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Design and Development of Topical Gel Bases Formulation of Cocoa (*Theobroma Cacao*) Shell extract for Hair Gel

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ABSTRACT

The mixture design was used to optimize the potential of hydrogenionic and the spreadability of topical hair gel from cocoa shell extract. The influence of the primary hair gel components thickener (0.25 – 0.75%), moisturizer (1-3%), styling polymer (1-3%), humectant (2-5%), adjusting PH agent (0.2-0.4%) and solvent (90.55-95.75%) were studied on two responses of the topical hair gel. The data obtained were fitted to the model with coefficient determination (R2= 0.994 for the pH and 0.9937 for the spreadability) and the lack of fit test shown not significant with a p-value bigger than 0.05. The optimized formulation with each significant factor was established at thickener (0.3%), moisturizer (0.25%), styling polymer (1.5%), humectant (2.99%), adjusting pH agent (0.4%), and solvent (94.57%) with the potential of hydrogenionic value and spreadability at 6.42 and 1.57 respectively. The predicted validation test shows that both values were comparable. This condition showed that the model development could be used to predict future observations within the design range.

Keywords:
Cocoa shell; cosmeceutical; hair gel; mixture design

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1. Introduction

Cocoa is one of the fruits that riches in good chemical composition and could give benefit to human health [17]. A recent study shows plant extract containing bioactive compounds such as flavonoids, curcumin, terpenes, and saponins, can be used to fight against various diseases [22] such as anti-cancer [18] and anti-inflammatory [11]. Despite the benefit of cocoa usually focusing on cocoa powder and cocoa bean, the cocoa waste has usually been left out. In the cocoa industry, cocoa shells

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and cocoa pod husk were under-utilized by-products [26]. Usually, this by-product was used as a soil compact and boiler fuel [19]. Another application of this by-product was activated carbon [2]. The biomass based activated carbon has the potential to be used as an alternative in the production of coal-based activated carbon whereas the production of biomass-based activated carbon gave a milder impact on the environment in terms of global warming potential and acidification potential. There were also many studies on the antioxidant (in vivo and in vitro) from cocoa plants such as cocoa pulp, cocoa butter, and cocoa nibs [3]. The previous study shows that extract from cocoa pod husk has the potential to be used in treatment to reduce wrinkles as its show comparable result to kojic acid [16]. Another study on unfermented cocoa bean from Malaysia, show that unfermented cocoa bean extracted have potential for cosmetic application as an anti-collagenase and anti-elastase that can give an anti-aging effect on human skin [1].

The chemical composition in the cocoa shell and other plants is quite variable due to several factors such as origin, clone, processing, and pre-processing. Moreover, different processing would give a different yield of a targeted natural product [7]. For example, a natural pigment such as anthocyanin from Hibiscus rosa-Sinensis L. and Clitoria Ternatea L gave different degradation rates during the drying process at different temperatures and duration [4]. Another example of variable chemical composition in cocoa is the yield of cocoa polyphenols from Malaysia was different than from Sulawesi, Ghana, and Ivory Coast [20] and the different chemical composition of different cocoa processes. Another study was done to discover that type of soil and growth stages play an essential role in obtaining a higher yield of targeted chemical composition [13]. Even though the previous study has shown promise, cocoa shell extract has many potentials, but the application on the cosmetic was not carried out.

To apply the natural product inside the cosmetic product, a proper technique should be chosen to study the specific process for cosmeceutical products. One of the techniques that can optimize the process and understand the complex interaction between ingredients is by applying mixture design in the process. However, cosmetic chemists rarely apply these tools. Mixture design is one of the tools that can be used to study the complex interaction between variables and reduced the number of experimental trials. It also can be used for future observation for a response within the design range [15].

2. Material and Methods

2.1 Material and Chemicals

The cocoa bean was collected from Pusat Penyelidikan dan Pembangunan Koko, Jengka, Pahang, Malaysia. Next beans were freeze-dried for three days and manually removed the dry shell from the bean and labeled as Malaysian Cocoa Shell (MCS). Other chemicals used in this study are distilled water, ethanol, thickener, moisturizer, styling polymer, humectant, and pH adjusting agent. All chemicals were in analytical grade and cosmetic grade. The brand and trade name ingredient of the formulation were not published due to a conflict of interest.

2.2 Ultrasound-Assisted Extraction

First, 1g of crushed MCS (1mm) was mixed with a 50mL solvent at 67% concentration. Next, the MCS was extracted using a sonication bath machine (Wise clean 40 kHz, 296 W, and Korea) at 60 °C for 25 minutes. As a result, the aqueous extract was filtered using Watman filter paper number 4, and the solvent was removed using a rotary evaporator (IKA, German) and freeze-dried (Labconco, USA) to get the crude extract and labeled as Malaysian Cocoa Shell Extract (MSCE). The crude extract was stored at -40 °C in storage vials.

