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RESEARCH ARTICLE

MICROENCAPSULATION OF FENUGREEK SEEDS OIL AND CURRY LEAVES OIL USING SIMPLE COACERVATION AND ITS APPLICATION IN SHAMPOO

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ABSTRACT

There is new trend in market for cosmetics prepared from herbal extracts. Natural and ecofriendly products are becoming increasingly popular. Fenugreek seeds and curry leaves are cheap source of herbal extract having very good properties for hair growth, hair strengthening, reducing hair loss and retaining hair conditioning. The essential oils of curry leaves and fenugreek seeds are volatile, it need to be coated with stable coating material for proper stability during storage and processing. In our present work we extracted the essential oil of fenugreek seeds by maceration method and curry leaves by hydrodistillation method and encapsulated the oils using coacervation technique. Gelatin is used as a coating material. The core to the wall ratio (C:W) was varied and parameters were optimized. 1% of microencapsulated oil was added in shampoo and the stability and sensory analysis of the shampoo were determined. The ratio of 1:3 (C:W) is found to be best among all other combination with maximum capsule yield (84.9 percent).The coacervates were stable for 4 months at 450C. Several tests such as colour, odor, visual inspection, pH, wetting time, % of solid contents, foam volume and stability, surface tension were performed to determine the physicochemical properties of prepared shampoo. The formulated shampoo has shown the better foaming productivity and the wetting effect of shampoo was taken 183 sec and all properties are within the standard limit of shampoos, which indicates its proper quality compared to other shampoos in market. Based on all the data, a good quality of shampoo can be formulated and can be introduced in the market.

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INTRODUCTION

Natural cosmetics are popular one all over the world as they are having better purity, safety and efficacy. Natural and eco-friendly products are becoming increasingly popular amongst the health and environmentally conscious shoppers of today. Everyone nowadays is realizing that continuous exposure of harmful chemicals to the hair causes hair damage. Hence herbal shampoos infused with natural oils and herbal extracts maintain moisture and improve the overall conditioning of hairs. Because it contains all natural ingredients it is a non-allergenic product which makes it suitable for all skin types including sensitive and allergy prone skin. Natural shampoos feature a more natural and mild aroma. Natural shampoos are environmentally friendly as they contain bio-degradable materials rather than harsh chemicals. Hair tonic and conditioners are formulated as shampoos contain a large number of cosmetic products (Wu *et al.,* 2010). Curry leaves are a treasure-trove of essential nutrients required for healthy hair growth.

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Rich in antioxidants and amino acids, they work to reduce hair fall, foster hair growth and strengthen hair shafts. Amino acids are essential for hair fall prevention. Antioxidants are known to combat free radicals, moisturise the scalp and aid in removal dead scalp flakes. Curry leaves are also a rich source of beta-carotene and proteins; beta carotene prevents hair loss while proteins prevent hair thinning. The fenugreek extract has important role in reducing hair loss and its conditioning mode (Wichtl, 1994).Fenugreek's proteins, nicotinic acids and large amounts of lecithinare extremely effective in strengthening the hair from the roots and treating follicular problems.

The seeds contain a hormone antecedent that improves hair growth and help in rebuilding the hair follicles. The natural tonic helps in moisturizing the hair and bringing back the luster and bounce. Therefore, this study was designed to formulate shampoos containing fenugreek extract and curry leaves oil and its physicochemical properties were studied. During storage or processing conditions, the oils from fenugreek seed and curry leaves might get evaporated or get reacted with other chemical components or get oxidized with air. Therefore, they should be protected or shielded from surrounding conditions.

Microencapsulation is the coating of active ingredient, called core material, within wall material, also called as shell material. (Xiao Jun-xia *et al.,* 2010). Microencapsulation forms a chemical, physical and physicochemical barrier. It prevents the core material from oxidation, evaporation, interaction with matrix composition etc. It is more attractive in making flowing liquid flavour into dry, stable, free flowing particles that are easy to handle. Coacervation is a phenomenon of separating liquid into polymer rich phase and polymer poor phase. The polymer rich phase is referred as the coacervate. Gelatine is widely used as a coating material in coacervation method(Vahabzadeh, 2004). It is a mixture of proteins and peptides obtained from animals. Due to its various properties, like colourless, solubility in water, britt flavourless, it is very efficient to use as a coating material in foods. The present study deals with the encapsulation of fenugreek seed extract and curry leaves oil within gelatine as a wall material using coacervation technique and utilization of microencapsulates in shampoo.

