

Formulation and Evaluation of Herbal Shampoo from *Couroupita Guianensis*

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Abstract: *The study was aimed at formulating and evaluating a herbal shampoo from *Couroupita guianensis* leaves. The shampoo contained aqueous extracts of *Couroupita guianensis* leaves, coconut oil, clove oil, castor oil, ethyl alcohol and lemon grass oil. Shampoos are the products which removes surface grease, dust from the hair shaft and scalp. The physicochemical parameters such as color, clarity, pH, and skin irritation, the percentage of solid contents, dirt dispersion, foaming ability and foam stability, wetting time and stability studies were studied using recommended procedures. The evaluation studies determine the characteristic features of specific Herbal shampoo. The organoleptic evaluations of the herbal shampoo showed good results. The pH of the herbal shampoo range between 5 to 6, which was near to the skin pH. The surface tension of developed shampoo was found to be 32.42 dynes/c respectively. The percentage of solid content of prepared shampoo was found to be b/w 28.05- 35.5. In the current scenario, the herbal shampoo is better in performance and safer than the synthetic ones. The main objective of this study was to eliminate harmful synthetic ingredient from herbal shampoo formulation and substitute them with a safe natural ingredient.*

Keywords: Shampoo, Herbal shampoo, physicochemical parameters, foam stability, and retention

I. INTRODUCTION

1.1 Herbal Formulation

Herbal technology is a tool for converting botanical materials into therapeutically useful products and medicines. Home grown innovation encircles all the propelling specialized wildernesses intended to tap bunches of methods of controlling plants around us. As extensive variety of advancements are created to gather the proper things that the plants turn out, including regular colors, biocompost, biopesticides and biofuel. Herbal formulations means a dosage form consisting of one or more herbs or processed herbs in specified quantities to provide specific nutritional ,cosmetic benefits meant for use to diagnose ,treat, mitigate diseases of human beings or animals, alter the structure or physiology of human beings or animals. Herbal formulations contain an active substance or herbal substance or herbal preparation or herbal substance in combination with one or more herbal preparations. Herbal formulation are obtained by subjecting herbal substances to treatments such as extraction, distillation, expression, fractionation, purification, concentration or fermentation include comminuted or powdered. Whole, fragmented or cut plants, plants parts, algae, fungi, lichen in an unprocessed, usually dried form but sometimes fresh were used in the preparation of herbal formulations. Herbal substances are precisely defined by plant part used and the botanical name according to the binomial system (genus, species, variety and author). Different herbal formulations are tinctures, extracts, essential oils, expressed juices and processed exudates. Markers are chemically defined constituents or groups of constituents of a herbal substance, a herbal preparation or a herbal medicinal product which are of interest for control purpose independent of whether they possess any therapeutic activity. Markers serve to calculate the quantity of herbal substance or herbal preparation in the herbal formulation if the markers have been quantitatively determined in the herbal substance or herbal preparations.

1.2 Advantages

- Low risk of side effect
- More effectiveness
- Lower cost
- Widespread availability

1.3 Herbal Shampoos

Herbal 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 and environmental pollution etc. It consists of several functions like lubrication, conditioning, hair growth, maintenance of hair colour, medication.

1.4 Advantages

- Pure and organic ingredients
- Free from side effect
- No surfactants like SLS
- No synthetic additives
- No animal testing
- Earth and skin friendly
- No petroleum based ingredient

Shampoo originally meant head massage in several North Indian languages. Both the words and concept were introduced to Britain from colonial India. The word is derived from Hindi “champo”. Before the advent of shampoos, people typically used soap for personal care. However, soap had the distinct disadvantages of being irritating to the eyes and incompatible with hard water which made it leave dull-looking film on the hair. In 1930s the first synthetic detergent shampoo was introduced, although it had some disadvantages. The 1960s brought the detergent we are using today. Over the years, many improvements have also been made to shampoo formulations.