2.3 Formulation of Topical Hair Gel using Mixture Design

A mixture of several ingredients and other chemical materials was used as the formulation for this topical hair gel. The mixture design (MD) was created and analyzed by using Expert Design Software (version 10, Stat-Ease Inc, USA). The thickener, styling polymer, humectant, moisturizer ingredient, and MSCE were diluted in distilled water and mixed using a commercial hand mixer. The mixture was then naturalized by using an adjusting pH agent to make it into gel texture and name a topical hair gel (THG). The range value of each ingredient and its combination for the experimental run was shown in table 1 and table 2. The other ingredient such as preservatives and fragrance, were mixed after the gel texture was obtained.

Table 1
Independent variables and their range set

Independent variables (%)	level	
	minimum	maximum
Thickener, (X_1)	0.3	0.5
Moisturizer, (X_2)	0.25	0.75
Styling Polymer, (X_3)	1	3
Humectant, (X_4)	2	5
adjustment agent, (X_5)	0.2	0.4
Solvent, (X_6)	90.55	95.75

2.4 Determination of the Potential of Hydrogenionic (pH) of THG

The sample was evaluated using a digital calibrator (Accumet AB200) for the potential of hydrogenionic (pH). The pH value of THG was recorded without any dilution until the screen shows a stable value as described by Slim Smaoui *et al*., [23] and Vijayakumar and Adedeji [25].

2.5 Determination of the Spreadability value of THG

The spreadability value was determined by using the procedure from Glibowski *et al*., [10], and the texture analyzer software with a probe TTC spreadability rig (HDP/SR) 90° male cone with matches female cone. The female cone was filled with 5g of THG by using a spatula, and air pockets (visible through Perspex cones) were eliminated by pressing down the gel. Next, the surface is leveled with a flat knife and recorded its high by adjusting the male cone from the software. The parameter was chosen from the software at a test speed of 3mms^{-1} for a distance of 23 mm. The spreadability value was calculated from the software.

2.6 Optimizing, Ramp Function, and T-test

The model of the mixture design was validated using a ramp function graph experiment, and T-tests were performed using Minitab software (version14, Minitab, USA) for the data collected from the experiment. Three random solutions were created from the mixture design model, with different conditions, as shown in Table 3. The desirability was fit to a value of 1.00. All of the experimental simulations were run in triplicate, and the data collected from the experiment ramp function were tested for statistical data using the T-test from the Minitab Software.

Table 3
Condition for the ramp function graph

Solution	Thickener (%)	Moisturizer (%)	Styling polymer (%)	Humectant (%)	Adjusted agent (%)	Solvent (%)	pH	Spreadability	Desirability
1	0.31	0.28	1.00	4.83	0.39	93.19	6.1[illegible]	1.82	1.00
2	0.30	0.25	1.50	2.99	0.40	94.57	6.42	1.57	1.00
3	0.30	0.26	2.31	5.00	0.20	91.93	6.11	1.92	1.00

2.7 Statistical Analysis

The significance was determined using analysis of variance testing (ANOVA) on Expert Design software, and the T-test for validation was performed on Minitab Software. Data are expressed as a mean ± standard error.

3. Result and Discussion

3.1 Model Fitting for THG

The complex interaction of six-ingredient for THG was optimized through the L-optimal mixture design approach. The variation response potential of hydrogenionic and spreadability was predicted through the mixture design by using equations 1, and 2 builds by the software. The comparison between actual data and predicted data were presented in table 2. The model for two responses shows significant with a p-value less than 0.0001 while the lack of fit test shows no significant with a p-value larger than 0.05. The ANOVA analysis shows the model fits the data and could be used to predict future observations within the design range. The complex interaction significant and not significant between ingredients, coefficient of determination, and lack of fit test were shown in table 4.