MATERIALS AND METHODS

Materials

Core materials, Fenugreek seeds and Curry leaves were procured from local market. Wall material, Gelatin and Phase inducing agent, purified anhydrous sodium sulphate were procured from S.D. Fine Chemicals Ltd. Mumbai. Hardening agent, Glutaraldehyde solution $C_5H_8O_2$ (25 percent w/w) was procured from S.D. Fine Chemicals Ltd. Mumbai.

Methods

Extraction of oils Isolation of Essential oil of Curry leaves by Hydro distillation

25gm of curry leaves were hydro distilled with 250ml of distilled water in Clevenger type apparatus without organic solvent for 5-6hr. The essential oil was dried over anhydrous Na_2SO_4 and stored in dark color glass bottle. The oil obtained through hydro-distillation gives rise to about 12.56% yield of curry leaves oil.

Isolation of Essential oil of Fenugreek seeds oil by Maceration

The extraction was performed by maceration method.100 g of the seeds were weighed and it was extracted using ethanol (50%) within 72 h. The Solvent was removed by rotary evaporation at temperature below 50°C.

Coacervation preparation

In coacervation gelatin was used as coating material. Gelatine was rehydrated for overnight at 10°C and 10 percent concentration of gelatine was dispersed in water at 45°C with continuous stirring. Fenugreek seeds oil and curry leaves oil (the core material) were added seperately into gelatine dispersed solution with continuous stirring at 40°C. Then, sodium sulphate solution (20 percent activity) (0.34 coating material) was added at 50 to 60 percent by final volume, in order to induce the coacervation. This system was cooled to 4°C. It was necessary to insolubilize the coacervate capsules suspended in the equilibrium liquid by the addition of a hardening agent such as glutaraldehyde and adjusting the acidic pH (Approx 4.0). The resulting microcapsules were washed with distilled water, dried and stored.

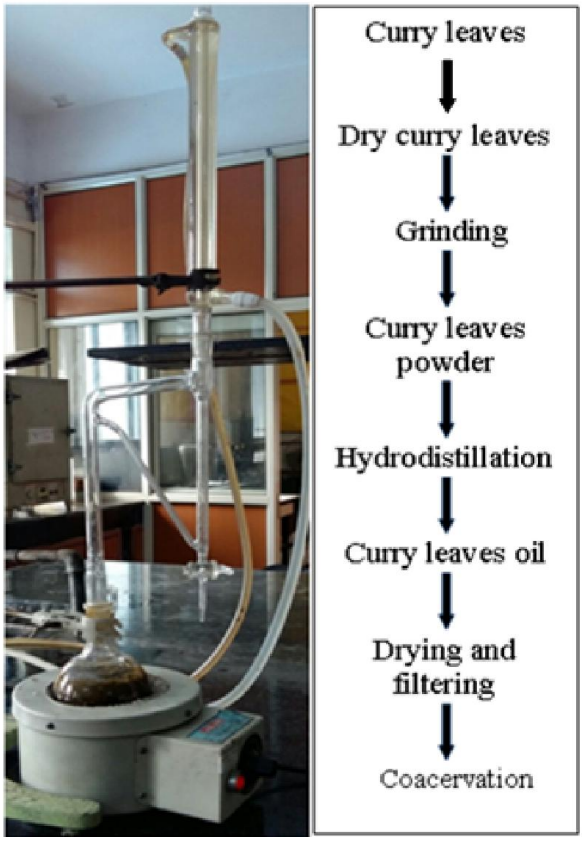

Fig. 1. Hydrodistillation of curry leaves

Fig. 2. Maceration of fenugreek seeds

Formulation of shampoo

To formulate a base of shampoo specific amount of sodium lauryl sulfate, cocobetain and cocodiethanol amide, citric acid and salt were added to an aqueous de-mineralized water solution containing methyl paraben.

