Indian Internet Journal of Forensic Medicine & Toxicology
Vol. 11, No. 2 April-June 2013, 35-40
DOI : 10.5958/j.0974-4487.11.2.008

Original Article

Forensic Analysis of some Tooth Pastes of Indian Brand

Roma Sharma[1], Vibhuti Narayan Singh[1], Anu Singla[1]*, Vinod Dhingra[2]

[1]Institute of Forensic Science and Criminology, Bundelkhand University, Jhansi-284128, Uttar Pradesh, India
[2]Department of Chemistry and Toxicology, Regional Forensic Science Laboratory, Gwalior-474001, Madhya Pradesh, India

*E-mail id: anusingla73@gmail.com

ABSTRACT

In the present work, studies have been carried out on the formulation and evaluation of various toothpaste brands available in Indian market. The designed toothpaste samples were analysed for various parameters and characteristics, which were conceded out for all the available samples. Physical parameters such as efficacy, performance, quality and sensitivity were tested according to the Bureau of Indian Standards, while chromatographic analysis of chlorates of alkali metals and chloroform were based on the European Directive. Our findings indicated that all the toothpaste formulations complied with the standards specified with Bureau of Indian Standards and are in agreement with manufacturer's specifications. All the products *in lieu* with forensic toxicology were found to be safe, compatible with packaging, compliant with current legislative constraints and capable of performing as they claimed.

Keywords: Toothpaste, Cosmetic, Cosmeceuticals, Bureau of Indian standards, Forensic Toxicology, Thin layer chromatography, Gas chromatography

INTRODUCTION

Earlier in India, oral hygiene was the domain of local homemade ayurvedic powders and natural herbs. Now the awareness regarding oral hygiene in the Indian society has increased and thus many companies have come up with their brands of toothpastes. As per the historic records, the First modern toothpaste in 1800 was homemade with soap, chalk and salt as common ingredients, which were sold in jars in the form of powders or pastes. The *first toothpaste tube* was invented by *Dr. Washington Wentworth Sheffield* of New London in 1892 who was a dentist by profession. He came up with this idea after his son travelled Paris and saw painters using paints from tubes. Packaging was done in lead and tin alloy tubes. In 1896, *Colgate Company launched a dental cream* which was packed in collapsible tubes imitating Sheffield tubes[1].

According to ISO 11609:1995(E) (International Organization for Standardization)[2], Toothpaste (also known as Dentifrice) is defined as **"***Any substance or combination of substances specially prepared for the public for cleaning the accessible surfaces of teeth."* Toothpaste contains three types of ingredients, viz., common ingredient, active ingredient and inactive ingredients. But nowadays there are two aspects of all Fast Moving Consumer Goods products that whether they belong to the category cosmetics or cosmeceuticals.

In recent years, the oral care market has shown an impressive growth rate of 18.6%. The growth is particularly prominent in the urban area. The widespread acceptance of using toothpaste for improved oral health has resulted in the use of dentifrices as an effective delivery system for both cosmetic and therapeutic agents[3].

In the present era of global marketing, national and international legislations have been implemented to resolve number of key issues as well as to govern manufacturing sailing, import and labeling of cosmetics. In India, Bureau of Indian Standards[4] governs all the requirements pertaining to safety, quality and performance of cosmetics. For toothpaste, the IS code number is IS 6356:1993, which

scrutinises the requirements for toothpaste. As Indian standards are not up to the acceptable level therefore upbringing of oral care products is quite difficult in our country as the confusion between cosmetics and cosmeceuticals still persists. The European (EU), USA, Japan and Canada are the major markets that provides regulatory framework for cosmetics in the major market. Hence, we had contributed our small carve up by discussing other standards for better efficacy in manufacture, sale, distribution and import of cosmetics.

