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# Formulation and development of SLS free toothpaste.

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#### Abstract

The focus of the research is to formulate and develop the SLS free toothpaste to reduce the adverse effects caused by the SLS. It is observed that this surfactant is" Moderate Hazard" that has been linked to Cancer, neurotoxicity, Skin irritation, Organ toxicity, endocrine disruption and can cause harm if taken in excess. In this work we have revealed the Natural Surfactants which can be helpful in replacement of SLS in toothpastes. 2 naturally occurring surfactants Liquorice and Hingot Fruit are used in desired combination to produce the foam in toothpastes. The foam produced is concrete foam and by using this natural saponins we can reduce the toxicity related to SLS. This work is also comprised of the extraction process of these natural surfactants along with evaluation parameters. **Keywords** – SLS, Liquorice, Hingot fruit, Carbopol, Foaming.

#### 1. Introduction

Most of the toothpaste nowadays contains sodium lauryl sulphate which is used as surfactant and gives the foam it removes the stain and plaque from teeth. SLS is consists of a 12 carbons tail which is attached to sulphate group and it is a sodium salt of a 12 carbons alcohol that has been esterified with sulphuric acid. SLS present in the toothpaste shows adverse effects like allergic reactions, mouth ulcers or it might be toxic also. If large amount of toothpaste is swallowed it may show the toxic effect, hence toothpaste containing SLS should be avoided in children and not intended to stay in mouth after brushing. Some of herbal toothpastes in India also uses SLS as a surfactant. Instead of using SLS other naturally occurring surfactants can be used in manufacturing of the toothpaste. Some natural surfactants which can be used are liquorice and hingot fruit.

#### 1.1. Liquorice

Liquorice is a common name of Glycyrrhiza glabra and belongs to family Fabaceae. Root of liquorice contains 10-25% of Glycyrrhizin which is saponin glycoside and gives foam. It is main component of the Liquorice which shows the saponin properties along with glycyrrhizitenic acid. It is also used as emulsifier and gel forming agent.MW of Glycyrrhizin – 822.92

Class	Present component	Effective against	
Chalcones	Licochalcone A	Candidiasis	
Flavonoids	Liquritigenin	Candidiasis	
Isoflavonoids	Glabridin	Candidiasis	
	Licoricidin	Periodoutal disease	
	Licorisoflavan A	Periodoutal disease	
Pterocarpens	Glyarrhizol A	Dental carries	
Saponins	Glycyrrhizin	Candidiasis	
	Glycyrrhizic acid	Dental carries	
	18 B-glycyrrhetinic acid	Candidiasis and periodoutaldisesase	
Stilbenes	Gancanin G	Dental disease	

 Table no. 01: Pharmacological actions of liquorice

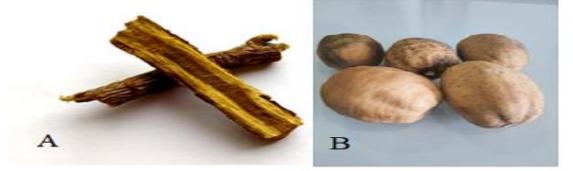


Fig. no.01: A. Liquorice, B. Hingot fruit

## 1.2. Hingot fruit

Hingot fruit (Balanites aegyptiaca) is a species of tree classified as a member of Zygophyllaceae. Basically, hingot fruit is used as Antibacterial agent. The seeds of the hingot fruit used as expectorant, antibacterial and antifungal. Fruit is used in whooping cough, also in leukoderma and other skin diseases.

This plant has been reported to be an antihelmintic, a purgative, vermifuge, febrifuge, emetic and can also cure other types of ailments like skin boils, leukoderma, malaria, wounds, colds, syphilis, liver and spleen disorders, and aches.

