

EVALUATION OF GEL STABILITY FROM *Coffea arabica* L. GROUNDS NANOPARTICLES

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ABSTRACT

In this study an evaluation of the stability of the pH and spreadability of the Arabica coffee nanoparticle gel preparation for 30 days at room temperature with an interval of 10 days. The method used to see the effect of factors on pH and spreadability on the 30th day is a Box-Behnken Design experimental design with 3 factors (carbopol 940, triethanolamine and nanoparticles) and 3 levels (low, medium and high). The results of the analysis showed that pH was influenced by the addition of triethanolamine and carbopol 940 and the spread was influenced by the addition of carbopol 940 and Arabica coffee pulp nanoparticles. Preparation of Arabica coffee pulp nanoparticle gel has a pH that is following the pH of the skin, which is 4.8 to 6.8 and has a good dispersal power, which is between 5.00 to 6.47 cm. The stability of the pH and spreadability of gel preparations during 30 days storage showed stable result: there were no significant changes.

Keywords: Nano Gel, pH, Spreadability, Box-behnken.

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INTRODUCTION

Arabica coffee grounds are residues from the processing of coffee drinks. Arabica coffee grounds contain many bioactive compounds¹, and secondary metabolite compounds such as alkaloids, flavanoids², saponins, phenolic³, caratenoid^{4,5}, chlorogenic acid⁶, oil⁷, polysaccharides⁸, as well as other active compounds.^{9,10} Arabica coffee grounds can be made into nanoparticles using nanotechnology. Arabica coffee pulp nanoparticles contain secondary metabolites that can be used as active compounds in antioxidant and anti-inflammatory gel preparations.^{11,12}

Gel or jelly is a semisolid form made of inorganic or organic suspense which is penetrated by liquid. Gel has many advantages over other semi-solid preparations because it is easy to use, practical and durable.¹³ Based on the composition and nature, the gel has several advantages including being able to penetrate deeper than the cream and is very well used in the hair area. A good gel characterization is having the same pH with skin pH which is around 4.5 to 6.5, if the gel has an acidic pH it will irritate the skin but if the gel has an alkaline pH it will cause dry skin.¹⁴ Good gel spread between 5-7 cm. The gel's dispersal power can affect the contact and spread of active compounds in the skin.¹⁵ In addition to the physicochemical properties of the gel preparation, it must also have good stability during storage.

The stability of preparation is a factor that must be considered because pharmaceutical preparations are usually produced in large quantities and take a long time to reach the user. Gel preparations that are stored for a long time can change either chemically, physically, or biologically so that it can damage the components of the preparation and will affect the dose received.¹⁶ Many factors that can affect the stability of the gel preparation include temperature, humidity, light, microorganisms, oxygen, and additional ingredients used.¹⁷ The main factors that can be used to evaluate the stability of gel

preparations are pH and spreadability. The pH of the gel preparation will affect the level of acidity, while the spreadability affects the thickness of the gel preparation.^{18,19}

Based on the description above, to produce a gel preparation that has good stability, this study evaluated the stability of the pH and spreadability of the Arabica coffee pulp nanoparticle gel preparation for 30 days at room temperature. To see the factors that affect the pH and spreadability, a Box-Behnken Design experimental model is used with 3 factors (x) and 3 levels.

EXPERIMENTAL

Materials

The tools used are laboratory equipment such as analytical balance, beaker glass, glass plates, scales, graph paper, measuring cups, volume pipettes, mortars and pastels and pH meters. The ingredients used are carbopol 940, aquadest, glycerin, methyl paraben, triethanolamine (TEA) (Pharmaceutical Grade) and Arabica coffee pulp nanoparticles (*Coffea arabica* L.).

Trial Design

The trial design uses Box-Behnken Design with 3 factors (x) and 3 levels (low, medium and high) as shown in Table-1.

Table-1. Design of Arabica Coffee Pulp Nanoparticle Gel Formulation with 3 Factors and 3 Levels.

