



FORMULATION & EVALUATION OF HERBAL TOOTHPASTE OF SYZYGIUM AROMATICUM.

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1. Abstract:

In India, there are 45000 old medicinal plant species in the Japanese chain, Eastern Himalayas, Western Ghats, and Andaman and Nicobar Islands. Although there are only 3000 officially recorded plants having Medicinal properties, ancient practitioners used around 6000. India is the world's largest producer of medicinal herbs, earning it the moniker "Botanical Garden of the World." There are currently 2, 50,000 Ayurveda medical practitioners registered in the United States. The goal of this study was to develop and test a polyhedral dentifrice that used commonly available healthy herbs in Luck now to treat dental disorders. Cloves are the fragrant flower buds of the Syzygium aromaticum tree, which belongs to the Myrtaceae family. Clove are the greatest medicinal value which is used in Ayurveda. Oils, dried flower buds, leaves, and stems are used to produce medication. Clove is most typically used directly to the gums for toothaches, pain relief during dental work, and other dental concerns. Clove oil contains a chemical called Eugenol that may help to decrease pain and fight infections. The main object of the present research is to provide a formulation useful as natural herbal tooth paste which obviates the drawbacks of the earlier tooth pastes. And its to provide a good tooth paste which can give effective protection to teeth and free them from any toxicity or toxic residue and irritation when regularly used. Brightening the teeth by removing stains would be a prerequisite of the formulation. It should be cosmetically acceptable having pleasant odour and should not leave stain on teeth or fingers after use⁽¹⁾

2. INTRODUCTION:

The word clove came from the Latin word caryophyllon and the Greek word karuophullon. These words were modified in old French to clou de girofle and in Middle English to clow of gilofer. The word was first used in English in the 15th century. ⁽²⁾

Cloves are the aromatic flower buds of a tree in the family Myrtaceae, syzygium aromaticum. Cloves are available throughout the year owing to different harvest seasons in different countries. Its small reddish brown flower buds used as a spice. Cloves were important in the earliest spice trade and are believed to be indigenous to the Moluccas, or Spice Islands, of Indonesia. strong of aroma and hot and pungent in taste, cloves are used to flavour many foods, particularly meats and bakery products in europe and the United States the spice is a characteristic flavouring in christmas holiday fare, such as wassail and mincemeat ⁽²⁾

Toothpaste have been used since ancient past and are one of the main irreplaceable component of oral health care. The design of toothpaste formulation began in china and India, as 300-500 BC. During that period, the squashed bone, pulverized egg and clam shells were utilized as abradives as a part of tooth cleaning. Modern toothpaste formulations were developed in 19th century. Later on, chalk and soap were incorporated to these formulation. After 1945, several formulation advancements of different detergents had began sodium laury sulphate had been used as emulsifying agent. In recent years, the focus has shifted towards the release of active ingredients during formulations developments to prevent and treat oral illness.

Toothpaste is dentifrice used to clean, maintain and improve the health of teeth. Toothpaste is mainly used to promote oral cleanliness and also acts as abrasive that helps to prevent the dental plaque and food particles from the teeth, aids in removing or veiling of halitosis and releases active ingredients such as fluoride to aid in preventing tooth and gum disease.⁽⁴⁾

Keyword-: Clove, Antifungal, Oral Cavity, Monopoly Devapuspa.

HISTORY:

As early as 200 BCE, envoys from Java to the Han-dynasty court of China brought clove that were customarily held in the mouth to perfume the breath during audiences with the emperor. During the late Middle Ages, clove on all islands except Amboina and Ternate in the order to create scarcity and sustain high prices. In the latter half of the 18th century the French smuggled cloves from the East Indies to Indian Ocean Islands and the new world, breaking the dutch monopoly. In the early 21st century, Indonesia was the world largest procedure of cloves, followed by Madagascar, Tanzania and Sri Lanka.⁽⁵⁾

DRUG PROFILE:

Synonym: Clove Buds, Clove Flower.

Biological Source: Clove Consists Of The Dried Flower Buds Of *Eugenia Caryophyllus* Thumb.