It shows the anti-bacterial, anti-diabetic, anti-fungal, Anti-helmintic, Anti-oxidant, Anti-inflammatory, Anticancerous, Molluscicidal. Extract of hingot fruit shows the properties of surfactant. Hingot Fruit is a source of saponin based surfactant.[4]

B. aegyptiaca contains different types of saponins, namely, balanitin -1, 2, 3, 4, 5, 6 and 7. This saponins are responsible concrete and stable foam.

# 2. Methodfor extraction

## 2.1. Extraction method for Liquorice

3. 50gm of liquorice powder was weighed and then 300ml of distilled water added in it. The mixture was on standing for 30min. After that residue was filtered and filtrate was collected. pH was adjusted to 2.8 by the addition of acid. Then precipitate was collected by filtration and washed with cold water to remove the excess of water from precipitate.



Fig. no.02: A. Filtration of liquorice and water mixture, B. pH adjustment to form precipitate of glycyrrhizin.

# 4. Characterization of prepared formulations

## 4.1. Purity and compatibility of ingredients

The purity and compatibility of glycyrrhizin and hingot was determined using FTIR technique. The graphs obtained and then compared with standard.

## 4.2. Foaming ability

1gm of prepared formulation was diluted with 9ml of distilled water in the test tubes. Then this test tube was shaken for 5 min and allow to stand for 15 min. then the length of foam was measured.

## 4.3. Spreadability

It is the test to check the spreading ability of semi solid formulation. Where 1 gm of sample was placed in between two glass slide and 1000gm weight was placed on it for 3 min.

After 3 min, the diameter of the formulation was measured thrice and mean was calculated. And then spredability was calculated using the formula,

$$S = MxL/T$$

Where, M is weight placed L is diameter and T is time

4.4. pH determination

By using digital PH meter, the pH of the prepared batches was determined. The pH meter was calibrate using phosphate buffer at pH 4, 7 and 9.2

4.5. Viscosity determination

The viscosity of formulations was determined using Brookfield's viscometer at different RPM using 94 spindle.

4.6. *Collapsibility of formulation.* 

Collapsibility of formulation was determined by applying the pressure on the tubes in which paste was filed earlier.

## 5. Formulation of batches

Table includes the formula for different batches having different concentration of drug and excipients. The formula represents % weight by weight of the components.

Tuble Ho.02. I official for balences ( 76 weight by weight)									
Sr. no.	Component	F1	F2	F3	F4	F5	F6	F7	F8
1.	Clove oil	3.74	3.74	3.74	3.74	3.74	3.74	3.74	3.74
2.	Carbopol	4	5	4	5	5	4	5	4
3.	PEG 400	75	75	75	75	75	75	75	75
4.	Glycerine	25	25	25	25	25	25	25	25
5.	Methyl paraben	9	9	9	9	9	9	9	9
6.	Propyl paraben	1	1	1	1	1	1	1	1
7.	Sodium saccharine	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
8.	Glycyrrhizin	-	-	0.3	0.1	-	-	0.3	0.1
9.	Hingot	0.8	0.5	-	-	0.8	0.5	-	-
10.	Distilled water	Q.S.							

 Table no.02:
 Formula for batches ( % weight by weight)

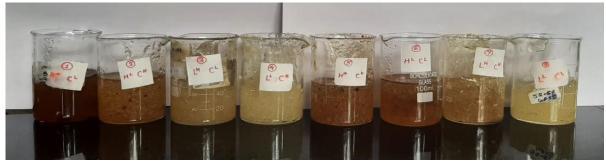


Fig. no.03: Formulated batches

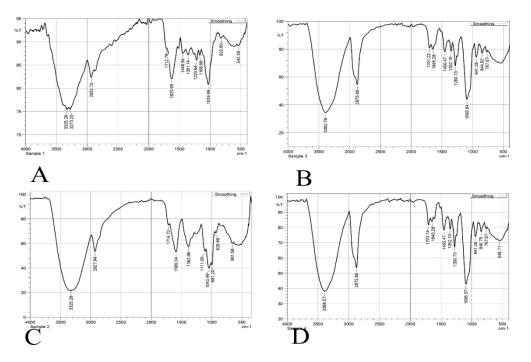
# 6. Result and discussion

# 6.1. Purity and compatibility of ingredients

FTIR(SHIMADZU) was performed for 4 samples. 2 samples were glycyrrhizin and hingot and other 2 are formulations developed by using glycyrrhizin and hingot. The graphs were obtained and compared with standards.

