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PREPARATION AND EVALUATION OF HERBAL DENTIFRICE

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ABSTRACT

Dentifrices are the products which are mainly used to maintain the oral hygiene such as freshness of mouth and to avoid tooth decay. The oral hygiene can be maintained throughout the day by using a variety of dentifrices prepared by herbal and synthetic ingredients. This work was carried out to prepare a tooth powder which can be used as a tool for proper oral hygiene and to overcome the side effects of the conventional toothpowder prepared by synthetic ingredients. The toothpowder was prepared by using various herbal ingredients which have the antibacterial, antiseptic properties. *Eugenia cryophyllus, Glycyrrhiza glabra, Pipernigrum, Cinnamonum zeylanicum, Foenuculum vulgare, Quercus infectoria, Piper longum, Accacia Arabica, Terminalia chebula* are the herbal ingredients were used in this work to formulate ideal tooth powder which can satisfy all the required properties to keep the mouth fresh and to prevent tooth decay caused by bacteria. The prepared tooth powder was evaluated for its organoleptic and physical characteristics such as colour, odour, taste, stability, foam ability and abrasiveness to ensure that it possesses all the desired features to use against the dental diseases. The result was found to be within the permitted limits.

KEYWORDS

Oral hygiene, Herbal ingredients and Anti-bacterial effect.

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INTRODUCTION

Oral hygiene is a vital key to maintain good appearance, impression of an individual and gives self-confidence. The tooth consists mainly of two parts, crown and the root. The crown of the tooth is enveloped by outer surface called enamel and it is the toughest tissue in the tooth. The chief composition of enamel is hydroxylapatite other than that it consists of keratin and water[1]. Dentine is the underneath part of the enamel, which is a composite

of hydroxylapatite. It also contains of 70% of the collagen water. Fluorine is the main component of dentine. Oral contain of not only tooth but also saliva for simple to swallow the food. Saliva is the main element which intended for lubricating the food and to maintain suitable environment in the mouth. Saliva is produced by various glands such as lingual, labial, buccal and palatal are the larger and smaller glands which produce saliva continuously to maintain the tooth environment in the dynamic state[2]. Proteins, bacteria, enzymes and mucco polysaccharide are present in the saliva and the inorganic materials like calcium, chloride, sodium, phosphate, potassium ions etc. The calculus, plaque, periodontal diseases are the most important issues related to tooth. It is mostly caused by bacterial action and mineralized deposition leads to form calculus. These diseases are mostly due to the negligence in proper caring of tooth, so it can be controlled and prevented by proper brushing by using effective and efficient toothpastes and tooth powders[3].

Dentifrice can be used as prophylactic cosmetic for tooth to prevent and control bad breath and tooth decay. Dentifrice can be prepared by herbal and synthetic ingredients. Now a day's herbal formulation are high in demand and require due to its efficiency to avoid the side effects when compared with synthetic ingredient formulations. Tooth powders and tooth paste are based on its abrasive property, the powder and paste applied on the tooth to rub against the tooth which helps to eradicate the deposited food debris and minerals from tooth[4].

The herbal dentifrices are available in different formulations such as tooth powder, toothpaste, mouthwashes etc. Plaques can be removed by effective toothpowder and toothpaste due to the presences of ingredients which possess the antibacterial, antiseptic property and it also gives fresh and cool feeling[5].

The present study was to prepare and evaluate the herbal dentifrice of organoleptic properties, moisture content, foaming character, swelling index, flow property, antibacterial potential and In-vitro antioxidant activity.

MATERIALS AND METHODS

Materials

Iingredients of herbal dentifrice

Methods

Herbal tooth powder was prepared using *Eugenia cryophyllus, Glycyrrhiza glabra, Piper nigrum, Cinnamonum zeylanicum, Foenuculum vulgare, Quercus infectoria, Piper longum, Accacia Arabica, Terminalia chebula.* All the herbal ingredients were dried and grounded using domestic mixer. Then the tooth powder passed into sieve and made fine powder.

EVALUATION

Identification of Organoleptic properties[6,7]

Colour

The prepared toothpaste was evaluated for its colour. The colour was checked visually.

