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Sergey Y. Yurish



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Design and Development of an Instrument to Determine the Fluoride Ion Concentration in Certain Tooth Pastes

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Abstract: The decline of dental caries in the industrialized countries can be attributed to wide spread use of fluorides. The Indian market share of dentifrices containing fluorides has increased day to day. For a fluoride dentifrice to be effective in the control of dental caries, an adequate concentration of the fluoride must be soluble. Several Indian fluoride dentifrices have in their packages as non-fluoridated tooth pastes and also in their formulations calcium phosphate as abrasive, which may react with fluoride. This study was designed to evaluate the determination of fluoride ion concentration in certain tooth pastes using the PC in the most consumed dentifrices in India. The fluoride ion concentration in certain tooth pastes was determined using the spectroscopic method and by using the PC. All dentifrices showed similar concentrations of total fluoride in all samples in accordance with the Indian legislation (Fluoride content of 1000 ppm). Some dentifrices showed in their packages as non-fluoridated tooth pastes. Although most of the Indian fluoridated dentifrices evaluated in this study contain fluoride they were found to have sufficient concentrations of fluoride ion to be effective in preventing dental caries. *Copyright © 2012 IFSA.*

Keywords: Tooth pastes, Optical fibre, Cuvette, Optical sensor, PC, DIOT Card.

1. Introduction

The prevalence of dental caries in developed countries has declined over the past several decades [1]. Several studies are in agreement that the main reason for the reduction of carries is the greater

availability of fluoride in the oral environment, particularly the increasing use of fluoridated dentifrice over the last 25 years [2].

In most western industrialized countries, the percentage of fluoridated dentifrices of all dentifrice sales is above 90 % with an upward trend. However, in order for the dentifrice to be efficient, it is important the fluoride is soluble in the formulation and it is regularly present in the oral cavity, so it can interfere with the phenomena of enamel – dentine demineralization and demineralization.

The Indian market share of dentifrices containing fluorides, such as sodium Mono Fluoro Phosphate (MFP) or Sodium Fluoride, has increased from 12 % in 1985 to 77 % in 2006. Until 1994, the market share of fluoridated tooth pastes were only 46 % but after the addition of fluoride into the most Popular Indian Tooth Pastes have in their formulations Declaim Phosphate as an abrasive, which may react with fluoride ions released from MFP.

The Indian legislation establishes the maximum fluoride content to be 1000 ppm which is permitted in a dentifrice. However the concentration of fluoride contained in the dentifrice is not indicated on the package.

Flora fluoridated dentifrice to be effective in controlling dental caries, and adequate concentration of fluoride must be soluble. The soluble forms of fluoride are able to interfere with the dynamics of the caries process, reducing the demineralization and activating the demineralization of dentine and enamel. Some forms of fluoride may link to the abrasive contained in the dentifrice formulation. Thus the total concentration of fluoride is not the concentration of the soluble and active fluoride contained in the Toothpaste.

The US Food and Drug Administration required that the soluble fluoride ion for NaF tooth pastes and soluble F⁻ ions for MEP tooth pastes shall not be less than 60 % of the total fluoride content. The Standards Association of Australia and the European Community have laid down rules for the minimum amount of soluble fluoride that must be present in fluoride Tooth paste, throughout its shelf life. These rules require that at least 60 % of the total fluoride content be present as a soluble ion either as F⁻ or Po₃⁻⁴, F₂⁻. The later ion is rapidly hydrolyzed in the mouth to provide F⁻. However some hydrolysis occurs slowly from the time of manufacture and combines with abrasive or other tooth paste constituents to form insoluble compounds so that the total soluble fluoride concentration falls with time. The minimum requirement for the anti-caries effect of a dentifrice is based on the available and stable fluoride in the formulation. However only the Indian legislation establishes the maximum fluoride to be 0.10 % to 0.15 % without specifying quality.

The measurement of fluoride ion is necessary in different fields of work. The fluoride ion is necessary to know the concentration levels in natural water since low fluorine i.e. < 1 ppm plays an important role in humans in the development of healthy bones and teeth. The estimation of fluoride ion gives valuable information. Therefore it is necessary to analyze the fluoride ion and to take proper and optimum precautions.

