

Design, Development and Optimization of Polyherbal Powder Shampoo using 3² Factorial Design

Abstract -

Objective: The aim was to formulate polyherbal powder shampoo containing natural ingredients. It is known to us that natural agents are more safer and effective than synthetic and semi-synthetic agents. Moreover, that synthetic ingredients may cause damage or risk to skin of human. It clears sebum, dirt, dandruff, promotes hair growth, strengthens, and darkens the hair. Also, it acts as a conditioning agent and performs all these actions without affecting or damaging hair. The herbs such as amla, fenugreek, green tea, bramhi, hibiscus, shikakai and aritha have been selected to formulate the polyherbal powder shampoo on the basis of the traditional system and scientific justification with modern uses.

Methods: Polyherbal powder shampoo were accurately weighed, passed through sieve no. 100. It was prepared by mixing in their ascending order of quantities with continuous trituration. Then it was stored in air tight containers and used for further studies. All the nine formulations (F1-F9) were subjected to organoleptic studies, general powder characteristics, physicochemical evaluation, ash and alcohol soluble extractives, moisture content determination, pH determination, cleaning action, foaming capacities, dirt dispersion, wetting time and studies on nature of hair after wash.

Results: All the nine formulations (F1-F9) complies the test. Also, general powder characteristics showed results in specified limits. Physicochemical evaluations, pH determination, ability to remove grease, foaming capacity, dirt dispersion, wetting time and nature of hair after wash were found to yield satisfactory results for F6 formulation.

Conclusion: The present work confirmed the successful preparation of polyherbal powder shampoo by mixing method without using other excipients in different concentrations. F6 is the optimized formula as it showed the best results.

Keywords: Cosmeceuticals, Polyherbal powder shampoo, Organoleptic properties, dirt, cleaning action, foaming index

Comment [VV1]: Abstract should cover only the short summary of your research work in one single paragraph and the Keywords. No need to put separate title for methods, results and conclusions. Method will come under introduction as subtitles.

Comment [VV2]: Please clarify by 'It' what do you mean? Is it the synthetic ingredient that clears sebum...etc or is it the herbal shampoo or just shampoo in general are you talking about?

Comment [VV3]: Same as above, please specify the term 'it'

Comment [VV4]: Define traditional system and provide some scientific justification for choosing these particular ingredients. The objective is not defined clearly. What is the significance, concept, properties of these herbs?

Comment [VV5]: Shampoo was not weighed, polyherbal powders were weighed initially. Shampoo would be formulated later.

Comment [VV6]: Have you also checked the physicochemical properties of the powders you procured?

Comment [VV7]: Have not seen anywhere in your abstract, on what relation are you putting this term?

Introduction -

The term 'cosmetic' originates from the Greek word 'Kosmtikos', defined as 'capable of arrangement, skilled in decoration', giving 'kosmein' to decoration, and 'kosmos' to order, harmony^[1]. Cosmetic is defined as a product that is applied to the human body for cleansing, beautifying, promoting attractiveness or altering the appearance without affecting the body structure or functions^[2]. Cosmetic includes powders, creams, paste, gel, lotions, emulsion, perfumes, lipsticks, fingernail and toe nail polish, eye and facial makeup, permanent waves, colored contact lenses, hair colors, hair sprays, and gels, deodorants, baby products, bath oils, bubble baths, bath salts, soaps, shampoo, sunscreen etc.^[3].

Comment [VV8]: Please remove one comma

Herbal cosmetics are denoted as products formulated using various permissible cosmetic ingredients to form the base in which one (or) more herbal ingredients are used to provide accurate cosmetic benefits^[4].

Hairs are the integral part of human beauty. People are using herbs for cleaning, beautifying and managing hair since the ancient era. During ancient times people used to trimmed, shaped and colored their hairs^[5]. People are aware about their personal hygiene and they keep interest towards it. More over, due to advancement in technology it is easy to maintain personal hygiene^[6]. Now-a-days washing the hair and scalp with shampoo has become a nearly universal practice^[7]. Shampoos are the most widely used hair products. Shampoo contains either synthetic ingredients or herbal ingredients. Shampoos are of various types, like powder shampoo, clear liquid shampoo liquid shampoo, lotion shampoo, solid gel shampoo, medicated shampoo, liquid herbal shampoo etc.^[8] Dandruff occur due to presence of fungi and can be eliminated by using anti-dandruff shampoos. There is a demand of polyherbal formulations in the world market. The natural remedies are more acceptable in market because it is safe and less side effect as compared to synthetic shampoo^[9]. The various ingredients used for the treatment of dandruff are such as zinc pyrithione, salicylic acid, imidazole derivatives, glycolic acid, steroids, and sulphur and coal tar derivatives. However, these agents show certain limitations due to poor clinical efficacy. Although, these drugs are unable to prevent recurrence^[10]. The synthetic shampoo contains cationic, anionic and non anionic surfactant which shows good foaming character. So, all synthetic shampoo shows toxic and caused irritation of eye. The surfactants leave a deposit of sodium, calcium and magnesium salts on the hair shaft. So, these synthetic shampoos shows side effects like hair too dry to handle (or) comb. On the other hand, polyherbal shampoos can be used to avoid this problems^[11,12].

