. The principle of this method is the measurement of the inhibition synthetic free radicals DPPH in polar organic solvents such as methanol by donating a hydrogen atom to stabilize purple DPPH radical to become yellow DPPH-H[7]. The result then was compared to the antioxidant activity of the aqueous fraction of *Clinacanthus nutans* leaf extract. Accorning to the test, the $EC_{50}$ value of the microcapsules was within the range of 703.37 ± 23.70 to 919.98 ± 80.48 ppm while the antioxidant activity of aqueous fraction of *Clinacanthus nutans* leaf extract was 532.24 ppm[8]. This reduction of

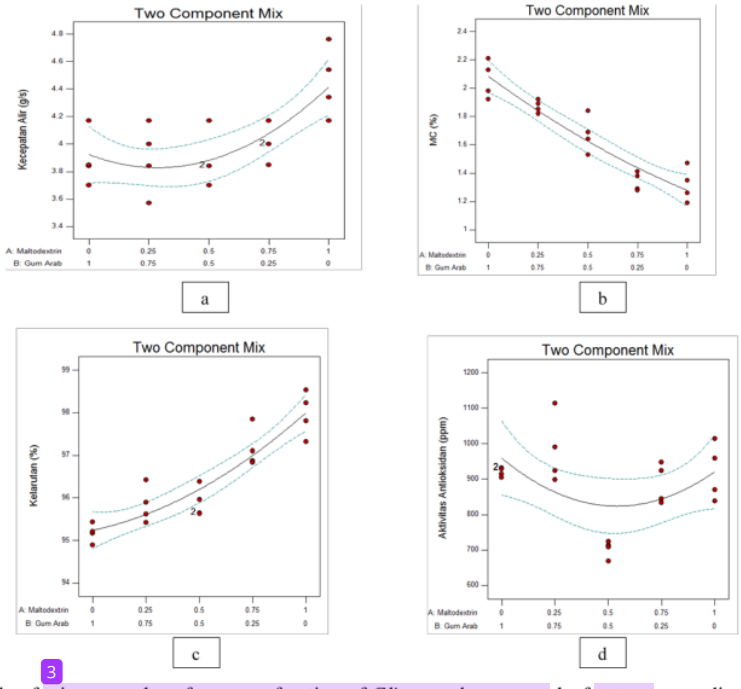



Figure 2: Profile of microcapsules of aqueous fraction of *Clinacanthus nutans* leaf extract according to the Simplex Lattice Design with (a) flow rate (b) moisture content (MC) (c) solubility (d) antioxidant activity.

**Constraints**

| Name | Goal | Lower Limit | Upper Limit | Lower Weight | Upper Weight | Importance |
|---|---|---|---|---|---|---|
| A:Maltodextrin | is in range | 0 | 1 | 1 | 1 | 3 |
| B:Gum Arab | is in range | 0 | 1 | 1 | 1 | 3 |
| Kecepatan Alir | maximize | 3.57 | 4.76 | 0.1 | 1 | 3 |
| MC | minimize | 1.19 | 2.21 | 0.1 | 1 | 4 |
| Kelarutan | maximize | 94.89 | 98.53 | 0.1 | 1 | 5 |
| Aktivitas Antio | is target = 669 | 669.22 | 1114.26 | 0.1 | 1 | 5 |

**Solutions**

| Number | Maltodextrin | Gum Arab | Kecepatan Al | MC | Kelarutan | Aktivitas Anti | Desirability | |
|---|---|---|---|---|---|---|---|---|
| 1 | 0.806 | 0.194 | 4.141 | 1.399 | 97.203 | 856.375 | 0.786 | Selected |

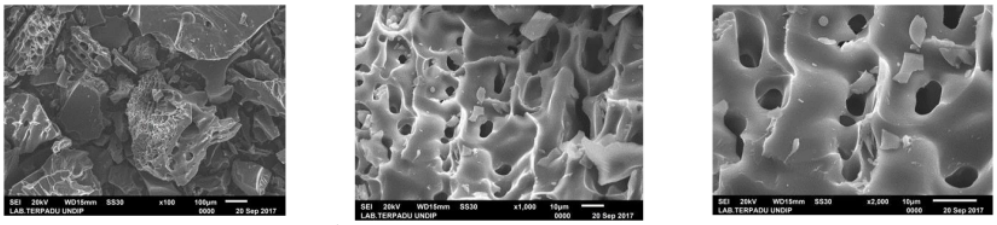
Figure 3: Formula optimum *Design Experts*.