Structure of glycyrrhizin contains groups like O-H, O=C-OH And C-C=O-C which shows peaks at 3325, 1712, 2933 frequency. This groups are retaining in the formulation which implies that excipients do not interfere with foam forming groups in the glycyrrhizin.

Similarly, in hingot groups like O-H, C-C=O-C, C=O, C-O shows peak at 3325, 2927,1714 and 1093 frequency which are also retained in formulation.



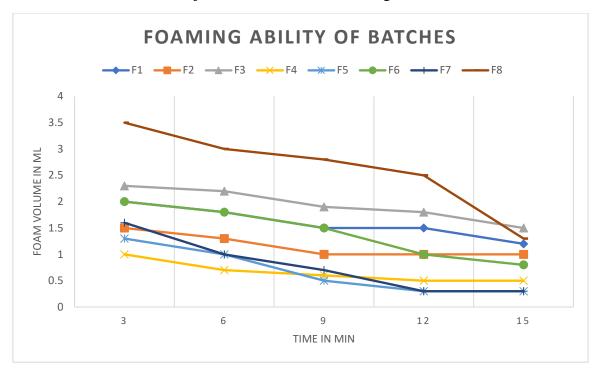
**Fig.no.04:** FTIR graphs for A Glycyrrhizin, B. formulation with glycyrrhizin, C. Hingot, D. formulation with hingot

## 6.2. Foaming ability

Foaming ability for the formulations were check and it is concluded that F3 has highest foaming, followed by F8 and F1.



Fig. no.05: Foaming ability

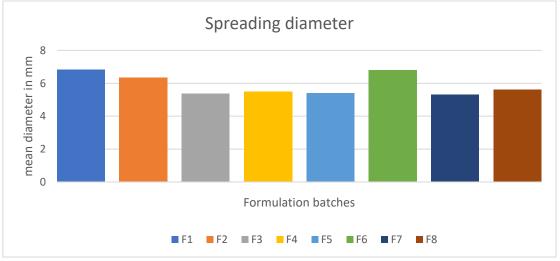


Graph no.01: Time in min Vs. Foam volume in ml

Foam of F3 was found to be highest, 1.5 cm after 15 minutes, followed by F1 and F8 was 1.3 cm. The foam of F5 and F7 was found to be lowest as 0.3 cm.

## 6.3. Spreadability

Spreadability and viscosity has corelation in between. Formulation with high viscosity shows less spreadability and low viscous formulation show high spreadability.



Graph no.02: Formulation batches Vs. Mean diameter in mm Table no.03: Readings for spreadability

Sr.no.	Formulation	Calculated
	batches	spreadability
		(gm.cm/sec)
1.	F1	37.94
2.	F2	35.16

3.	F3	29.77
4.	F4	30.55
5.	F5	30
6.	F6	37.77
7.	F7	29.44
8.	F8	31.11

Batch F5 and F7 shows less spredability this is because of high viscous nature of the formulation. Formulation with high viscosity shows less spreading. Both batches contain high concentration of Carbopol and liquorice or hingot this imparts the high viscosity to the formulation.

# 6.4. pH determination

pH for all the formulation was determined using pH meter and the values of formulation lies in acidic pH which shows the suitability of formulation for oral flora.

Sr.No.	Formulation batches	pH
1.	F1	4.16
2.	F2	3.85
3.	F3	3.83
4.	F4	3.87
5.	F5	3.85
6.	F6	3.88
7.	F7	3.60
8.	F8	3.97

Table no.04: Readings for pH

The ideal pH for gel toothpaste lies in between 3.6 to 6.5. All the formulation batches have the pH I in between 3.6 to 6.5 expect the F7 this is because of high concentration of glycyrrhizin, as glycyrrhizin contains carboxylic group in structure.