Comment [VV9]: Add the below para highlighted in green here. Then the next sentence will start – Shampoos are the most widely used hair products.

Comment [VV10]: What brings you to put this statement here? Are you focusing of developing polyherb shampoo for dandruff?

Comment [VV11]: Instead of 'and less' write- 'with less side effect'

Comment [VV12]: Again, what is the relevance of this statement here? Are you proving that your formulation prevents from dandruff and it's reoccurrence? If yes, please mention that in order to provide some relevance to your statement regarding dandruff.

Comment [VV13]: Do you mean good foaming character shows toxic and eye irritation? Define the advantages and disadvantages of synthetic surfactants properly and then talk about the objective of your work.

Comment [VV14]: removes

Comment [VV15]: instead write- there is no residue on scalp & hair when rinsed with water

Comment [VV16]: does not cause excessive drying

Comment [VV17]: these days consumers prefer non fragrant or less fragrant products so write accordingly

Comment [VV18]: are

Comment [VV19]: what is the proof / justification? Mention the reason for its safety & minimum side effects

Comment [VV20]: shine, but when it is used for long term

A shampoo powder is said to be ideal^[13] when it

- (1) Effectively and completely remove dust particles and excessive sebum from the scalp and hair,
- (2) Easily remove when rinsed with water
- (3) Leave the hair non-dry, soft and manageable
- (4) Impart a pleasant fragrance to the hair
- (5) Cause no side effect or irritation to the skin or hairs

Natural products showed more safer and causes minimum side effects as compared to synthetic products. This is one reason that the herbal products are among the consumers. Synthetic hair products may provide good hair growth and shine. When it is used for long term causes damages to the hair which may even lead to baldness, premature hair graying and hair loss. Some of the

synthetic shampoo includes sodium directly sulphate, N-nitrosodiethanolamine, EDTA, disodium EDTA, formaldehyde causes toxic etc. Thus synthetic shampoo causes more hair damage as compared to polyherbal powder shampoo^[14].

It has been known to us that women use herbals such as Shikakai, reetha and soil that are natural cleansing agents without harmful effects.

A shampoo is a preparation of surfactant in a suitable liquid, solid or powder which when used under the specific conditions will remove surface grease, dirt and skin debris from the hair shaft without adversely affecting the user's hairs. Polyherbal shampoos are the cosmetic preparations that with the use of traditional ayurvedic herbs are meant for cleansing the hair and scalp just like the regular shampoo. They are used for removal of oils, dandruff, dirt, environmental pollutions etc.^[15]

The advantages of this polyherbal formulation are that it is pure and organic ingredients. Thus, it is free from side-effects and good stability. They are less harmful as compared to commercial shampoos.

In the present study, herbal shampoo was formulated containing various types of herbs and suitable ingredients, such as - Emblica officinalis, Acacia concinna,

Lowsonia inermis, Greentea, Coffee, Aloe barbadensis, Brahmi, Shikakai, Reetha, Methi powder, Ocimum sanctum, Azadirachta indica etc.

Herbal products are affordable and they are having very little negative effects. They are used not just cleaning of hair but for lightening and keeping hair under control.^[16]

MATERIALS AND METHODS OF PREPARATION OF HERBAL SHAMPOO:

Different parts of plants were selected to study hair care property. The plants are Amla (Fruits), Fenugreek (Leaves), Green Tea (Leaves), Brahmi (Root), Hibiscus (Leaf and Flower), Shikakai (Fruits), and Aritha (Fruit). All the required powders of these crude drugs were collected from the local herbal drug store market. These powders were accurately weighed, passed through sieve no. 100 and then mixed in their ascending order of quantities with continuous trituration and stored in airtight containers until it was used for further studies.^[17,18] The preparation formulas are given in table 1.

