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COMPARATIVE PHYTOCHEMICAL SCREENING OF DIFFERENT EXTRACTION TECHNIQUE AND FORMULATION, CHARACTERIZATION OF HERBAL LIPSTICKS CONTAINING BETA VULGARIS LINN

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ABSTRACT

The comparative study of different extraction techniques and phytochemical screening of *Beta* Vulgaris performed. The objective achieved were to use natural dyes, to prepare natural lipstick formulation, to reduce cost of product. to avoid side effect of synthetic dye, to avoid irritation of synthetic and chemical dyes to lip skin, to avoid drying of lips, to use and beautification purpose or as cosmetics which is widely used in women, to avoid hydration of lipstick, to know different method of extraction. Extraction of plant by sonication and by soxhlation done. Selection of lipstick ingredients and selection of method of preparation of lipstick carried out. Selection of lipstick mold and preparation of lipstick along with evaluation of lipstick done before filling of lipstick in the container.

KEYWORDS: *Beta* Vulgaris, lipstick formulation, Extraction, soxhlation.

INTRODUCTION

Lipstick is a type of cosmetics which is applied on lip to color it. It is mainly used by female; in the early period general people did not use lipstick. Only actresses used lipstick. In 1884 a French perfume company introduced commercial lipstick. But it took a long time to attract the general population. By 1921 lipstick was started to be used widely in Britain. In the united states, carmine dye was used as lipstick. But it could not give a natural look. When carmine dye was mixed with oil and wax base in 1890s, then it was capable to make a natural look. After that, many women started to use lipstick. The main ingredients of lipstick are pigments, oil, waxes and emollients. These are used for color, texture and protection of the

lips. In the early period, limited colors of lipstick were produced. Red was the only color of lipstick in its verge. There were hardly some pink colored lipsticks in the market, but nowadays, different colored lipsticks are available in the market. Red, pink, orange, violate, wood color even black colored lipsticks are also seen. If you want to make your face attractive, you have to put lipstick. without lipstick your make up will not be completed. but everybody must have a good sense about choosing the color of lipstick. When you apply lipstick, firstly you have to consider the color of your dress. you have to choose light colored lipstick if your dress's color is light and you have to choose dark colored lipstick if your dress's color is dark. for this reason, you should have a good collection of different colored light and dark lipsticks. similarly you should also consider the place where you are going on. If you are going to a party, use dark colored lipstick and light colored lipstick suits best if you are going to your workplace. Which types of lipstick should you chooses; matte or glossy? you have to consider the weather of your area. Use matte lipstick when it is summer. You can make your lipstick matte by mixing some powder with it and use glossy lipstick in winter as our skin become dry in this season. To make your lipstick glossy you can mix some lip gel with it. Now many women use lipstick with SPF (sun protection formula) to save their lip from sunburn. Many women also use water proof lipstick so that their lipstick will not bevanished even after eating. these types of lipsticks are mainly used in beauty parlor. They use it in bridal make up.

The beet (***Beta vulgaris***) is a plant in the Chenopodiaceae family which is now included in Amaranthaceae family. It is best known in its numerous cultivated varieties, the most well-known of which is the root vegetable known as the beetroot or garden beet. Beetroot (*Beta vulgaris*) is the main source of natural red dye, known as "beetroot red". Betanine is the main component of the red colorant extracted from *Beta vulgaris.* The roots are most commonly deep red-purple in colour, but come in a wide variety of other shades, including golden yellow and red-and-white striped. The color of red/purple beetroot is due to a variety of betalain pigments, unlike most other red plants.

MATERIALS AND METHODS

Methods of Extraction

a) Soxhlet extraction

When a compound low solubility needs to be extracted from a solid mixture a Soxhletextraction can be carried out. The technique places a specialized piece of glassware in

– between a flask and a condenser. The refluxing solvent repeatedly washes the solid extraction the desired compound into the flask. Soxhlet extraction was carried out for colorant identification. In this work dried plant parts were put into thistle of soxhlet extractor and methanol was used as solvent. Temperature of the instrument was maintained well under boiling point to the used solvent. Several cycles of solvent were run so as to extract all the compounds from plant parts.

b) Sonication methods

Sonication is the act of applying sound energy to agitate particles in a sample, for various purposes. Ultrasonic Frequencies (>20 kHz) are usually used, leading to the process also being known as ultrasonication or ultra-sonication.