Shampoo is a hair care product, typically in the form of viscous liquid that is used for cleaning hair. Less commonly, shampoo is available in bar form, like a bar of soap. Shampoo is used by applying it to wet hair, massaging the product into the scalp, and then rinsing it out. Some users may follow a shampooing with the use of hair conditioner. The typical reason of using shampoo is to remove the unwanted build up of sebum in the hair without stripping out so much as to make hair unmanageable. Shampoo is generally made by combining a surfactant, most often sodium lauryl sulfate or sodium lauryl sulfate, with a co-surfactant. Specially shampoos are marketed to people with dandruff, color-treated hair, gluten or wheat allergies, an interest in using an organic product, and infants and young children. There are also shampoos intended for animals that may contain insecticides or other medications to treat skin conditions or parasite infestations such as fleas.

Couroupita guianensis Commonly known as cannonball tree grown in the Indian gardens as an ornamental tree for its beautiful flowers. It also known as Nag lingam tree in Tamil and Kailaspati in Hindi. The trees are grown extensively in Shiva temples in South India. The cannonball tree so called because of its brown cannon ball like fruit. The majority of these trees outside their natural environment have been planted as a botanical curiosity, as they grow very large with distinct flowers. The fruit contain small seeds in a white unpleasant smelling jelly, which are exposed when the upper half of the fruit goes off like a cover. The trees are used to cure cold and stomach ache. Juice made from leaves is used to cure skin diseases and Shamanas of South America have even use tree parts for treating Malaria, while the flowers used to cure cold, intestinal gas formation and stomach ache. This plant is very important in traditional veterinary medicine. Cold relief bam, flower dried and powdered used as snuff, cure scabies, bleeding piles, scorpion poison, dysentery, arthritis, fragrance of flowers used for curing asthma.

II. MATERIALS AND METHODS

Collection and Authentification of Plant Material:

The leaves of *Couroupita guianensis* were collected in August 31, 2019 from Aluva (shiva temple). Botanical identification and authentification were done by Mr. PRADEEP KUMAR, CALICUT UNIVERSITY, KOZHIKODE.

Preparation and Extraction of Plant Material:

After collection, the leaves were initially washed using running tap water to remove dirt or dust and are dried under sunshade for about 30-35 days and made to coarse and to fine powder and stored in a container for further studies. The

leaves were then chopped into small pieces . The powder sample was weighed and stored in airtight container until extraction commenced.

Extraction of Couroupita:

Dry leaves under sunshade for 4 weeks to make it suitable for grinding and also to avoid the degradation of active constituents. Grinding of leaves into coarse powder and put in airtight container. Weigh about 5 gram and perform maceration using ethanol and water solvents. Remove the solvent under reduced pressure at 50 degree Celsius. Filtered with filter paper and allowed to heat until extract is obtained. The extract was collected and incorporated in the shampoo base formula.

III. PHYTOCHEMICAL SCREENING

Qualitative Tests

Table 1: Chemical tests for leaf extracts

Phyto constituents	Test	Observation
AlkaloidsP	Hager's Test: 2ml extract+ few drops of Hager's Reagent	Yellow precipitate
Flavanoids	Ammonia test: Filter paper dipped in alcoholic solution of drug was exposed to ammonia vapour	Formation of yellow spots on filter paper
Carbohydrates	Molisch's test: 2ml extract + 10ml water + 2 drops ethanolic alpha naphthol (20%) + 2ml con sulphuric acid	Reddish violet ring at the junction
Glycosides	Liebermann's test: 2ml extract + 2ml chloroform + acetic acid	Violet to blue to green color
Tannins	Braymer's test: 2ml extract + 2ml water + 2-3 drops ferric chloride (5%)	Green precipitate
Steroids	Salkowski test: 2ml extract + 2ml chloroform + 2ml con sulphuric acid	Reddish brown ring at the junction
Proteins	Ninhydrin test: 1ml extract + 2ml Ninhydrin reagent	Violet precipitate
Saponins	Foam test: 5ml extract + 5ml water + heat	Froth appears
Phenols	Ferric chloride test: extracts were treated with 3-4 drops of ferric chloride	Formation of bluish black colour

