Preparation, Evaluation And Comparison Of Herbal Toothpaste With Markedly Available Tooth Pastes

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ABSTRACT: The main aim of the present work is to prepare, evaluate and compare Lab Made Herbal toothpaste with commercial Herbal toothpastes. In the present study, commercial herbal toothpastes such as Meswak, Dauber red, sudanta have been evaluated for their quality. All the marketed Herbal tooth pastes and Lab Made Herbal toothpastes were evaluated and complied with the standards specified by the Bureau of Indian Standards. The formulations were evaluated by different tests like pH, spreadability, abrasiveness, foaming ability, fineness, test for F3, Ph and stability studies. All the results showed that Labmade formulation is comparatively equal and rarely better in terms of results than marketed formulation. Hence the selected Labmade formulation was found to be of good quality.

Key words: Commercial Herbal toothpastes, Bureau of Indian standards, Toothpaste

I. INTRODUCTION:
Oral hygiene is an important key to maintain good appearance, impression of an individual and gives confidence. The tooth consists of two parts, crown and the root. The crown of the tooth is covered by outer surface called enamel and it is the hardest tissue in the tooth. The major composition of enamel is hydroxyl apatite other than that it consists of water and keratin. Dentine is the beneath part of the enamel, which is a composite of hydroxylapatite. It also consists of 70% of the collagen water. Fluorine is the major component of dentine. Oral consists of not only tooth but also saliva for easy to swallow the food. Saliva is the major element which intended for lubricating the food and to maintain proper environment in the mouth. Saliva is produced by various glands such as Labial, lingual, buccal and palatal are the larger and smaller glands which produce saliva continuously to keep the tooth environment in the dynamic state. Proteins, enzymes, bacteria and mucopolysaccharide are present in the saliva and the inorganic materials like calcium, sodium, potassium, chloride, phosphate ions.

Toothpastes have been used since the ancient past and are one of the main irreplaceable components of oral health care. The design of toothpaste formulations began in China and India, as 300-500 BC. During that period, squashed bone, pulverized egg and clam shells were utilized as abrasives as a part of tooth cleaning. Modern toothpaste formulations were developed in the 19th century. Later on, chalk and soap were incorporated to those formulations. After 1945, several formulation advancements of di chloro- phosphates had been used as emulsifying agent. In recent years, the focus has shifted towards the release of active ingredients during formulation developments to prevent and/or treat oral illness. Toothpaste is a dentifrice used to clean, maintain and improve the health of teeth. Toothpaste is mainly used to promote oral cleanliness and also acts as an abrasive that helps to prevent the dental plaque and food particles from the teeth, aids in the removing and/or veiling of halitosis, and releases active ingredients such as fluoride to aid in preventing tooth and gum disease (eg. Gingivitis). The majority of the cleaning is performed by the mechanical utilization of the toothbrush with the help of excipients used in toothpaste. The main aim of this investigation is to evaluate the Herbal toothpaste formulations and comparing with three popular commercial toothpastes.

Ideal properties:
- Good abrasive effect
- Non irritant and non toxic
- Impart no stain in tooth
- Keep the mouth fresh and clean
- Prolonged effect
- Cheap and easily available

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II. MATERIALS AND METHODS

Materials
The weight of every ingredient was decided by reviewing previous herbal toothpaste formulations. The combination of percentage by weight of all the ingredients of this is 100%, which means the sum of quantity of toothpaste will formulate 100gm of toothpaste. The ingredients of the toothpaste prepared in lab are given in table 1 and compared with marketed herbal tooth pastes Meswak and Sudanta and Dauber red.

Herbal tooth paste was prepared using guava leaves extract, tulsi, banyan, acacia, calcium carbonate and sodium lauryl sulphate. Guava extract gives relief from toothache, Neem leaf has antibacterial activity, Ginger gives antiseptic property and bad breath of mouth is prevented by tulsi. Banyan is used against toothache. Acacia to prevent gingivitis and also acts as gelling agent. Sodium lauryl sulphates is used as a foaming agent & Para hydroxy benzoic acid is used as a preservative. Saccharin sodium acts as sweetening agent; amaranth is used as colourant and water as vehicle.

