RESEARCH ARTICLE

Development of Face Pack Powder of Arabica Coffee (*Coffea arabica L.*) Using Variations of Binders with D-Optimal Mixture Design

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ABSTRACT

Cosmetics like face pack powder are popular preparations used by men and women. Hydroxypropyl methylcellulose (HPMC), sodium carboxymethyl cellulose (CMC-Na) and carbopol are commonly used binders and are suited to dispersal by water. Many natural ingredients such as arabica coffee (*Coffea arabica L.*) are useful for cosmetics. Chlorogenic acid and α-tocopherol is a bioactive compound for antioxidant activity and is important in its application in face pack powder. This study aimed to determine the ratio comparison of the composition of the three polymers used as the binders for preparations of the coffee face pack powder using a D-optimal mixture design, analyze the evaluation results of the optimum face pack powder formula, and determine its antioxidant activity. The study began with coffee extraction with 70% ethanol as solvent, wet granulation process with binders and other excipients, evaluation of organoleptic properties, pH, and water content, stability observation for 28 days, and *in-vitro* measurement of antioxidant activity. One-sample t-test was applied to analyze the results. The obtained optimum formula was with the ratio of HPMC (8.43): carbopol (1.16): CMC-Na (5.41). The evaluation results of the optimum formula showed that the face pack powder has ash brown color, weak aromatic typical coffee fragrance, homogeneous texture, pH value of 5.93 ± 0.06, and water content of 0.89 ± 0.15%. The resulting study shows the coffee face pack powder and crude extract have antioxidant activity (34.94 ppm ± 0.75 and 15.78 ppm ± 2.53). The analysis with the one-sample t-test showed that the experimental results were not significantly different from the software predictions, so the Design-Expert® software using the D-optimal mixture design is applicable to predict the optimum formula of coffee face pack powder.

Keywords: Face pack powder, *Coffea arabica*, D-optimal mixture design.

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INTRODUCTION

Cosmetics are popular preparations that are used by both men and women.[1] Cosmetics are defined as a preparation applied on the skin surface to protect or maintain skin conditions.[2] Face masks, commonly called face pack, is one of the widely used cosmetics. Face pack is a form of cosmetic commonly used for skin care such as removing dead skin cells. Face pack powder can make skin younger, and clean and reduce oil content.[4] Face pack powder or powder masks are examples of commonly used face masks.[3] Face packs, especially face pack powder, are continuously developed by the industry because of their fairly long shelf life,[5] and this causes the need for optimization of the face pack powder formula to become a qualified preparation. The formulation of the mask consists of active substances, fillers, binders, and wetting agents.[6]

A commonly used filler in mask preparations is rice starch. It contains amylose and kojic acid which can lighten the skin.[7] The binder is a component used in the manufacture of pharmaceutical preparations. Wet binders are generally used in the wet granulation method.[8] The wet granulation method can be applied in powder mask manufacture.[6] The wet granulation method uses a water-soluble binder, so it is suitable for preparations that are easily washed in water.[8] Hydroxypropyl methylcellulose (HPMC), sodium carboxymethyl cellulose (CMC-Na) and carbopol are commonly used binders and are suited to dispersal by water. The suitable compositions of HPMC, carbopol, and CMC-Na for binders are in the range of 2 to 5%, 0.75 to 3%, and 1 to 6%, respectively.[9] The use of each of these binders at a concentration of 15% results in a mask powder that is qualified for the preparation evaluation. The use of these materials as a single binder has a weakness, thus should be combined. HPMC produces the lowest dispersion among carbopol and CMC-Na. Carbopol has the highest humidity of the others when used as a dry preparation so that