Experimental Design of Polyherbal Powder Shampoo

A 3² full factorial design was used in the present study. On the basis of extensive literature survey, the amount of Amla (X₁) and the amount of Green tea (X₂) were chosen as independent variables in 3² full factorial design, while Cleaning action and % Foaming Index were taken as dependent variables. Thus to achieve the formulation with desired Cleaning action and % Foaming Index, the formulation prepared by using different combination of Amla and Green tea were optimized and evaluated using 3² - full factorial design.

Full factorial design

This design is useful when a detailed analysis of higher order interactions among the factors is needed. Runs are made at all possible combinations of factor levels. As the number of runs required increases rapidly as the number of factors increases, full factorials are usually

Comment [VV21]: not needed, delete this

Comment [VV22]: causes toxic what???

Comment [VV23]: Not clear on your justification why herbal is better than synthetic? Define the reasons clear and crisp to the point. Do not write in general terms. Why can't herbal powder shampoo cause dryness or premature graying or any other hair problem? Put your answer in this paragraph to justify your statement

Comment [VV24]: Please provide justification. It could be clays may be for scalp clarification but where is soil coming from? Does any research claims this?

Comment [VV25]: Instead write - that contains

Comment [VV26]: Put this entire paragraph in the above para before SHAMPOOS highlighted in green

Comment [VV27]: How?? These days adulteration is very common so how do you know it's purity?

Comment [VV28]: These are botanical herbs so always write them in their botanical form. For eg : *Emblica officinalis*
All herbs should be in Italic form and write botanical names of every herb in your entire paper

Comment [VV29]: Irrelevant statement on lightening and under control. Please either delete or provide necessary justification

Comment [VV30]: Please mention the complete process of trituration, was there any solvent involved to purify the crude powder?

Comment [VV31]: Please mention the actual process of formulation of the shampoo. You have addressed the fundamental behind taking the quantities of powder in each formula but how was it done? Through mixing /sieving or what was the experimental setup?

used when a relatively small set of factors that are known to be important are available or when collecting a large number of observations is feasible. More information is obtained with less work and effects are measured with maximum precision.

The number of experiments required for these studies is dependent on the number of independent variables selected. The response (Y) is measured for each trial.

$$Y = b_0 + b_1 X_1 + b_2 X_2 + b_{12} X_1 X_2 + b_{11} X_{12} + b_{22} X_{22}$$

In The 3^2 - full factorial design 2 independent factors were evaluated, each at 3 levels, and experimental trials were performed for all 9 possible combinations. The design layout of 3^2 - full factorial design as shown in table 1 and table 2.

Two independent variables were selected as below:

$X_1 = \text{Amla(g)}$

$X_2 = \text{Green tea (g)}$

Table 2: Variables with coded and exact values for 3^2 full factorial design

Independent Variables	Low	Medium	High
Coded values	-1	0	+1
$X_1 = \text{Amla(g)}$	1.6	2.0	2.4
$X_2 = \text{Green tea (g)}$	0.8	1.0	1.2
Dependent Variables			
$Y_1 = \text{Cleaning action}$			
$Y_2 = \% \text{ Foam Index}$			

Table 3: Formulations showing factors optimized by 3^2 full factorial design (n = 9)

Ingredients	F1	F2	F3	F4	F5	F6	F7	F8	F9
Amla (Fruits)	1.6	2.0	2.4	1.6	2.0	2.4	1.6	2.0	2.4
Fenugreek (Leaves)	1.1	1.1	1.1	1.1	1.1	1.1	1.1	1.1	1.1
Green Tea (Leaves)	0.8	0.8	0.8	1.0	1.0	1.0	1.2	1.2	1.2
Brahmi (Root)	1.4	1.4	1.4	1.4	1.4	1.4	1.4	1.4	1.4
Hibiscus (Leaf and Flower)	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0

Comment [VV32]: Where is Table 1?

Comment [VV33]: What is the reason for choosing amla and green tea only as independent variable and cleaning action & % foam index as dependent variables? Mention the reason in detail in the paper.