Figure 4: The Optimized microcapsules

antioxidant activity probably caused by the possibility of not all the fraction had been encapsulated. Although there was a reduction of antioxidant activity, the microcapsule can give protection to the aqueous fraction of *Clinacanthus nutans* leaf extract during the storage begause the active components are coated so their contact with air and light is reduced. According to the analysis result of *Design Expert version 10.0 Trial*, neither maltodextrin nor Arabic gum did not give significant effect to the antioxidant activity with the prob>F value larger than 0.05 (0.1331) which

Table 3: The experiment result compared to the theoretical value.

| Response | Actual value | Prediction value | Significance | Conclusion |
|---|---|---|---|---|
| Flow rate | 4.375 g/s | 4,141 g/s | 0,088 | Did not differ significantly |
| Moisture content | 1.495 % | 1,39 % | 0,007 | Differed significantly |
| Solubility | 97.47 % | 97,203 % | 0,572 | Did not differ significantly |
| $EC_{50}$ | 842.499 ppm | 856,371 ppm | 0,010 | Differed significantly |



a b c

Figure 5: The micrograph of aqueous fraction of *Clinacanthus nutans* leaf extract microcapsules (a) 100x magnification (b) 1000x magnification (c) 2000x magnification

means it was not significant. The relating equation can be seen at Equation 9

Y = 921.19 (M) + 959.08 (G) – 462.09 (MG) ..... (8)

The evaluation profile of the aqueous fraction of *Clinacanthus nutans* leaf extract microcapsules according to the Simplex Lattice Design can be seen at Figure 2.

The optimization process was done using *Design Expert version 10.0 Trial*. Then it would process all of the response variable according to the chosen criteria and give the solution formula as the chosen encapsulant formula. The formula which was chosen was the formula with desirability value namely the formula with 0.806 parts maltodextrin, 0.194 part Arabic gum. The formula had 4.141 g/s flow rate, 1.399 % moisture content, 97.203% solubility, and $EC_{50}$ 856.375 ppm. The resulting optimum formula can be seen at Figure 3.

The resulting optimum formula then was validated to find whether the theoretical formula equation created by *Design Expert* would match the result of the experimental one. The theoretical result was compared to the experimental one with *one sample T-test* to find the validity of the optimization equation. The optimized formula for the microcapsules can be seen at Figure 4.

When the experiment results of each test parameters were compared to the theoretical ones, it was found that in the parameter of flow rate and solubility, it had significance larger than 0.05. It was then concluded that it did not differ significantly and the optimization equation was valid and could be used to predict the results.

SEM analysis then was done to the optimized microcapsules. This analysis intended to find the shape and the morphology of the microcapsules. The shapes and morphology can be seen at Figure 5. The microcapsules were not spherical, their surface was uneven, and they were porous. These shape was caused by the mixing and grinding process which could break the microcapsules to smaller particles with unspherical shapes. The uneven surface was caused by the imperfect process of polymer crosslinking.

## CONCLUSION

Based on the experiment result, it could be concluded that aqueous fraction of *Clinacanthus nutans* leaf extract microcapsules could be made with the ratio of 0.804 part maltodextrin and 0.194 part Arabic gum. The resulting microcapsules had 1.49% moisture content; 97.47 % solubility; 4,375 g/s solubility; 842.499 ppm antioxidant activity.


## ACKNOWLEDGEMENT

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