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it makes microorganisms grow more easily. CMC-Na has the lowest water absorption of the other polymers, thus reducing its ability to be paste.[6] The combination of HPMC, carbopol and CMC-Na can be synergistic so as to improve the physical properties of the preparation.[10] Many natural ingredients such as arabica coffee (*Coffea arabica* L.) are useful for cosmetics. Coffee contains bioactive compounds which has therapeutic activity.[11-13] Green arabica coffee beans have a 44.27 0.01% face brightening activity[14] and an IC_{50} value of 0.050, 0.01 mg/mL, which inhibits L-Tyrosinase. A study on the ethanolic extract of arabica coffee beans, which has highly potent antioxidants with an IC_{50} value of 12.427 ppm,[15] lends credence to this. Numerous phytochemicals found in coffee have antioxidant activity, which includes the capacity to neutralize free radicals, donate electrons and hydrogen, promote reduction activity, and function as pro-oxidant metal ion chelates. Coffee additives can promote the production of tissue antioxidant genes and defend against gastrointestinal oxidative stress.[16]

Based on the description above, it is necessary to conduct a study aiming to obtain the optimum formulation of a mask in the form of a coffee powder face pack. This optimization was done by combining three types of binders to produce qualified face-pack powder preparations. In addition, the study was carried out to obtain the best coffee powder face pack formula to be combined with active ingredients so that it is useful for the development of pharmaceutical science, particularly pharmaceutical technology. The scope of this research topic was pharmacy with cosmetic products developed from natural ingredients, and it aimed to determine the ratios of the composition of carbopol, HPMC, and CMC-Na as the binders for the preparations of the coffee face pack powder using the D-optimal mixture design and analyze the evaluation results of the optimum preparation formula, stability test and its antioxidant activity. The novelty of this research is that this study used the active compounds of arabica coffee from an area in West Kalimantan, and it was formulated in face pack powder with an optimal formula.

MATERIAL AND METHODS

Materials

The instruments used in this study are a, micropipette Socorex® (0,5 – 10; 5–50; 50–200, 200–1000 μL), Eppendorf tube, rotary evaporator (Heldolph tipe Hei-VAP), oven (Memmert), water bath (Memmert tipe WNB14), sieve size 120 mesh, aluminum foil, thermometer, vaporizer cup, *Arabica* coffee beans (Nanga Pinoh, Melawi, West Kalimantan), ethanol 70% (Dwicentra), aquadest (Dwicentra), HPMC (Kimia Jaya Labora), Carbopol (Kimia Jaya Labora), amylum oryzae (Anak Raja), CMC-Na (Kimia Jaya Labora), parfum.

Methods

Preparation of arabica coffee extraction

A total of 322 g of roasted and ground coffee was macerated with 1 L of 70% ethanol for 3x24 hours. Then, every 3x stirring in 24 hours. The maceration results were collected. At a temperature of less than 60°C, the products of the maceration are filtered and evaporated.[17]

Preparation face pack powder formulation

Utilizing Design Expert software and the D-Optimal Mixture Design approach, formulas are created. With the lowest and maximum values, a sample set of data will be optimized. An amalgam of HPMC, carbopol, and CMC-Na was used in this study's optimal sample, which had a minimum limit of 1 and a maximum of 13. There were fifteen mixes altogether.

The responses used to determine the optimum formula were pH, moisture content, and dry time tests. Obtained 16 runs which is the optimum composition ratio. The run data obtained is a comparison of HPMC: Karbopol: CMC Na with a ratio of 4.98:5.17:4.85; 1:13:1; 7.07:3.24:4.69; 7.51:1:6.49; 1:1:13; 12.99:1.01:1; 9.02:3.48:2.5; 6.48:7.52:1; 2.67:9.18;3.15; 1:1:13; 1:7.09:6.91; 12.99:1.01:1; 6.48:7.52:1; 3.34:2.29:8.75; 1:13:1; 1:7.09:6.91. In addition to a variety of binders, the coffee mask formula also involves other ingredients, including rice starch, coffee extract, and aquadest. Making coffee face pack powder begins with making mucilago. The powder material is mixed with the binder until it is homogeneous. Then a felt mass is made, followed by wet sifting and drying. After drying, dry sieving is carried out.[6]

Observation of physical evaluation of face pack powder

The optimum formula that has been made is then carried out a physical evaluation to determine its physical properties. The test parameters for the optimum formula are:

