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Preparation And Evaluation Of Herbal Toothpaste And Compared With Commercial Herbal Toothpastes: An *Invitro* Study

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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 Himalaya, Meswak and Dent county have been evaluated for their quality. All the marketed Herbal tooth pastes and Lab Made Herbal toothpaste which had been evaluated complied with the standards specified by the Bureau of Indian Standards. The formulations were subjected to various evalution tests like pH, Spreadability, abrasiveness, foaming ability, cleaning ability, fineness, moisture and volatile content, tube inertness,Test for F-, Pb, As, and stability studies. All the Results of evaluating parameters 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 Ingredients.

INTRODUCTION

Toothpastes have been used since the ancient past[1] and are one of main irreplaceable components of oral health care[2].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[3]. 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 different detergents had begun, sodium lauryl sulfate had been used as emulsifying agent [(2,3)]. In recent years, the focus has shifted towards the release of active ingredients during formulation developments to prevent and /or treat oral illness [(1,3)].

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) [4]. 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.

MATERIALS AND METHODS

Materials

The weight of every each ingredient was decided by review previous study formulation of Herbal toothpaste. 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 formulation. The ingredients of all toothpaste formulations are given in table 1 and Marketed Herbal tooth pastes Himalaya. Meswak and Dant kanti are used.

Formulation of toothpaste.[5]

Table 1.Herbal Toothpaste Formulation Ingredients

SNO	INGREDIENTS	QUANTITY USED (%)	MANUFACTURER
1	Gauva extract	19.5	Powder Extract from plant of Guava Leaves
2	Papain	10	Crude drug from local market.
3	Calcium carbonate	35	Sd fine Chem. Limited,Mumbai,India.
4	Sodium lauryl sulfate	1.50	Thermofisher Scientific India Limited,Mumbai,India.
5	Glycerin	30.00	Thermo Electron LLS India Pvt Limited,Mumbai,India.
6	Methyl cellulose	01.00	Sd fine Chem. Limited,Mumbai,India.
7	Sodium saccharine	00.30	Sd fine Chem. Limited,Mumbai,India.
8	Methyl paraben	00.10	Sd fine Chem. Limited,Mumbai,India.
9	Propyl paraben	00.02	Sd fine Chem. Limited,Mumbai,India.
10	Titanium dioxide	00.50	Sd fine Chem. Limited,Mumbai,India.
11	Menthol	1.50	Sd fine Chem. Limited,Mumbai,India.
12	Purified water	q.s	Made from Laboratory.

Methods

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

Method: 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 4 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 6 hours at 50 degrees C with ethanol. After the extraction process, the product was collected and shade dried for 10 days and the extract was powdered. The standard toothpaste base was formulated. Extracts of Guava leaves and papain powders were incorporated in the base in various Concentrations as given in Table 1. All the formulations were Filled in regular metal tubes used in toothpastes. The storage in tubes was done to correct the problem of Crusting and drying of extruded toothpaste during evaluation and stability studies.[6]

Antibacterial Activity of *Psidium guajava*

Extraction Methods Used on Guava: 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 extraction process. The leaf powder was incorporated

into each of solvents to prepare 20% concentration. The mixture was mixed in Erlenmeyer flask, wrapped the flask with aluminium foil to avoid solvent evaporation and then expose 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 with the revolutions of 4000rpm at room temperature. Finally, the supernatant liquid was separated and stored in refrigerator at 4^0C until it becomes useful in the procedure[7].

Antibacterial Activity: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^oC. 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 [7].

Evaluation of Toothpaste [8]

According to the guidelines, the standards were prescribed for each evaluation test of non-fluorinated (type I) or fluorinated (type II) toothpastes.

Composition

Toothpaste is not composed of mono or disaccharides such as sucrose or fermentable carbohydrates. All ingreadients should comply with the Indian standards.

Homogenecity

The toothpaste shall extrude a homogenous mass from the collapsible tube or any suitable container by applying of normal force at 27±20C. In addition, bulk of contents shall extrude from the crimp of container and then rolled it gradually.