Shikakai (Fruits)	2.8	24	20	26	22	18	24	20	16
Aritha (Fruits)	1.3	1.3	1.3	1.3	1.3	1.3	1.3	1.3	1.3
Total (g)	10	10	10	10	10	10	10	10	10

Comment [VV34]: Missed adding points in between each numbers otherwise total gram will exceed 10

EVALUATION OF HERBAL POWDER SHAMPOO:

1. Organoleptic studies: Organoleptic evaluation studies were performed by taking the samples randomly for the parameters like color, taste and texture.^[19,20]

2. General Powder Characteristics: General powder characteristics included particle size, angle of repose, bulk density and tapped density, which in turn affects properties like flow property. Hence they are evaluated.^[21,22,23]

Comment [VV35]: Have you not checked the hygroscopicity of powder raw material and the finished product?

Particle Size: The particle size of herbal shampoo powder was determined by using microscope method. Place the stage micrometer on the microscope and initially focus on lower power by positioning the object to the center of the object. Focus the object, measure the size of each particle in terms of eyepiece division. Select two points one on left side other on right side. Calculation can be done by using calibration factor.^[17,24]

$$\text{Calibration factor} = \text{Number of stage divisions} / \text{Number of eye piece divisions} * 10$$

Angle of Repose: A glass funnel was held in place with a clamp on ring support over a glass plate. The glass plate was placed on a micro lab jack. Approximately, 10gm of the powder was transferred into the funnel keeping the orifice of funnel blocked by the thumb. As the thumb was removed, the lab jack was adjusted so as to lower the plate and maintain about 2 cm gap between the bottom of the funnel stem and the top of the powder pile. When the powder was emptied from the funnel, the angle of the heap to the horizontal plane was measured with a protractor. The height and radius were measured using a ruler. The angle of repose was thus estimated by the following formula. It is expressed in g/cm^3 .^[17,25]

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

Where,

α = Angle of repose

h = Height of the pile formed

r = Radius of the base of pile

Bulk Density: The bulk density of a powder is the ratio of the mass of an untapped powder sample and its volume, including the contribution of the inter-particulate void volume. Hence, the bulk density depends on both the density of powder particles and the spatial arrangement of particles in the powdered. The bulk density is expressed in g/cm^3 . A volume of 100 ml graduated cylinder was taken and required amount of herbal shampoo powders (F1-F9) was added to the graduated cylinder. This was transferred to bulk density apparatus and bulk density was calculated. It is an important property for packaging and uniformity in the bulk of the product.^[26]

Bulk Density = Mass of powder/ Bulk volume of the powder

Tapped Density: Required amount of herbal shampoo powders (F1-F9) was taken and placed in 100 ml graduated cylinder and tapped for 2 minutes until little change in volume was observed.^[27,28]

It is expressed in g/cm³. The tapped density is calculated by using the following formula:

Tapped density = Mass of the powder/ Tapped volume of the powder

3. Physicochemical evaluation:

Comment [VV36]: Where is non-volatile alcohol soluble matter test?

Extractive values:

Determination of water soluble extractive:

4g each of the dried herbal shampoo powder was weighed and macerated with 100ml of chloroform in a 250 ml conical flask for 24hrs shaking frequently during 6 hrs of shaking and allowed to stand for 18 hrs. Filter into a 50 ml cylinder. When sufficient filtrate has collected; transfer 25ml of the filtrate to a weighed, thin porcelain dish, as used for the ash values determinations. Evaporate to dryness on the water bath and complete the drying in an oven at 105 °C for 6hrs. Cool in desiccators for 30 minutes and weigh immediately.^[18,19] Calculate the percentage w/w of extractive with reference to air-dried shampoo powders (F1-F9).

Water soluble extractive value of the sample = 80*%

Ash value:

This value is used to determine quality and purity of herbal shampoo powder and to establish the identity of it.

Determination of total ash

A flat, thin, porcelain dish or a tarred silica crucible was weighed and ignited. About 2g of herbal shampoo powder formulation (F1-F9) were weighed and taken into a dish. Support the dish on pipe-clay triangle placed on a ring of retort stand. Heat the dish about 7cm above the flame, with the help of a burner, using a flame about 2cm high, heat till vapors almost cease to be evolved, then lower the dish and calculate the percentage of total ash with reference to air dried shampoo powders.^[18,29]

Determination of acid insoluble ash:

After determining total ash value using 25ml of dilute hydrochloric acid, wash the ash from the dish used for total ash into 100ml beaker. Place mere gauze over a Bunsen burner and boil for 5 minutes. Filter through an ash-less filter paper; wash the residue twice with hot water. Ignite a crucible in the flame, cool and weigh. Put the filter paper and residue together into crucible, heat gently until vapors cease to be evolved and then more strongly until all carbon has been removed. Cool using desiccators.^[18,31] Weigh the residue and calculate acid insoluble ash with reference to the air dried herbal shampoo powders (F1-F9).