Factor	Parameters	Levels		
		Low(-)	Medium(0)	High(+)
x_1	Carbopol 940 (%)	0.50	0.75	1.00
x_2	TEA (%)	0.40	0.50	0.60
x_3	Nanoparticles (%)	1.50	2.25	3.00

Gel Formulation

Weighed carbopol 940 according to the design then put into a beaker glass and added with 100 mL of distilled water stirred using a magnetic stirrer for 30 minutes at a temperature of 80°C. The expanded carbopol base is then put into a mortal, TEA is added according to the design, 7.5 ml of glycerin is added and stirred until smooth. Methyl paraben 0.1 g was added while stirring until homogeneous, then the Arabica coffee pulp nanoparticles were added according to the design, crushed until homogeneous and completely dispersed.

Characterization and Evaluation

The pH test is carried out utilizing weighing 10 grams of gel into the beaker glass, plus 100 ml of distilled water and stirring thoroughly, then dipping the pH meter into the solution to the mark. The gel dispersion test is carried out by weighing 0.5 g of gel preparations and then placed on a glass plate on graph paper. the glass plate is covered with another glass plate and weighed 500 g above. Evaluation of pH stability and spreadability of gel preparations was carried out for 30 days at room temperature with an interval of 10 days. On the 30th day, ANOVA results were analyzed using Box-Behnken Design.

RESULTS AND DISCUSSION

Gel Formulation Design

Design gel formulations using Box-Behnken Design on Software Design Expert Version 10.0.3.0 with 3 factors (x) and 3 levels. This results in 17 runs as shown in Table-2. The resulting gel has a semisolid (gel) shape, smells typical of coffee grounds and has a brownish-black color.

Table-2: Design of Arabica Coffee Pulp Nanoparticle Gel Formulation with 3 Factors and 3 Levels

Run	Factor 1 A: Carbopol 940 (%)	Factor 2 B: TEA (%)	Factor 3 C: Nanoparticles (%)
1	1.00	0.50	1.50
2	0.75	0.50	2.25
3	1.00	0.40	2.25

4	0.50	0.40	2.25
5	0.50	0.50	3.00
6	0.75	0.60	1.50
7	0.75	0.40	1.50
8	0.75	0.50	2.25
9	0.75	0.50	2.25
10	0.75	0.50	2.25
11	0.75	0.40	3.00
12	0.75	0.60	3.00
13	0.50	0.50	1.50
14	0.75	0.50	2.25
15	1.00	0.50	3.00
16	0.50	0.60	2.25
17	1.00	0.60	2.25

pH Stability

The gel is a topical preparation used on the skin, therefore the stability of the gel dosage was very important. During storage, the gel preparation must have a stable pH. The human coolies pH of 4.5-6.5 used to avoid irritation and dryness on the skin of gel preparations,¹⁴ the pH of the gel preparation range from 4.8 to 6.8 was the best range of skin pH. Figure-1 shows the pH results of a stable gel preparation, there was no significant pH change in all variations of the gel preparation during 30 days storage.²⁰

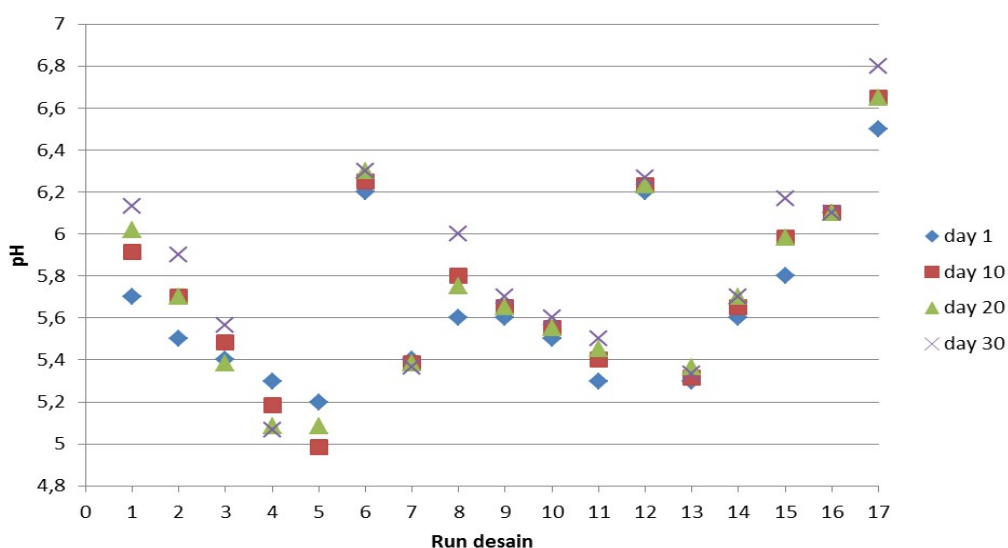


Fig.-1: Graph of the pH Stability Test Results of Arabica Coffee Pulp Nanoparticle Gel Preparation for 30 Days.

