

Development and Optimization of Cocoa-Based Herbal Face Pack Using Design of Experiments

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ABSTRACT

Background: Skincare products featuring natural ingredients are widely favored for their perceived efficacy and safety. This study aimed to develop and evaluate cocoa fruit-based formulations for skincare, enriched with synergistic herbal constituents such as rose petals, guava peel and papaya leaf. **Materials and Methods:** The cocoa fruit underwent processing and fermentation to yield cocoa beans. In this study, 12 facial pack formulations were developed using JMP 17 software, with variations in the quantities of cocoa powder, papaya leaf powder, guava peel powder and rose petal powder. The custom design generated by JMP facilitated the creation of optimized formulations, considering responses such as bulk density and angle of repose. The final optimized formulation was assessed for organoleptic properties, moisture content, washability, bulk density, angle of repose, total ash value and phytochemical constituents. **Results:** Optimal formulations were determined for the product based on desired attributes and skincare benefits. The DoE-generated twelve facial pack formulations exhibited acceptable model fit, with the optimized formulation demonstrating positive outcomes in organoleptic and phytochemical analyses. **Conclusion:** This study successfully formulated skincare products using cocoa bean and herbal constituents, demonstrating their potential for natural skincare formulations with desirable properties and therapeutic benefits. DoE enabled a data-driven approach to optimize the herbal facial pack formulation efficiently, ensuring robust physical properties and potential skincare benefits. Future research endeavors could focus on further refining formulations and exploring additional applications in skincare product development.

Keywords: Herbal face pack, Jmp pro-18, Optimization, *Theobroma cacao*.

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INTRODUCTION

Cosmetics are products designed to cleanse, beautify and enhance appearance, commonly applied to the face, hair and body. Since ancient times, herbs have been essential in cosmetics for their natural benefits, offering properties like antioxidant, anti-inflammatory, antiseptic and antibacterial effects. The rising demand for natural and cruelty-free ingredients has popularized herbal cosmetics, which are eco-friendly and generally free from the side effects of synthetic agents (Aglawe *et al.*, 2018; Shivanand *et al.*, 2010; Hii *et al.*, 2009).

Cocoa (*Theobroma cacao*) is rich in polyphenols like catechin and epicatechin, which are potent antioxidants that protect the skin from free radicals and aging. Cocoa butter, derived from cocoa seeds, contains essential fatty acids and phytosterols that improve

skin elasticity and provide UV protection. Beyond cosmetics, cocoa is widely valued for health benefits related to heart, brain and mental wellness (Halim *et al.*, 2020; Pal *et al.*, 2017; Karole *et al.*, 2019).

Face packs are an effective method for cleansing and revitalizing the skin. Herbal face packs are straightforward to use, nourishing the skin with essential vitamins and increasing blood circulation for a brighter complexion. Polyherbal face packs combine various herbal ingredients to enhance therapeutic effects while reducing toxicity, making them beneficial for diverse skin conditions (De Souza *et al.*, 2018; Ribeiro *et al.*, 2015; Hernández *et al.*, 2019).

Key ingredients in these face packs include

Cocoa Seed: Cocoa's flavonoids provide antioxidants that reduce signs of aging, protect against UV damage and improve skin texture.

Guava Peel: Rich in vitamin C, guava promotes collagen production, treats acne, evens skin tone and guards against UV damage (Mane *et al.*, 2018; Aglawe *et al.*, 2018; Yadav *et al.*, 2015).



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Papaya Leaf: Contains papain enzyme, which exfoliates dead cells, boosts cell turnover and has a moisturizing effect that prevents dryness.

Rose Petals (*Rosa indica*): High in antioxidants, rose petal powder slows aging, tightens pores, reduces oil and soothes the skin with its natural (Sinde *et al.*, 2020; Scapagnini *et al.*, 2014; Sundriyal *et al.*, 2022; Song *et al.*, 2020; Ashawat *et al.*, 2009; Hariono *et al.*, 2021).

These herbal components contribute to healthy, glowing skin and are increasingly preferred for their effectiveness and natural origin, aligning with the growing trend toward Ayurvedic and holistic skincare solutions (Kumar, 2021; Pratiwi *et al.*, 2023; Grace *et al.*, 2014).

MATERIALS AND METHODS

Materials

Cocoa fruit, Rose petals, Guava peel, Papaya leaf, Coconut oil, Glycerin, Cocoa butter.