MATERIALS USED

Chemicals: Benzene, Chloroform, Acetone, Ethanol, Water.

Apparatus: Ultra sonication, Soxhlet apparatus, Hot air oven, Heating mantle, weighing balance, colorimeter, pH meter.

Glass wares: RBF, Condenser, beaker, pipette, conical flask, petri dish, stirrer, test tubes.

Fig: 1 Sonication Extractions

Procedure for Sonication Extraction

100gm of beet and 10 gm of dried powder of are taken and then taken beet and it is crushed in girder then it was taken in 250ml beaker to it add 50 ml of 1.benzene. 2.Chlorofrom. 3.Acetone. 4.Ethanol.5.Water and kept it for extraction in sonicator. For 1 hour extract are

kept in a different petriplates for evaporation in a room temperature. Then after evaporation kept the petriplate in the refrigerator. Thenfinaly the coloured extract is taken ou

Extract Made By Sonication Method

Fig. 2

Table 1: Percent Yield of extract by sonication

Sr no	Solvent	% Yield (W/W)
1	Benzene	5.5
2	Chloroform	12.6
3	Acetone	21.86
4	Ethanol	79.4
5	Water	18.16

Soxhlet Apparatus

Figure: no 3

Procedure

Finely ground crude drug is placed in a porous bags or thimble made of strong filter paper which placed chamber of soxhlet apparatus. The extracting solvent (1.Benzen, 2.Chloroform,

3.acetone, 4.Ethanol.5.Water) is heated &its vapour is condensed in condenser. The condensed extractant drips into the thimble containing crude drug. When the level of liquid in chamber rises to the top of siphon tube, the active constituents of Chamber siphon tube into the flask, thus emptying the body of extractor. This alternation of filling & emptying the body of extractor goes on continuously. The soluble active constituents of a drug remain in the flask while the solvent is repeatedly voltilised. The process of filling & emptying of the extractor is repeated until the drug is exhausted

Extract Made By Soxhlet Apparatus

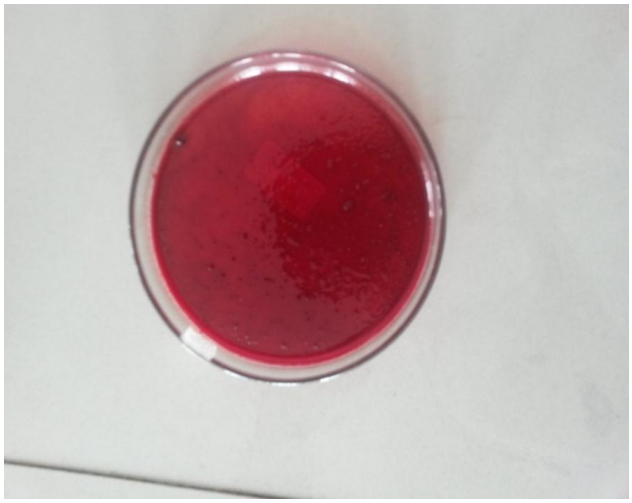


Fig. 4

Table: 2 Percent Yield by Soxhlation

Sr. no	Solvent	% Yield
1	Benzene	4.3
2	Chloroform	7.80
3	Acetone	12
4	Ethanol	70.13
5	Water	15.8

Advantages

- The main advantage of soxhlat extraction is that it is a continuous process.
- Isolate desired oil when it has limited solublity in a solvent and when impurity is insoluble in a solvent.

SELECTION OF HERBS

The various herbs used in the formulation of natural dye lipsticks wereselected on the basis of literature survey.