Tube inertness

The toothpaste container shall not produce any corrosion or deterioration in normal storage conditions like heating temperature at 45 ± 2^0C for ten days. Tube inertness can be examined by cutting the internal surface, open and observing whether any sign of deterioration or chemical attack occurred in the container.

Determination of sharp and edge abrasive particles

Extrude the contents 15-20 cm long on the butter paper, repeat the same process for at least ten collapsible tubes. Press with the contents of the entire length with finger tip for the presence of sharp and hard edged abrasive particles. Toothpaste shall not contains such particles.

Determination of spreadability

One gram of toothpaste placed on a glass slide (10 x 10 cm), cover with another glass slide. Then carefully place two kg weight of on covered glass slide (sliding, shall not take place). Measure the spreading (in cm) of the toothpaste after 3 minutes. Repeating the experiment and note the average value of three readings.

Determination of fineness

Weigh accurately about 10 gm of toothpaste placed in a 100 ml beaker. Allow 50 ml of water, stand for 30 min with stirring until the paste gets completely dispersed. Transfer the solution to 150 micron IS sieve and wash with a slow stream of tap water. Allow running tap water drained the on sieve and dry (at 105 ± 2^0C) the sieve by place it in an oven. Transfer any residue particle is present on the sive to a watch glass and weigh it.

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

ii. Weigh accurately about 10 gm of toothpaste placed in a 100 ml beaker. Allow 50 ml of water, stand for 30 min with stirring until the paste gets completely dispersed. Transfer the solution to 75 micron IS sieve and wash with a slow stream of tap water. Allow running tap water drained on the sieve and dry (at 105 ± 2^0C) the sieve by placing it in an oven. Transfer any residue particle is present on the sieve to a watch glass and weigh it.

pH determination

Weigh 10 g of toothpaste placed in 150 ml beaker. Allow 10 ml of boiled and then cooled water. Stir vigorously to make a suspension. Measure 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.

Determination of arsenic

Yellow color stains produced when arsine gas reacts with mercuric bromide paper. Sample stain is comply with the standard stain.

Foaming power

Take a suspension of the material in measuring cylinder and shake the suspension for 12 times. And measure the volume of the foam produced after shaking for 5 minutes.

Procedure: weigh 5 g of toothpaste in a 100 ml glass beaker. Add 10 ml of water, cover the glass beaker with a watch glass and stand for 30 minutes. Heat the suspension gently to dissolve the detergent if present in it. Stir the suspension with glass rods and transfer it to 250 ml measuring cylinder. Examine 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 30^0C. after shaking, keep the cylinder stand for 5 minutes. And final note the volume obtained with foam + water.

Determination of fluoride ion

Fluoride ions can be 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) $M = 2\ a \times 10000$

a = mg of fluoride ion calculated from graph

M= Mass of sample taken in gram

Stability

The toothpaste shall be stable, but not to be deteriorating, ferment and segregate during normal storage conditions and usage. Stability of toothpaste can be tested when it exposes to 45±20C for a period of 28 days. After storage, no phase separation, fermentation and gassing can be observed. Also exposed to cool conditions such as 5^0C for 1 hour, no obstruction of extrudable form from the container is observed.

Determination of moisture and volatile matter

Weigh 5 g of sample placed in a porcelain dish containing 6-8 cm in diameter and 2- 4 cm depth in it. Dry the sample in an oven at 105^0C.

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.

RESULTS AND DISCUSSION

Use of fluorides has been the foundation of caries counteractive action and the use of fluoridated toothpaste is the most widely recognized types of caries control being used today. The advantages of fluoridated products are well documented in the literature. Many commercial toothpastes claim to have abrasive, spreadability, foaming ability and have caries counteractive action, very little research has been conducted to compare these properties in Lab made dental product. Hence the purpose of this study was to evaluate and compare the efficacy of commercial toothpastes with Lab made toothpaste. Evaluation tests of toothpastes were carried out according to the standards specified by the Bureau of Indian standards IS 6356-1993 (table.3.3) for Herbal tooth pastes samples (Himalaya, Meswak and Dant kanti) 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 and pH. 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 formulationhasmoreabrasiveness than marketed pastes. And also, given significant result for cleaning ability which is similar to the results obtained in the commercial formulations.