Methods

Processing of Cocoa Fruit

The ripe cocoa fruit pods from the cocoa trees were collected from a nearby source. These pods were cracked open to extract the cocoa beans surrounded by the sweet, white pulp inside. The beans with the pulp were subjected to a 4-5 days fermentation process where they were piled and covered, allowing microbial and enzymatic reactions to develop precursor flavor compounds. Every day, the beans were mixed by hand to ensure proper fermentation. After fermentation, the cocoa beans were sun-dried for 6-7 days by spreading them out evenly. This step reduced the moisture content and the dried fermented beans were further milled.

Formulation of face pack using design of experiment

The development of a facial pack incorporating cocoa bean and synergistic herbal constituents such as rose petals; guava peel and papaya leaf involves a methodical approach. Key parameters governing the formulation process include ingredient quantities and their impact on critical responses such as bulk density and angle of repose. Using SAS JMP Pro 18 software, a custom design was constructed within defined input ranges; yielding 12 unique formulations (see Table 1). These ingredients were selected based on their documented skincare benefits. Cocoa bean's antioxidant attributes promote skin hydration and microcirculation, while rose petals exhibit anti-inflammatory and antimicrobial properties. Guava peel's high vitamin C content contributes to skin brightening and papaya leaf enzymes facilitate gentle exfoliation. Fresh rose petals, guava peel and papaya leaves were obtained locally and underwent a meticulous cleaning, drying and grinding process to create fine powders. The ingredients

were mixed according to the design outlined in Table 2 and subsequently analyzed for bulk density and angle of repose.

Evaluation

The optimized formulation was evaluated for following parameter:

Organoleptic evaluation

The organoleptic characteristics, including texture, color and odor, were assessed through manual evaluation.

Moisture content

The powders were initially weighed and then stored in desiccators at 37°C for 24 hr. Subsequently, the powders were reweighed at intervals until a constant weight was achieved. The moisture content percentage was then determined using the following formula (Avinash *et al.*, 2019).

$$\frac{\text{Initial weight} - \text{final weight}}{\text{Initial weight}}$$

Washability

Washability was evaluated by applying the formulation to the skin and manually assessing how easily and thoroughly it washed off with water (Rokade *et al.*, 2017).

Bulk density

Bulk Density (B.D) of a powder was determined by dividing the total Mass of the powder (M) by the bulk volume of the powder (V_0). The bulk volume was obtained by pouring the weighed powder, which had passed through a standard sieve #20, into a measuring cylinder. The initial volume recorded was considered the bulk volume (V_0) and the bulk density was calculated using the formula provided (Jain *et al.*, 2024).

$$\text{Bulk Density} = \frac{\text{Mass of powder}}{\text{Bulk volume}}$$

Angle of Repose

The maximum angle that can exist between the surface of a pile of powder or granules and its horizontal plane is termed the angle of repose. This angle was determined through the funnel method, where a funnel was fixed at a set height (2.5 cm) on a burette stand. Powder samples were then poured through the funnel until they formed a heap and pouring ceased once the heap touched the funnel's tip. A circle was drawn around the heap without disturbing it and measurements of the heap's radius and height were recorded. This process was repeated three times and the average values were calculated. Finally, the angle of repose was determined using a specific equation (Anilkumar *et al.*, 2020).

$$\tan \theta = h/r$$

$$\theta = \tan^{-1}(h/r)$$

Table 1: Formulation Design.

Run	Cocoa powder	Papaya leave powder	Guava peel powder	Rose petal powder
	Quantity of powders in grams			
1	6	2	1	1
2	5	2	1	2
3	4	2	2	2
4	5	1	3	1
5	3	2	3	2
6	7	1	1	1
7	4	1	3	2
8	6	1	2	1
9	5	1	2	2
10	4	2	3	1
11	5	2	2	1
12	6	1	1	2

Where, θ =angle of repose, h =Height of the heap, r =Radius of the heap.