Organoleptic evaluation

Organoleptic tests were carried out by observing the texture, color and smell of face pack powder preparations with a combination of CMC-Na, HPMC, and carbopol binders.[6]

- *pH evaluation*

By making a paste consisting of 1 gram of the preparation and water, the pH test was conducted. This preparation typically has a pH between 4.5 and 6.518, which is in line with the pH of skin.[18]

- *Water content evaluation*

The moisture balance method was used to test the powder's moisture content. In order to use this technique, weigh 1 gram of the preparation before placing it into measuring equipment. Observe until there is %water content on the tool. A good water content is less than 10%.[19]

- *Antioxidant activity evaluation*

The stages of testing are as follows:[20]

Preparation 0.05 mM DPPH solution.

Place 0.0098 grams of DPPH powder in a 250 mL measuring flask and gradually dissolve in 250 mL (0.1 mM) of methanol.

Preparation of coffee extract measurements

To get a stock solution containing 100 ppm, weigh 0.0025 gram of arabica coffee ethanol extract that has been dissolved in 25 mL of methanol. Then, concentration series of 3, 6, 9, 12, and 15 ppm were generated from the mother liquor.

Determination of the maximum wavelength

By measuring the absorbance of 0.05 mM DPPH up to 4 mL using a spectrophotometer with a wavelength of 500 to 525 nm to get an absorbance of 0.2 to 0.8, the maximum wavelength may be determined. The maximum wavelength is determined by the absorbance that is greatest.

Antioxidant activity test

Weigh 1 g of face pack powder and put it in an Erlenmeyer, then dissolve it in methanol p.a until the volume becomes 25 mL, heat it over a water bath until the face pack powder becomes homogeneous, then cool it in ice cubes. Do this 3 times into a test tube, then centrifuge at 3000 rpm for 10 minutes, filter and let stand. In 1-mL of sample (with a concentration of 10, 20, 30, 40, 50 ppm) was added to 2 mL of 0.1 mM DPPH and kept in a dark place for 30 minutes. Read the absorbance using a UV-vis spectrophotometer at a wavelength of 516.65 nm. The same treatment was also carried out for the blank solution (DPPH solution which did not contain the test material). The blank solution consisted of 2 mL of 0.1 mM DPPH and 1-mL of methanol p.a.

Data Analysis

Analysis of free radical scavenging activity with DPPH.

Data from absorbance measurement results were analyzed for the percentage of antioxidant activity in the inhibition percentage value (%inhibition) using equation 1:

$$\%\ \text{Inhibition} = \frac{A_{blanko} - A_{sampel}}{A_{blanko}} \times 100\% \quad \ldots\ldots\ldots(1)$$

Next, the IC_{50} value is calculated, which is a value that describes the concentration of the test fraction that can capture free radicals by 50% through the equation of a linear regression line which states the relationship between the concentration of the compound (sample) tested (x) and the percent activity of the average radical scavenger. (y).

RESULT AND DISCUSSION

pH and Water Content

pH is one of the criteria that must be met as it relates to the safety of preparations, especially face pack powder, when skin-applied. It is important to do a pH test to ensure that the preparation is safe and comfortable when applied on the skin. The ideal topical preparation pH is 4.5 to 6.5. If the preparation is too acidic, it is feared that irritation will occur, and if it is too basic, it is feared that dry skin occurs.[18]

The pH test results indicate (Table 1), of the 16 runs, the permissible pH values for the face pack powder preparations is in the range of 4.5 to 6.5, except for run 1: HPMC (1): carbopol (7.09): CMC-Na (6.91) and run 16: HPMC (1): carbopol (1): CMC-Na (13). The difference in pH at each run indicates the effect of the binder combinations on the preparation pH. Based on the results of the highest pH, the high use of CMC-Na can increase the preparation pH. Table 2 shows the analysis using Design-Expert® software.

The linear model in the equation is a valid and suitable model for the pH based on the ANOVA test, This shows that the experimental data and the data that the proposed model predicts do not differ noticeably. Figure 1 shows the contour plot of the pH responses.