ANTIMICROBIAL STUDY

ANTIBACTERIAL STUDY

- Media : Nutrient Agar Media
- Ingredients : Peptic digest of animal tissue 5.00gms/litre
- Sodium chloride 5.00gms/litre
- Beef extract 1.50gms/litre
- Agar 1gm/litre
- Organism used : E. coli
- Drug used : *Couroupita guianensis*
- Standard used : Penicillin

Preparation of Nutrient Agar Media: The Nutrient Agar medium is prepared by mixing the ingredients and is dissolved in distilled water. Heat if necessary.

Sterilization: 28gm Nutrient Agar was dissolved to a 1000 ml standard flask containing distilled water and cover it with cotton. Autoclaved at pressure of 15 psi(121 C)for 15 minutes.

Working: Using horizontal Laminar flow under aseptic condition.

IV. FORMULATION AND EVALUATION OF HERBAL SHAMPOO

Preparation of Shampoo:

Coconut oil, olive oil and castor oil were saponified with potassium hydroxide using reflux condenser. After complete saponification, glycerin was incorporated with stirring. Ethyl alcohol, methyl paraben used as preservative and lemon grass oil used for masking the pungent smell of extract. Six different formulations F1, F2, F3, F4, F5 and F6 were prepared by taking 5%, 10%, 15%, 20%, 25% and 30%w/w of extract respectively as shown in table 1 given below.

Table 1: Composition of formulations of shampoo (alcoholic extract)

Composition of shampoo formulations	F1 %w/w	F2 %w/w	F3 %w/w	F4 %w/w	F5 %w/w	F6 %w/w
Coconut oil	3.0	3.0	3.0	3.0	3.0	3.0
Olive oil	0.9	0.9	0.9	0.9	0.9	0.9
Castor oil	0.9	0.9	0.9	0.9	0.9	0.9
Potassium hydroxide	0.9	0.9	0.9	0.9	0.9	0.9
Glycerin	0.6	0.6	0.6	0.6	0.6	0.6
Ethyl alcohol	1.2	1.2	1.2	1.2	1.2	1.2
Couroupitaguianensis extract	5.0	10.0	15.0	20.0	25.0	30.0
Lemon grass oil	0.015	0.015	0.015	0.015	0.015	0.015
Methyl paraben	0.003	0.003	0.003	0.003	0.003	0.003
Distilled water q.s	30	30	30	30	30	30

Table 2: Composition of formulation of shampoo(aqueous extract)

Composition of shampoo formulations	F1 %w/w	F2 %w/w	F3 %w/w	F4 %w/w	F5 %w/w	F6 %w/w
Coconut oil	3.0	3.0	3.0	3.0	3.0	3.0
Olive oil	0.9	0.9	0.9	0.9	0.9	0.9
Castor oil	0.9	0.9	0.9	0.9	0.9	0.9
Potassium hydroxide	0.9	0.9	0.9	0.9	0.9	0.9
Glycerin	0.6	0.6	0.6	0.6	0.6	0.6
Ethyl alcohol	1.2	1.2	1.2	1.2	1.2	1.2
Couroupitaguianensis extract	5.0	10.0	15.0	20.0	25.0	30.0
Lemon grass oil	0.015	0.015	0.015	0.015	0.015	0.015
Methylparaben	0.003	0.003	0.003	0.003	0.003	0.003
Distilled water q.s	30	30	30	30	30	30

V. CHARACTERISATION

Prepared 1%v/v solution of shampoo formulations by mixing, 2 milliliters (40 drops) of shampoo with sufficient distilled water. The shampoo was taken in the beaker and then slowly added distilled water. After thorough mixing of shampoo and water all the evaluation parameters were determined.