Determination of total ash value

The total ash value helps determine the quality of low-grade or exhausted products and is essential for identifying excessive sandy or earthy matter in a drug. Approximately two grams of the powdered sample were placed in a previously ignited and tarred crucible. The sample was spread evenly in the crucible and gradually heated until it turned into a white powder, indicating it was free from carbon. After cooling the sample in a desiccator, the weight was recorded. The percentage of total ash was then calculated based on the weight of the air-dried sample. The total ash value is determined using the following formula (Gopikrishna *et al.*, 2024).

$$\% \text{ Total ash} = \frac{(\text{ashed weight}) - (\text{crucible weight})}{(\text{crucible and sample weight}) - (\text{crucible weight})} \times 100$$

Phytochemical evaluation

The aqueous extract of the herbal face pack was evaluated for the presence of different phyto constituents as per the standard procedures.

Detection of alkaloids

To detect the presence of alkaloids, several reagents were employed. Dragendorff's reagent was added to the sample, with the formation of an orange-red precipitate indicating alkaloid presence. Hager's reagent was used similarly, where a yellow precipitate served as a positive indication. Wagner's reagent was also applied, with a reddish-brown precipitate indicating a positive result. Lastly, Mayer's reagent was added and the appearance of a cream-colored precipitate was taken as confirmation of alkaloids.

Table 2: Results of responses.

Run	Bulk Density (g/cm ³)	Angle of Repose (°)
1	0.36± 0.004	35±0.289
2	0.323±0.002	30±0.577
3	0.3±0.047	30±0.231
4	0.32±0.001	30±0.173
5	0.35±0.001	35±0.289
6	0.32±0.0141	32±0.577
7	0.32±0.002	35±0.173
8	0.35±0.0005	35±0.404
9	0.34±0.0005	35±0.231
10	0.32±0.0014	30±0.404
11	0.35±0.0009	35±0.520
12	0.35±0.0014	35±0.462

Detection of carbohydrates and glycosides

Carbohydrates were identified using Molisch's test. A few drops of α -naphthol were added to the sample, followed by sulfuric acid along the tube wall to create a purple ring at the interface, indicating the presence of carbohydrates. To further confirm, Benedict's and Fehling's tests were conducted: the sample was mixed with Benedict's reagent and heated, observing a reddish-brown precipitate; in a separate test, Fehling's solution was added and boiled, with a brick-red precipitate indicating carbohydrate presence.

Detection of flavonoids

Flavonoids were detected by Shinoda's test, which formed a crimson-red color with ethanol, hydrochloric acid and

magnesium. The sodium hydroxide test produced an intense yellow color, indicating flavonoids.

Detection of phenolic compounds and tannins

Phenolic compounds were identified by a color change with FeCl_3 . Tannins were detected by a white precipitate with lead acetate, red precipitation with sodium hydroxide and a buff precipitate in the gelatin test (Bhutkar *et al.*, 2019; Sundriyal *et al.*, 2022).

RESULTS

Design evaluation and Optimization

The bulk density and angle of repose for all 12 formulations are depicted in Table 2, which was utilized in the design. The study found that the amount of cocoa powder significantly affected both the bulk density and angle of repose of the facial pack, whereas the quantity of other ingredients did not exhibit a significant effect, as shown in Figure 1. From the actual vs predicted plot (Figures 2 and 3), it was evident that the regression model utilized was robust, with high R-squared values (0.90 for bulk density and 0.77 for angle of repose) and low *p*-values (0.0013 for bulk density and 0.0231 for angle of repose), indicating that the results were statistically significant. Additionally, a straight line indicated that the model fit well. By using a prediction profiler (Figure 4), an optimized formulation was identified with a predicted bulk density of 0.33 and an angle of repose of 33, achieving 81% desirability. This mix includes 4.5 g of cocoa powder, 1.5 g of papaya leaf powder, 2 g of guava peel powder and 1.5 g of rose petal powder, offering a balanced blend likely to produce a product with favorable physical properties. The optimized Formulation (FA) was then formulated and evaluated for all parameters.

Organoleptic Evaluation

The optimized formulation, FA, exhibited a powdery nature, accompanied by a musty odor and a semi-smooth texture to the touch.

Moisture content

The optimized formulation's moisture content exceeded 5%, indicating its hygroscopic nature and emphasizing the need for special storage techniques to prevent degradation.

Washability

The formulated product successfully passed the washability test, indicating that it can be easily removed from the skin with water. This suggests that the product offers good cleansing properties and does not leave a significant residue on the skin after washing.

Powder Rheology

The rheological evaluation of the optimized formulation showed a close resemblance to the predicted values, further highlighting the relevance of the design. These results are illustrated in Table 3.