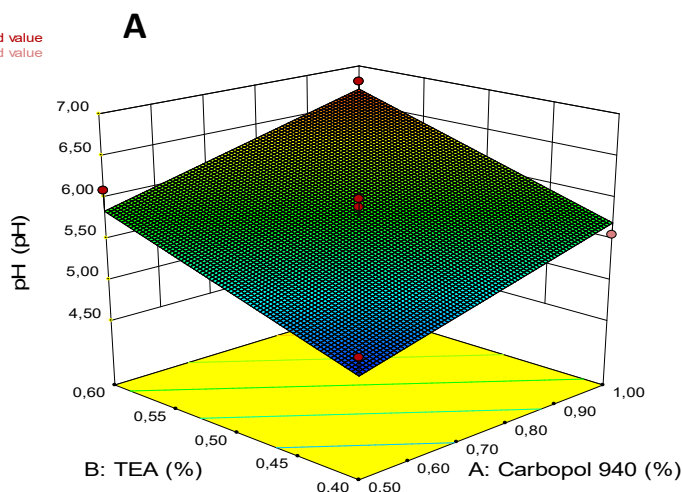
The linear design model was chosen because it has a significant effect on the pH of the gel preparation with a p-value of <0.0001 as shown in Table-3. Significant factors on pH are A and B because they have a p-value of 0.0001, respectively; and <0.0001. The insignificance of lack of fit with an F-value of 2.14 and a p-value of 24.18%, shows that the linear design model is appropriate for the analysis of pH testing data on the 30th day.

Table-3: ANOVA Analysis For Linear Models of pH of Gel Preparations on Day 30

Source	Sum of Squares	df	Mean Square	F Value	p-Value Prob > F	Characterization
Model	3.44	3	1.15	23.74	< 0.0001	significant
A-Carbopol 940	1.45	1	1.45	29.96	0.0001	
B-TEA	1.97	1	1.97	40.77	< 0.0001	

C-Nanoparticles	0.023	1	0.023	0.49	0.4977	
Residual	0.63	13	0.048			
Lack of Fit	0.52	9	0.058	2.14	0.2418	not significant
Pure Error	0.11	4	0.027			
Cor Total	4.06	16				

Design-Expert® Software
Factor Coding: Actual
pH (pH)
● Design points above predicted value
● Design points below predicted value
6,80
4,77
X1 = A: Carbopol 940
X2 = B: TEA
Actual Factor
C: Nanoparticles = 2,25



Design-Expert® Software
Factor Coding: Actual
pH -30 (pH)
● Design points above predicted value
● Design points below predicted value
6,80
4,77
X1 = C: Nanoparticles
X2 = A: Carbopol 940
Actual Factor
B: TEA = 0,50

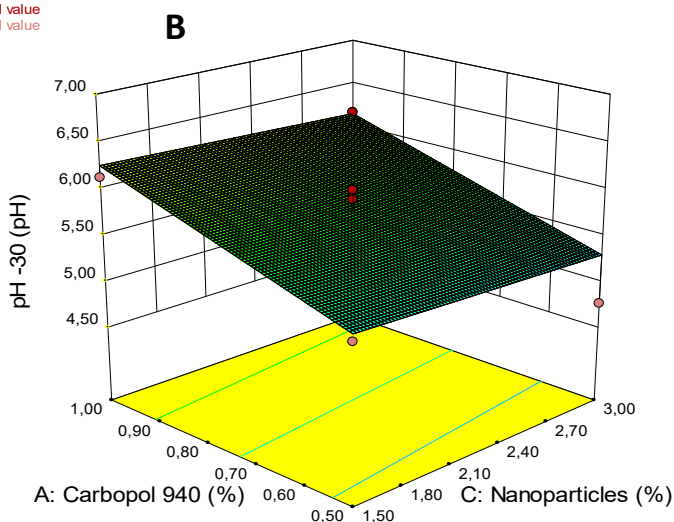


Fig.-2: Plot 3D Relationship between (A) Carbopol 940 and TEA on pH, (B) Carbopol 940 and Nanoparticles on pH, at 30 Days Storage.