- **Physical Appearance:** Physical appearance can be determined by visual inspection method.
- **PH:** 1% of shampoo solution was used to determine the pH by using the pH meter.
- **Percent of Solids:** Weighed a clean dry evaporating dish and recorded the initial weight of the evaporating dish. Shampoo formulation was taken in the evaporating dish. Weighed the dish and shampoo and recorded initial weight of shampoo and dish. Calculated the exact weight of the shampoo only and recorded the initial weight of shampoo only. Put the evaporating dish with shampoo on the hot plate until the liquid portion has evaporated. After drying, weighed the dish and shampoo solids and results were noted.
- **Foam Formation (Shake Test):** Took 15ml of the solution shampoo in a 50ml graduated cylinder and recorded the volume. Then covered the cylinder with hand and shake 10 times. The total volume of the content was recorded after shaking. Calculated the volume of the foam and recorded the size of the bubbles.

- **Foam Quality and Retention:** Immediately after the Shake Test time was recorded. Recorded the volume of foam at 1 minute intervals for 5 minutes as depicted in fig 1.
- **Surface Tension:** Prepared shampoo solution by mixing 2 ml of shampoo with 200ml of distilled water. The shampoo was taken in the beaker and then slowly added distilled water. After thorough mixing of shampoo and water the surface tension was measured by using Stalagmometer.
- **Skin Irritation Test:** Applied the solution of prepared shampoo on skin and kept for 5 min and observed for redness of skin and irritation there, were no any red coloration and the irritation to the skin.
- **Visual Stability:** The prepared shampoo was tested for the visual stability for 21 days at room temperature with relative humidity 65, and observed for colour change and Ph. There were no changes in colour and pH of shampoo within 21 days and no any phase separation between oil and water.
- **Viscosity:** Viscosity was determined by using the Ostwald viscometer.

VI. RESULTS AND DISCUSSION

EXTRACTION OF ACTIVE INGREDIENT

Extraction was carried in conical flask using distilled water.

PHYTOCHEMICAL SCREENING

The screening was carried out as follows:

Table 3: Phytochemical screening

S. No	Name of the Compounds	Name of the Test	Aqueous extract	Alcoholic extract
1.	Carbohydrates	Fehling's Benedict's	+ +	+ +
2.	Alkaloids	Mayer's Hager's DragenDroff's	- - -	- - -
3.	Steroids	Chloroform+ Acetic acid+ sulphuric acid	-	-
4.	Saponins	Foam test	+++	+++
5.	Tannins	10% Lead acetate 5% Ferric chloride 1% gelatin	- + +	+ + -

+++ - High rich amount, + - Minimum amount, - Absent

Physical Appearance

The prepared shampoo showed good characteristics and the appearance on visual inspection of the formulation. The results of visual inspection of series of formulations are listed in Table 1 and Table 2. As can be seen, all formulation was clear and had the good characteristics with respect to foaming. The main challenge with this formulation was of characteristic colour of *couroupitaguianensis* extract. With less concentration in both aqueous as well as alcoholic extract which is free from characteristic smell and were acceptable, but as the concentration increases in both the extracts the smell were observed.

Table 4: Evaluation of Formulations for physical appearance, pH, solids and viscosity (aqueous):

Formulation Code	Physical appearance	pH	Solids (%)	Surface tension (dy./cm)	Viscosity (Poise)
F1	Clear, no characteristic smell	6.01±0.1	13.21±0.02	28.43±0.32	3.5±0.2

F2	Clear, good foaming, no smell	6.00±0.002	13.58±0.08	27.25±0.45	3.1±0.0
F3	Clear, slight smell, good foaming	5.82±0.03	14.37±0.04	28.34±0.12	2.3±0.5
F4	Clear, slight smell, foaming	5.75±0.01	14.77±0.06	28.5±0.21	2.2±0.7
F5	Clear, foaming, characteristic smell	5.53±0.04	15.25±0.07	29.23±0.13	1.8±0.1
F6	Clear, good foaming, characteristic smell	5.01±0.02	15.50±0.04	29.87±0.2	1.4±0.2

pH:

The pH of the shampoo has been shown to be important for improving and enhancing the qualities of hair, the current trend to promote shampoos of lower pH is one of the ways to minimize damage to the hair. Mild acidity prevents swelling and promotes tightening of the scales, thereby inducing shine. All shampoos were acid balanced and were ranged at pH near to the skin PH. On increasing the extract concentration the acidity of the formulation increased.