Moisture content determination:

10g of herbal shampoo powder formulations (F1-F9) was placed in a tarred evaporating dish and kept in hot air oven for 105 ° C. The weight loss was observed at an interval of 15 minutes until constant weight was obtained.^[32,33]

pH:

1g each of herbal shampoo powder formulations (F1-F9) was taken and dissolved in 10ml of water. Their pH was checked with the help of pH paper.^[34]

Skin/eye irritation test:

The eye and skin irritation tests revealed that the herbal shampoo powder shows no harmful effect on skin and eye. This is due to the absence of synthetic surfactants. Most of the synthetic surfactants produce inflammation of the eyelid and corneal irritation. But in this formulation of herbal shampoo powder, the uses of all ingredients were obtained naturally. So it does not produce any harmful effect on skin and eye.^[35]

Cleaning action:

2g of wool was taken and placed in grease; the same was then placed in 200 ml of water containing 1g of each herbal shampoo powder formulations (F1-F9) in a flask and was shaken for 4 minutes. The solution was removed and sample was taken out, dried and weighed. The amount of grease removed was calculated.^[36]

Foaming capacity:

2g of each herbal shampoo powder formulations (F1-F9) were taken in 250ml graduated cylinder, 50ml of water was added and shaken for 5-10 times. The foaming capacities of all the nine formulations (F1-F9) after 1 minute shaking and % foaming capacities of all the nine formulations (F1-F9) for a time period of 60 minutes were performed.^[37]

Dirt dispersion:

Two drops of each 1% herbal shampoo powder formulations (F1-F9) were added in a large test tube containing 10ml of distilled water. A drop of Indian ink was added; the test tubes were stoppered and shaken. The amount of ink in the foam was estimated as none, moderate, heavy.^[38]

Wetting time:

A canvas was taken and cut into 1 inch diameter discs. The discs were floated on the surface of each formulation (F1-F9) of 1% herbal shampoo powder solution and time was noted. The time required for the disc to begin sink was measured accurately and noted as wetting time.^[39]

RESULTS:

Organoleptic properties:

The results of visual inspection for all herbal shampoo powders were observed and evaluated for color, odor, taste and in terms of their appearance, flow property and texture. They somewhat shows distinct change in color. The results were reported in Table 4.

Comment [VV37]: As per BIS, for powder shampoo, you need to mix 1g sample into 9ml of water. Below you have mentioned the pH readings of all formulations so I assume you have measured them on pH meter and not pH paper, so write accordingly. PLEASE REPEAT THE PH VALUES AGAIN AS PER BIS METHOD

PLEASE STRICTLY ADHERE TO BIS SPECIFICATIONS WHILE CARRYING OUT ANY TESTS MENTIONED AS PER BIS

Comment [VV38]: How did you perform this test? Mention that

Comment [VV39]: Instead write : all ingredients were naturally obtained. But again provide justification for its safety through physicochemical analysis data of each ingredient used in the formula.

Comment [VV40]:

Comment [VV41]: How was it calculated mention if you have used any formula?

Comment [VV42]: Please check BIS of Shampoo and see if you have performed the test on the same lines

Comment [VV43]: You can mention the expectation after this statement. "Shampoos that cause the ink to concentrate in the foam are considered poor quality. The dirt should remain in the water portion. Dirt that remains in the foam will be difficult to rinse away and will be redeposited on the hair."

TABLE 4: THE EVALUATION PARAMETERS OF ORGANOLEPTIC STUDIES

Evaluation parameters	Colour	Odour	Texture
F1	Faint Brown	Characteristic	Fine and smooth
F2	Faint Brown	Characteristic	Fine and smooth
F3	Brown	Characteristic	Fine and smooth
F4	Faint Brown	Characteristic	Fine and smooth
F5	Faint Brown	Characteristic	Fine and smooth
F6	Brown	Characteristic	Fine and smooth
F7	Faint Brown	Characteristic	Fine and smooth
F8	Faint Brown	Characteristic	Fine and smooth
F9	Faint Brown	Characteristic	Fine and smooth

General powder characteristics:

The particle size, angle of repose, bulk density and tapped density results were determined. All the herbal shampoo powder shows the results in the specific limits for the respective evaluation parameters. The results were reported in Table 5.

