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RESEARCH ARTICLE

COMPARATIVE EVALUATION OF EXPERIMENTAL TOOTHPASTE FORMULATION AND THREE POPULAR COMMERCIAL TOOTHPASTES: AN INVITRO STUDY

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ABSTRACT

The aim of the present work is to evaluate and compare experimental toothpaste with commercial toothpastes. In the study, commercial toothpastes such as colgate, close up and pepsodent have been used and also can be evaluation studies were performed for their quality. All the marketed tooth pastes and experimental toothpaste which had been evaluated the complied with the standards specified by the Bureau of Indian Standards. The formulations were subjected to various evolution tests like pH, spreadability, abrasiveness, fineness, foaming ability, cleaning ability, moisture and volatile content, F, Pb, As, tube inertness and stability studies. All the Results of evaluated parameters showed that experimental formulation is comparably equal and rarely better in terms of results than marketed formulation. Hence, the selected experimental formulation was found to be of good quality.

INTRODUCTION

Toothpastes have been used since antiquity (Davies R *et al*., 2010) and is one of main indispensable elements of oral health care (Ersoy *et al.,* 2008). The development of toothpastes began in China and India, as 300-500 BC. Initially, crushed bone, crushed egg and oyster shells were used as abrasives in tooth cleaning (Jardim *et al*., 2009). Modern toothpastes were developed in the 1800's. Later, soap and chalk were added to their formulations and after World War II, due to advances in the synthesis of different detergents, the soap was replaced by emulsifying agents such as sodium lauryl sulphate (Ersoy *et al.,* 2008; Jardim *et al.,* 2009). Toothpaste is a paste or geldentifrice used with a toothbrush as an accessory to clean and maintain the aesthetics and health of teeth. Toothpaste is used to promote oral hygiene: it acts as an abrasive that aids in removing the dental plaque and food from the teeth, assists in the elimination and/or masking of halitosis, and delivers active ingredients such as fluoride or xylitol to help prevent tooth and gum disease (gingivitis) (The History of Toothpaste and Toothbrushes.,2013). It is important to note that most of the cleaning is done by the mechanical use of the toothbrush, along with excipients in used toothpaste. Most toothpaste contains trace amounts of chemicals that may be toxic when ingested; toothpaste is not intended to be swallowed.

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Fluoride toothpaste

Toothpastes are pastes, also called dentifrices, that help to remove plaque and strengthen tooth enamel and dentine. Plaque is a film of bacteria that forms on teeth and gums, which can cause damage to both teeth and gums. The major anti-caries effect of fluoride toothpaste results from small but protracted elevations in levels of fluoride in plaque and saliva. It is thought that fluoride has exerted a caries reduction benefit largely through three mechanisms: (1) inhibition demineralization; (2) promotion of demineralization and (3) interference with bacterial growth and metabolism (Brambilla *et al* 2001). OTC fluoride toothpaste containing fluoride at up to 1,000 or 1,500 ppm F, depending on local regulations, can directly to consumers.

Use and Application: Fluoride toothpaste is generally used at home, but there are successful examples of community programs often based around schools to prevent dental caries. Many organizations the world over suggest the patient" brush teeth at least twice a day using fluoride-containing toothpaste."Over the past five decades, professional dental organizations have come to endorse the use of fluoride-containing toothpaste as safe and effective for preventing tooth decay and improving oral hygiene. In order to mitigate a small risk of fluorosis that might occur due to excessive fluoride ingestion during the development period of the dentition, most toothpaste manufacturers now

Include the following language on toothpaste container: use only a pea-sized amount for children under six. To prevent swallowing, children under six years of age should be supervised in the use of toothpaste. "The U.S FDA also required a new label on all fluoride toothpastes stating, "If you accidentally swallow more than used for brushing, seek professional help or contact a poison control center immediately" (ADA., 1997).

Safety: different countries use different limits and suggest different concentrations of fluoride acceptable for oral health for general use by consumers (Scheifele *et al.,* 2002). Broadly, in different parts of the worlds the maximum permissible of level of fluoride available in toothpaste for general sale is either 1000 or 1500 ppm F. Fluoride toothpaste is not intended to be swallowed, and toothpaste tubes should be kept out of the reach of young children. Even though it is very rare, young children are at some risk of ingesting toxic doses of fluoride from a standard toothpaste tube. Warning labels on the tube are intended to help reduce the risk of mild fluorosis, which is a cosmetic defect noticeable as very light spots on permanent teeth that develop while the teeth are still forming. Fluorosis only occurs when more than the recommended daily amount of fluoride is ingested. The children at greatest risk of fluorosis are those 6 years of age and younger when the front teeth are developing and the body weight is relatively low compared to the amount of toothpaste ingested. Currently the Food and Drug Administration of the united states requires the warning label "if you accidentally swallow more than used for brushing, seek professional help or contact a poison control center immediately" on all fluoride toothpastes. High-quality toothpastes that are endorsed by professional organizations should be used to ensure safety and efficacy (FDA., Id/18985512).