Solid Content

Shampoo with high solid content will be very difficult to rinse and hard to work with the hair. The prepared shampoo contain 15.50% of solid content. Thus, they considered easy to washout when having less solid content during preparation of shampoos. The result of percent of solids contents is tabulated in Table 3. As a result, they were easy to wash out. If the shampoo has too many solids it will be hard to work into the hair or too hard to wash out.

Table 5: Evaluation of Formulations for physical appearance, pH, Solids and viscosity (alcoholic)

Formulati on Code	Physical appearance	pH	Solids	Surface tension (dy./cm)	Viscosity (Poise)
F1	Slight turbid, no characteristic smell	6.05±0.012	14.54±0.12	21.02±0.05	4.8±0.08
F2	Slight turbid, slight foaming, no characteristic smell	6.002±0.14	14.45±0.132	21.42±0.06	4.5±0.04
F3	Turbid, foaming, slight smell	5.03±0.046	14.35±0.24	21.89±0.08	3.8±0.04
F4	Turbid, foaming, slight smell	5.012±0.03	13.89±0.03	21.97±0.12	3.4±0.03
F5	Turbid, good foaming, characteristic smell	5.001±0.023	13.65±0.023	22.24±0.21	2.5±0.01
F6	Turbid, good foaming, characteristic smell	5.00±0.012	13.32±0.003	22.26±0.43	2.1±0.03

Foaming Ability and Foaming Stability

For the consumer point of view, foam stability is one of the important needs of shampoo. Important parameter that was considered in the shampoo evaluation was determination of foaming stability. The prepared shampoo generates uniform, small sized, compact, denser and stable form.

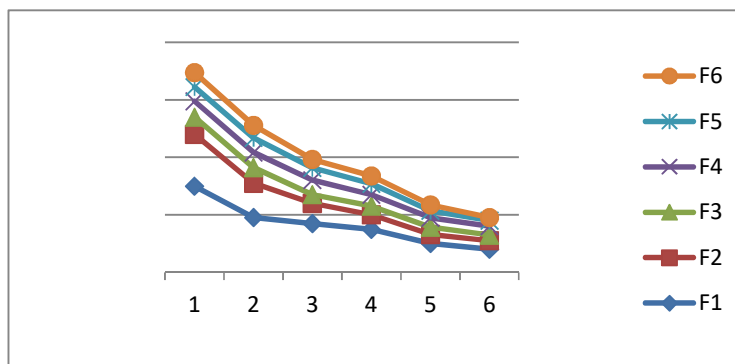


Figure 13: Change in Foam retention volume with respect to time (alcoholic)

The foam quality and stability of formulations were observed as depicted in Table 2, the volume changed from 30 to 8 for F1, 18 to 3 for F2, 6 to 2 for F3, 5.5 to 3 for F4, 5 to 2 for F5, 5 to 1 for F6. All the formulations showed good foam

quality and retention but the range was $F_6 > F_5 > F_4 > F_3 > F_2 > F_1$. As foam production and retention is very important with respect to consumer palatability and acceptance hence it becomes important evaluation parameter for the formulation

Surface Tension:

The surface tension reduction in the prepared shampoo was found to be 29.87 dynes/cm (Table.3). One of the mechanism in the detergency property is the lowering of surface tension, this will be the marker for a good detergency effect of the shampoo. It has been mentioned that a proper shampoo should be able to decrease the surface tension of pure water. As observed in Figure 2 maximum reduction was by formulation F2. The reduction in surface tension of water by the herbal shampoos is an indication of their good detergent action.