EXTRACTION OF COLOUR PIGMENTS

The shade dried coarsely powdered seeds of *Beta vulgaris* (100gms) were extracted with methanol (60-80ºC) for half hrs. After completion of extraction, the defatted extract was filtered while hot through Whatman filter paper (No.10) to remove any impurities if present. The extract was concentrated by vacuum distillation to reduce the volume to 1/10; the concentrated extract was transferred to 100 ml beaker and the remaining solvent was evaporated in petriplates. Dark reddish coloured extract was obtained. The concentrated extract was then kept in desiccators to remove the excessive moisture. The dried extract was packed in air tight glass container for further studies.Coloring agent betanine can be obtained from beetroot by milling followed by pressing, filtration and evaporation of the resulted juice

FORMULATION

- **Wax mixture**

Gloss and hardness of lipsticks are hardly depends on the characteristics and the quantity of the waxes used. So the composition of the wax mixture is of prime importance.

- **Oil mixture**

The oil mixture is required to blend properly with the waxes to provide a suitable film on the applied lip. It's also acts as a dispersing agent for insoluble pigments. Ideal mixture is one which enables the product to spread easily.

- **Colour**

The color of the lipstick is most important from commercial and appealing view. The color is imparted to the lips in two ways;By straining the skin with a solution of dyestuff which can penetrate the outer layer of the lip skin .by covering the lips with a colored layer.

- **Preservatives**

These are used prevent growth of microorganisms.

- **Perfume:** To mask theodor.

Table 3: Formulation Table

Sr.No	Ingredient		Quantity Taken	Uses
1	Bees wax		14 g	Glossiness
2	Whites soft paraffin	6 g	Glossiness	
3	Oilve oil	6 ml	Blending	
4	Pigment	0.7 g	Colouring agent	

5	Acacia	-	surfactant
6	Lemon juice	1ml	Anti-oxidant
7	Vitamin E	1 ml	Anti-oxidant
8	Vanilla essence	1 ml	Preservative
9	Perfume	q.s	Perfume

GENERAL MANUFACTURING PROCEDURE

First, the raw ingredients for the lipstick are melted and mixed—separately because of the different types of ingredients used. One mixture contains the solvents, a second contains the oils, and a third contains the fats and waxy materials. These are heated in separate stainless steel or ceramic containers. The solvent solution and liquid oils are then mixed with the colour pigments.

After the pigment mass is prepared, it is mixed with the hot wax. The mixture is agitated to free it of any air bubbles. Then it is poured into tubing molds, cooled, and separated from the moulds.

SOXHLET

Table 4

Sr.No	Ingredient	Quantity Taken
1	Bees wax	14 g
2	Whites soft paraffin	6 g
3	Oilve oil	6 ml
4	Pigment	0.5 g
5	Acacia	-
6	Lemon juice	1ml
7	Vitamin E	1 ml
8	Vanilla essence	1 ml
9	Perfume	q.s

Fig. 5

Table: 5

Sr.No	**Ingredient**	**Quantity**
1	Bees wax	14 g
2	Whites soft paraffin	6 g
3	Oilve oil	6 ml
4	Pigment	1gm
5	Acacia	-
6	Lemon juice	1ml
7	Vitamin E	1 ml
8	Vanilla essence	1 ml
9	Perfume	q.s

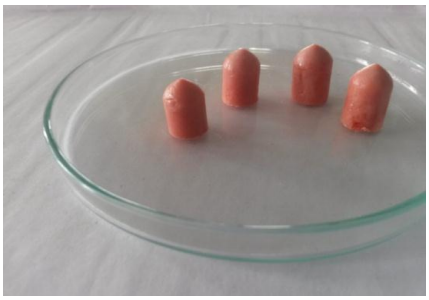


Fig. 6

Table 6

Sr.No	Ingredient	Quantity Taken
1	Bees wax	14 g
2	Whites soft paraffin	6 g
3	Oilve oil	6 ml
4	Pigment	1.5 g
5	Acacia	-
6	Lemon juice	1ml
7	Vitamin E	1 ml
8	Vanilla essence	1 ml
9	Perfume	q.s