Mechanism of Action of Fluoride: (Silverstone. LM, *et al*., 1977, Wefel. JS, *et al*., 1982, Moreno. EC, *et al* 1977, Brown. WE *et al* 1977 and Posner, 1985)

The development of newer dentifrice formulations has paralleled the increased understanding of the caries process and how fluoride works. The original belief of a continual dissolution of tooth surface has been replaced by the acceptance of subsurface demineralization and the maintenance of a relatively intact surface layer (probably by demineralization). Demineralization occurs when there is an imbalance between processes of mineral gain and loss. Fluoride may interact with these processes in several ways. It is now widely accepted that fluoride has both a systemic and topical mode of action. The interaction of fluoride with the mineral component of teeth produces a FHAP, by substitution of F^- for OH^-. This results in increased hydrogen bonding, smaller crystal lattice, and an overall decrease in solubility. The incorporation of fluoride into HAP lattice may occur while the tooth is forming or by ion exchange after it has erupted. A decrease in solubility increases with greater amounts of fluoride incorporation, but rarely do we exceed several thousand ppm of fluoride in the outer enamel. Thus, only limited protection from fluoride substitution would be expected as compared to pure FHAP that has 40,000 ppm fluoride. Another means of acquiring fluoride into the enamel is from topical applications and ion exchange.

This surface oriented exchange could also affect the solubility of the bulk solid. The exception to limited protection may be the crystallite surface, where a thin coating of pure FHAP would make the bulk solid appear to be less soluble than the degree of substitution would predict. Therefore, a limited incorporation of fluoride into the crystal lattice or on the surface may have a significant impact on solubility. The systemic "solubility reduction effect" was thought to be the only mechanism of action until studies revealed a significant topical effect on mineralization as well as a bacterial effect. Fluoride found in solution can also affect the dissolution rate without changing the solubility of tooth mineral. As little as 0.5 mg/L in acidic solutions causes a reduction in the dissolution rate of apatite. This mechanism also involves absorption and/or ion exchange at the crystal surface. Thus, the surface may act more like FHAP than HAP and have a different dissolution rate. When the enamel dissolves, it may also contribute fluoride to the solution. Under sink conditions this would not have much of an effect, but the solutions normally bathing the teeth are always partially saturated with respect to apatite. A fluoride level as low as 230 µg/g has been shown to significantly reduce the dissolution rate of apatite. Thus, both the concentration of fluoride at the crystal surfaces and the fluoride concentration in the liquid phase during acariogenic challenge are important. In addition to protecting against demineralization, another way in which fluoride interacts with enamel to reduce dissolution is through demineralization. This is a process in which partially dissolved enamel crystals act as a substrate for mineral deposition from the solution phase that enables partial repair of the damaged crystals. Therefore, remineralisation will counteract some of the demineralization and equilibrium will develop between the two processes.

The carious lesion is the result of demineralization outweighing remineralisation. One of the benefits of the demineralization/remineralisation interplay is the creation of less soluble mineral in enamel. This occurs by dissolution of the more soluble calcium deficientmagnesium containing carbonated apatite which makes up enamel when first formed. The remineralisation process results in formation of a less soluble form of apatite. When fluoride is also present, formation of FHAP results in a mineral with an enhanced level of acid resistance. The remineralisation process is one controlled by the super saturation of fluids bathing the teeth - plaque fluid or saliva. The degree of super saturation will, in part, determine the rate of precipitation of minerals from the solution. Too high of a super saturation will result in the rapid formation of calcium phosphate and block the surface pores of enamel. This precipitation then limits the diffusion of calcium, phosphate and fluoride into the interior of the lesion resulting in lesion arrestment but not also lesion repair. The interior of the lesion is partially saturated with respect to HAP and can become supersaturated with respect to FHAP if small levels of fluoride are present or diffuse into the lesion. The use of low concentration fluoride products, such as dentifrices on a daily basis, will help maintain this favourable saturation.

Thus, remineralisation of the lesion may result in the repair of the existing lesion with less soluble mineral and render this portion of the tooth less susceptible to future episodes of demineralization (Fig.1). This is probably one of the most important modes of action of fluoride. Fluoride, at a relatively low concentration, may also interact with the oral bacteria to reduce plaque acid production. Several mechanisms have been proposed to account for this end result.