TABLE 5: THE GENERAL POWDER CHARACTERISTICS

Evaluation parameters	F1	F2	F3	F4	F5	F6	F7	F8	F9
Particle size (μm)	1-10	1-10	1-10	1-10	1-10	1-10	1-10	1-10	1-10
Angle of repose ($^{\circ}$)	41.98	41.02	39.35	39.69	38.56	37.50	43.10	41.96	40.12
Bulk density (g/cm^3)	0.36	0.39	0.41	0.38	0.41	0.46	0.40	0.43	0.47
Tapped density (g/cm^3)	0.49	0.55	0.53	0.54	0.53	0.57	0.59	0.61	0.65

Comment [VV44]: Mention the exact particle size?

Physicochemical evaluation: The results of extractive values, ash values and moisture content were reported in Table 4.

Comment [VV45]: Should be Table 6

Determination of pH:

All the formulations (F1-F9) of herbal shampoo powders were acid balanced and all formulations showed slightly acidic pH which may not cause damage to hairs. The results were reported in Table 6.

TABLE 6: THE PHYSICOCHEMICAL EVALUATION STUDIES

Evaluation Parameters	F1	F2	F3	F4	F5	F6	F7	F8	F9
Extractive values:	20.2	32	22	18.8	20.2	32	22	18.8	18.8
Water soluble (%w/w)									
Ash value:	17.8	17.2	16.8	19.8	17.8	17.2	16.8	19.8	19.8
Total ash (%w/w)									
Acid insoluble ash (%w/w)	5.4	4.2	4.6	7	5.4	4.2	4.6	7	7
Moisture content:	91.65	89	89.85	89.4	91.65	89	89.85	89.4	89.4
(%w/w)									
pH	5.6	5.5	5.4	5.4	5.6	5.5	5.4	5.8	5.4

Cleaning action:

As cleaning is the primary action of a shampoo powder, cleaning action was tested on wool in grease. As seen from the results there is a significant difference in the amount of grease removed by all the five batches of formulated herbal shampoo powders. The cleaning action of F1- F9 formulations were observed. F6 formulations showed better ability in the removal of grease compared to other formulations. These were reported in Table 7.

Comment [VV46]: How was it calculated, mention the formula if any used

TABLE 7: THE CLEANING ACTIVITY EVALUATION PARAMETERS

Evaluation Parameter	F1	F2	F3	F4	F5	F6	F7	F8	F9
Cleaning action (%)	78	83	86	91	95	98	79	84	89

Foaming capacity:

All the four formulations of herbal shampoo powders have comparable foaming characteristics in distilled water. The total foam volume of herbal shampoo powders after 1 minute shaking ranged from mild to good was recorded. The average percentage foaming capacities for F1-F9 formulations for a time period of 60 minutes were observed and were reported in Tables 8 and 9.

Comment [VV47]: Check if this was as per BIS method, also check for foam stability

TABLE 8: THE FOAMING CAPACITIES OF ALL FORMULATIONS

Evaluation Parameter	F1	F2	F3	F4	F5	F6	F7	F8	F9
Foaming capacity (%)	Good foam	Good foam	Mild foam	Good foam	Good foam	Good foam	Mild foam	Good foam	Good foam

TABLE 9: THE % FOAMING CAPACITY OF HERBAL SHAMPOO POWDERS

Time (Minutes)	Foaming Capacity								
	F1	F2	F3	F4	F5	F6	F7	F8	F9
0	126	132	141	148	155	156	159	168	183
5	122	130	139	144	152	153	157	166	180
30	119	126	134	138	147	149	154	163	176
60	113	120	130	134	142	146	150	159	173
Average foaming capacity	120	127	136	141	149	151	155	164	178

Dirt dispersion:

Shampoo powders that cause the ink to concentrate in the foam are considered as poor quality, the dirt should stay in water. Dirt that stays in the foam will be difficult to rinse away and will be re-deposited on the hair. The amount of ink in the foam of F1-F9 formulations of polyherbal powder shampoo were evaluated. It was found to be ranged from moderate to light respectively. The results were reported in Table 10.

TABLE 10: THE DIRT DISPERSION PARAMETERS OF ALL THE FOUR FORMULATIONS

Evaluation Parameter	F1	F2	F3	F4	F5	F6	F7	F8	F9
Dirt dispersion	Light	Light	Light	Mode rate	Mode rate	Mode rate	Light	Light	Light

Wetting time:

Wetting time of a substance is a function of its concentration. The wetting time of the F1-F9 formulations were observed. F1 formulation showed less wetting time when compared to other three formulations. The results were reported in Table 11.