Geographical Source:

Clove Tree Is A Native Of Indonesia. It Is Cultivated Mainly In Islands Of Zanzibar, Pemba, Brazil, Amboiana, And Sumatra. It Is Also Found In Madagascar, Penang, Mauritius, West Indies, India, And Ceylon.

Family: Myrtaceae

Kingdom: Plantae

Division: Magnoliophytes

Class: Magnoliopsida

Clade: Tracheophytes

Order: Myrtales

Genus: *Syzygium*

Species: *Aromaticum*

Vernacular Name:

Sanskrit: Bhadrasriya, Devakusuma, Devapushpa,

Hindi: Laung, Laumg, Lavang

Malayam: Grampu, Karampu, Karayampu⁽⁵⁾



Collection and Cultivation:

Clove Tree Is Evergreen And 10 To 20 M In Height. The Plant Requires Moist, Warm And Equable Climate With Well-Distributed Rainfall. It Is Propagated By Means Of Seeds. The Seeds Are Sown In Well-Drained Suitable Soil At A Distance Of About 25 Cm. The Plants Should Be Protected Against Pests And Plant Diseases. Initially It Has To Be Protected From Sunlight By Growing Inside A Green House Or By Constructing Frames About 1 M High And Covering Them With Banana Leaves. As The Banana Leaves Decay Gradually More And More Sunlight Falls On The Young Seedlings And The Seeds Are Able To Bear Full Sunlight When They Are About 9 Months Old. The Seedlings When Become 1 M High, They Are Transplanted Into Open Spaces At A Distance Of 6 M Just Before The Rainy Season. The Young Clove Trees Are Protected From Sun Even For A Longer Period By Planting Banana Trees In Between. The Drug Can Be Collected Every Year Starting From 6 Years Old Till They Are 70 Years Old.

Clove Buds Change The Colour As They Mature. At The Start Of The Rainy Season Long Greenish Buds Appear Which Change To A Lovely Rosy Peach Colour And As The Corolla Fades The Calyx Turns Yellow And Then Red. The Buds Are Collected During Dry Weather In The Month Of August To December. The Collection Is Done Either By Climbing On The Tree Or By Using Some Ladders Or With The Help Of Mobile Platforms. In Some Places The Trees Are Even Beaten Using Bamboo Sticks For The Collection Of The Bud. The Drugs Which Are Collected Are Then Separated From The Stalks And Then Placed On Coconut Mats For Drying Under Sun. The Buds Loose About 70% Of Its Weight, Whereas Drying And Change Their Colour To Dark Reddish-Brown. The Dried Clove Is Graded And Packed.⁽⁷⁾

Chemical Constituents:

Clove Contains 14–21% Of Volatile Oil. The Other Constituents Present Are the Eugenol, Acetyl Eugenol, Gallotannic Acid, and Two Crystalline Principles; A- And B- Caryophyllenes, Methyl Furfural, Gum, Resin, and Fibre. Caryophyllin Is Odourless Component And Appears To Be A Phytosterol, Whereas Eugenol Is A Colourless Liquid. Clove Oil Has 60–90% Eugenol, Which Is The Cause Of Its Anesthetic And Antiseptic Properties.

Use:

- Clove Is Used As An Antiseptic, Stimulant, Carminative, Aromatic, And As A Flavouring Agent.
- It Is Used As Anodyne, Antiemetic.
- Dentists Use Clove As An Oral Anesthetic And To Disinfect The Root Canals.
- Clove Kills Intestinal Parasites And Exhibits Broad Antimicrobial Properties Against Fungi And Bacteria.
- It is used In the Treatment of Diarrhea, Intestinal Worms, and Other Digestive system.
- Relief pain and inflammation, Regulate Blood Sugar.⁽⁷⁾

3. RATIONAL AND SIGNIFICANCE OF STUDY:

It must be aware of the various benefits of clove and clove oil when it comes to oral health. It may not be possible for you to gain these benefits every day while taking care of oral hygiene level. There are various options available in a clove toothpaste, often in combination with various natural ingredient to let take care of your teeth and gums properly.

Regular use of this toothpaste can prevent cavities, yellow teeth, toothache, gum troubles, bad breath, and more to ensure that your teeth and gums remain healthy.