One is the Well-known interaction of fluoride with the enzyme enolase which could reduce acid production directly. There is also an indirect effect on the PTS pathway that decreases the amount of sugar entering the cell by limiting PEP. Another possibility is diffusion of fluoride into the cell occurs as HF which then dissociates and lowers the intercellular pH. Fluoride may also affect the ability of the cell to remove excess H+ and less acid production may result from cytoplasmic acidification. The overall effect is less acid and a less acidic environment that should lower the driving force for dissolution.

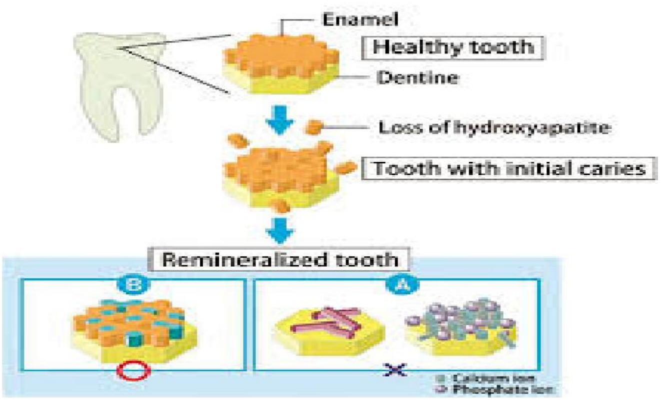

Fig.1. Remineralization-mechanism of action of fluoride

Classification of toothpastes

While many types of toothpaste have multiple ingredients and additives that differentiate the dental professional's recommendations for use, one can categorize toothpastes into seven major categories based upon patient needs and desires (Sanjivani *et al.,* 2010).

These broad categories of toothpastes are

- Caries prevention—cavity protection
- Antitartar activity (reduction of calculus formation)
- Gingivitis reduction
- Plaque formation reduction
- Remineralizing—calcium-phosphate-fluoride containing
- Cosmetic effect—tooth whitening, stain removal
- Reduction in tooth sensitivity (Multicaretoothpastes)
- Natural toothpaste—natural ingredients
- Toothpastes for patients with dry mouth—xerostomia

Tooth pastes are preferred over other dental preparations because of following reasons:

1.Easy to take and spread on the tooth brush
2.No spillage or wastage
3.Attractive consistency
4.Proper distribution in mouth
5.Available in wide varieties

A good tooth paste should have the following characteristics:

- It must clean the dental surface properly without any scratches.
- Consistency should be such that it can be easily squeezed out of the tube to spread on the brush, but should not penetrate into the brush.
- The consistency should remain constant in a wide range of temperature during shelf life.
- It should be non-toxic and should not sensitize buccal membrane.
- It should not interact with the container material.
- It should have pleasant taste and odour.
- It should have good appearance.

Toothpaste ingredients

Toothpastes usually contain excipients, represented in Table 1, and active ingredients.

- Common fluorides in toothpastes include stannous fluoride, sodium monophosphate fluoride and sodium fluoride. Fluoride's primary action is to be incorporated into the tooth substrate (enamel and dentin) making the tooth more resistant to acid attack by cariogenic bacteria. Fluoride is also bactericidal and has additional antiplaque effects.
- Desensitizing agents are active ingredients, usually potassium nitrate, in toothpaste that reduce dentin hypersensitivity through a depolarizing effect on the odontoblastic processes in the dentinal tubules. The nerve endings of the odontoblastic processes then repolarise and have a reduced pain sensing ability. Also, desensitizing effects of arginine bicarbonate/ calcium carbonate complex and stabilized stannous fluoride have been demonstrated to provide a dentine desensitizing effect.
- Anti plaque agents reduce plaque growth. This can have a positive effect in reducing plaque growth on teeth, reducing gingivitis, and potentially reducing carines. Some antiplaque agents include triclosan, papain, and sanguinary extract. Triclosan has been accepted by the FDA as an antiplaque- antigingivitis therapeutic additive to toothpastes.
- Ant tartar ingredients that reduce calculus buildup on teeth include tetrapotassium pyrophosphate, tetra sodium pyrophosphate, disodium pyrophosphate, papain and citroxaine.
- Remineralizing agents have recently been added to toothpastes. These remineralizing agents are based upon amorphous calcium phosphate. This soluble calcium and phosphate are described as enhancing remineralization, preventing dental caries, reducing enamel and/or dentin erosion, and reducing dentin hypersensitivity. The mode of action that has been hypothesized for these agents is the calcium and phosphate in soluble form which allows it to bind to enamel and dentin and to dental plaque.