TABLE 11: THE EVALUATION PARAMETERS OF WETTING TIME OF ALL THE FOUR FORMULATIONS

Evaluation Parameter	F1	F2	F3	F4	F5	F6	F7	F8	F9
Wetting time (min)	2	2	2	2	2	2	3	3	3
(sec)	10	24	35	41	48	56	15	22	31

Nature of hair after wash:

Nature of hair after wash was carried out with the help of application of polyherbal powder shampoo formulations (F1-F9) to volunteers observed the hairs as soft and manageable.

Statistical Analysis

The results of 3² full factorial design were analyzed. A considerable information was gathered by using statistical design to optimize the formulation. All the responses were fitted to a quadratic model and compatibility of the model was verified by ANOVA, lack of fit and co-efficient of determination (R²). To optimize the responses, every response should be interconnected with each other and the most supportive zone must be required for every response to exclude bias.

Comment [VV48]: Please mention the journal name/ reference number from which you have copied this line. It is from <https://pmc.ncbi.nlm.nih.gov/articles/PMC5783807/>
Please add this reference

Comment [VV49]: Please mention the title as Qualitative Evaluation. Define every volunteer's opinion in a proper column as you have done for other tests. You can perform ease of combing as well.

Desirability function was supported by much literature to optimize the multiple responses [40,41]. The statistical analysis of the factorial design batches was performed by multiple linear regression analysis. The Cleaning action (Y_1) and Foaming Index (Y_2) were selected as dependent variables.

The fitted equations (full model) relating the responses that is, Cleaning action (Y_1) and Foaming Index (Y_2) to the transformed factor are shown in Table 5. The polynomial equations can be used to draw conclusions after considering the magnitude of coefficient and the mathematical sign it carries (i.e. positive or negative). Data were analyzed using Design of Expert version 13. R^2 values for Cleaning action (Y_1) and Foaming Index (Y_2) were 0.9956 and 0.997 respectively indicating good correlation between dependent and independent variables. There was no need to develop reduced models because response variable were significant i.e. $P < 0.05$. The terms with $P < 0.05$ were considered statistically significance and retained in the full model. The results of ANOVA suggested that F values calculated for Cleaning action (Y_1) and Foaming Index (Y_2) were 134.76 and 1945.41 respectively. Calculated F values were greater than tabulated for all dependent variables therefore factors selected have shown significant effects. From the results of multiple regression analysis, it was found that both factors had statistically significant influence on all dependent variables as $p < 0.05$ (Table 6).

Table 12: Summary of regression analysis of the responses

Quadratic Model	R^2	Adjusted R^2	SD	Adequate Precision	p-value
Cleaning action	0.9956	0.9882	0.7454	33.1372	< 0.0010
Foaming Index	0.9970	0.9992	0.5358	131.826	< 0.0001

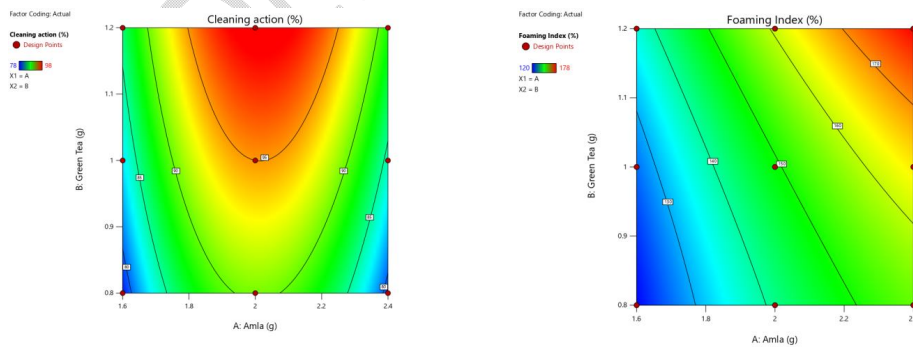


Fig. 1: 2D Response surface contour plot showing effect of Amla and Green tea

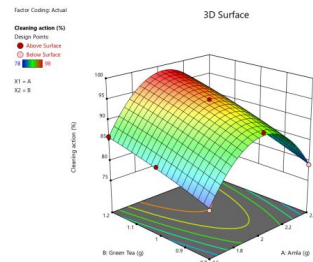


Fig. 2: 3D Response surface contour plot showing effect of Amla and Green tea on Cleaning action