Toothpaste is one item that nearly everyone uses today and there is somewhat within this concoction that makes it a special one. *In lieu* with Forensic Toxicology**,** an attempt has been made to scrutinise few aspects of this concoction that takes into account: the general toxicological profile of the ingredients, their chemical structures and the potential levels of exposure. While using this standard commodity we came across that most of the toothpaste packages warn: "Do Not Ingest!" This is so because some harmful ingredients are also there in promising toothpaste formulations and after doing so, the manufacturers positioned their products as dental care products without explicitly stating about the use of harmful ingredients. Some researchers provide useful information while working like Hattab[5] worked upon various parameters of fluoride toothpastes; Goldemberg[6] investigated about the usage of formaldehyde toothpastes in Japan and Thailand; Vermeer *et al*.[7] reported about the widespread usage of the term Cosmeceutical and its legitimacy; Mandel[8] described about the common use of fluoride toothpastes and the current competition among various products; Foxall[9] provides the toxicological overview of chloroform. Keeping in view these facts the present researchers have examined various parameters by physical and chemical examination, viz, identification of chlorates of alkali metals by thin-layer chromatography (TLC) and analysis of chloroform by gas chromatography in toothpastes available in Indian market.

EXPERIMENTAL PROCEDURE

Materials

In the present study, a total of 20 toothpaste samples of various brands were studied, which were procured from the local market of Jhansi, U.P. (Table 1). All the chemicals and reagents used were of analytical reagent grade and used without further purification. Deionised distilled water was used throughout.

Table 1: Brands of toothpastes

Sl. No.	NAME OF BRAND
1.	NEEM
2.	SENSODYNE
3.	THERMOSEAL
4.	BABOOL
5.	DABUR RED
6.	PROMISE
7.	ANCHOR WHITE
8.	HOMODENT
9.	CREST SALT
10.	DENTOBAC
11.	STOLIN
12.	COLGATE STRONG
13.	SACH
14.	CLOSE-UP
15.	INFINITE TOOTHPASTE
16.	PEPSODENT
17.	VICCO
18.	AQUAFRESH
19.	LORDENT
20.	HIMALAYA

Instrument

Gas chromatographic determinations were performed on Hewlett-Packard 7620A instrument equipped with Flame Ionisation Detector (manufactured by Hewlett Packard Corporation, Palo Alto, California). Detectors The column was made up of glass and packed with Propak Q, Chromosorb 101 (manufactured by Restek Corporation, 110 Benner Circle, Bellefonte, PA) or equivalent 80 to 100 meshes with aid of a vibrator. Carrier gas used was nitrogen at a flow rate of 65 ml/min.

Physical Analysis (Table 2)

1. Determination of hard and sharp-edged abrasive particles

In this process, toothpaste was extruded out on the surface of butter paper and was tested by pressing the entire length with the help of the fingers for the presence of hard and sharp-edged abrasive particles.

Table 2: Assessment by physical examination

Sl. No.	NAME OF BRAND	APPEARANCE	HARD AND SHARP-EDGED ABBRASSIVE PARTICLE	SPREADABILITY	pH	FOAMING POWER
1.	NEEM	LIGHT GREEN	ABSENT	3.9	7	65
2.	SENSODYNE	BLUE	ABSENT	4.2	7	60
3.	THERMOSEAL	PINK	ABSENT	6.4	7	25
4.	BABOOL	WHITE	ABSENT	3.7	7	57
5.	DABUR RED	BRICK RED	ABSENT	5.1	7	56
6.	PROMISE	LIGHT BLUE	ABSENT	4.8	7	70
7.	ANCHOR WHITE	WHITE	ABSENT	5.2	7	54
8.	HOMODENT	WHITE	ABSENT	4.5	7	25
9.	CREST SALT	WHITE SOFT MASS WITH BLUE SPOT	ABSENT	4.3	7	20
10.	DENTOBAC	BLACK	ABSENT	3.4	8	5
11.	STOLIN	WHITE	ABSENT	4.0	6	1
12.	COLGATE STRONG	WHITE	ABSENT	4.7	7	55
13.	SACH	RED SOFT MASS WITH MULTI COLOURED SPOT	ABSENT	5.8	7	45
14.	CLOSE-UP	RED	ABSENT	6.8	6	54
15.	INFINITE TOOTHPASTE	PALE YELLOW	ABSENT	4.2	7	0
16.	PEPSODENT	LIGHT PINK	ABSENT	4.9	7	58
17.	VICCO	GREY	ABSENT	3.5	7	52
18.	AQUAFRESH	WHITE, RED AND GREEN	ABSENT	6	7	49
19.	LORDENT	WHITE	ABSENT	6.1	7	70
20.	HIMALAYA	GREY	ABSENT	5.3	7	62
	STANDARDVALUE		**ABSENT**	**(Max.) 8.5**	**(Max.) 0.5**	**(Min.) 50**

2. Determination of spread ability

About 1 g of sample was taken on the glass plate and was pressed with the glass plate and a 2-kg weight was put thereon. The spread ability was measured along the diameter after 30 min.