Table 1. Toothpaste excipients

Abrasives	Gelling Or Binding Agents	Surfactants	
Alumina	Carboxymethyl cellulose	Amine fluorides	
Aluminium	trihydrate Carrageenan	Dioctyl sodium sulfosuccinate	
Bentonite	Hydroxyethyl cellulose	Sodium lauryl sulfate (SLS)	
Calcium carbonate	Plant extracts (alginate, guar gum,)	Sodium N lauryl sarcosinate	
Calcium pyrophosphate	Silica thickeners	Sodium stearylfumarate	
Dicalcium phosphate	Sodium alginate	Sodium stearyl lactate	
Kaolin	Sodium aluminum silicates	Sodium lauryl sulfoacetate	
Silica Xanthan gum	**Colours**	**Sweeteners**	
Sodium bicarbonate	Chlorophyll	Aspartame	
Sodium metaphosphate	Titanium dioxide	Saccharine	
Humectants	**Preservatives**	**Flavours**	**Film Agents**
Glycerol	Alcohols	Aniseed	Cyclomethicone
PEG 8	Benzoic acid	Clove oil	Dimethicone
Pentatol	Ethyl parabens	Eucalyptus	Polydimethylsiloxane
PPG	Formaldehyde	Fennel	
Sorbitol	Methylparabens	Menthol	
Water	Phenolics (methyl,ethy, propyl)	Peppermint	
Xylitol	Polyaminopropylbiguanide	Vanilla	

- Abrasive also called as polishing agents Cleaning and polishing (function) Solid, insoluble particles. It has potential for fluoride interaction It. used in dentistry for abrading, grinding, polishing Remove debris & residual strain from teeth. There are types and examples of Abrasives Phosphates help the product to leave the teeth looking white and feeling clean such as Dicalcium phosphate dihydrate Calcium pyrophosphate Carbonates.
- Function of surfactant to produce foam and aid in the removal of debris Emulsifies flavouring agents Characteristics: It may react with other toothpaste components High level may cause mucosal irritation. Sodium Lauryl Sulfate Sodium N-Lauryl Sarcosinate and Sodium Dodecyl Benzene Sulfonate PEG examples of type of surfactant.
- Humectant used in toothpaste to prevent loss of water and subsequent hardening of the product upon exposure to air. Characteristics of these properties affect taste perception. Thus, proper usage level produces clear translucent toothpaste. Examples of humectant compound in toothpaste formulation is Glycerine, Sorbitol, Polyethylene Glycol, Xylitol, and Propylene Glycol
- Binder provides consistency, shape and keeps the solid phase properly suspended in the liquid phase to prevent separation of the liquid phase out of the toothpaste. They also provide body to the dentifrice, especially after extrusion from the tube onto the toothbrush. A binder or thickener can prevent the toothpaste from drying out. They control the viscosity and contribute to give the toothpaste a creamy consistency. Types and examples of Binders are Natural Polymers, CMC,Carrageenans, Xanthan Gum, Synthetic Polymers, and others
- Function of Preservative to prevent the growth of micro-organisms in toothpastes. It also should non-irritating, compatible with other ingredients and it should be used in combination for example Sodium benzoate, Methylparaben, and Propylparaben.
- Flavoring agents are added to improve the taste of toothpastes. They can range from minty flavors to fruity flavours.Sweeteners also improve the taste of toothpaste. Most toothpaste sweeteners are artificial and are not able to be used by cariogenic bacteria.
- Colouring agents are added to provide toothpastes with a pleasing appearance.

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- Colouring agents are added to provide toothpastes with a pleasing appearance.
- All this property is combine is very low concentration of each properties. toothpaste formulation due to medicinal effect and its function as active ingredient. All of this components should be well mix to formulate a toothpaste and several test have to be done to make sure the stability of the toothpaste product and it antimicrobial activity. Anti-bacterial or active ingredients agent is a main component in toothpaste formulation that takes action to treat and prevent toothache.

All this property is combine is very low concentration of each properties. toothpaste formulation due to medicinal effect and its function as active ingredient. All of these components should be well mixed to formulate a toothpaste and several test have to be done to make sure the stability of the toothpaste product and it antimicrobial activity.

Antibacterial or active ingredients agent is a main component in toothpaste formulation that takes action to treat and prevent toothache. Nowadays, toothpastes are produced to serve multiple purposes at the same time and, thus, possess a complex chemical composition. The ideal toothpaste must have the following properties: slight abrasion, froth, sweetening, bleaching and prevention of plaque, calculus and decay (Ersoy *et al.,* 2008, Gunsolley *et al.,* 2006). Moreover, toothpastes may also function as vehicles for antimicrobial agents that may have a preventive/therapeutic role in periodontal disease (Maltz *et al.,* 2009, Gunsolley *et al.,* 2006). The complex composition of toothpastes implies that it is necessary to ensure that the active ingredients are not inactivated (Davies *et al.,* 2010). For example, calcium carbonate binds to sodium fluoride rendering it ineffective as an anti-caries agent. So, the elaboration of a correct composition of toothpaste is of crucial importance, regarding its effectiveness of oral health maintenance.