$$Y_{ph} = 389.8223x_1 - 122.304x_2 + 2.404321x_3 + 0.344147x_4 + 96.80492x_5 + 0.066353x_6 - 5.04041x_1x_2 - 6.06443x_1x_3 - 3.11934x_1x_4 - 1.80482x_1x_5 - 3.95566x_1x_5 + 0.895819x_2x_3 + 1.237137x_2x_4 + 7.15344x_2x_5 + 1.231749x_2x_6 - 0.02332x_3x_4 - 1.80277x_3x_5 - 0.01038x_3x_6 - 0.90386x_4x_5 - 0.00708x_4x_6 - 0.96764x_5x_6$$

$$Y_{Spreadability} = -5912.58482x_1 - 120.80792x_2 + 12.21304x_3 - 1.70686x_4 - 3119.21655x_5 - 0.077216x_6 + 57.87132x_1x_2 + 63.10817x_1x_3 + 59.43754x_1x_4 + 118.50082x_1x_5 + 59.52933x_1x_5 + 0.93014x_2x_3 + 1.31623x_2x_4 + 32.52024x_2x_5 + 1.23266x_2x_6 - 0.14682x_3x_4 + 31.14239x_3x_5 - 0.13710x_3x_6 + 30.92483x_4x_5 - 0.019213x_4x_6 - 31.29207x_5x_6$$

where $x_1, x_2, x_3, x_4, x_5, x_6$ were thickener, moisturizer, styling polymer, humectant, adjusting agent, and solvent, respectively.

Table 3
Analysis of variance for a fitted quadratic model of ingredient ratio THG

Source	Potential of Hydrogenionic (pH)			Spreadability Value		
	Mean Square	F-Value	p-value	Mean Square	F-Value	p-value
Model	0.380858664	82.46985	1.52E-08	1.073204	79.23328	1.85E-08
Linear Mixture	1.275026387	276.0899	2.24E-10	3.224986	238.0966	4.66E-10
X_1X_2	0.009171293	1.985921	0.189098	1.208999	89.25889	2.67E-06
X_1X_3	0.014407799	3.119816	0.107797	1.56023	115.1898	8.28E-07
X_1X_4	0.003829682	0.829266	0.38391	1.390459	102.6558	1.41E-06
X_1X_5	0.00047519	0.102896	0.754985	2.04852	151.2396	2.32E-07
X_1X_6	0.006199083	1.342328	0.273543	1.403948	103.6517	1.35E-06
X_2X_3	0.013149741	2.8474	0.122414	0.014177	1.046644	0.330393
X_2X_4	0.025865538	5.600836	0.039505	0.029279	2.161615	0.172245
X_2X_5	0.018733281	4.056441	0.071687	0.387161	28.5836	0.000325
X_2X_6	0.026009918	5.6321	0.039063	0.026049	1.923135	0.195645
X_3X_4	0.001746337	0.378146	0.552332	0.069225	5.110795	0.047312
X_3X_5	0.001159496	0.251073	0.627165	0.346015	25.54583	0.000496
X_3X_6	0.000497051	0.10763	0.749625	0.086679	6.399404	0.029886
X_4X_5	0.00028931	0.062646	0.807429	0.338666	25.0033	0.000537
X_4X_6	0.001108136	0.239952	0.634809	0.00816	0.602461	0.455606
X_5X_6	0.000330398	0.071543	0.794539	0.345521	25.50934	0.000499
Residual	0.004618157	Not Defined	Not Defined	0.013545	Not Defined	Not Defined
Lack of Fit	0.005916313	1.782022	0.27071	0.01073	0.655857	0.67263
R^2	0.994			0.9937		
Predicted R^2	0.8084			0.8201		

3.2 Optimizing the Ingredient THG ratio

The pH value played an essential role in determining the function of cosmetic hair products. It also affects hair cuticles. The cuticle is the most crucial component of human hair to determine the structure of the hair shaft. It covers the hair thread from the scalp to the end as overlapping 8 to 11 layers and it may be more or less affected by cosmetic treatments [24]. On the cuticle, cosmetic products, such as conditioners, hair sprays, mousses, and gels, are deposited. The condition and function of cuticles were determined from the pH value. For example, at the acidic pH level, the cuticles attempt to close to each other. The effect of acidic pH will have a shimmering effect on the hair. At the alkaline level, the cuticles attempt to be open and it is easier to change their structure, form, and integrity from perm and straightening treatment [6]. Besides that, the thickener used in the formulation affects the properties of the product. Many types of thickeners can be used to make cosmetic products such as natural thickeners, mineral thickeners, lipid thickeners, and ionic thickeners. The thickener used in this formulation was an ionic type. The ionic type of thickener was commonly used in a cosmetic product due to its flexibility to manipulate the pH value through the pH adjusting agent. Figure 1 shows that the pH value increased from 5.15 to 7 when the moisturizer and styling polymer was increased. Even though the main reasons the presented moisturizer in the formulation were intended to give a shiny look and to moisturize the hair, it probably also affects the interaction between styling polymer and the thickener. Besides that, the ionic thickener can be adjusted by using the salt derivative to form an anionic surfactant [21].