3. Determination of pH

Five gram of all samples were accurately weighed and placed in a 150-ml beaker. To this about, 45 ml of freshly boiled and cooled water was added at 27ºC and stirred well. The pH was determined within 5 min.

4. Determination of foaming power

In this process, about 5 gm of each sample was weighed and placed in a 100-ml beaker. To this, 10 ml of water was added and the beaker was covered with a watch glass and allowed to stand for 30 min. This operation was carried out to disperse the toothpaste in water. As soon as the contents of the cylinder reached 30ºC and 12 complete shakes were given to it, foaming power was determined for the samples, respectively.

TLC Analysis (Table 3)

TLC analysis was performed to identify chlorates of alkali metals in the toothpaste formulations. It was performed on Sigma Aldrech TLC plates (manufactured by Sigma Aldrech Co. LLC, St. Louis, MO) coated with cellulose of 0.25 mm thickness, the plate was activated by heating in an oven at 110 ºC for about 1 h. One gram of toothpaste sample was extracted with water, filtered and diluted. Two-microlitre aliquots of the three reference solutions, i.e., potassium chlorate, bromate and iodate were spotted on the TLC plate, which were developed with ammonia:acetone:1-butanol (60:10:30) in a presaturated TLC chamber to a height of 10 cm. The plate was removed from the chamber, dried in air and sprayed with potassium iodide, starch solution and then with hydrochloric acid. The chlorates of alkali metals, if present in the toothpaste, would appear as *blue spot* after half an hour with Rf value approximately 0.7–0.8.

Gas Chromatographic Analysis

In present study, chloroform content in various brands of

Table 3: Rf value for chlorates of alkali metals

Sl. No.	NAME OF BRAND	Rf VALUE FOR CHLORATES OF ALKALI METAL
1.	NEEM	0.83
2.	SENSODYNE	0.80
3.	THERMOSEAL	0.78
4.	BABOOL	0.81
5.	DABUR RED	0.80
6.	PROMISE	0.84
7.	ANCHOR WHITE	0.80
8.	HOMODENT	0.75
9.	CREST SALT	0.71
10.	DENTOBAC	0.79
11.	STOLIN	0.73
12.	COLGATE STRONG	0.81
13.	SACH	0.86
14.	CLOSE-UP	0.70
15.	INFINITE TOOTHPASTE	0.75
16.	PEPSODENT	0.75
17.	VICCO	0.71
18.	AQUAFRESH	0.85
19.	LORDENT	0.73
20.	HIMALAYA	0.80
	REFERENCE	**0.70**
	Standard value	**0.70-0.80**

toothpaste was analysed by performing Gas Chromatography according to European Directive 80/1335/EEC[10].

Principle

The toothpaste was suspended in a dimethyl formamide/methanol mixture to which a known quantity of acetonitrile was added as an internal standard. After centrifuging, a portion of liquid phase was subjected to Gas chromatographic (GC) column.

Sample preparation

The sample for analysis was taken from an unopened tube. One-third of the contents were removed and the cap was replaced on the tube. It was mixed carefully in the tube and the test portion was taken.

Method evaluation

In GC, the analytical characteristics of the method such as retention time, height, area and concentration were investigated to evaluate the efficiency of the method. Figures 1 and 2 show the chromatograms for standards of chloroform with retention time of 4.100, height 15,808, area 4,03,371.5 and concentration 3.183, and in Figure 3 the chromatogram for sample does not show any chloroform peak at this retention time.

RESULTS AND DISCUSSION

In the present study, an attempt has been made to scrutinise various parameters related with toothpaste to determine whether the samples available in Indian market meet the legal requirements and liabilities or not. The present methodologies underscore various physical as well as chemical examinations of the 20 dentifrices, which were taken for the purpose of study. In physical