FORMULATION OF HERBAL TOOTHPASTE

Table 1. Herbal Toothpaste Formulation Ingredients

<table>
<thead>
<tr>
<th>S. No.</th>
<th>Common name</th>
<th>Botanical name</th>
<th>Parts used</th>
<th>Category</th>
<th>Quantity(%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Gauva extract</td>
<td>Psidium guajava</td>
<td>Leaves</td>
<td>Relieve the toothache pain</td>
<td>10.5</td>
</tr>
<tr>
<td>2</td>
<td>Banyan</td>
<td>Fiscus bengalensis</td>
<td>Bark</td>
<td>Prevent toothache</td>
<td>6</td>
</tr>
<tr>
<td>3</td>
<td>Neem</td>
<td>Azadirachta indica</td>
<td>Leaves</td>
<td>Antibacterial</td>
<td>5</td>
</tr>
<tr>
<td>4</td>
<td>Tulsi</td>
<td>Ocimum tenuiflorum</td>
<td>Leaves</td>
<td>Prevent bad breath</td>
<td>5</td>
</tr>
<tr>
<td>5</td>
<td>Ginger</td>
<td>Zingiber officinale</td>
<td>Root</td>
<td>Antiseptic</td>
<td>5</td>
</tr>
<tr>
<td>6</td>
<td>Acacia</td>
<td>-</td>
<td>-</td>
<td>Prevention and treatment of gingivitis</td>
<td>3</td>
</tr>
<tr>
<td>7</td>
<td>Calcium carbonate</td>
<td>-</td>
<td>-</td>
<td>Abrasive</td>
<td>31</td>
</tr>
<tr>
<td>8</td>
<td>Glycerin</td>
<td>-</td>
<td>-</td>
<td>Humectant</td>
<td>30</td>
</tr>
<tr>
<td>9</td>
<td>Sodium lauryl sulphate</td>
<td>-</td>
<td>-</td>
<td>Detergent</td>
<td>1.50</td>
</tr>
<tr>
<td>10</td>
<td>Sodium saccharin</td>
<td>-</td>
<td>-</td>
<td>Sweetening agent</td>
<td>0.30</td>
</tr>
<tr>
<td>11</td>
<td>Para hydroxy benzoic acid</td>
<td>-</td>
<td>-</td>
<td>Preservative</td>
<td>0.15</td>
</tr>
<tr>
<td>12</td>
<td>Menthol</td>
<td>-</td>
<td>-</td>
<td>Flavouring agent</td>
<td>1.5</td>
</tr>
<tr>
<td>13</td>
<td>Amaranth</td>
<td>-</td>
<td>-</td>
<td>Colouring agent</td>
<td>0.50</td>
</tr>
<tr>
<td>14</td>
<td>Water</td>
<td>-</td>
<td>-</td>
<td>Vehicle</td>
<td>Q.S</td>
</tr>
</tbody>
</table>

METHODS

Preparation of extracts:
The tender Leaves (100 grams) were extracted by two methods.

Method-1: Continuous hot extraction is performed with absolute alcohol at 50°C. Procedure: Gauva leaves were taken and washed in order to take out impurities from them. They were shade dried for about 5 days, after proper drying, they were grounded to a fine powder which was passed through sieve No. -6. The powder was packed in soxhlet apparatus and continuously extraction process was done for about 7 hours at 50°C with ethanol. After the extraction process, the product was collected and shade dried for 10 days and the extract was powdered.
All the herbal ingredients were dried and grounded using domestic mixer. The required quantities of the ingredients were weighed and taken in mortar. Calcium carbonate, sodium lauryl sulphate, glycerine and saccharin sodium were mixed in water. Para hydroxy benzoic acid, menthol and acacia were added into the above mixture. This solution was added drop wise into mortar containing herbal ingredients and triturated well until a paste consistency is formed.

**Method-2:** The leaf samples were washed, dried and blended into powder. Increasing polarity solvents such as methanol (>95%), n-hexane (>95%), ethanol (>99.5%), and boiling distilled water were used in the maceration process. The leaf powder was incorporated into each of solvents to prepared 20% concentration. The mixture was mixed in Erlenmeyer flask, wrapped the flask with aluminium foil to avoid solvent evaporation and then exposed it to light for three consequent days at room temperature. The contents were shaken with platform shaker at 70 rpm. The mixture was soaked for 3 days, the contents were transferred to 50 ml test tube and again centrifuged for 10 min at 4000rpm in room temperature. Finally, the supernatant liquid was separated and stored in refrigerator at 40°C until it becomes useful in the procedure.