# 6.5. Viscosity determination

The viscosity of all formulations was determined using Brookfield's viscometer. The viscosity of F4, F5 and F7 was found to be very high. This are the formulation which has high concentration of Carbopol. Carbopol gives the viscosity to the formulation. And the viscosity of other formulation found to be appropriate.

# 6.6. Collapsibility of formulation

The collapsibility of formulations was determined. All formulation expects F4, F5 and F7 were collapsible. Collapsibility is also related to the viscosity of the formulation. Hence, those formulations have high viscosity were not collapsible.

#### Conclusion

Toothpaste containing Glycyrrhizin and hingot was found to have good foaming properties, along with appropriate pH, spreadability, viscosity and collapsibility. From FTIR determination it is concluded that ingredients used as surfactant are compatible with other excipients. Batches with low concentration of Carbopol also shows good viscosity and collapsibility as well as compatibility.

Groups present in the glycyrrhizin and hingot retains their properties of foaming even in presence of excipients and gives stable foam. These 2 ingredients together will give more foaming and cleaning action. Along with there foaming action, other pharmacological actions which plays important role in maintaining oral hygiene are also taken in to consideration.

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#### **Confect of Interest**

All authors declare there is no conflict of interest.

#### References

- 1. Kokate.C, Purohit.A, Gokhale.S, Pharmacognosy, NiraliPrakashan,2006. 34: p.212-213.
- 2. Dr.Mukherjee.P. Quality control of herbal drugs, an approach to evaluation of botanicals, Business horizon, New Delhi, 2012. 1.
- 3. Dr.Khandelwal.K, Practical pharmacognosy techniques and experiments, Niralipublicatrions, Pune, 2013. 22.
- 4. Barminas.K, Nkafamiya.J, Akiterinwa.A,Extraction and evaluation of a saponin based surfactant from Balaniteaegyptiaca plant as an emulsifying agent, IJISET,2015. **2**.
- 5. Michael O. Ugwaha, Chinenye J. Ugwah-Oguejioforb, Emmanuel U. Etukc, Shaibu O. Belloc, Adamu A. Alierod, Evaluation of the antiulcer activity of the aqueous stem bark extract of Balanites aegyptiaca L Delile in Wistar rats, Journal of Ethnopharmacology, May 2019.
- Bishnu P. Chapagain, ZeevWiesman, Leah Tsror (Lahkim), In vitro study of the antifungal activity of saponinrich extracts against prevalent phytopathogenic fungi, Industrial Crops and Products, August 2007. 26(2): p.109-115.
- 7. SelijaKhwairakpam, Indu Siva Ranjani Gandhi, Assessment of the potential of a naturally available foaming agent for use in the production of foam concrete, Materials Today: Proceedings 32, May 2020.
- Shahira Mohammed Ezzat, Amira Abdel Motaal& Sally Abdel Wanees El Awdan, In vitro and in vivo antidiabetic potential of extracts and a furostanol saponin from Balanites aegyptiaca, Pharmaceutical Biology, 2017. 55(1): p. 1931-1936.
- Priyal Sharma, Mahesh Kumar Saini, Jagdish Prasad, Vinod Singh Gour, Evaluation of Robustness of the Biosurfactant Derived from Balanites aegyptiaca (L.) Del., Journal of Surfactants and Detergents, March 2019. 22(2): p. 403-408.
- Dr. A. H. Mohamed, K. E. H. Eltahir, M. B. Ali, M. Galal, I. A. Ayeed, S. I. Adam and O. A. Hamid, Some Pharmacological and Toxicological Studies on Balanites aegyptiaca Bark, phytotherapy Research, 1999. 13(5): p. 439-441.
- 11. Bishnu P. Chapagain and ZeevWiesman, Determination of Saponins in the Kernel Cake of Balanites aegyptiaca by HPLC-ESI/MS, Phytochemical Analysis, 2007. **18**(4): p. 354-362.
- 12. Chen Sun, YuchunXie, Qinglai Tian, Huizhou Liu, Separation of glycyrrhizic acid and liquiritin from Liquorice root by aqueous nonionic surfactant mediated extraction, Colloids and Surfaces A: Physicochemical and Engineering Aspects, 2007. **305**(1-3): p. 42-47.
- 13. Yu Fu, Jun chen, Yan jing li, Yun feng Zheng, Ping Li, Antioxidant and anti-inflammatory activities of flavonoids separated from Liquorice, Food Chemistry, 2013. **141**(2): p. 1063-1071.