Table 1: Response to pH and water content value at 16 runs

Run	*HPMC (%)*	*Carbopol (%)*	*CMC Na (%)*	*pH*	*Water Content (%)*
1	1	7.09	6.91	6.8	1.04
2	4.98	5.17	4.85	5.5	1.03
3	12.99	1.01	1	5.6	2.14
4	1	1	13	6.4	0.9
5	3.34	2.90	8.75	5.9	0.57
6	1	13	1	4.8	0.79
7	7.51	1	6.49	5.9	0.76
8	7.07	3.24	4.69	6	0.93
9	2.67	9.18	3.15	5.6	1.55
10	1	7.09	6.91	6	1.9
11	6.48	7.52	1	5.2	2.46
12	12.99	1.01	1	5.8	2.94
13	9.02	3.48	2.5	5.9	2.16
14	6.48	7.52	1	5.5	1.19
15	1	13	1	5.3	1.21
16	1	1	13	6.7	1.01

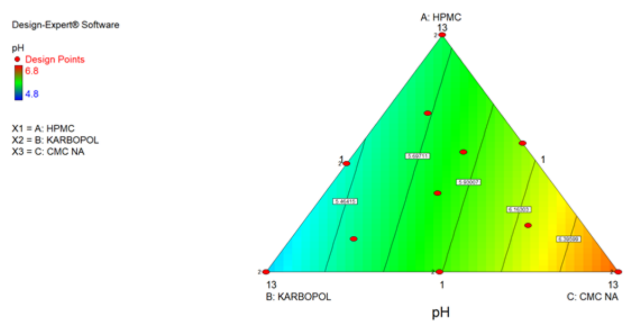

Figure 1: Contour plot pH response

The D-optimal mixture design equation obtained can be seen in equation 1.

Y = 0.37 (A) + 0.34 (B) + 0.46 (C) ……………… (1)

Note:

Y = pH

X1 = A: HPMC

X2 = B: Carbopol

X3 = C: CMC Na

It is known that the pH value is decided by each optimized component based on the pH, the data from the contour plot, and the equation. The size of the coefficient in the equation shows that CMC-Na has the biggest impact. The pH value is more affected by HPMC than by carbopol. Figure 1 shows that the greater the CMC-Na composition in the face pack powder preparation, the higher the pH of the preparation. The orange-colored area is an area with a higher pH response. The large

CMC-Na composition in the figure shows the more basic pH result (6.39), and the blue area produces a lower pH. According to Ismail (2014), the pH values resulting by carbopol, HPMC, and CMC-Na are 6- 6.5, respectively so that it fits the skin pH range for topical preparations. In this study, the water content test was carried out to determine the percent water content in each produced preparation. The water content in the face pack powder greatly affects the preparation quality. The higher the water content of the preparation, the greater the opportunity for the growth of microorganisms in it.[19] The permissible water content in the face pack powder is not more than 10%.[21] Table 1 shows the results of the water content test, indicating that of the 16 runs, the permissible water content allowed for face pack powder is less than 10%. The difference in water content at each run indicates the effect of the binder combinations on the water content of the preparations. The range of water content values is 0.76 to 2.94%. The highest water content value was seen at run 12 with HPMC (12.99): carbopol (1.01): CMC-Na (1). The high use of HPMC could increase the water content of the preparations, but it still meets the requirements of the permissible water content.

The quadratic model in the equation is a valid and suitable model for the water content based on the ANOVA test. This demonstrates that there is no discernible difference between experimental data and data that the suggested model predicts. The quadratic model's *p-value* is 0.05, indicating a significant variation in the water content due to the employment of several binders with various compositions. The contour plot of the water content responses is shown in Figure 2.

The D-optimal mixture design equation obtained can be seen in equation 2.