Evaluation of Coacervation

Capsule yield and Encapsulation efficiency

The overall yield of the process is defined as the ratio of mass of the final microcapsules obtained to the initial mass of wall and core material used in the formulation. The mass used in the calculation was an adjusted mass i.e. (total mass – water content). The capsule yield was expressed in terms of percent yield. The results for coacervate yield are summarized in table 1. Data shows the optimizing parameters for the coacervation on the basis of capsule yield. In the present study fixed ratio of 10 percent w/v concentration of gelatine was used along with different levels of core to wall ratio to prepare the coacervate particles. A wide range of variation was observed in the capsule yield when the core to wall ratio was varied. The ratio of core to wall i.e. 1:3 gave maximum yield (84.9 percent). Encapsulation efficiency is one of the imperative parameters in encapsulation. It is calculated by following formula 1. Encapsulation Efficiency (percent) = (Total Oil content – Surface Oil content)/ (Total Oil content) Encapsulation efficiencies are given in Table 1. Also, proper performance of the encapsulation in coacervation was achieved by dropwise addition of the phase inducing agent (sodium sulphate) at 50-60 percent dosage. Excess addition resulted into the formation of the solid mass and hence affecting the yield and quality of the coacervates.

Table No. 1. Optimization of coacervate on the basis of the capsule yield

Sr. No.	Core material (C)	Wall material (W)	C:W Ratio	Yield (%)	Encapsulation efficiency (%)
1	1	1	1:1	56.8	12.2
2	1	2	1:2	79.2	53.7
3	1	3	1:3	84.9	64.5
4	2	1	2:1	39.0	10.0
5	3	1	3:1	42.4	22.6

Microscopy

The structure, the shape and the formation of the microcapsules were determined by mounting the coacervates on the microscope slide and then observing using a light microscope (KRUSS Optronic, Germany), lens 10x/0.25. Microscopy, the light microscope was used to see the integrity of prepared spheres. Also, the rate of addition of phase inducing agent was decided by microscopy. Upon the visualization of the slides under the light microscope if the phase separation was observed the addition of the sodium sulphate was stopped. The technique was basically used to study the morphology of the wall as well as the coacervate phase formation

Bulk density

Bulk density was determined by tapping method. 10 gm of capsules was loosely weighed in a 100 ml graduated cylinder. Cylinder with the capsules was tapped on soft surface. The final volume was recorded.

Bulk density was calculated by dividing the sample weight by the volume. It has been observed that there is no correlation between C:W ratio and bulk density of microcapsule. The bulk densities range from 0.299 g/ml for 1:5 ratio to 0.535 g/ml for 1:3 ratio. The bulk density of the sample having C:W ratio of 1:2 was found to be 0.372 g/ml.

Evaluation of Herbal Shampoo

Physical appearance/visual inspection

The formulations prepared were evaluated in terms of their clarity, foam producing ability and fluidity.

Determination of pH

The pH of 10% shampoo solution in distilled water was determined at room temperature 25°C. The pH of a basic shampoo (formulation without extract) and shampoo with extract were measured by pH meter. The measurements were performed in triplicate and mean values and standard deviation (SD) were used for analysis. The experiment was performed in 1% shampoo solution at 25°C

Solubility

Solubility is defined as the ability of the substance to soluble in a solvent. One gram of the powder is weighed accurately and transferred into a beaker containing 100 ml of water. This was shaken well and warmed to increase the solubility. Then cooled and filter it, the residue obtained is weighed and noted.

Determine percent of solids contents

A clean dry evaporating dish was weighed and added 4 grams of shampoo to the evaporating dish.

The dish and shampoo was weighed. The exact weight of the shampoo was calculated only and put the evaporating dish with shampoo was placed on the hot plate until the liquid portion was evaporated. The weight of the shampoo only (solids) after drying was calculated.

Wetting time

The canvas was cut into 1-inch diameter discs having an average weight of 0.44g. The disc was floated on the surface of shampoo solution 1%w/v and the stopwatch started. The time required for the disc to begin to sink was measured accurately and noted as wetting time.

Foaming ability and foam stability

Cylinder shake method was used for determining foaming ability. 50 ml of the 1% shampoo solution was put into a 250 ml graduated cylinder and covered the cylinder with hand and shaken for 10 times.