- 14. Zjian Wang, Xiuhua Zhao, Yuangang Zu, Weiwei Wu, Yuanyuan Li, Zijing Guo, Li wang, Lingling Wang, Liquorce flavonoids nanoparticles prepared by liquid antisolvent recrystallization exhibit higher oral bioavailability and antioxidant activity in rat, Journal of Functional Foods, 2019. **57**: p. 190-201.
- 15. Fangilian yang, Tiantin Chu, Yujing Zhang, Xitong Liu, Guoxiang Sun, Zhenhovgchan, Quality assessment of Liquorice from different sources by multiple fingerprint profiles combined with qualitative analysis, antioxidant activity of chemomortile method, Food Chemistry, 2020. **324**.
- 16. Jun-Gang Ma, Zhi-Long Xiu, Dai-Jia Zhang, Ling-Yun Jia, Concentration and separation of glycyrrhizic acid by foam separation, Journal of Chemical Technology and Biotechnology, 2002. **77**(6): p. 720-724.
- 17. C Messier, F Epifano, S Genovese, D Grenier, Licorice and its potential beneficial effects in common ora-dental diseases, Oral Diseases, 2012. **18**(1): p. 32-39.
- 18. Somayeh Nazari, Maryam Rameshrad, HossienHossienzadeh, Toxicological effects of Glycyrrhiza glabra (Licorice): A Review, Phytotherapy Research, 2017. **31**(11): p. 1635-1650.
- 19. A. E. Badr, N.Omar, F. A. Badria, A laboratory evaluation of the antibacterial and cytotoxic effect of Liquorice when used as root canal medicament, International Endodontic Journal, 2011. **44**(1): p. 51-58.
- 20. Zhenzuo Jiang, Yuefei Wang, Yufeng Zheng, Jing Yang, Lei Zhang, Ultra high performance liquid chromatography coupled with triple quadrupole mass spectrometer and chemometric analysis of Licorice based on the simultaneous determination of saponins and flavonoids, Journal of Separation Science, 2016. **39**(15): p. 2928-2940.
- 21. Jia Cai, Paul Li, Chemical separation of Bioactive Licorice compounds using capillary electrophoresis, Journal of Liquid Chromatography & Related Technologies, 2007. **19**: p. 2805-2817.
- 22. Qian Chen, Youg sun, Jiangjun Wang, Guowen Yan, Zhaoyuan Cui, Hongli Yin, Haitaian Wei, Preparation and characterization of glycyrrhetinic acid-modified steric acid-grafted chitosan micelles, Artificial cells, Nanomedicine, and Biotechnology, 2015. **43**(4): p. 217-223.
- 23. Na Li, Ting Zhou, Fei Wu, Rui Wang, Qing Zhao, Ji-Quan Zhang, Bai-Can Yang, Bing-Liang Ma, Pharmacokinetic mechanism underlying detoxification effect of Glycyrrhiziae Radix et.Rhizoma (Gancao) : drug metabolizing enzymes, transporters and beyond, Expert Opinion on Drug Metabolism & Toxicology, 2019. **15**(2): p. 167-177.