The increasing concentrations of carbopol 940 and the TEA, also the pH of the gel preparation increased, it was used following the p-value of 0.0001 and <0.0001 as shown in Figure-2. At room temperature, TEA was difficult to evaporate. The relatively basic nature of TEA with a pH of 10.5 can be used as a base agent¹³ and carbopol 940 has a pH of 7.7 as a base agent. While nanoparticles have a pH of 5.33, the Arabica coffee nanoparticles gel preparation was not give a significant effect on the pH.

Stability of Spreadability

The greater the surface area of the skin, the greater the diameter of the dispersal power, it can be reached by the gel.¹⁹ The best of spreadability has a diameter of 5-7 cm^{14,15}. The results of the gel dispersion test showed a good value of 5.00-6.47 cm. As well as the stability of the spreadability of gel preparations during 30 days storage showed stable results and were still in the range of good spreadability values as shown in Figure-3.

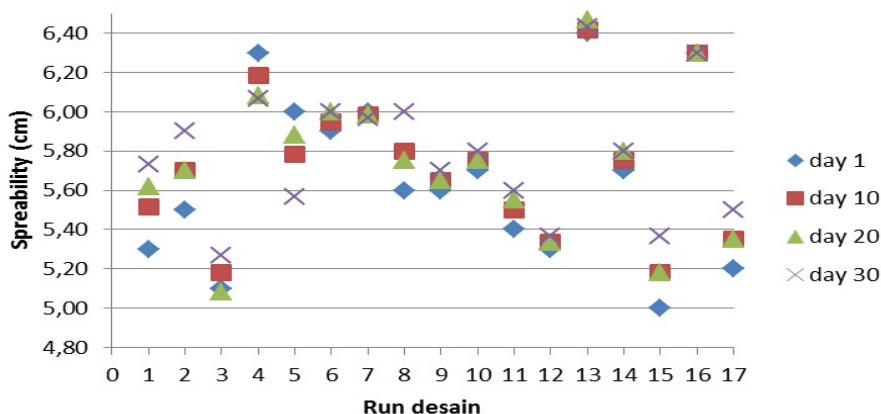


Fig.-3: Graph of the Test Results For the Stability of the Spreadability of Arabica Coffee Pulp Nanoparticle Gel Preparation For 30 Days of Storage.

The result of ANOVA analysis for the linear model of the gel dispersion was shown in Table-4. Significant factors for the spreadability are A and C with p-values of <0.0001 and 0.0001, respectively. Then, the model shows significant results with a p-value < 0.0001. The lack of fit value is not significant with an F-value of 1.94 and a p-value of 27.26%, it shows that the linear design model is appropriate for the analysis of the scattering power test data at 30 days storage.

Table 4. ANOVA Analysis For the Linear Model of the Spreadability of Gel Preparations on the 30th Day

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	characterization
Model	1.41	3	0.47	21.92	< 0.0001	significant
A-Carbopol 940	0.79	1	0.79	36.55	< 0.0001	
B-TEA	8.450E-003	1	8.450E-003	0.39	0.5415	
C-Nanoparticles	0.62	1	0.62	28.84	0.0001	
Residual	0.28	13	0.021			
Lack of Fit	0.23	9	0.025	1.94	0.2726	not significant
Pure Error	0.052	4	0.013			
Cor Total	1.69	16				

Scattering power is strongly influenced by carbopol 940 because carbopol 940 has properties as thickener, surfactant and stabilizer^{13,14}, the more carbopol 940 is used, the smaller the dispersion value of gel preparations. In addition to carbopol 940, coffee pulp nanoparticles also affect the spreadability of gel preparations, this effect is seen in the ANOVA analysis results and the 3D plot in Fig.-4.

CONCLUSION

The preparation of Arabica coffee pulp nanoparticle gel has a pH that is following the pH of the skin, which is 4.8 to 6.8 and has a good dispersion of 5.00 to 6.47 cm. The stability of the pH and spreadability of gel preparations during storage for 30 days showed stable results, without significant changes. The results of the analysis showed that pH was influenced by the addition of TEA and carbopol 940 and the dispersion was influenced by the addition of carbopol 940 and Arabica coffee pulp nanoparticles.