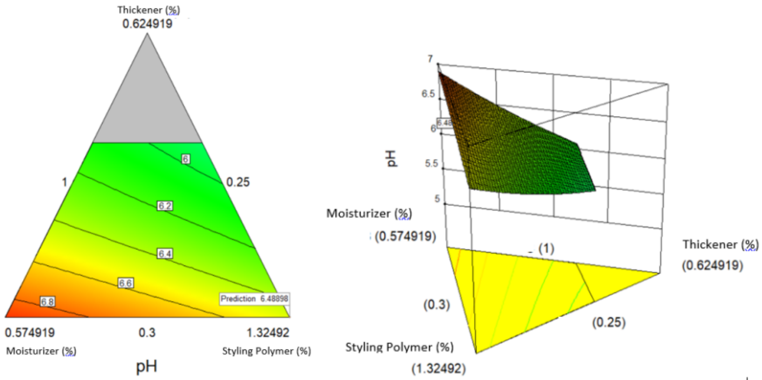

Fig. 1. Contour plot and surface plot of thickener, moisturizer, and styling polymer concentration

The spreadability of a semisolid formulation is the ability of a cosmetic product to spread on the targeted area evenly. It plays a vital role in the administration of the standard and efficacy of cosmetic products. Figure 2 shows the spreading values of the THG through the surface and contour plot. The value obtains from the MD was between 1.501 and 4.591, with the coefficient determination R^2 = 0.9937. The predicted coefficient determination was 0.82 showing that the model could predict future observations within the design range. The value obtained from the spreadability was in the range of five market product spreadability values (data not published). The previous study on the spreading semisolid formulation shows that several factors influenced the spreading values such as formulation characteristics including viscosity, elasticity, rheology, temperature, and area of application [9]. For example, increasing the viscosity of the delivery ingredient will increase its retention time in the targeted area but also decreases its spreadability [5]. Another study shows there is a good correlation between spreadability and cohesiveness because strong, cohesive forces within a formulation would reduce its flowability and thereby affecting its spreadability profile on the substrate [8].

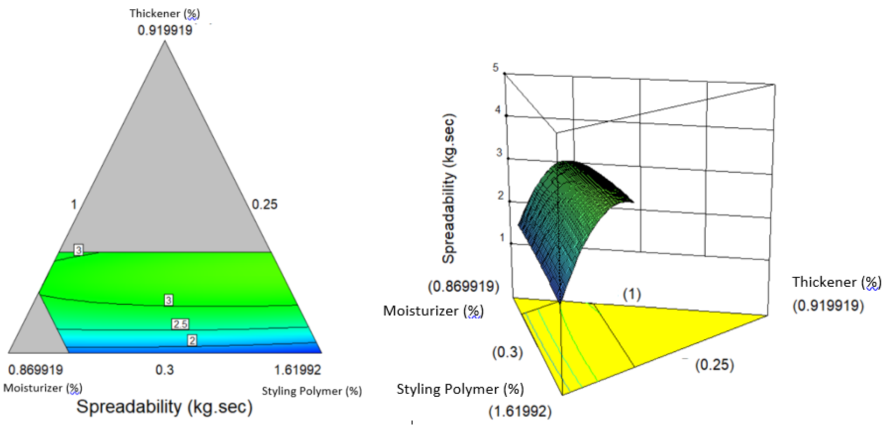

Fig. 2. Contour plot and surface plot of thickener, moisturizer, and styling polymer concentration

3.3 Verification of the Model

This experiment was used to verify the model that can be used with any set of ingredients within the design range. Using the desirability function approach for THG by choosing lower thickener, low moisturizer, low styling polymer, high humectant, high adjusting agent, and low solvent with the desirability value yields a final result of 1.00 for the simultaneous optimization. The T-test shows that the data is not significant ($p>0.05$) between predicted and experimental values (Table 5 and Table 6). Based on the results, the predicted and experimental values were comparable. Therefore, the formulation obtained by the mixture design model is satisfactory. T-test from the ramp function shows not significant given the model can be repeated at any point within the design range. The best formulation was selected as follows: thickener (0.3%), moisturizer (0.25%), styling polymer (1.5%), humectant (2.99%), adjusting PH agent (0.4%), and solvent (94.57%) with the potential of hydrogenionic value and spreadability at 6.42 and 1.57.