Y = 0.22 (A) + 0.04 (B) + 0.07 (C)+ 3.31 (A)(B) – 0.036 (A)(C) + 0.012 (B)(C)…………………………………(2)

Note:

Y = Water Content

X1 = A: HPMC

X2 = B: Carbopol

X3 = C: CMC Na

The coefficient values of HPMC, carbopol, and CMC-Na; the interaction of HPMC with carbopol; and the interaction of carbopol and CMC-Na are positive, meaning that each component and the combination of those components can increase the water content responses. The HPMC coefficient is greater than the CMC-Na coefficient, and the carbopol coefficient is smaller than the CMC-Na coefficient. The interaction of HPMC and carbopol and the interaction of carbopol and CMC-Na result in a positive coefficient, meaning that the interactions have increased the water content values. The interaction of HPMC and CMC-Na has decreased the water content value. The interaction of carbopol and CMC-Na has increased the water content value, but the effect is not greater than the interaction of HPMC and carbopol. The coefficient of each component is positive, but it is smaller than the HPMC - carbopol coefficient.

In dry preparation such as powders, a high and low water content can affect the quality of the preparation. The higher the water content of the preparation, the easier microorganisms overgrow in it, causing shorter shelf life of the preparation. On the other hand, the lower the water content of the preparation, the more difficult for microorganisms to grow, resulting in a longer shelf life of the preparation.[19] Using Figure 2, the contour plot of the water content of the face pack powder, the colored area explains the occurring component interactions that can affect the water content of the preparations. When the addition of the material increases the water content, the color shown leads to an orange color with a value of 2.23; on the contrary, when the water content decreases, it leads to a blue color with a value of 0.72. Based on the color parameters contained in the contour plot, it is known that the combination of HPMC and carbopol shows an orange color. This indicates an increase in the water content of the preparations. Likewise, the addition of carbopol and CMC-Na has increased the water content of the preparations. Nevertheless, despite the high composition of carbopol, the water content of the preparations gets lower. The addition of carbopol, the combination of carbopol and CMC-Na, and the combination of carbopol and high CMC-Na can provide the best water content for powder preparations.

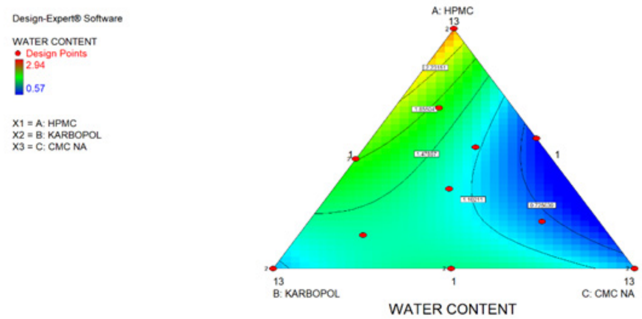

Figure 2: Contour plot water content response

Optimum Formula of Face Pack Powder

The ratio of HPMC (8.43), carbopol (1.16), and CMC-Na (5.41) with a desirability value of 0.842 makes up the best formula discovered using the D-optimal mixture design. If the value is close to one, the chosen response variables for formula optimization can reach the optimal point in accordance with the desired goal. The formulation of the face pack powder has the most HPMC, according to the findings of analyzing the compositions of the binders utilized. Figure 3 displays the contour plot of the ideal formula for the preparations, and displays the desirability value of the binder compositions.

The optimum formula was generated with Design-Expert® version 7.0 using the D-optimal mixture design. The resulting contour plot is superimposed on the responses of pH and water content (Figure 4). It gives a yellow area that provides the optimum responses. The best formula prediction is made in this area, with a desirability of 0.842.