Odour

Odour was found by smelling the product. Taste was checked manually by tasting the product.

Determination of moisture content[8]

Moisture content was determined by Infra-Red moisture balance. The scale lamp was turn on by means of toggle switch. By turning the scale adjusting knob the scale was rotated until the 100% mark coincides with the index. The pointer was moved to the index by turning the pointer adjusting knob in a direction opposite to that in which the pointer must move to coincide with the index. The scale was rotated until 0% mark coincides with the index. Then, the pointer was above the scale .The lamp housing was raised and carefully distributed the test each powder on the sample pan until the pointer returned to the index. That weight was then corresponds to 100 divisions of the scale. The lamp housing was lowered and I.R. was turned on by means of the toggle switch. The autotransformer was readjusted to control the proper setting for the powder material. The sample was placed on the sample pan. The autotransformer control was set an arbitrary setting and the I.R. lamp was turn on. The result was observed and final moisture content was recorded.

Determination of foaming character[8]

1gm of drug was taken in 500ml conical flask containing 100ml of boiling water. Moderate boiling temperature was maintained for 30minutes. Cooled and filtered in 100ml volumetric flask and volume was made up to 100ml with water. The decoction was poured into 10 test-tubes in successive portions of 1-10ml and the volume of each test-tube was made up to 10ml with water. Then test-tubes were shaken for 15 sec and allowed to stand for 15 min and the height of the foam was measured. The foaming index was calculated according to the height of foam observed in every test-tube.

Determination of swelling index[8]

1gm powder was accurately weighed and carefully introduced in 100ml glass - stopper measuring cylinder and 25ml of distilled water was added (measure the volume as form of initial volume) and mixture was shaken thoroughly in every 10 min for 1 hour and then, allowed to stand for 3 hours at room temperature. Volume was measured in ml occupied by the drug. All marketed herbal tooth powders were taken separately into experiment. The mean value of the individual determinations was calculated, related to 1g of drug.

S.I. = Final vol. - Initial vol.

$$S.F. = \frac{\text{Swelling index} \times 100}{\text{Initial vol.}}$$

Determination of flow property[9]

A funnel was taken and was fixed with clamp to the stand. A graph paper was kept below the funnel and the height between graph paper and bottom of the funnel stem was measured. Then, 50gm of powder was weighed and poured into funnel by blocking the orifice of the funnel by thumb, the thumb was removed. The powder started flowing down onto the graph paper and formed a cone shaped pile until the peak of pile become touched to the bottom of the funnel stem. All marketed herbal tooth powders were taken separately into experiment. Then, the angle of repose was calculated by following formula.

$$\tan\theta = \frac{H}{R}$$

The flow property was observed as -

(Powder flow property when $\theta<25$ Excellent, 25-30 Good, 30-40 Passable, >40 very poor.

Determination of Bulk density[10]

50gm of powder was accurately weighed and carefully introduced into a 100ml graduated (1ml) measuring cylinder. The cylinder was dropped at 2-seconds interval onto a hard surface three times from a height of a 1 inch to equalize upper surface of powder. All marketed herbal tooth powder was taken separately into experiment. Then, the volume of powder was noted and the bulk density in gm/ml was calculated as

$$\text{Bulk density} = \frac{\text{Wt. of drug}}{\text{Bulk volume}}$$

Determination of Tapped density[10]

50gm of powder was accurately weighed and carefully introduced into a 100ml graduated (1ml) measuring cylinder. Measuring cylinder was fitted on the tapped density apparatus. The instrument was switched on. It raised the cylinder on the base from a height of about 4 inches. Number of strokes given until further bulk volume was changed. Then, volume of powder was noted and the tapped density in gm/ml was calculated as.

$$\text{Tapped density} = \frac{\text{Wt. of drug}}{\text{Tapped vol.}}$$

Determination of Particles size by mechanical sieve shaker[10]

Select standard sieve set (IP or USP). Arranged them in such a manner that the coarsest at the top and finest at the bottom. 50gm powder was weighed and placed on the coarsest sieve set. Above sieve set fixed on a mechanical shaker and clamp it tightly. Switch on the mechanical shaker and timer was set for 15min. When the shaker automatically stops, sample was collected which retained on each sieve into a paper and weighed. All marketed herbal tooth powders were taken separately into experiment. Average particle size was calculated as

$$\text{Avg. particle size} = \frac{\Sigma nd}{\Sigma d}$$

Where, Σnd = Sum of arithmetic mean × wt. retained on a sieve

Σd = Sum of wt. retained on a sieve.