The extract clove, clove oil, charcoal, salt gives various benefits to teeth and gums. The clove extract in these preparation can protect teeth and gums from bacterial attacks and ensure strong teeth and gums. These will ensure fresh breath and give you a refreshing sensation every time.

4. LITERATURE REVIEW:

- A. Treating Toothache With Clove Oil-By-Shawn Watson (2020) Use Of Clove And Its Picture.
- B. Chemical Composition, Antioxidant And Antifungal Potential Of Clove (Syzygium Aromaticum) Essential Oil, Its Major Compound And Its Derivatives-By-Kamal Preet Kaur And Et.Al. (2019) From This Studied the Phytochemical Analysis and Some Biological Activities.
- C. Clove: A Champion Spice -By-Parle Milind and Et.Al. (2011) Vernacular Name, Biological Names Etc.
- D. Role of Herbal Medicine in Dental Health -A Detailed Review-By-P. Anushya and Et.Al. (2020) Get The Introduction About Clove.
- E. Formulation Of Clove Toothpaste – A Detailed Review By – Suraj Maurya, Shashikant Maurya Et Al (2021) Get from Formulation Of clove paste.
- F. Preparation and evaluation of toothpaste –A detailed research By –Asha M.Jagtap, Sudhir R.Kaulage.Et Al 2018 get the evaluation parameter of toothpaste.

5. PLAN OF WORK:

1. Identification.
2. Collection
3. Processing of raw materials.
4. Materials & Methods
5. Extraction of Crude Drug.
6. Formulation of Product
7. Evaluation of Drug.
8. Result & Discussion.

6. AIM AND OBJECTIVES

Aim: Formulation & Evaluation Of herbal Toothpaste of Syzygium Aromaticum.

Objectives:

The objectives of the present article as follow,

1. To study a drug profile of clove
2. To study extraction method
3. Study of different phytochemical screening
4. Study of evaluation parameters.



7. MATERIAL AND METHODS:

Clove, Mortar And Pestle, Sieve, Weighing Balance, Iodine Flask, Measuring Cylinder, Ethanol, Distilled Water, Calcium Carbonate, Glycerine, Sodium Lauryl Sulphate, Acacia gum, Sodium Chloride, Sodium Saccharin, Methy paraben etc

Identification of the Drug:

For this project work buds from Pharmacognosy lab of Matoshri Miratai Aher College of Pharmacy, Karjule Harya, Tal-Parner, Dist-A.Nagar.

EXTRACTION:

The clove reduced to moderately coarse powder and extracted successfully with solvent ethanol by using Maceration.

Method of Extraction:

Maceration:

In this process, the whole or coarsely powdered crude drug is placed in a stoppered container with the solvent and allowed to stand at room temperature for a period of at least 3 days with frequent agitation until

The soluble matter has dissolved. The mixture then is strained, the marc (the damp solid material) is pressed, and the combined liquids are clarified by filtration or decantation after standing

- In this process solid ingredients are placed in a stoppered container with the whole of the solvent and allowed to stand for a periods of at least 3 days with frequent agitation, until soluble matter is dissolved.
- The mixture is then strained the mare pressed and combined liquid clarified or by decantation after standing.
- Weigh about 30 gm of crude powder then add 150 ml ethanol kept for 4 hours with continuous shaking on magnetic stirrer then filter it.
- For preparation of filtrate 1 add residue then add 50 ml ethanol kept for four hours with continuous shaking.
- For prepartion of fitrate 2 add residue add 50 ml ethanol kept for overnight.
- For preparation of filtrate 3 add residue 50 ml ethanol kept for a night.⁽⁸⁾

FORMULATION OF CLOVE PASTE:

Using a home mixer, all herbal material were dried & pulverised. The required quantity of ingredients were weighed and taken in mortar. Calcium is a mineral that is found in sodium lauryl sulphate, carbonate, methyl in water, cellulose, honey and glycerine were combined. The aforesaid mixture was supplemented with acacia. These is answer was added drop wise into mortar containing herbal ingredient and triturated well unstill a paste consistency is formed ^(8,9)

Table No 1: Chemical Composition of Formulation

Sr.No	Ingredients	Quantity In (gm)		
		F1	F2	F3
1	Clove Extract	2	4	6
2	Glycerine	1	1	1
3	Base Material	27	25	24
4	Distilled Water	q.s	q.s	q.s