Toothpaste is by far the most common form of caries control in use today. The intensive promotion of fluoride toothpastes by the oral healthcare industry has been a major factor in their increased use and in the developed world since 1980s, nearly all commercially available toothpaste formulations contain fluoride (Dean *et al.,* 1942). Commercially available toothpaste has the properties like ant plaque, antimicrobial activity, tooth whitening. In addition of these properties in our project efforts are made to bring new formulation of toothpaste with more stable in terms of rheological behaviour, long term protection and stability in diverse conditions.

MATERIALS AND METHODS

Materials and ingredients

The weight of every each ingredient was decided by review previous study formulation of medicinal 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 100ml of toothpaste formulation. The ingredients of all toothpaste formulations are given in table 2 and Table 3.

Apparatus

Hot plate

Hot plate is being used to heat the several mixing compounds of toothpaste. The mixture temperature is control around 40 to 50^{o}C.Scilogex MS-H-S Analog Magnetic Hot Plate comprising of magnetic stirrerused for preparing chemicals in scientific research.

Mixer

This formulation is done by using a mixer (Sell Electric Lab Mixer). In this experiment, the ingredients are mix according to their phase. The mixing is done in 100ml beaker for each formulation and it mixed until homogenous.

Incubator

During in vitro method, nutrient agar plates were prepared by pouring 20 ml of nutrient in sterile petri dishes for antibacterial assay. Sterile petridish is used to inoculate the formulation on the toothpaste and incubator is used to controlled temperature for the cultivation of bacteria. Bacteria are obtained from oral swab by using lawn culture technique. The sterile petri dishes incubating at 37^{0}C for 24 hours and the observation is record for next three days. During this observation, the zone of inhibition of microorganism in petri discs is also measured.

Viscometer

Viscometer is an instrument that being used to measure the viscosity of toothpaste formulation. In general, the toothpaste remains stationary and a spindle moves through it.
The drag caused by relative motion of the toothpaste and a surface is a measure of the viscosity.

Formulation of toothpaste (Vidya Namdeo Dange *et al.,* 2014)

Method

The binder is premixed with solid abrasive, which is then mixed with the liquid phase, containing humectant, preservative and sweetener into a mixer. After formation of homogeneous paste, the flavour and detergent are added, mixed, milled deairated and tubed.

Some active toothpaste ingredients are

- Calcium carbonate is an abrasive agent that reduces stains on the teeth. It neutralizes acids in the saliva and provides a foaming action in the mouth.
- Calcium phosphate has been mineralization agent that enhances is mineralization, preventing dental caries, reducing enamel and/or dentin erosion, and reducing dentin hypersensitivity. The mode of action that has been hypothesized for these agents is the calcium and phosphate in soluble form which allows it to bind to enamel and dentin and to dental plaque. While there are laboratory studies demonstrating these effects, there is little clinical evidence to support these claims.

Table 2. Toothpaste Formulation Ingredients

Ingredients	Quantity used (w%)	Properties
Calcium Carbonate	28.5	Abrasive
Calcium phosphate	38.0	Remineralising agent
Sodium fluoride	0.7	Anticaries agent
Glycerine	25.0	Humectant
Soduim lauryl sulphate	1.5	Surfactant
Tragacanth	1.5	Binder
Sodium benzoate	0.007	preservative
Sodium saccharin	0.8	sweeteners
Menthol	1.50	Flavoring Agent
Titanium dioxide	0.6	Colouring agent
Water	1.52	Diluent

Table 3. Toothpastes used in the study

Group	Toothpaste	Manufacturer ingredients
G1	Colgate	Sodium fluoride, triclosan, sorbitol, hydrated silica, sodium lauryl sulphate, cellulose gum, mint, sodium bicarbonate, sodium saccharine, titanium dioxide
G2	Close up	Sodium fluoride, hydrated silica, sodium lauryl sulphate, cellulose gum, sorbitol, sodium saccharine, cocamidopropylbetaine.
G3	pepsodent	Sodium fluoride, sorbitol, PEG 32, Hydrated silica, sodium lauryl sulphate, cellulose gum, mint, zinc citrate trihydrate, sodium saccharine, titanium dioxide

Table 4. Standard values for non-fluorinated and fluorinated toothpastes

S.NO	Characteristics	Standard Values	
		Non-Fluorinated Type	Fluorinated Type
1	Hard & sharp edge abrasive particles	Absent	Absent
2	Spreadibility (cm), (max.)	8.5	8.5
3	Fineness (Particle retained on 150µ sieve.)	0.5	0.5
4	pH of 10% aq. Suspension	5.5-10.5	5.5-10.5
5	Test for Pb (maximum)	20 ppm	20 ppm
6	Arsenic (As2O3) 2 ppm 2 ppm		
7	Foaming part in ml, min. (applicable to foaming toothpaste only	50(minimum)	50(minimum)
8	Fluoride ion, ppm, max.	50	50

- Sodium fluoride is an anticaries agent; the primary action of fluoride is to make the tooth substrate (enamel and dentin) more resistant to acid attack by cariogenic bacteria. Fluoride is also bactericidal and has additional antiplaque effects.