Verification of the Optimum Formula of Face Pack Powder

Verification of the optimum formula was done by comparing the optimum face pack powder experimental results with the software prediction results. Table 2 shows the verification

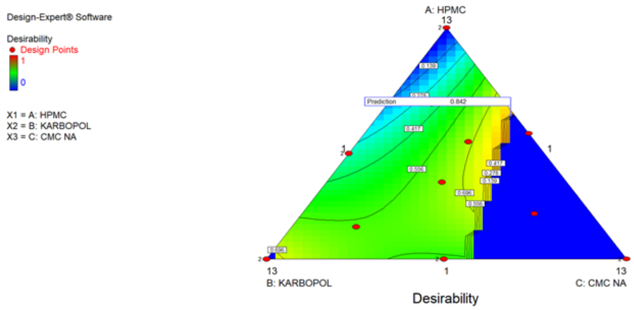

Figure 3: Contour plot desirability

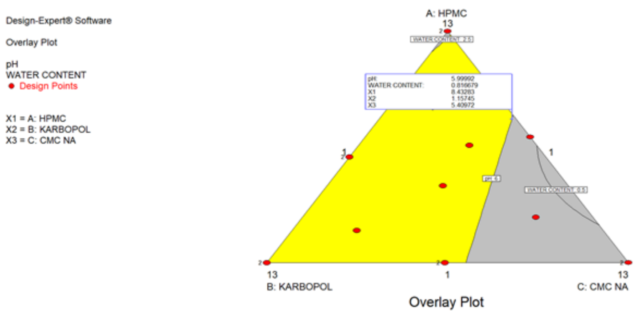

Figure 4: Superimposed optimal formula

Table 2: Face pack powder optimal evaluation

Sample	*pH*	*Water content (%)*
Face pack powder optimal prediction from software	5.99	0.82
Face pack powder optimal test	5.93 ± 0.06	0.89 ± 0.15

(Figure 5).

The resulting *p-value* is larger than 0.05 based on the probability value of each response. This explains why there is no discernible difference between the outcomes of the experimental observations and those anticipated by the program. Comparing the findings of the face pack powder observations with those of the projected responses generated by the D-optimal mixture design's optimal formula. The OpenStat software's one-sample t-test was then used to do the verification. The one-sample t-test was used to assess the data. The calculated *p-value* for the pH test parameters is 0.321 > 0.05. Based on the probability value of each response, the resultant *p-value* is more than 0.05. This explains why the results of the experimental observations and those predicted by the program cannot be distinguished from one another. comparing the results of the observations of the face pack powder with the expected responses produced by the D-optimal mixture design's optimal formula. The verification was then performed using a one-sample t-test in the OpenStat program. The one-sample t-test was used to assess the data. The Design-Expert software's prediction results with the D-optimal mixture design and the experimental findings on the ideal Face pack do not differ in the pH test parameters, as indicated by the resulting *p-value* of 0.321 > 0.05, demonstrating that there is no difference between the trial findings on the ideal face pack powder formula and the forecast results of the Design-Expert program with the D-optimal mixture design. The water content test yields a *p-value* of 0.461 > 0.05, indicating that there is no difference between the two groups. The software prediction and the experimental results. The pH of the optimum preparation is still in the pH range (4.5–6.5) for face pack powder preparations or powder masks that were acceptable to human skin. The pH of the preparations must meet the requirements, either not too acidic or too basic, in order to avoid skin irritation which can cause dryness, itching and redness. The pH test outcomes pH value 5,93 ± 0,06 it indicates that the pH of the optimum face pack powder formula is within the range required for topical preparations (Figure 6).

The water content of the preparations has an effect on the oven temperature when drying them. The preparation composition during the drying process allows for differences in the water shrinkage time. A good water content of the face pack powder mask preparations is less than 10%.[17] The less the water content of the powder preparations, the more difficult microorganisms to grow.[19] Table 2 displays the outcomes of the water content test of the optimum preparation formula and shows that the water content is in the range required for face pack powder preparations (Figure 7).

Stability Evaluation of Coffee Face Pack Powder

The real-time stability testing of the coffee mask optimal formula includes an organoleptic test, pH test, and water content test. In organoleptic testing, the preparation is stable: it has an ash brown color, common coffee aroma, and fine powder texture before stored until the 31 storage day. Based on the stability testing on the pH and the water content, significant changes do not occur from day 0 to day 31 of the storage. On the pH test, the results of statistical analysis with The results of the ANOVA reveal a *p-value* of 0.57 > 0.05. This explains why there was no discernible change in pH levels between day 0 and day 31 of storage. The findings of the statistical analysis using the ANOVA method on the water content test reveal a *p-value* of 0.57 > 0.05, suggesting that there is no significant variation in the pH values from day 0 to day 31 of storage.