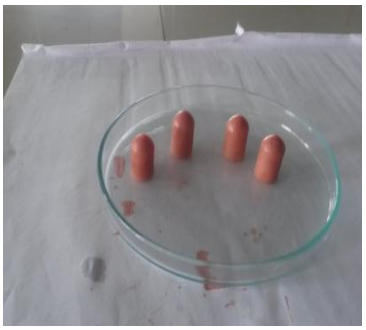


Fig 7

Table 7

Sr.No	Ingredient	Quantity Taken
1	Bees wax	14 g
2	Whites soft paraffin	6 g
3	Oilve oil	6 ml
4	Pigment	2 g
5	Acacia	-
6	Lemon juice	1ml
7	Vitamin E	1 ml
8	Vanilla essence	1 ml
9	Perfume	q.s

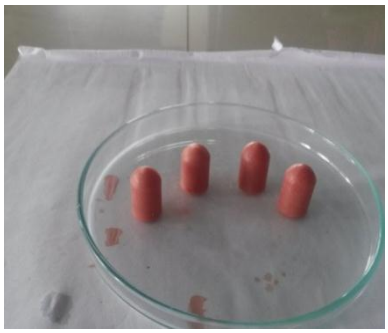


Fig. 8

Table 8

Sr.No	Ingredient	Quantity Taken
1	Bees wax	14 g
2	Whites soft paraffin	6 g
3	Oilve oil	6 ml
4	Pigment	2.5g
5	Acacia	-
6	Lemon juice	1ml
7	Vitamin E	1 ml
8	Vanilla essence	1 ml
9	Perfume	q.s

Fig. 9

SONICATION

Table 9

Sr.No	Ingredient	Quantity Taken
1	Bees wax	14 g
2	Whites soft paraffin	6 g
3	Oilve oil	6 ml
4	Pigment	0.7 g
5	Acacia	-

6	Lemon juice	1ml
7	Vitamin E	1 ml
8	Vanilla essence	1 ml
9	Perfume	q.s

Fig.10

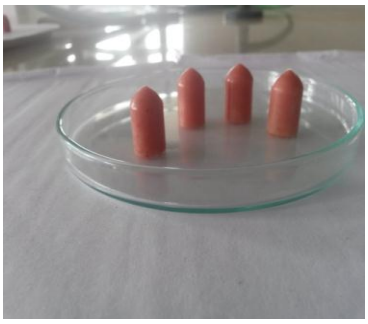


Table 10

Sr.No	Ingredient	Quantity Taken
1	Bees wax	14 g
2	Whites soft paraffin	6 g
3	Oilve oil	6 ml
4	Pigment	1 g
5	Acacia	-
6	Lemon juice	1ml
7	Vitamin E	1 ml
8	Vanilla essence	1 ml
9	Perfume	q.s

Fig. 11

Table 11

Sr.No	Ingredient	Quantity Taken
1	Bees wax	14 g
2	Whites soft paraffin	6 g
3	Oilve oil	6 ml
4	Pigment	1.5 g
5	Acacia	-
6	Lemon juice	1ml
7	Vitamin E	1 ml
8	Vanilla essence	1 ml
9	Perfume	q.s

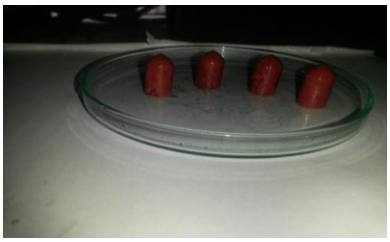


Fig. 12

Table 12

Sr.No	Ingredient	Quantity Taken
1	Bees wax	14 g
2	Whites soft paraffin	6 g
3	Oilve oil	6 ml
4	Pigment	2 g
5	Acacia	-
6	Lemon juice	1ml
7	Vitamin E	1 ml
8	Vanilla essence	1 ml
9	Perfume	q.s

Fig. 13

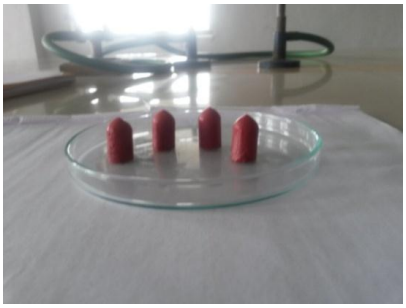


Table 13

Sr.No	Ingredient	Quantity Taken
1	Bees wax	14 g
2	Whites soft paraffin	6 g
3	Oilve oil	6 ml
4	Pigment	2.5 g
5	Acacia	-
6	Lemon juice	1ml
7	Vitamin E	1 ml
8	Vanilla essence	1 ml
9	Perfume	q.s