Table No 2: Formulation of Base Materials

Sr No	Ingredients	Quantity (gm)
1	Calcium Carbonate	60 gm
2	Glycerine	15 ml
3	Sodium Lauryl Sulphate	3 gm
4	Acacia Gum	1.5 gm
5	Sodium Chloride	1.5 gm
6	Sodium Saccharine	1.5 gm
7	Methyl Paraben	1.5 gm
8	Distilled Water	q.s
9	Total	100 gm

PROCEDURE:

In a mortar- pestle 2 gm of clove extract where triturated with 1 gm of methy paraben and 0.5 gm of sodium chloride (as a preservatives)

- As foaming agent 1 gm of sodium lauryl sulphate is used, and sodium saccharine is used as sweetener.
- Glycerine was added as humectant and acacia gum was employed as binder. The mixture was triturate well, and qs distilled water was added to bring the total weight to 30 gm.
- A solution of sodium hydroxide is used to alter the ph. clove oil is used as mask the bitterness of the taste^(8,9)

FIGURES:

Fig no 1: clove Powder.



Fig no 2: clove toothpaste.

8. PHYSICAL PARAMETERS:**A) PREFORMULATION STUDY:**

It is the principal investigation in the drug development to obtain information on the known properties of compound and the proposed development schedule so, this pre formulation investigation may merely conform that there are no significance barrier to compound development.

- **General Appearance:** colour, odour, taste
- **Bulk Density:**

For determine the bulk density, powder mixture was weighted accurately poured into 100 ml glass cylinder without compacting. The volume of powder mixture was recorded and then calculate by equation.

$$\text{Bulk density} = \text{mass bulk of powder} / \text{bulk volume of powder}$$



- **Tapped Density:**

Weigh accurate quantity of powder sample was transfer into a graduated measuring cylinder. Volume occupied by the powder was noted down. Then cylinder was subjected to 100-300 taps in tap density apparatus. ^(9,11)

$$\text{Tapped density} = \text{mass of powder} / \text{final tapped volume}$$

➤ **Carr's Index:**

the compressibility index and hausners ratio was measured the property of powder to compress. the packing ability of powder material was evaluated from change in value which is due to rearrangement of packing occurring during tapping. it was indicated as cars compresibilty index was calculated by formula.

$$\text{Carr's index} = \frac{\text{tapped density} - \text{bulk density}}{\text{tapped density}} \times 100$$

➤ **Hausners Ratio:**

Measurment of frictional resistance of powder. ideal range 1.2 to 1.5

$$\text{Hausners ratio} = \frac{\text{tapped density}}{\text{bulk density}}$$

➤ **Angle Of Repose(Θ):**

It was determine by using fixed funnel method. Specified amount of powder drug was transfer to the funnel keeping the orifice of the funnel blocked by thumb. When powder was cleared from funnel then measured it's angle of repose and measure in Θ .^(10,11)

$$\text{Angle of repose } (\Theta) = \tan^{-1} \frac{h}{r}$$

➤ **Flow Rate:**

Weighed accurate qunntity of powder sample. place a cotton plug at the neck of a clean and dry funnel stem diameter 1-2.5cm. Place powder sample in the funnel. remove plug from neck and record the total time required for all the powder to flow.

$$\text{Flow rate} = \frac{\text{weight of powder}}{\text{time required to flow}}$$

➤ **Water Soluble Extractive :**

Useful for the evaluation of crude drug. give idea about the nature of the chemical constituents present in a crude drug. weigh about 5 gm of the coarsely powdered drug and transfer it to a dry 250 ml conical flask. fill a 100ml graduated flask with a water and transfer it into a conical flask. cork the flask a side for 24 hours, shaking frequently. filter into 50 ml cylinder. when sufficient filtrate has collected, transfer 25 ml of filtrate to a weigh thin porcelain dish. Evaporate to dryness on a water bath complete the drying in an oven at 105°C for 6 hours. cool and weigh immediately. calculate the percentage w/w of extractive with the reference to the air dried drug.

$$\text{W.S.E} = \frac{\text{Weight of residue} \times 100 \times 100}{\text{Wt of drug taken} \times \text{volume of filterate}}$$

➤ **Alcohol Soluble Extractive:**

It is same as water soluble extractives only water is replace by alcohol.