Spread ability

Spread ability was evaluated by spreading the powder manually.

Abrasiveness

It was evaluated manually.

Foamability

The foamability 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.

Stability

The product was maintained in different temperature conditions to check its stability.

***In-vitro* Antibacterial activity**[11]

In-vitro antibacterial activity of all the four extracts was evaluated by using agar well diffusion method.

Agar well diffusion method

Preparation of Agar media

Suspended 9.5gm Mueller Hinton Agar media (MHA) in a 500ml conical flask and 250ml distilled water was added. Then, it was heat on hot plate with frequent agitation until it completely dissolved. Then, media was sterilized in autoclave at 121ºC for 1 hour.

Procedure

Approximately 25ml of Mueller-Hinton Agar (MHA) was poured into sterile petri-dish and allowed to solidify. 50µl of bacterial inoculums was spread on the solidify MHA media by using sterile spreader. In each of these plates two wells (5mm diameter) was punched into the agar by using sterile cork borer. Then, working concentration of 100mg, 150mg, 200mg and 250mg dilution were prepared from 500mg/ml of stock solution of each extracts and 150µl of each extract was separately added into wells and allowed to diffuse at room temperature. Equal volume of alcohol was used as negative control and standard antibiotic (Chloramphenicol) was used as positive control. The plates were incubated for 24hours at 37°C and the diameter (in mm) of clear zone of growth inhibition was recorded and measured with the help of radius scale.

***In-vitro* antioxidant activity by β-Carotene-linoleate bleaching assay**[12]

The antioxidant activity of the extract was assayed based on the β-carotene bleaching (BCB) method developed by Velioglu *et al.* β-carotene (0.2mg in 1ml chloroform), linoleic acid (0.02ml) and Tween 20 (0.2ml) were transferred into a round-bottomed flask. The mixture was then added to 0.2ml of sample extract or standard (Quercetin) or ethanol (as control). Chloroform was removed at room temperature under vacuum at reduced pressure using a rotary evaporator. Following evaporation, 50ml of distilled water was added to the mixture, and then shaken vigorously to form an emulsion. Two milliliter (2ml) aliquots of the emulsion were pipetted into test tubes and immediately placed in a water bath at 50°C. The absorbance was read at 20 min intervals for 2 h at 470nm, using UV/VIS spectrometer T70. Degradation rate (DR) was calculated according to first order kinetics, using the following equation based on Al-Saikhan, Howard, and Miller.

$\ln (a / b) \times 1/t = DR_{sample}$ or $DR_{standard}$

Where,

ln is natural log, *a* is the initial absorbance (at 470nm) at time 0,

b is the absorbance (at 470nm) at 0, 20, 40, 60 min,

t is the initial absorbance (470nm) at time 0.

Antioxidant activity (AA) is expressed as percent of inhibition relative to the control, using the following formula:

$$AA = \frac{DR_{control} - DR_{sample\ or\ standard}}{DR_{control}} \times 100$$

RESULTS AND DISCUSSION

Organoleptic characteristics of herbal tooth powder were showed in the Table No.2. The prepared herbal tooth powder showed characteristic odour, sweet and sour taste. The percentage of moisture content showed in the Table No.3. The prepared herbal tooth powder was containing percentage of moisture (3.8%). Foaming index in powder form when compared alcoholic extracts, herbal tooth powder showed in Table No.4, less than 1cm. Swelling index and swelling factor were found 2ml and 9.52% respectively. Flow property of herbal

tooth powders was calculated by angle of repose method. θ˚ = 22.52, which signifies excellent flow property. Both of tapped density = 0.39gm/ml and Bulk density = 0.51gm/ml were found for herbal tooth powder. Carr's index 22.52% and Hausner's ratio 1.20 were also shown good flow property.