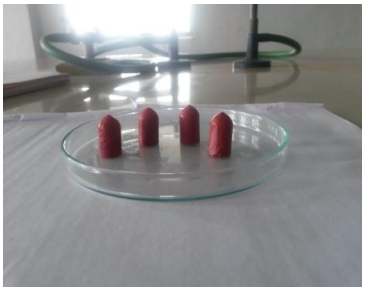


Fig. 14

Preliminary Phytochemical Screening:

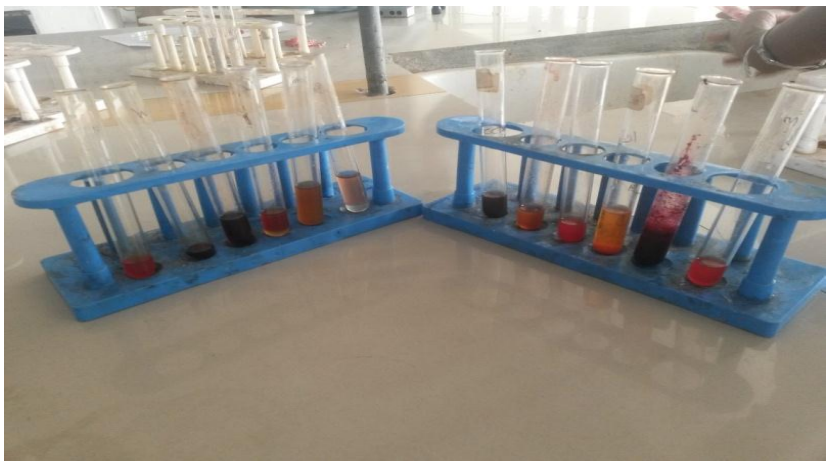


Figure: No 16

CHEMICAL TESTS

Table 14

A. Test for Alkaloids	**Inference (Sonication)**	**Inference (Soxhlet)**
1. Mayer's Test	+	+
2. Dragandroff's Test	+	+
3. Wagner's Test	+	+
4. Hager's Test	+	+
B. Test for Tannins		
1. 5% $FeCl_3$	+	+
2. Lead Acetate	+	+
3. Bromine water	+	+
C. Test for Steroids		
1. Liebermann Test	+	+
2. Salkowaski Test	+	+
D. Test For Glycosides		
1. Antharquinone Test	_	_

2. Killer-Killani Test	_	_
3. Brontrager's Test	_	_
4. Modified Brontrager's Test	_	_
5. Saponin Test	_	_

EVALUATION OF TEST

Melting point

Determination of melting point is important as it is an indication of the limit of safe storage. The melting point of formulated lipstick was determined by capillary tube method, the capillary was filled and keep in the capillary apparatus and firstly observed the product was slowly-slowly milted. After sometimes observed product wascompletely melted. The above procedure was done in 3 times and the melting point ratio was observed in all formulation.

Breaking point

Breaking point was done to determine the strength of lipstick. The lipstick was held horizontally in a socket inch away from the edge of support. The weight was gradually increased by a specific value (10 gm) at specific interval of 30 second and weight at which breaks was considered as the breaking point.

Force of application

It is test for comparative measurement of the force to be applied for application. A piece of coarse brown paper kept on a shadow graph balance and lipstick was applied at 45º angle to cover a 1 sq. Inch area until fully covered. The pressure reading is an indication of force of application.

Surface anomalies

This was studied for the surface defects, such asno formation crystals on surfaces, no contamination by moulds, fungi etc.

Aging stability

The products were stored in 40ºC for 1 hrs. Various parameters such as bleeding, crystallization of on surface and ease of application were observed.

Solubility test

The formulated herbal lipstick was dissolved in various solvents to observe the solubility.

pH parameter

The pH of formulated herbal lipsticks were determined using pH meter.

Skin irritation test

It is carried out by applying product on the skin for 10 min.