Table 5
T-test pH table of verification of the model

Solution	Number of samples	Mean	Standard deviation	Standard error mean	95% coefficient of the interval	T-value	P-value
1	3	6.11	0.06	0.035	5.96, 6.26	0.29	0.80
2	3	6.44	0.05	0.0289	6.32, 6.56	0.69	0.56
3	3	6.10	0.04	0.023	6.00, 6.19	-0.43	0.71

Table 6
T-test spreadability table of verification of the model

Solution	Number of samples	Mean	Standard deviation	Standard error mean	95% coefficient of the interval	T-value	P-value
1	3	1.76	0.07	0.040	1.59, 1.93	-1.48	0.276
2	3	1.62	0.10	0.058	1.37, 1.87	0.87	0.478
3	3	1.86	0.04	0.023	1.76, 1.96	-2.60	0.122

4. Conclusion

The MD was used in the formulation of THG with MSCE and optimized its variable by using L-optimal. The results determined that the variables are significant only concerning moisturizer, humectant, and solvent for the potential of hydrogen. For spreadability, thickener plays an important role as an influencer compared to other variables. However, data obtained were fit to the model with significant ($p<0.001$), allowing the model can be used to predict future observations. Therefore, in this study, the THG was successfully formulated, and the spreadability and potential of hydrogenionic of the formulations were evaluated properly.

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Table 2
Mixture design of six-ingredient to formulate THG and their observed responses

Run	Thickener (%)	Moisturizer (%)	Styling Polymer (%)	Humectant (%)	Adjustment pH agent (%)	Solvent (%)	pH_{Actual}	$pH_{Predicted}$	Spreadability (Kg.sec) $_{Actual}$	Spreadability (Kg.sec) $_{Predicted}$
1	0.50	0.25	2.85	5.00	0.20	91.20	5.49	5.48	3.53	3.56
2	0.30	0.61	2.02	2.91	0.34	93.83	6.70	6.74	1.96	2.11
3	0.41	0.75	1.67	2.31	0.40	94.45	6.61	6.55	3.17	3.18
4	0.50	0.75	1.22	3.35	0.23	93.94	5.35	5.27	2.51	2.50
5	0.30	0.25	2.03	3.71	0.20	93.51	6.04	6.13	2.07	1.92
6	0.30	0.75	3.00	5.00	0.40	90.55	7.00	7.00	1.76	1.72
7	0.30	0.25	3.00	2.00	0.40	94.05	6.96	6.98	1.88	1.90
8	0.44	0.25	1.96	2.00	0.26	95.10	5.68	5.70	3.30	3.41
9	0.45	0.31	1.87	4.19	0.40	92.78	6.12	6.09	3.34	3.40
10	0.40	0.75	2.65	3.35	0.20	92.65	5.46	5.48	3.30	3.31
11	0.30	0.58	1.59	5.00	0.20	92.33	5.83	5.80	2.05	2.11
12	0.48	0.45	3.00	2.98	0.30	92.78	5.64	5.60	4.59	4.53
13	0.30	0.25	1.00	2.68	0.20	95.57	5.90	5.81	1.77	1.91
14	0.30	0.25	1.00	5.00	0.40	93.05	6.10	6.09	1.50	1.49
15	0.50	0.25	1.00	3.15	0.40	94.70	5.98	5.98	2.88	2.91
16	0.40	0.50	1.00	2.00	0.40	95.70	6.45	6.47	3.00	2.98
17	0.50	0.75	2.39	2.00	0.40	93.96	5.92	5.94	3.82	3.85
18	0.50	0.75	2.29	5.00	0.30	91.16	5.57	5.60	3.59	3.56
19	0.44	0.25	1.96	2.00	0.26	95.10	5.71	5.70	3.50	3.41
20	0.34	0.25	2.88	4.55	0.28	91.69	6.30	6.28	2.95	3.01
21	0.30	0.61	2.02	2.91	0.34	93.83	6.78	6.74	2.23	2.11
22	0.43	0.30	1.00	4.95	0.26	93.06	5.65	5.67	3.05	3.00
23	0.48	0.45	3.00	2.98	0.30	92.78	5.55	5.60	4.49	4.53
24	0.30	0.25	1.00	2.68	0.20	95.57	5.77	5.81	1.97	1.91
25	0.30	0.75	1.00	2.00	0.20	95.75	5.84	5.87	2.09	2.06
26	0.50	0.25	2.67	2.64	0.40	93.54	5.73	5.73	4.36	4.30
27	0.40	0.50	1.00	2.00	0.40	95.70	6.48	6.47	3.03	2.98
28	0.50	0.61	1.03	2.68	0.20	94.98	5.15	5.19	2.14	2.15
29	0.40	0.75	1.00	4.11	0.40	93.33	6.60	6.63	2.91	2.93
30	0.42	0.43	2.27	4.46	0.40	92.01	6.26	6.26	3.51	3.50
31	0.30	0.40	3.00	2.00	0.20	94.10	6.80	6.76	2.21	2.22