Total ash value

The total ash value was 4.5% for the optimized formulation, indicating a moderate presence of mineral content. Lower ash values often suggest higher ingredient purity and product quality, essential for meeting regulatory standards

Phytochemical evaluation

The phytochemical evaluation confirms the presence of alkaloids, carbohydrates, glycosides, flavonoids and phenolic compounds/tannins in the sample. These compounds offer diverse health

Effect Summary

Source	Logworth	PValue
Cocca Powder(3,6)	3.934	0.00012
Rose petal(1,2)	0.864	0.13674
papaya leave(1,2)	0.671	0.21329
Guava peel(1,3)	0.278	0.52771

Figure 1: Effect summary of the ingredient on the responses.

Table 3: Results of powder rheology.

Sl. No.	Parameter	FA	
		10 g	15 g
1	Bulk Density (g/cm^3)	0.33±0.0005	0.33±0.0015
2	Angle of Repose (°)	33.52±0.15	

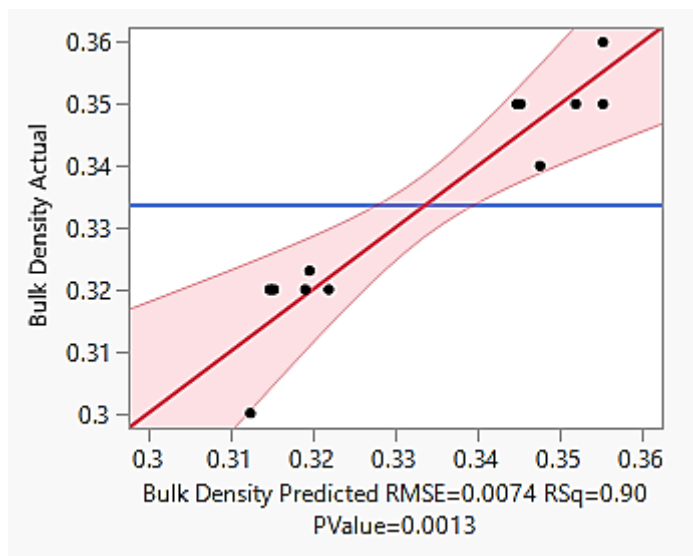


Figure 2: Actual vs predicted plot for bulk density.

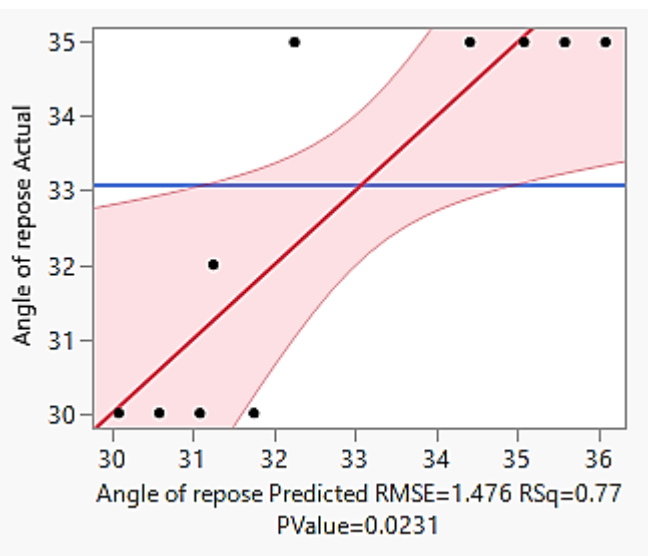


Figure 3: Actual vs predicted plot for Angle of repose.