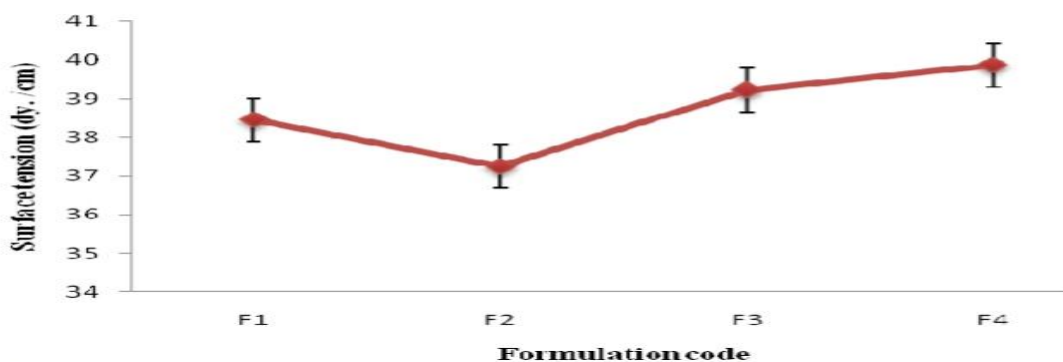


Figure 14: Surface tension of various shampoo formulations (aqueous)

The viscosity was in the range 1-3 poise which indicates the great fluidity, which makes the formulation easy to apply on and easy to spread on hair. Formulated shampoos did not show any skin irritation on application showing the safety of the constituents present.

ANTIMICROBIAL STUDY

Antibacterial study:

The antibacterial activity of the plant extract *Couroupita guianensis* was observed using the Disc Diffusion method. It is done by measuring the diameter of the growth inhibition zone of *E. coli* bacteria in the culture media. The results are shown in the following table:

Table 6: Antibacterial study

SL.NO	ORGANISM USED	SAMPLE	ZONE OF INHIBITION (cm)	
			TEST	STANDARD
1	E.COLI	<i>Couroupita guianensis</i>	2	2

The result obtained from the antimicrobial study shows that the plant has significant antibacterial activity. The strain used for antibacterial activity was *E.coli*.

From the evaluation test given below the extract (aqueous) of *Couroupita guianensis* is much more effective than that of aqueous one.

Table 7: Evaluation study for aqueous extract.

Evaluation test	Formulated shampoo (aqueous)
Colour	Light brown
Transparency	Clear
Odour	Good
Ph	5.01
Solid content	15.50
Foam type	Dense, small
Surface tension	29.87

Here the aqueous extract containing the sixth formulation of the given plant which would perform the antimicrobial activity against various disease condition as a Herbal shampoo.

Stability Study:

Stability and acceptability of organoleptic properties (odor and color) of formulations during the storage period indicated that they are chemically and physically stable. The stability of herbal formulation is listed in table no. 10.

Sr. No	Parameters	01 month	02 month	03 month
01	Physical appearance/visual inspection	Clear	Clear	Clear
02	pH	5.21	5.23	5.31
03	Solids contents (%)	21.41	23.15	24.63
04	Surface tension measurement (dy. /cm)	32.77	31.69	34.70
05	Rheological evaluations (cps)	1.21	1.24	1.27
06	Detergency ability (%)	64.21	66.21	53.77
07	Foaming ability and foam stability (ml)	168	178	168

VII. SUMMARY AND CONCLUSION

The herbal shampoo preparation was formulated based upon traditional knowledge and emphasis was to formulate a stable and functionally effective. The formulated shampoos were not only safer than the chemical conditioning agents, but also greatly reduce the hair loss during combing as well as strengthen the hair growth. The pH of the shampoos was adjusted to 5.5, to retain the acidic mantle of scalp. It was found to be harmless, more effective and economical