**Antibacterial Activity of Psidium guajava:** The well-diffusion method was used to identify the antibacterial activity of toothpaste as per the standard of the National Committee for Clinical Laboratory Standards. The antibacterial activity of plant extracts was carried out using Mueller Hinton II plates. Initially, plates were streaked with bacteria, punches were made with 5mm diameter into the medium using a sterile cork borer. The test bacterium was inoculated into all places, a sterile cotton swab dipped using sterile forceps into the suspension, rotated multiple times and excess inoculums removed by pressing the swab firmly above the fluid level inside the tube. The surface of the agar plate was rotated to ensure an equal distribution of inoculums present around the rim. Fifty test extract aliquots were dispensed into each well present in the plates after inoculation with bacteria. The plates are dried for 3 to 5 min to remove excess moisture present in it. Triangle shaped wells were prepared with a distance of 2 inches apart. The same extract was incorporated into three plates for each selected bacterial strain. Controls were prepared with pure solvents for each bacterium. The plates are wrapped with parafilm, labelled, and stored in an incubator at 37°C. Each plate was examined after incubation for 24 hrs, identified inhibition zones measured (in millimeters) with a ruler. Experimental results were noted in parallel, and took the average results of three independent experimental results. Initially, the binder is mixed with solid abrasive, then it is added to liquid phase, contains sweetener, humectants and preservative into a mixer. As a result, homogeneous paste was formed, then added the detergent and flavor, mixed vigorously, finally deairated and tubed.

**Evaluation of Toothpaste**

**Colour:**
The prepared toothpaste was evaluated for its colour. The colour was checked visually.

**Odour:**
Odour was found by smelling the product.

**Taste:**
Taste was checked manually by tasting the product.

**Stability**
Stability of toothpaste is checked by exposing the product at 45±2°C for a period of 28 days. After storage, no phase separation, fermentation and gassing was observed. Also exposed to refrigerator conditions at 2-8°C for 24 hours. The product was observed to be stable even at refrigerated conditions.

**Determination of sharp and edge abrasive particles**
Took the contents on to the finger and scratched on the butter paper for 15-20 cm long to check for the presence of any sharp or abrasive particles. Repeated the same process for at ten times. No sharp or abrasive particles were found.

**Determination of spreadability**
Took one gram of toothpaste, placed on a glass slide (10 x 10 cm) and covered with another glass slide. Then carefully placed two kg weight on covered glass slide (sliding, shall not take place). Measured the spreading (in cm) of the toothpaste after 3 minutes. Repeated the experiment and took the average value of three readings.
**Determination of fineness**

Weighed accurately about 10g of toothpaste and placed in a 100 ml beaker. Added 50 ml of water, and allowed to stand for 30 min with stirring until the paste is completely dispersed. Transferred the solution to 150µ & 75µ IS sieves and washed with a slow stream of tap water. Allowed the running tap water to drain completely and dried the sieve at 105±2° C by placing it in an oven. Transferred the residue particle present on the sieve on to a watch glass and weighed it.

**Calculation:**

Material on the sieve % by (Retained mass / Material taken) x 100.

**pH determination**

Weighed 10 g of toothpaste placed in 150 ml beaker. Added 10 ml of boiled and then cooled water. Stirred vigorously to make a suspension. Measured the pH of the suspension using pH meter.

**Determination of lead**

The color produced with sample solution containing hydrogen sulfide is compared with standard lead solution.

**Foaming power**

Took a suspension of the material in measuring cylinder, shaken the suspension for 12 times and measured the volume of the foam produced after shaking for 5 minutes.

**Procedure:** Weighed 5g of toothpaste in to a 100 ml glass beaker. Added 10 ml of water, covered the glass beaker with a watch glass and kept aside for 30 minutes. Heated the suspension gently to dissolve the detergent if present in it. Stirred the suspension with glass rods and transferred it to 250 ml measuring cylinder. Examined if no foam is produced (more than 2 ml). Transfer the residue retained in the beaker to measuring cylinder by adding of 5-6 ml of water. Then make up the cylinder with 50ml of water. Stir the contents with up-down movements to get uniform suspension at 30° C. After shaking, kept the cylinder aside for 5 minutes. And final note the volume obtained with foam + water.

**Determination of moisture and volatile matter**

Weighed 5g of sample placed in a porcelain dish of 6-8 cm in diameter and 2- 4 cm depth. Dried the sample in an oven at 105° C.

**Calculation**

% by mass = 100 M1 / M

M1 - loss of mass (in grams) on drying

M - Mass (in grams) of the material taken for the test.

**Determination of fluoride ion**

Fluoride ions are determined using potentiometer containing fluoride ion sensitive electrodes.

**Calculation:** a graph is plotted on a log scale, taking the concentration of fluoride (x-axis) Vs potential in mV (y-axis). From the calibration curve, the fluoride ion concentration (in mg) of test solution is measured.

fluoride ion concentration (ppm) M = 2 a × 10000

a = mg of fluoride ion calculated from graph

M= Mass of sample taken in gram

### III. RESULTS AND DISCUSSION

Evaluation tests of toothpastes were carried out according to the standards specified by the Bureau of Indian standards IS 6356-1993 for Herbal toothpastes samples (Meswak, Dauber red, Sudanta) and Lab made toothpaste sample. All the samples were complied with BIS and they found to be of good quality.