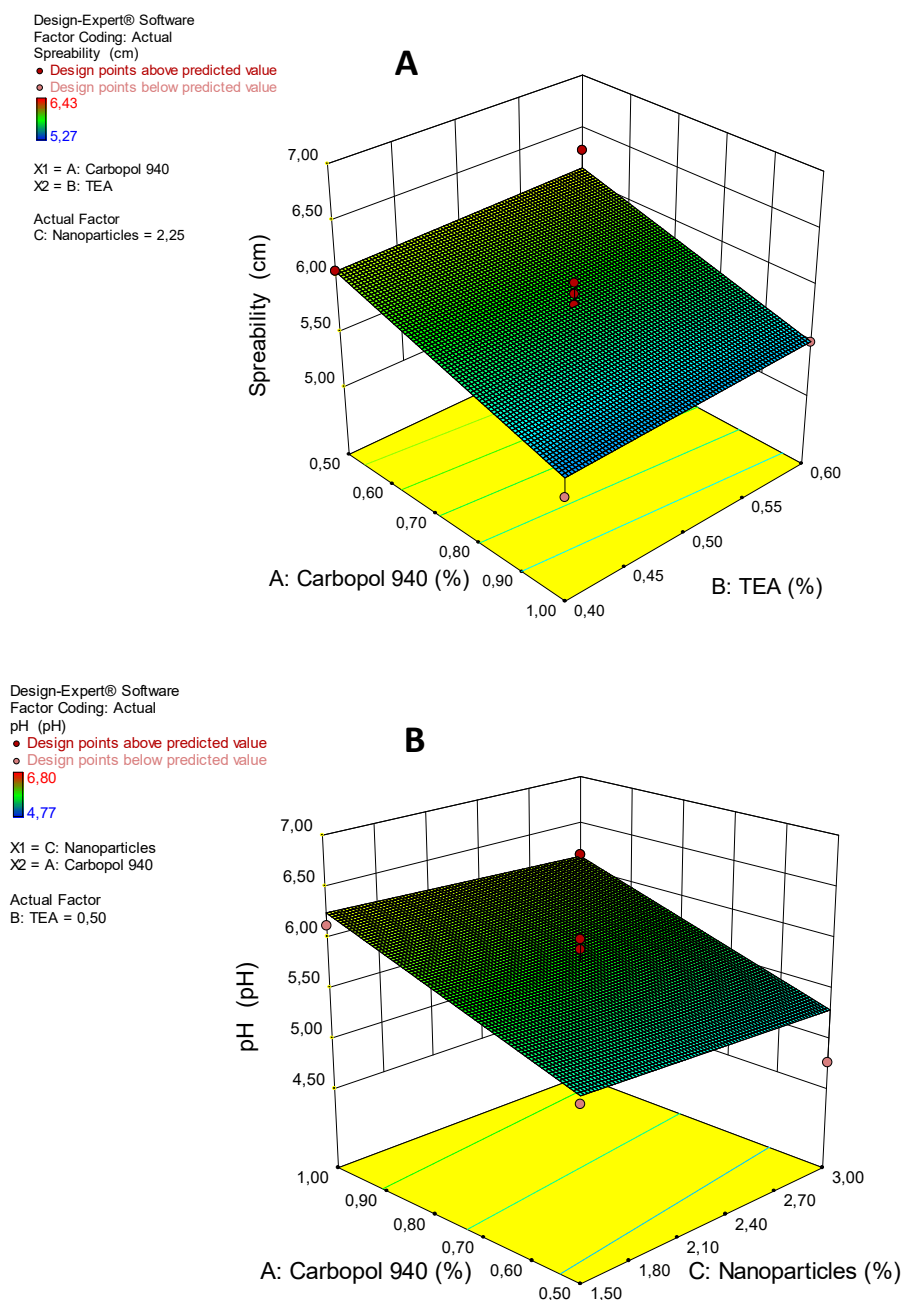


Fig.-4: 3D Plot of the Relationship between (A) Carbopol 940 and TEA on the Dispersion, (B) Carbopol 940 and Nanoparticles on Dispersion, at 30 Days Storage.

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