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±20C for a period of 28 days. We found extrudables from the all tubes after placing it in cool temperatures (5^0C) for 1 hour. So it confirmed that all toothpastes have good stability.

The internal part of all collapsible tubes have given no sign of corrosion or damage during normal storage conditions at a temperature of 45 ± 2^0C for 10 days except for Himalaya Herbal toothpaste tube has slightly affected by corrosion. So it was confirmed that the containers of Lab made, Meswak and Dant kanti have shown good tube inertness.

The preferable amount of residue has retained on sieve for Lab made formulation which is better than the residue obtained by Himalaya Herbal and Meswak toothpastes but little less to the residue of Dant kanti toothpaste. So it was found that Lab made preparation has shown reasonably good % of fineness (fig.2).

The moisture and volatile matter present in Himalaya Herbal was significantly more than the rest of the formulations. The percent of moisture and volatile content in Lab made formulation is given the same value as that of Dant kanti and Meswak got the least value. These results explaining all the formulations have good moisture and volatile content (fig..3).

The color produced with hydrogen sulfide in test solution is less than obtained with standard solution that indicates all the samples have limited amount of lead impurities.

Stain produced by Himalaya Herbal sample is more than standard stain that indicates Himalaya Herbal toothpaste is having a little more amount of arsenic impurities. All the remaining formulations passed the limit test of arsenic.