Non-active ingredients include

- Sodium lauryl sulfate is a surfactant that promotes foaming action. The foaming action is equated with cleaning.
- Tragacanth gum is binding agents to maintain the consistency of toothpaste for mouth feel properties.
- Sodium benzoate is a preservative to prevent growth of micro-organisms.
- Humectant, such as glycerine, holds moisture so the toothpaste does not dry up.
- Flavouring is added to impart a pleasant taste.
- Sodium saccharine is added to give the toothpaste a sweet taste.
- Titanium dioxide is considered to be a colouring agent. It makes the toothpaste opaque and gives it a white colour.

Evaluation of Toothpaste (Abirami *et al.,* 2005)

This standard prescribes requirement & methods of sampling & test for toothpaste. As per guidelines toothpaste shall be of either type I (Non-fluorinated) or type II (Fluorinated).

Composition

Toothpaste shall not contain mono or disaccharides for e.g. sucrose or other readily fermentable carbohydrates. All the raw materials used shall confirm to respective Indian standard wherever they exist.

Homogenecity

The paste shall extrude from the collapsible tube or any other suitable container in which it is packed at 27±20C in the form of homogenous mass with the application of normal force & without using excessive force which could cause injury to the tubes or the container. It shall be possible to extrude bulk of contents from the container (tube) starting from crimp & then tube rolling it gradually.

Stability

The toothpaste shall not segregate, ferment or physically deteriorate during normal condition of storage & uses. When heated to a temperature of 45±20C for a period of 28 days the toothpaste shall not undergo phase separation, gassing, fermentation or otherwise deteriorate aesthetically. When cooled to a temperature of 5^0C for 1 hour after taking out & pressing the tube, the paste shall be found extrudable from the tube.

Tube inertness

Toothpaste packed in collapsible tubes or any other suitable container shall not corrode or deteriorate or cause contamination in the toothpaste during normal condition of storage & use. When heated to a temperature of 45 ± 2^0C for 10 days, the paste shall then be examined visually by extruding part of the contents. The internal surface of the tube shall be examined after slitting (cut) it, open & removing. The open contents, there should be no sign of corrosion, chemical attack or other damage. The toothpaste shall also comply with the following requirements represent in table 4)

Determination of sharp and edge abrasive particles

Extrude the paste 15-20 cm long, each from at least 10 collapsible tubes on a butter paper, test the paste by pressing it along its entire length by a finger for the presence of hard & sharp edge abrasive particles. In all cases, the material shall be free from such particles.

Determination of spread ability

Weigh about 1 g of toothpaste at the centre of glass plate (10 by 10 cm) & place another glass plate over it carefully place 2 kg wt. at the centre of the plate (avoid sliding of the plate). Measure the diameter of the paste in cm after 3 minutes. Repeat the experiment thrice & report the average.

Determination of fineness

Place about 10 gm of toothpaste, accurately weight, in a 100 ml beaker. Add 50 ml of water & allow standing for 30 minutes with occasional stirring until the toothpaste is completely dispersed. Transfer to the 150 µ IS sieve & wash by means of a slow stream of running tap water. Let the water drained from the sieve & then dry the sieve containing the residue in an oven, if there is any residue on the sieve carefully transfer it to a tare watch glass & dry it to constant mass in an oven at 105±20C.

Calculation

i. Material retained on sieve % by (Mass / Material taken) x 100
ii. Sieve is now 75µ weigh accurately 10 gm of toothpaste & proceed as above & if there is any residue on the sieve. Transfer it to a tare watch glass & dry it to constant mass in an oven at 105±20C.

Determination of pH

Take 10 g of toothpaste in 150 ml beaker. Add 10 ml of freshly boiled & cooled water (at 270C). Stir well to make a thorough suspension. Determine the pH of the suspension within 5 minutes using pH meter.

Determination of lead

The colour produced with hydrogen sulphide solution is matched against that obtain by standard lead solution.

Determination of arsenic

Arsenic is present in solution of material reduced to arsine, which is made to react with mercuric bromide paper. Stain produced is compared with a standard stain.

Foaming power

A suspension of the material in water is taken in a graduated cylinder & given 12 shakes under prescribed condition. The volume of the foamed is observed after keeping the cylinder for 5 minutes.