➤ **Moisture Content :**

Weigh 1.5 gm of sample in a porcelain dish containing 6-8 cm diameter and 2-4 cm depth in it. dry the sample in an oven at 105°C. cool and weigh. calculate the moisture contents by using formula

$$\text{Moisture content } (\%) = \frac{\text{final weight} - \text{initial weight}}{\text{initial weight}} \times 100$$

➤ Total Ash Value :

Used to determine quality and purity of crude drug and to establish the identified of it. Weigh 2 gm of powder drug into the crucible.ignite sample on burner until all the carbon is burn off. Cool it and weighed the ash. Calculate the percentage of total ash with reference to their air dried sample of crude drug.⁽¹²⁾

Weight of Empty Dish = X

Weight Of the Drug Taken = Y

Weight of the Dish with Ash = Z

Weight of Ash = (Z-X)

$$\text{Total Ash} = 100(Z-X) / Y$$

Evaluation of Formulation:

Prepared clove toothpaste were evaluated for the following evaluation parameter.

A) Evaluation:

Colour:

Colour of the prepared toothpaste was evaluated for its colour .The colour was checked visually.

Odour:

Odour was found by smelling the product.

Taste:

The taste was checked manually by tasting product.

B) Physical characterisation test:

Determination of pH:

1 gm of toothpaste in 150 ml beaker and 10 ml of freshly boiled and cooled water (at 27°C.). Stir well to make a through suspension. Determined the pH of the suspension within 5 minutes, using digital pH meter.The result were mentioned.

Foamibility

The foam ability of the product was evaluated by taking small amount of preparation with water in a measuring cylinder initial volume was noted and then shaken for 10 times. Final volume of foam was noted.

C) Study of rheological properties:

Spreadibility:

The spreadibility is term express to denote the extent of area to which the paste readily spreads on application area. One of the criteria for the paste to meet ideal quality is that it should possess good spreadability. About 1 gm of medicated dental paste was weighted and kept at the center of the glass plate (10×10 cm) and another glass plate was placed over it carefully. 1 kg weighted was placed at the center of the plate. The diameter of the paste in cms, after 15 min was measured.

Tube extrudability:

The formulation under study was filled in a clean, lacquered aluminium collapsible one-ounce tube with a nasal tip of 5mm opening and applies the pressure on tube by the help of finger. Tube extrudability was taken determined by measuring the amount of the paste extruded through the tip when a pressure was applied on tube paste.

Viscosity:

Paste viscosity measurement were evaluated using a Brookfield digital viscometer UltraS programmable Remotor, USA using spindle no.3 by applying increasing value of the shear rate, in order to reveal possible flow behavior of the pastes. All viscosities measurements were performed at controller temperature of 30 °C.⁽¹³⁾

Solubility:

Soluble in boiling water, miscible with alcohol and ether.

Stability Study:

The stability study was performed as per ICH guidelines. Physical stability of the prepared toothpaste was carried out for 3 month at various temperature condition like 20°C, 25°C, 37°C.⁽¹⁴⁾

9. RESULT:

Table No 1 Preformulation Study of powder sample:

SR NO	Parameters	Sieve no #60	Sieve no #66	Sieve no #80
1	Colour	Dark Brown	Dark Brown	Dark Brown
2	Bulk density (gm/ml)	0.410	0.421	0.421
3	Tapped Density(gm/ml)	0.643	0.666	0.644
4	Carr's index (%)	11.00	12.19	13.04
5	Hauser's ratio	1.14	1.16	1.17
6	Porosity (%)	61.76	62.20	60.75
7	Angle of repose(Θ)	20.12	21.32	22.14
8	Moisture Content (%)	9	10	10
9	Flow Rate(gm/sec)	0.66	0.46	0.68
10	Ash value (NMT4%)	14.2	11.10	12.00
11	Water soluble Extractive (NLT 35%)	14.50	16.21	17.63
12	Alcohol soluble Extractive (20%)	9.86	10.62	11.00

From Above Preformulation Data Powder From Sieve No#66 Shows Acceptability Angle Of Repose, Tapped Density, Bulk Density, Carr's Index & Hausner's Ratio, Flow Rate, Moisture Conclude The Powder From Sieve No #66 Consider As An Optimize Batch .