Perfume stability

The formulated herbal lipsticks were tested after 30 days, to record fragrance.

Table 15: Evaluation test results for lipstick prepared by using colour pigments obtained by Soxhlet extract.

S.NO	Parameter	F1	F2	F3	F4	F5
1	Colour	Orange -red	Pale pink	Pale pink	Pink	Red
2	Melting point(^{0}C)	55-60	60-61	50-60	59-61	59-60
3	Breaking point(gm)	30	30	32	31	30
4	Ease of application	Good	Good	Poor	Good	Good
5	Aging stability	Smooth	Smooth	Smooth	Smooth	Smooth
6	pH parameter	6	5	6	6	6
7	Perfume stability	++	+	++	++	++
8	Solubility test	+	+	+	+	+
9	Skin irritation	No	No	No	No	No

Table No 16: Evaluation test results for lipstick prepared by using colour pigments obtained by Sonication extract.

S.NO	Parameter	S1	S2	S3	S4	S5
1	Colour	White	Pale pink	Pale pink	Pink	Deep red
2	Melting point (^{0}C)	55-60	60-61	50-60	59-61	62-63
3	Breaking point (gm)	30	30	32	31	30
4	Ease of application	Good	Good	Poor	Good	Easy
5	Aging stability	Smooth	Smooth	Smooth	Smooth	Smooth
6	pH parameter	6	5	6	6	6
7	Perfume stability	++	+	++	++	++
8	Solubility test	+	+	+	+	+
9	Skin irritation	No	No	No	No	No

RESULT

Table 17: Percent of extract by different extraction techniques in different solvent

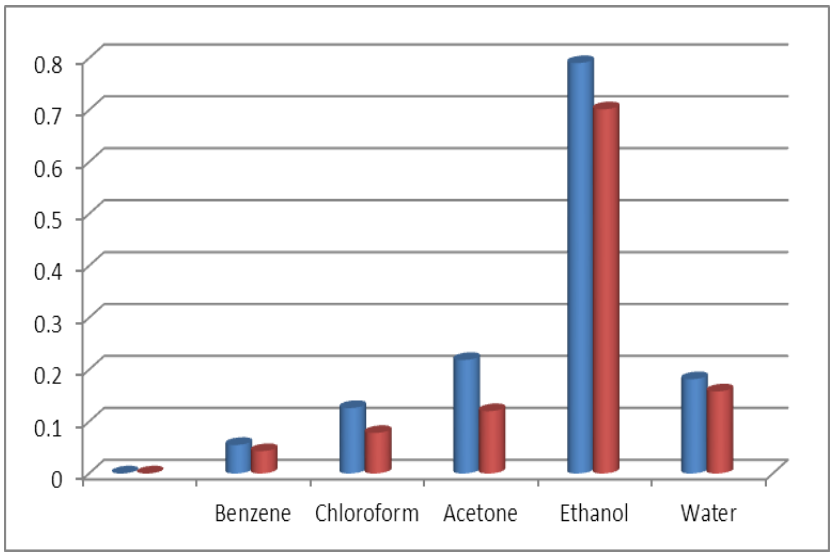
	Sonication	Soxhlation
Benzene	5.5%	4.3%
Chloroform	12.6%	7.80%
Acetone	21.86%	12%
Ethanol	79.04%	70.13%

Water	18.16%	15.8%

From the observation of the table

The sonication process was given the highest percentage yield (%) of extract as compared to the soxhlation method as per showing above table.

By Statistical Analysis



Graph Figure No -18

From the above statistical analysis

The soxhletion method was given less percentage of extract as compared to the sonication process as per shown.

CONCLUSION

By comparative extraction technique study we concluded that by sonication extraction technique % yeild of extract increases presence of phytochemical contituents also maximum in the same extract. The objectives achieved were to minimize side effects and develops a standard formulation of lipstick, to prepare standard lipstick formulation develops new formula for lipstick, to promote use of natural dyes and natural formulations, to increase use of natural formulation over the synthetic, to formulate cost effective product by using natural dyes, to utilize vast range of different natural colors and imparted better properties over synthetic dye.

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