Procedure

Weigh about 5 grams of toothpaste, accurately in a 100ml glass beaker. Add 10 ml of water cover the beaker with a watch glass & allow it to stand for 30 minute. This operation is carried out to disperse the toothpaste. Ensure that the detergent is completely dissolved, warming the aqueous suspension if necessary. Stir the contents of the beaker with glass rod and transfer the slurry to the 250ml graduated cylinder, ensuring that no foam (more than 2 ml) is produced and no lump paste goes into the cylinder. Repeat the transfer of the residue left in the beaker with further portions of 5-6 ml of water ensuring that all the matter in the beaker is transferred to the cylinder. Adjust content in the cylinder to 50ml by adding sufficient water and bring the contents of the cylinder to 30^0C. Stir the content of the cylinder with a glass rod or thermometer to ensure a uniform suspension as soon as the temperature of the contents of the cylinder reaches 30^0C. Stopper the cylinder and give it 12 complete shakes, each shake comprising of upside-down movements. After the shakes allow the cylinder to stand still for 5 minutes and read the volume of foam + water.

Determination of fluoride ion

Fluoride ions are determined potentiometrically with the help of fluoride ion sensitive electrodes. Calculation: a graph is plotted for conc. of fluoride ion against potential (mV) on semi log graph paper. The potential is plotted on the X-axis and milligram of fluoride ion on Y-axis (on log scale). From calibration curve is determined the mg of fluoride ion in the test solution.

Concentration of fluoride ion in toothpaste (ppm) $M = 2\ a \times 10000$

a = mg of fluoride ion calculated from calibration graph

M= Mass of sample taken in gram

Stability

The toothpaste shall not segregate, ferment or physically deteriorate during normal condition of storage & uses. When heated to a temperature of 45±20C for a period of 28 days the toothpaste shall not undergo phase separation, gassing, fermentation or otherwise deteriorate aesthetically. When cooled to a temperature of 50C for 1 hour after taking out & pressing the tube, the paste shall be found extricable from the tube.

Determination of moisture and volatile matter

About 5gm of each sample material was weighed accurately and placed in a porcelain or glass dish, about 6-8cm in diameter and about 2-4cm in depth. It was dried in an air oven at a temperature of 105°C to a constant mass.

Calculation

The moisture and volatile matter of all samples were calculated by using the following formula. Moisture and volatile matter, percent by mass =100 M1 / M M1 - loss in mass on drying and, M - Mass in grams of the material taken for the test

RESULTS AND DISCUSSION

Use of fluorides has been the cornerstone of caries prevention programmes and the use of fluoridated toothpaste is by far the most common form of caries control in use 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 preventive action, very little research has been conducted to compare these properties in the experimental dental product. Hence the purpose of this study was to evaluate and compare the efficacy of commercial toothpastes with experimental 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 tooth pastes samples (Colgate, Close up, Pepsodent) and experimental toothpaste sample. All the samples were complied with BIS and they found to be of good quality. Evaluation tests were performed to view the different properties of experimental and commercial toothpastes. All the Results of evaluated parameters were given in Table 5.

In the present study, comparatively equal and rarely better results have been observed with experimental 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 experimental formulation (fig. 2). Comparison of the abrasiveness of marketed pastes with experimental formulation suggests that experimental formulation has more abrasiveness than marketed pastes. And also given significant result for cleaning ability which is similar to the results obtained in the commercial formulations.

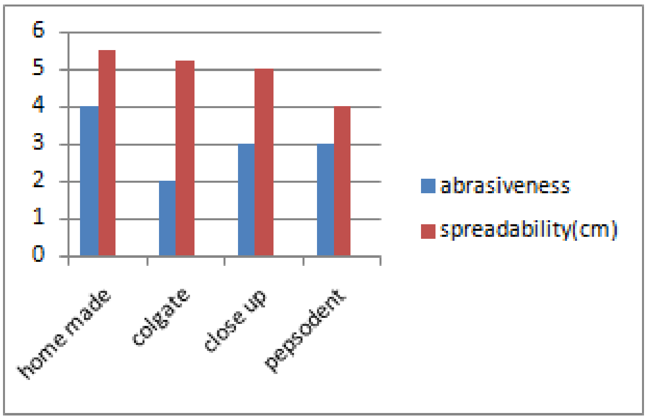

Fig. 2. Abrasiveness and Spreadability of experimental and commercial toothpastes