In-vitro antibacterial activity on prepared herbal tooth powder was performed against *Staphylococus aureus* (gram positive) and *E.coli* (gram positive) by agar well diffusion method using different doses results were shown in Table No.6. Antibacterial activity against *Staphylococus aureus* (gram positive) more than *E.coli* (gram negative).

In- vitro antioxidant activity on prepared herbal tooth powder by beta carotene - linoleate bleaching assay was performed to calculate percentage inhibition of free radicals formed by linoleic acid thus 100mg/ml concentration of prepared tooth powder were taken into experiments against quercetin (standard). The percentage inhibition was of antioxidant activity among all herbal tooth powder calculated as 69.35%. Sufficient quantity should be applied with tooth brush and to be used twice daily early in the morning and before going to bed or as advised by the dentist for best result. It is useful against bacterial infections and to maintain freshness of mouth.

Table No.1: Ingredients of herbal dentifrice

S.No	Name of the drug	Biologial source	Family	Cheical constituent	Medicinal uses
1	CLOVE	*Eugenia cryophyllus*	Myrtaceae	Eugenol, Gallotannic, Eugintin	Antioxidant Toothache Dental plaque
2	LIQUORICE	*Glycyrrhiza glabra*	Fabaceae	Glycyrrhizic acid, Glycyrrhizin	Antiviral Antibacterial Sweetening agent
3	BLACK PEPPER	*Piper nigrum*	Piparaceae	Volatile oil, Piperine, Peperidine, pungent resin	Carminative Aromaic stimulent
4	CINNAMON	*Cinnamonum zeylanicum*	Lauraceae	Cinnamic acid Cinnamic aldehyde	Flavouring agent Germicide
5	FENNEL	*Foenuculum vulgare*	Umbeliferae	Anethole fenchone	Aromatic Flavoring agent
6	GALL NUT	*Quercus infectoria*	Fagaceae	40-60% of tannic acid	Astringent
7	THIPILI POWDER	*Piper longum*	Piperacae	Piperine Piperidine Pungent resin	Aromatic stimulant
8	INDIAN GUM	*Accacia arabica*	Leguminacae	Arabin Arabinoes	Binding agent
9	MYROBALAN	*Terminalia chebula*	Combretaceae	Galic acid Chebulic acid	Astringent
10	HIMALAYAN SALT	----	------	Sodium chloride Magnesium Potassium	Flavouring, Sweetening agent

Table No.2: Evaluation of Herbal Dentifrice

S.No	Parameters	Observations
1	Colour	Brown
2	Odour	Characteristic
3	Taste	Sweet and sour
4	Stability	Stable
5	Spread ability	Easily spreadable
6	Abrasiveness	Good abrasive
7	Formability	Good

Table No.3: Determination of moisture content

S.No	Temperature (˚c)	Time (min.)	Moisture content (%)
1	100^{o}	3.10	3.8

Table No.4: Determination of foaming character

Foaming character	Powder	Extract
	Less than 1cm	Less than 1cm

Table No.5: Determination of swelling index

S.No	Sample amount (gm)	Volume (ml)		Swelling Index (ml)	Swelling Factor (%)
		Initial vol.(ml)	Final vol. (ml)		
1	1	21	23	2	9.52

Table No.6: Determination of flow property

S.No	Tapped density (gm/ml)	Bulk density (gm/ml)	Hausner's ratio	Carr's index (%)	Avg. particle size (µm)
1	0.39	0.51	1.20	22.52	316

Powder flow property when θ < 25 Excellent, 25-30 Good, 30-40 Passable, >40 very poor.
Carr's index < 23%, Hausner's ratio <1.25 to good in flow