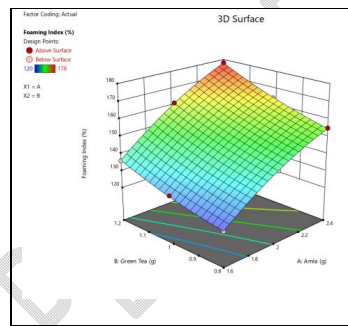


Fig. 3: 3D Response surface contour plot showing effect of Amla and Green tea on Foaming Index

Full and reduced model for Cleaning

The contour plot and 3D response surface graph for cleaning action was observed in Fig. 3 and Fig. 4 respectively and revealed that a corresponding decrease of cleaning action was observed with increase in concentration of Amla (X_1). Moreover, the results also indicated that the effect of Amla (X_1) was less significant as compared to Green tea. From regression, it was observed that X_1 and X_2 was significant model term which affect the effectiveness of polyherbal powder shampoo. Interaction and non-linearity was not observed.

For cleaning action, the significant levels of the coefficients b_2 , b_{12} and b_{22} were found to have P value of 0.0714, 0.2722 and 0.4128. So, it was omitted from the full model to generate a reduced model. The coefficients b_0 , b_1 and b_{11} were found to be significant at $P < 0.05$. Hence, they were retained in the reduced model.

The reduced model for cleaning action was:

$$\text{Cleaning action} = +95 + 0.83*X_1 + 4.17X_2 - 11.50*X_1^2$$

Full and reduced model for Foaming Index

The contour plot and 3D response surface graph for Foaming Index was observed in Fig.3 and Fig. 5 respectively and revealed that a corresponding increase in the Foaming Index was observed with increase in concentrations of Green tea. Moreover, the regression coefficient values of both factors can be concluded that the Foaming index appeared to increase more with an increasing amount of the Green tea and increase the amount of Green tea. Interaction and non-linearity was observed.

For Foaming index, the significant levels of the co-efficients b12, b11 and b22 were found to have P value of 0.0073, 0.0023 and 0.0168. So, it was omitted from the full model to generate a reduced model. The coefficients b₀, b₁ and b₂ were found to be significant at P < 0.0001. Hence, they were retained in the reduced model.

The reduced model for Foaming Index was:

$$\text{Foaming Index} = +149.11 + 19.0*X_1 + 9.83*X_2$$

Validation by Check point batch

A check point batch was prepared to confirm the validity of response surface plot and equation generated by multiple regression analysis. An overlay plot was obtained by adding desired range of evaluation parameters from Design Expert 13. The overlay plot is shown in Fig. 6. Yellow colour area in overlay plot showed optimum concentration range for desired result. A batch was prepared by taking concentration of Amla (X₁) and concentration of Green tea (X₂) observed in overlay plot and the actual responses were evaluated from the prepared check point batch. The overlay plot indicated that optimum concentration which showed the best result. The practically obtained values were closer to the predicted values. Thus, it justified the validation of design.

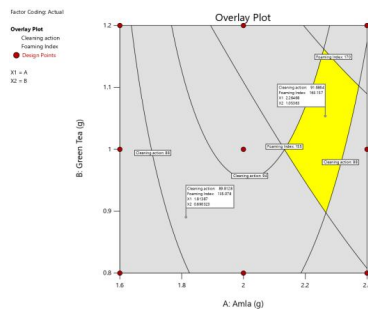


Fig 4: Overlay plot of Check point batch

CONCLUSION:

The polyherbal powder shampoo were successfully prepared, optimized and evaluated using Design Expert software by 3^2 full factorial design. The present investigation showed quick Cleaning action of the polyherbal powder shampoo .Amla and Green tea were used as cleaning and foamingagents. These formulations were evaluated for the parameters like organoleptic properties, powder characteristics, pH, physiochemical evaluation, cleaning action, foaming index, dirt dispersion, wetting time . On the basis of preliminary results, the amount of Amla(X_1) and the amount of Green tea (X_2) were selected as independent variables in 3^2 full factorial design, while Cleaning action and Foaming Index were taken as dependent variables. Multiple linear regression analysis, ANOVA and graphical representation of the influence of factor by contour plots and 3D response surface graphs were performed using Demo version of Design Expert 13. Check point batch was prepared to validate the evolved model. Batch F6 was selected as an optimized batch.

Comment [VV50]: formulated

Comment [VV51]: any studies in any research paper on their foaming property? Because Shikakai and Areetha are better foaming agents than amla and green tea.

Comment [VV52]: Why? Give the reason in brief details.

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