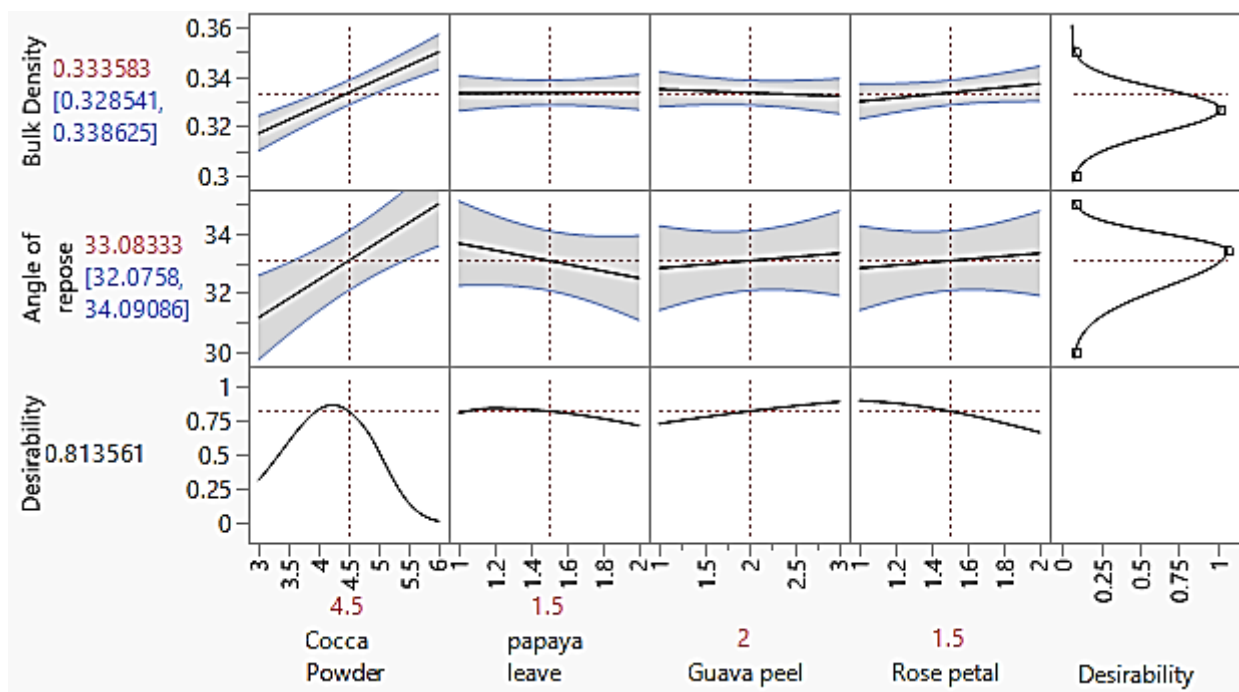


Figure 4: Prediction profiler.

benefits, including pharmacological effects, antioxidant properties and potential therapeutic value, highlighting the sample's potential for traditional medicine and functional product development.

DISCUSSION

The study successfully employed Design of Experiments (DoE) to optimize the formulation of a cocoa-based facial pack. The bulk density and angle of repose were identified as critical parameters influenced primarily by cocoa powder quantity. The high R-squared values and statistically significant *p*-values validated the robustness of the model, while the prediction profiler provided

an optimized formulation that balanced desirable physical and functional properties.

The organoleptic, moisture content and washability assessments affirmed the optimized product's potential for skincare applications, highlighting its ease of use and minimal residue post-application. The powder rheology results aligned with predictions, reinforcing the design's effectiveness.

The total ash value and phytochemical evaluation confirmed the product's quality and therapeutic potential, suggesting that it could serve as a functional skincare formulation with antioxidant and health benefits.

CONCLUSION

This study successfully formulated and optimized a cocoa-based herbal facial pack by integrating cocoa powder, papaya leaf, guava peel and rose petal powders to harness their skincare benefits. Using Design of Experiments (DoE) through SAS JMP software allowed for a systematic and efficient approach to evaluate the effects of multiple ingredients on key formulation attributes like bulk density and angle of repose. The DoE approach enabled the identification of an optimized blend with desirable physical properties, providing a bulk density of 0.33 g/cm³, an angle of repose of 33° and an 81% desirability score. Moreover, DoE facilitated the development of a robust statistical model with high predictive accuracy (R² values of 0.90 and 0.77), ensuring that the optimization was both data-driven and statistically significant. Based on these results, an optimized formulation was derived and further assessed for organoleptic properties, moisture content, washability, bulk density, angle of repose, total ash value and phytochemical constituents. The evaluation of the optimized blend revealed positive results for chemical tests detecting carbohydrates, glycosides, alkaloids and flavonoids, indicating promising skincare properties.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interest.

ABBREVIATIONS

DoE: Design of Experiments; **FA:** Optimized formulation; **BD:** Bulk Density; **θ:** Angle of repose.

ETHICAL APPROVAL

No ethical approval required as there were no animal or human studies done.

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