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

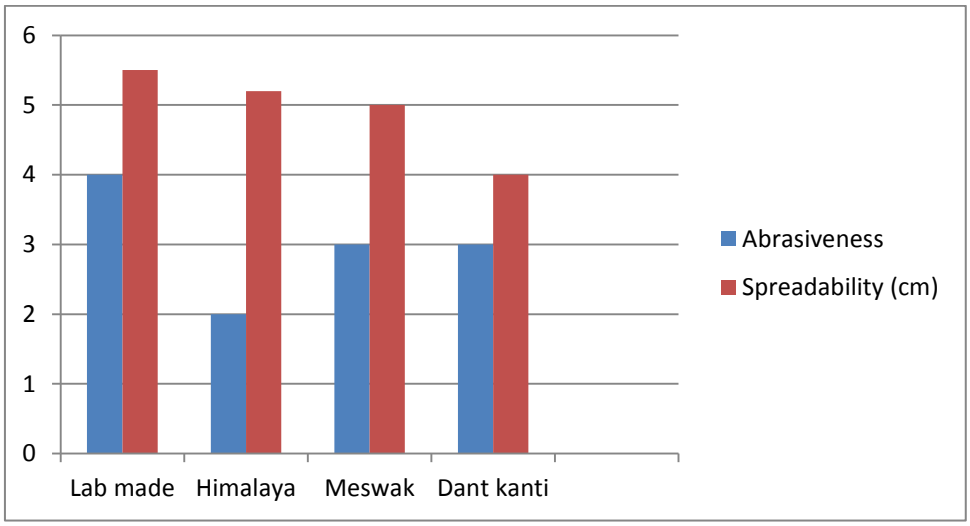

Fig.1. Abrasiveness and Spreadability of Lab made and commercial toothpastes

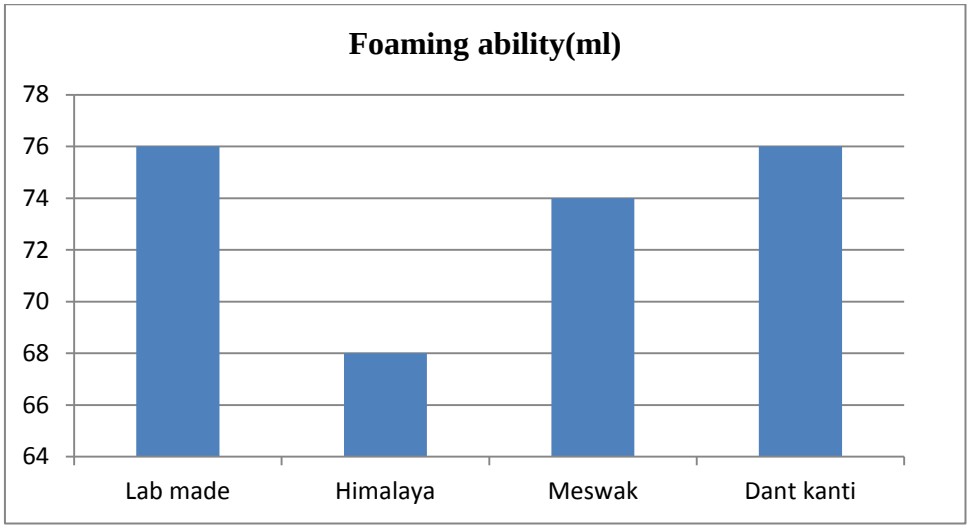

Fig.2. Foaming ability of Lab made and commercial toothpastes

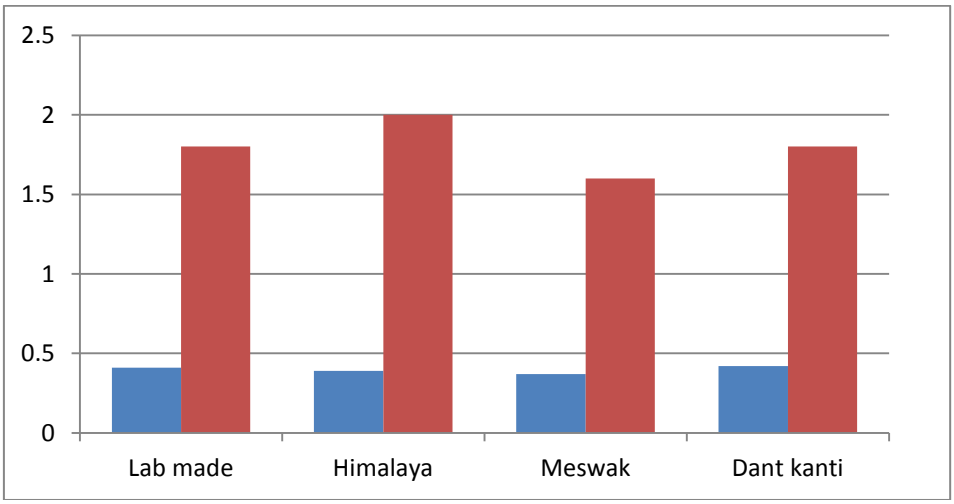

Fig.3. Fineness (%), moisture and volatile matter (%) of Lab made and commercial toothpastes

Table 2. Evaluation tests for Lab made and commercial Herbal toothpastes

S.NO	PROPERTIES	LAB MADE	HIMALAYA	MESWAK	DANT KANTI
1	Hard and sharp edged abrasive particles	Absent	Present	Absent	Present
2	Abrasiveness	4	2	3	3
3	Spreadability (cm)	5.5	5.2	5.0	4.0
4	pH of 10% aq. suspension	8.6	8.6	7.3	8.2
5	Foaming ability	76	68	74	76
6	Cleaning ability	Good	Good	Good	Satisfactory
7	Stability (45 ± 2^0C for 28days & at 5^0C for 1 hour)	Good	Good	Good	Good
8	Tube inertness (at 45 ± 2^0C for 10 days)	No corrosion	Slight corrosion	No corrosion	No corrosion
9	Fineness (% by mass)	0.41	0.39	0.37	0.42
10	Moisture and volatile matter (% by mass)	1.8	2.0	1.6	1.8
11	Test for Pb (ppm)	6	8	12	10
12	Arsenic (As_2O_3)	Within limits	Exceeding limit	Within limits	Within limits
13	Fluoride ion, ppm, max.	42	36	55	48

CONCLUSION

Eventually Herbal toothpastes having an emphasized role in the 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 are demonstrated that Lab made toothpaste is having Equal patronizing and engrossing passion over the marketed formulations (Himalaya, Meswak and Dant kanti).All the marketed Herbal tooth pastes and La bmade Herbal toothpaste which had been evaluated compared with the standards specified by Bureau of Indian standards. This preliminary *in vitro* study demonstrated that La bmade 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 La bmade Herbal toothpaste formulated in a laboratory was found to be of good quality.

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