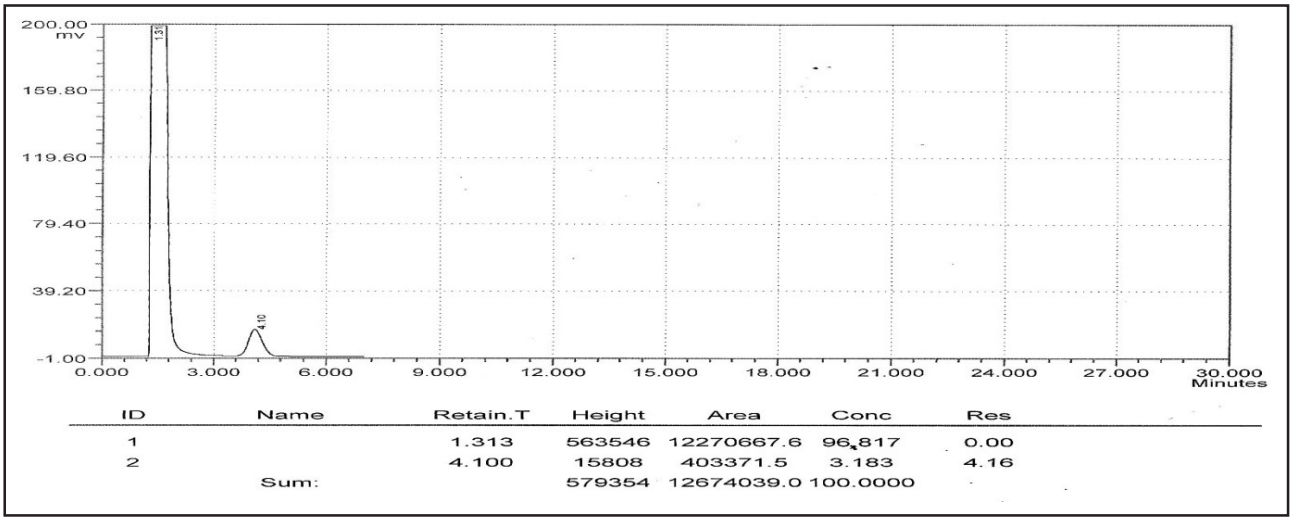
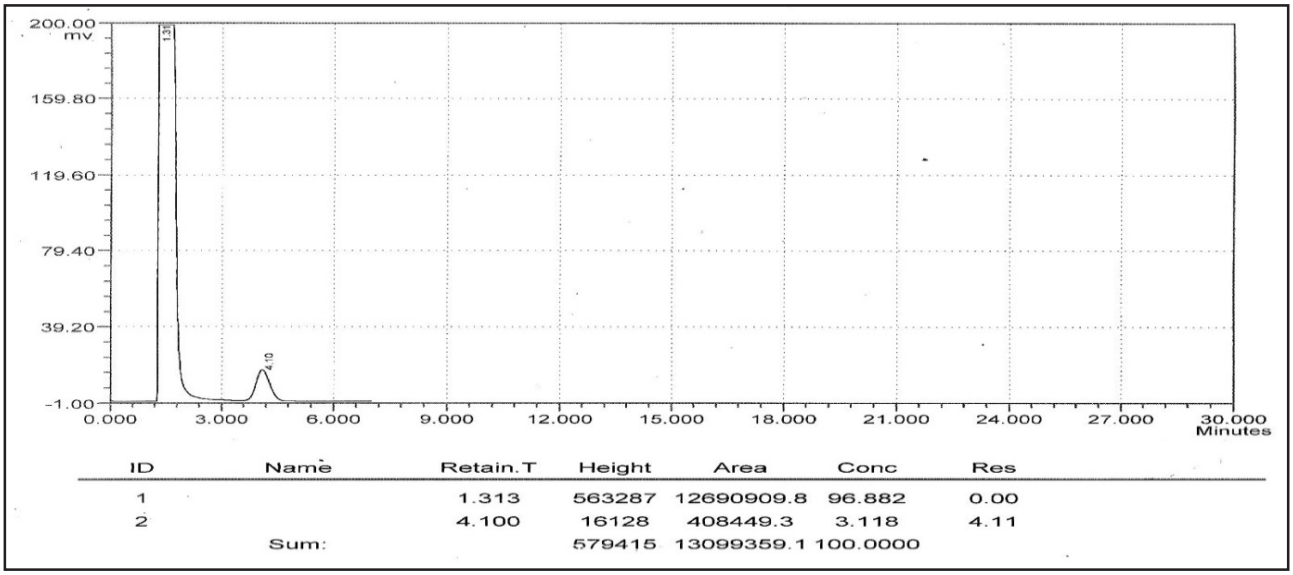
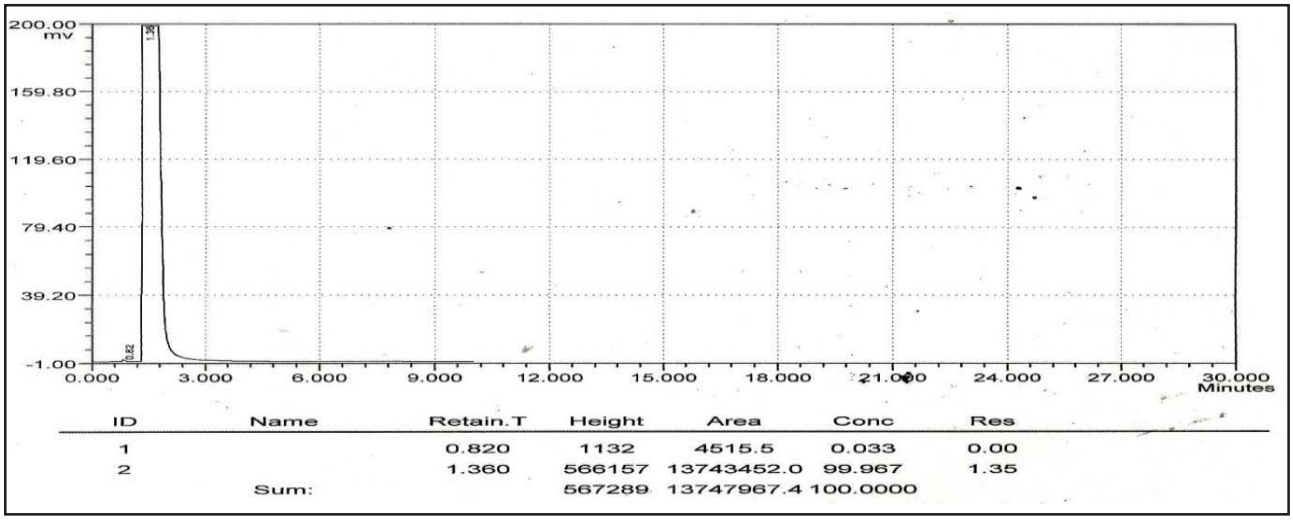