Table No: 2 Description

Sr.No	Parameter	Observation		
		F1	F2	F3
1	Colour	Yellowish Brown	Yellowish brown	Yellowish brown
2	Odour	Characteristics	Characteristics	Characteristics
3	Taste	Sweet	Sweet	Sweet
4	Stability	Stable	Stable	Stable
5	Spreadability	Easily Spread	Easily Spread	Easily Spread
6	Abrasiveness	Good Abrasive	Good Abrasive	Good Abrasive
7	Foamability	Good	Good	Good

Table No: 3 Physical evaluation of Formulation

Sr. No	Parameter	Observation		
		F1	F2	F3
1	pH	8.5	8.5	8.5
2	Spredability (cm)	7.7	7.7	7.7
3	Viscosity (CPS)	39751.6	39751.6	39751
4	Tube Excludability	Good	Good	Good

10. CONCLUSION: -

The research concluded that herbal toothpaste more acceptable in dental research and they are safer with minimum side effect than synthetic preparation. The formulated toothpaste capable to the tooth and oral hygiene.

The conclusion can be drawn from the result obtained in the present work of investigation. The dental paste preparation of herbal toothpaste designed using bases for the treatment of dental plaque. During our physicochemical evaluation studies all the formulation were found to have pH, good tube extrudability, good spreadability and viscosity characteristics. The Above Evaluation Parameter It Can Be Conclude That Overall Batches The F3 Batch Shows All Parameter In Acceptable Limit. Therefore It Consider As Good Formulation.

The formulation compare with market preparation therefore it shows the equal patronizing and engrossing passion over the marketed formulation (Colgate, Daber red, Dantkanti). The formulated herbal tooth paste has been good scope in future in nature remedies research and dental health of public.

11. REFERENCE:

1. L. Merr. And L.M. Perry. Kew Science, Plants Of Syzygium Aromaticum The World Online. 2021. Retrieved 28 February 2021.
2. Yun, Wonjung (13 August 2018). "[Tridge Market Update] Tight Stocks of Quality Cloves Lead To A Price Surge". Tridge. Archived From The Original On 14 August 2018. Retrieved 13 August 2018.
3. Dr. kamal Pathak, Dr. Ankur Vaidya textbook of cosmetics science concept and principle. Oral cavity herbal toothpaste page no.6.21
4. The drug profile aromaticum syzygium <https://www.britannica.com/plant/clove>
5. The chemical constitution and uses of clove <https://www.newworldencyclopedia.org/entry/Clove>.
6. The history of plant clove <http://www.pharmacy180.com/article/clove-247/>
7. The method of extraction of clove and its procedure <https://g.co/kgs/X9y99R> (Wikipedia).
8. The formulation of clove paste procedure, Suraj Maurya, 2021, Formulation of clove toothpaste, JETIR May 2021, Volume 8, Issue 5.
9. Formulation <https://www.jpsr.pharmainfo.in/Documents/Volume/vol12issue01/jpsr12012020.pdf>.
10. Aulton ME: Pharmaceutics the science of dosage design .Second ed. Churchill Livingstone; 2002
11. Lackman Leon, Lieberman Herbert A, Kanig Joseph L. The theory and practice of industrial pharmacy. 3rd ed. Varghese publishing house; 2009
12. Ritesh Kumar Tiwari, Lalit Singh .the pre- formulation study of clove in Asian food Science journal 2018.
12. Pavan Deshmukh - Formulation and evaluation of toothpaste: compared with marketed preparation march 2017, Indian Council of Medicinal Research, Delhi 25/26 march 2017.
13. Asha M. Jagtap, Sudhir R. Kaulage –formulation and evaluation of toothpaste Asian Journal of pharmaceutical science 2018.
14. Roshan Telerandhe, Pavan Deshmukh - Formulation and evaluation of toothpaste: compared with marketed preparation march 2017, Indian Council of Medicinal Research, Delhi 25/26 march 2017