The total volumes of the foam contents after 1 minute shaking were recorded. The foam volume was calculated only. Immediately after shaking the volume of foam at 1 minute intervals for 4 minutes were recorded.

RESULTS AND DISCUSSIONS

Optimization of core to wall ratio

The ratio of both the oils to gelatine ranging from 1:1 to 1:3. Experimental design of core to wall material ratio used for coacervation microencapsulation is shown in the Table 1. Hence, it was observed that shampoo base with coacervates was stable for 4 months, with no change in appearance and good odour profile at 45°C, which would be equivalent to 1 year stability at ambient temperature.

Formulation of shampoo

To formulate a base of shampoo specific amount of sodium lauryl sulfate, cocobetain and cocodiethanol amide and citric acid were added to an aqueous de-mineralized water solution containing methyl paraben. Sodium lauryl sulfate and coco diethanol amide, citric acid and salt were added into demineralized water and were mixed gently to avoid making any foam. Then, methyl paraben was added and mixed gently. Previously prepared coacervates of both the oils were added to the basic shampoo formulation (1%), after which it was mixed gently and then topped up with water where needed.

Table No. 2. Formulation of base for shampoo

Sr. No.	Ingredients	Quantity (%)
1	Sodium lauryl sulphate	44
2	Cocodiethanol amide	2.5
3	cocobetain	2.0
4	Methyl paraben	0.2
5	Citric acid	0.15
6	water	51.25
7	salt	0.2

Table No.3. Physiochemical evaluation of formulated shampoo

Evaluation Tests	Results
colour	white
odor	good
transparency	Milky white
pH(10% solution)	7.01
% solid contents	21.65
Foam volume (ml)	112
Foam Type	Small, dense
Wetting time (sec)	183
Surface Tension(dynes/cm)	32.64

Physical appearance/visual inspection

A shampoo should have good appealing physical appearance. The formulated shampoo was evaluated for physical characteristics such as color, odor and transparency (Table 3). The prepared shampoo was white in colour and had good odor

pH

Most shampoos are slightly alkaline to protect the hairs from damaging. The pH of shampoo also helps in minimizing irritation to the eyes, enhances the quality of hair.

The pH of formulated shampoo was found to be nearly neutral (7.01) and is presented in (Table 3).

% of solid contents

Good shampoos usually have 20-30% solid content as it is easy to be applied and rinse out from the hair. If it doesn't have enough solid it will be too watery and wash away quickly, similarly too many solids will be hard to work into the hair or too hard to wash out. The percent solid contents of all the tested shampoo was found within the range of 21.65 % and are expected to wash out easily.

Wetting time

The wetting ability of a surfactant is used to test its efficacy. The wetting time of the formulated shampoo shampoo was found to be 183 seconds. It can be concluded that the formulated shampoo exhibited good wetting time so, it contains minimum concentration of detergents.

Foaming ability and foaming stability

Foaming is very important parameter in evaluation of shampoo. The foams generated by formulated shampoo were small, uniform and stable similar to commercial samples. The higher foaming property of formulated shampoo may be due to the saponins in Fenugreek seeds extract.

Surface tension

Lesser surface Tension indicate stronger cleaning ability of the shampoo. The formulated shampoo reduced the surface tension to 32.64 dyne/cm indicating good cleaning ability.

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Conclusion

Good quality of essential oil is obtained by using hydro distillation and maceration method under laboratory conditions. The main component of fenugreek seeds oil and curry leaves essential oil are found to be very effective in formulated herbal shampoo. In the present study, microencapsulation of essential oils within gelatine (coating material) using coacervation technique was successfully carried out. The optimization was done by varying core to wall material ratio. During the characterization of microcapsules, it was found that 1:3 (C:W) ratio gives the highest capsule yield i.e. 84.9 percent.

The stability of microcapsule in shampoo base is very significant. It was stable for 4 months at 45°C which is equivalent to 1 year at ambient condition. Several tests were performed to evaluate and compare the physicochemical properties of the prepared shampoo. The prepared shampoo showed comparable result with that of marketed shampoo for quality control tests. Thus a good quality of herbal shampoo is formulated which will be beneficial for hair growth and strengthening.

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