Antioxidant Activity Face Pack Powder Optimal Formula and Ethanol Extract *C. arabica*

The results of the UV-vis spectra on the test show a wavelength of 528.25 nm which is included in the visible light wavelength range. The maximum wavelength of 0.1 mM DPPH is 517 nm.[21] The time used for the interaction between the test sample and

Figure 5: *Face pack powder of C. arabica* optimal formula

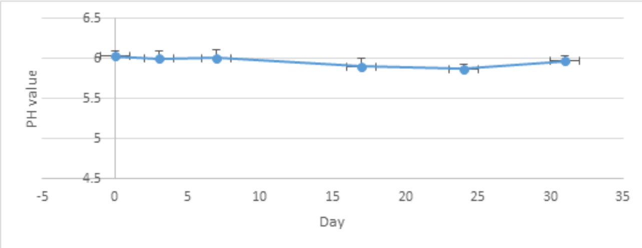

Figure 6: Face pack powder optimal formula pH stability evaluation

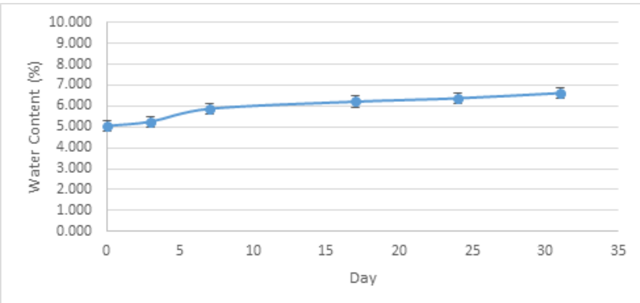

Figure 7: Face pack powder optimal formula water content stability evaluation

Table 3: Antioxidant activity with DPPH method (mean ± SD; n=3)

Sample	*IC_{50} DPPH Assay*
Face pack powder of *C. arabica* optimum formula	34.94 ± 0.75
Ethanol extract *C. arabica*	15.78 ± 2.53

DPPH in this study was 30 minutes. It is also visually visible with a purple discoloration that is fading and becoming slightly yellowish after a 30-minute incubation time. This color change occurs due to the presence of a compound in the sample that donates a hydrogen atom to the DPPH radical so that it is reduced to a more stable form, namely 1, 1-diphenyl-2-picrylhydrazine. Based on several studies, the reaction time is 30 minutes, and it has been investigated.[22]

According to the study's findings, face pack powder of *C. arabica* has an extremely high antioxidant activity level of 34.94 g/mL 0.75, which is less than 50 g/mL. If the IC_{50} value of a substance is less than 50 g/mL, it is classified as a very strong antioxidant; if it is between 50 and 100 g/mL, it is classified as a strong antioxidant (Table 3). The strength of the antioxidant action increases with decreasing IC_{50} values.[23] Because they feature a hydroxyl group that may release protons in the form of hydrogen ions, flavonoid compounds function as antioxidants. The radical electrons in the nitrogen atom in the DPPH molecule attach to hydrogen ions and generate the reduced hydrogen ion since the hydrogen ion only has one proton and no electrons decreased DPPH. Radicals in the DPPH can be reduced by interacting with hydrogen donors found in phenolic substances.[24]

CONCLUSION

To produce face pack powder of *C. arabica* optimal formula using the D-optimal mixture design. The ratio comparison of the HPMC, carbopol, and CMC-Na used as the binders is 8.43: 1.16: 5.41.

The evaluation results of the optimum formula of the coffee face pack powder have shown organoleptic properties: ash brown color, weak aromatic typical coffee fragrance, homogeneous texture; pH value of 5.93 ± 0.06; and the water content of 0.89 ± 0.15%.

Face pack powder of *C. arabica* optimum formula has very strong antioxidant activity with IC_{50} value of 34.94 µg/mL ± 0.75.

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