Figure 1: Chromatogram of chloroform standard



ID	Name	Retain.T	Height	Area	Conc	Res
1		1.313	563287	12690909.8	96.882	0.00
2		4.100	16128	408449.3	3.118	4.11
	Sum:		579415	13099359.1	100.0000	

Figure 2: Chromatogram of chloroform standard



ID	Name	Retain.T	Height	Area	Conc	Res
1		0.820	1132	4515.5	0.033	0.00
2		1.360	566157	13743452.0	99.967	1.35
	Sum:		567289	13747967.4	100.0000	

Figure 3: Chromatogram of toothpaste formulation

examination, it has been observed that the hard and sharp-edged abrasive particles were absent in all the brands. The spread ability of all the samples evaluated was within the range, pH of all the 20 brands was within the standard pH range of 8.5 and the foaming power calculated shows affirmative results. This shows that the analysed samples compliant with current legislative constraints and are capable of performing as claimed. Qualitative and quantitative examinations were performed by TLC and GC according to European legislation. Chlorates of alkali metals were analysed by using TLC and it has been observed that the R_f value for all the 20 brands is within the permissible range, i.e., 0.7–0.8 as mentioned in European legislation. The presence of chloroform in toothpaste was analysed by GC and a comparison of retention time, height, concentration and area was done. Out of all the 20 samples analysed, the quantity of chloroform was found to be nil in all the toothpaste formulations.

CONCLUSION

Continued surveillance of such dental products is essential in order to better educate people and inform about the risks associated with the ingredients as it is a matter of public health concern. Twenty toothpaste samples that have been evaluated shows that the toothpaste formulations are in agreement with the manufacturer's specification.

In lieu with *Forensic Toxicology*, it has been concluded that the products:

a) Compliant with Current Legislative constraints.

b) Are safe to use as a daily commodity

c) Performed as claimed.

d) Compatible with their Packaging.

The suggested protocol for testing toothpaste in forensic toxicology laboratory is easy and less time consuming, this protocol can be applied in routine forensic identification and examination of genuine and duplicate toothpastes and toxic substances in to them.

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