Evaluation tests were carried out to view the different properties of Lab made and commercial toothpastes. All the results of evaluating parameters were given in table 2.
In the present study, comparatively equal and rarely better results have been observed with Lab made formulation than marketed formulations. Both preparations have shown equal efficacy in terms of foaming ability. But increased activity in terms of abrasiveness and spreadability was appeared in Lab made formulation (fig.1). Comparison of the abrasiveness of marketing pastes with Lab made formulation suggests that Lab made formulation has more abrasiveness than marketed pastes.

All the toothpastes were having good consistency and smooth texture. Also shown no symptoms for deterioration such as phase separation, gassing, fermentation when all the samples were placed at a temperature of 45±2°C for a period of 28 days. It confirmed that the toothpaste is stable.

The preferable amount of residue has retained on sieve for Lab made formulation which is better than the residue obtained by commercial tooth pastes. So it was found that Lab made preparation has shown reasonably good % of fineness (fig.2).

The moisture and volatile matter present in Meswak was significantly more than the rest of the formulations. The percent of moisture and volatile content in Lab made formulation has the same percent of moisture as that of Dauber red and Sudanta. These results explain that moisture and volatile content are within the limits (Fig.3).

The color produced with hydrogen sulfide in test solution is less than obtained with standard solution indicating that amount of lead is within the acceptance criteria.

Fluoride ions present in the sample were potentiometrically determined by fluoride ion sensitive electrodes. The concentration (ppm) of fluoride ion in Lab made formulations is less than the standard values mentioned in table 2.

![Fig.1. Abrasiveness and spread ability of Lab made and commercial tooth pastes](image1.png)

![Fig.2. Foaming ability of Lab Made and commercial tooth paste.](image2.png)
Preparation Evaluation And Comparision Of Herbal Toothpaste With Markedly Available Toothpaste

![Fig.3. Fineness(%), Moisture and volatile matter(%) of Lab made and commercial tooth paste](image)

<table>
<thead>
<tr>
<th>S.No.</th>
<th>PROPERTIES</th>
<th>LAB MADE</th>
<th>MESWAK</th>
<th>DAUBER RED</th>
<th>SUDANTA</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Hard and sharp edged abrasive particles</td>
<td>Absent</td>
<td>Present</td>
<td>Absent</td>
<td>Present</td>
</tr>
<tr>
<td>2</td>
<td>Abrasiveness</td>
<td>4</td>
<td>3</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>3</td>
<td>Spread ability</td>
<td>5.6</td>
<td>5.2</td>
<td>5.0</td>
<td>4.0</td>
</tr>
<tr>
<td>4</td>
<td>pH</td>
<td>8.5</td>
<td>8.6</td>
<td>7.3</td>
<td>8.1</td>
</tr>
<tr>
<td>5</td>
<td>Stability(45±2C for128 days &amp; at 5 C for 1 hour)</td>
<td>Good</td>
<td>Good</td>
<td>Good</td>
<td>Good</td>
</tr>
<tr>
<td>6</td>
<td>Fineness(%by mass)</td>
<td>0.41</td>
<td>0.38</td>
<td>0.36</td>
<td>0.41</td>
</tr>
<tr>
<td>7</td>
<td>Moisture and volatile matter(%by mass)</td>
<td>1.8</td>
<td>2.0</td>
<td>1.6</td>
<td>1.8</td>
</tr>
<tr>
<td>8</td>
<td>Foaming ability</td>
<td>75</td>
<td>66</td>
<td>75</td>
<td>70</td>
</tr>
<tr>
<td>9</td>
<td>Test for Pb(ppm)</td>
<td>5</td>
<td>6</td>
<td>13</td>
<td>11</td>
</tr>
<tr>
<td>10</td>
<td>Fluoride ion, ppm,max.</td>
<td>40</td>
<td>35</td>
<td>54</td>
<td>46</td>
</tr>
</tbody>
</table>

IV. CONCLUSION

Herbal toothpastes have an emphasized role in maintaining the oral hygienic nature as well as preventing dental caries. Based on this pattern, Lab made Herbal toothpaste was formulated by selecting suitable ingredients to get the formulation more stable. Evaluation and comparison of results with commercial Herbal toothpaste, demonstrated that Lab made toothpaste is having equal patronizing and engrossing passion over the marketed formulations (Meswak, Dauber redand Sudanta). All the marketed Herbal tooth pastes and Labmade Herbal toothpaste are evaluated and compared with the standards specified by Bureau of Indian standards. This preliminary in vitro study demonstrated that Labmade Herbal toothpaste was equally efficacious as three commercially popular toothpastes in terms of all evaluation properties of toothpaste. Hence, by the evidence of in vitro studies, it is concluded that Labmade Herbal toothpaste formulated in a laboratory was found to be of good quality.

REFERENCES


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