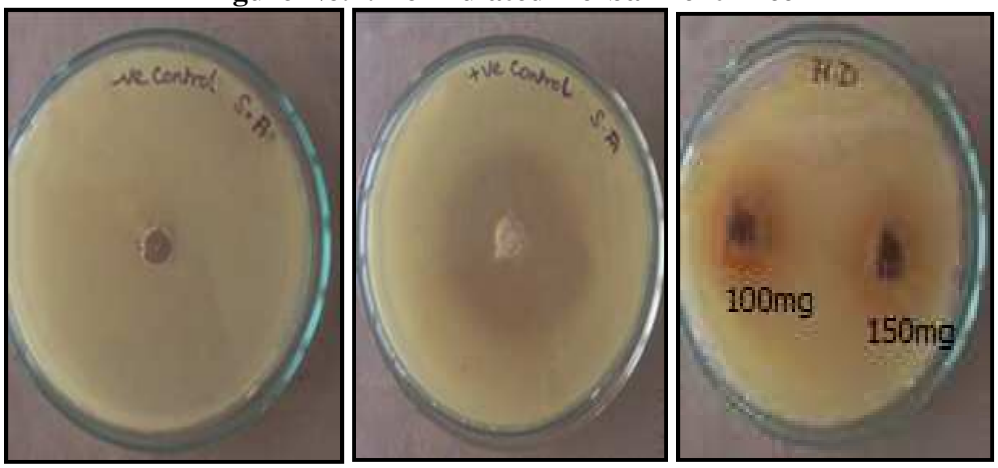
Table No.7: *In-vitro* Antibacterial activity against *S.aureus and E.coli* on herbal tooth powder by Agar well diffusion method

S.No	Concentration (mg)	Zone of Inhibition (mm)	
		S.aureus	*E.coli*
1	100	10.7	9
2	150	12.3	11
3	Alcohol (Negative control)	0	0
4	Chloramphenicol -100mg (Positive control)	23	20

Table No.8: Determination of *In-vitro* antioxidant activity by β-Carotene-linoleate bleaching assay

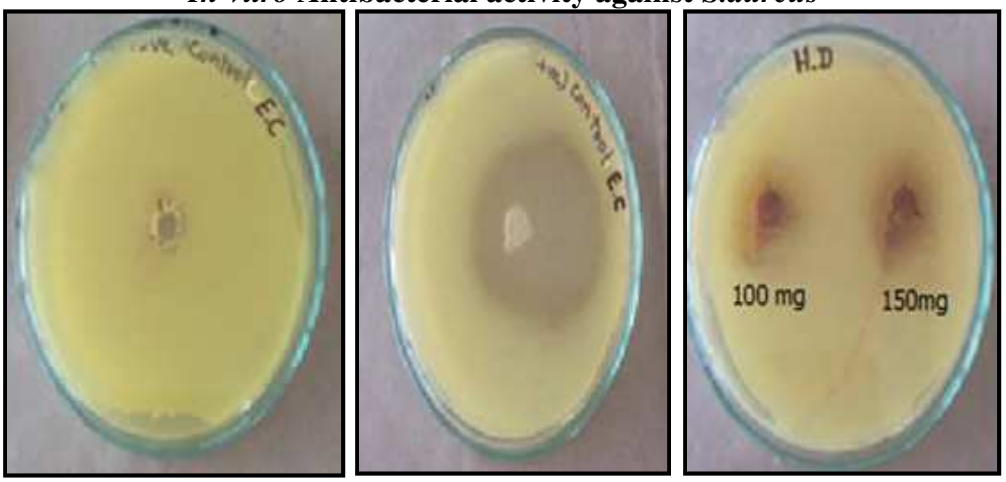
S.No	Sample	Absorbance at 470nm		% inhibition of Antioxidant activity
		At O min.	Over 60 min.	
1	Herbal tooth powder	0266	0.298	69.35
2	Quercetin	0.270	0.295	74.19

Figure No.1: Formulated Herbal Dentifrice



Negative control **Positive control** **Samples**

In-vitro* Antibacterial activity against *S.aureus



Negative control **Positive control** **Samples**

In-vitro* Antibacterial activity against *E.coli

CONCLUSION

The ingredients used in the present work, was screened and selected to have antibacterial effect and to maintain oral hygiene as it can be claimed by its results as efficient and successful tooth powder. Any herbal tooth powder is considered safe to use twice a day and it does not cause any harmful effects, instead imparts good freshness and away from bad odour. Oral hygiene can be maintained in a reliable, safe and inexpensive way by using herbal tooth powder.

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CONFLICT OF INTEREST

Authors declare no conflict of interest.

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