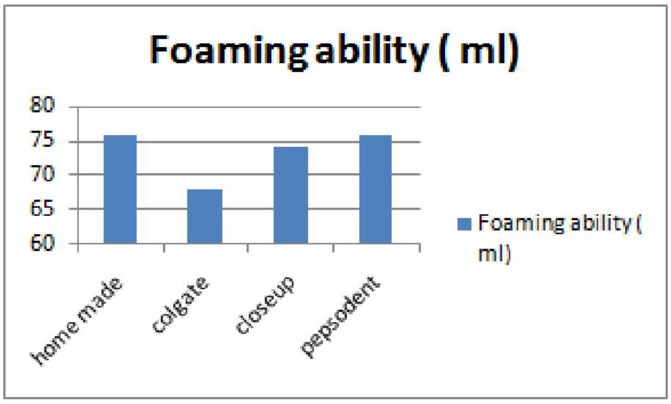

Fig.3. Foaming ability of experimental and commercial toothpastes

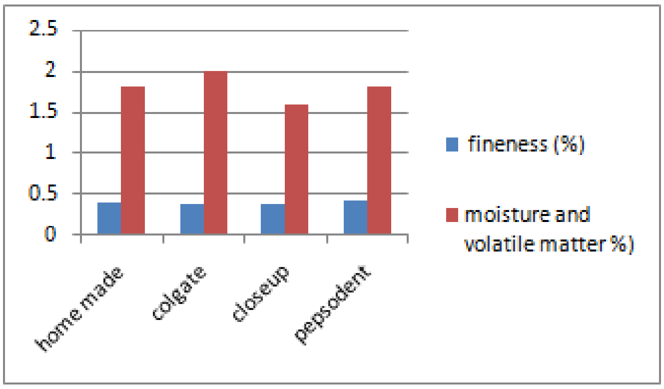

Fig. 4. Fineness (%), moisture and volatile matter (%) of experimental and commercial toothpastes

All the toothpastes were having good consistency and smooth texture. Also shown no symptoms of 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 has given no sign of corrosion or damage during normal storage conditions at a temperature of 45±2^0C for 10 days except for colgate toothpaste tube has slightly affected by corrosion. So it was confirmed that the containers of experimental, close up and pepsodent have shown good tube inertness.

Table 5. Evaluation tests for experimental and commercial toothpastes

S. No	Properties	Experimental	Colgate	Close Up	Pepsodent
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^{0}C for 28days & at 5^{0}C for 1 hour)	Good	Good	Good	Good
8	Tube inertness (at 45±2^{0}C for 10 days)	No corrosion	No corrosion	No corrosion	No corrosion
9	Fineness (% by mass)	0.41	0.39	0.37	0.42
10	Moisture & 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 limit	Exceeding limit	Within limit	Within limit
13	Fluoride ion, ppm, max.	42	36	55	48

The preferable amount of residue has retained on sieve for experimental formulation which is better than the residue obtained by colgate and close up toothpastes but little less to the residue of pepsodent toothpaste. So it was found that experimental preparation has shown reasonably good % of fineness (Fig. 3). The moisture and volatile matter present in colgate was significantly more than rest of the formulations. The percent of moisture and volatile content in experimental formulation is giving the same value as that of pepsodent and close up got the least value. These results explaining all the formulations have good moisture and volatile content (Fig. 4). The colour produced with hydrogen sulphide in test solution is less than obtained with standard solution that indicates all the samples have limited amount of lead impurities.

Stain produced by colgate sample is more than standard stain that indicates colgate 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 experimental formulations is less than standard values mentioned in Table 5.

Conclusion

Multicare toothpastes have become available. These toothpastes contain all the ingredients that would make that toothpaste multicare to include anticaries, antigingivitis, antitartar, desensitizing, and whitening. Tooth whitening is an important criterion for many of our patients when they choose toothpastes. An important role for the dental health professional is the recommendation of oral care products based upon the patient's oral conditions. For patients needing toothpaste to remove plaque, stain and for whitening, the abrasive particle in the toothpaste plays an important role.When recommending toothpaste, one should recommend toothpastes that are kinder to enamel, dentin and restorative materials. Currently, the advances in the ingredients and additives to toothpastes offer the patient some good clinical choices.

It is the responsibility of the oral care professional to understand the ingredients in toothpastes and direct patients to different products based upon their individual needs. Eventually toothpastes having an emphasized role in the maintaining the oral hygienic nature as well as preventing dental caries. Based on this pattern, experimental toothpaste was formulated by selecting suitable ingredients to get the formulation more stable. Evaluation and comparision of results with commercial toothpaste are demonstrated that experimental toothpaste is having great patronizing and engrossing passion over the marketed formulations (colgate, close up and pepsodent).

All the marketed tooth pastes and experimental toothpaste which had been evaluated complied with the standards specified by BIS. This preliminary *invitro* study demonstrated that experimental toothpaste was equally efficacious as three commercially popular toothpastes in terms of all evaluation properties of toothpaste. Hence, by the evidence of *invitro* studies, it is concluded that experimental toothpaste formulated in laboratory was found to be of good quality.

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