No doubt, several investigators developed techniques for the measurements for different ion concentrations in different samples and several manufacturers are producing a variety of instruments for estimation of ions [4]. But the attempts to design and develop the computer based integrated system for determination of fluoride ion in tooth paste is rather scarce particularly in India though they offer many advantages. Hence in the present study an attempt is made to design a computer based instrument for determination of fluoride ion concentration in tooth pastes. No studies about the percentage of fluoride ion concentration in Indian dentifrices were found in the literature survey, for justifying this study.

2. Experimental Setup

The block diagram of a PC based fluoride ion concentration measurement system used in the present work is shown in Fig. 1. It consists of the following:

1. Regulated power supply;
2. LED;
3. Sample holder;
4. Optical sensor (photo diode);
5. Feedback PD amplifier;
6. Data acquisition card;
7. PC.

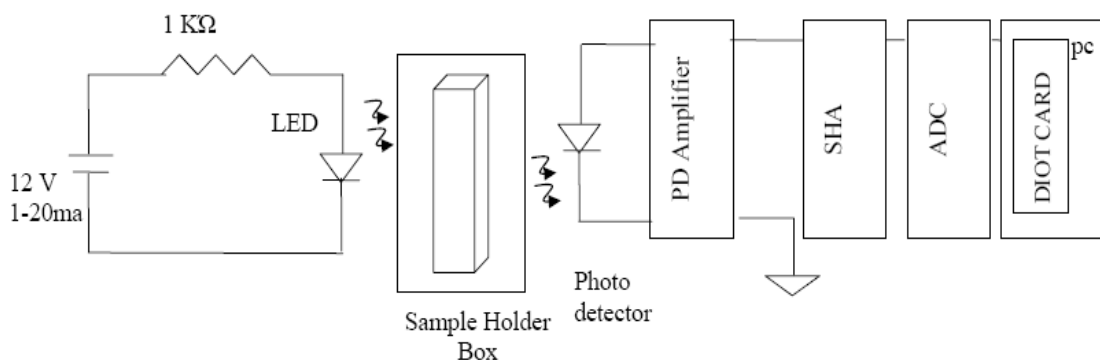


Fig. 1. Block diagram of PC based fluoride ion concentration measurement system.

2.1. Regulated Power Supply

A d.c power supply which maintains output voltage constant irrespective of a.c. mains fluctuations or load variations is known as regulated d.c. power supply. The regulated power supply is constructed in the laboratory using the standard components.

2.2. LED

In the present study LED [3] is used to emit the light and then incident on the solution through optical fibre. Light emitting diode (LED) is a semiconductor diode that emits incoherent narrow- spectrum light when electrically biased in the forward direction of the P-N Junction. This effect is a form of electro luminescence. An LED is usually a small area source often with extra optics added to the chip that shapes its radiation pattern.

The color of the emitted light depends on the composition and condition of the semi conducting material used, and can be infrared, visible or near – ultraviolet. An LED can be used as a regular house hold light source.

2.3. Sample Holder

In the present study cuvettes [4] are used for the purpose of filling the sample and blanks solutions. A cuvette is a kind of laboratory glass ware, usually a small tube of circular or square cross section,

scaled at one end, made of plastic glass or optical grade quartz and designed to hold samples for spectroscopic experiments. The set up was mounted on box and cuvette is inserted from the top face, sides opposite faces are connected with LED and photo detector and remaining face is closed. Total set up covered with a black photographic film.

2.4. Optical Sensor (Photo Diode)

Devices that convert the signal into electric form e.g. vacuum photo detectors, thermal detectors PIN junction photo detectors, photo transistors, photo resistors. In the present study, pin photo diode is used, as a detector.

2.5. Feedback PD Amplifier

Trans impedance Feedback Photo detector amplifier [5] is used for converting an input current signal which has AC and DC component from a photo diode into an output voltage. The input current signal varies, by producing a constant current source for supplementing the DC feedback current. Fig. 2 shows the feedback PD amplifier used in the present study. This comprises a photodiode, an operational amplifier and feedback network. At the input photodiode receives the current I_{pd} , the amplifier converts the input current I_{pd} producing a differential output voltage V_{out} . The linearization is accomplished by the feedback network including feedback amplifier, low pass filter combination and a bypass circuit.

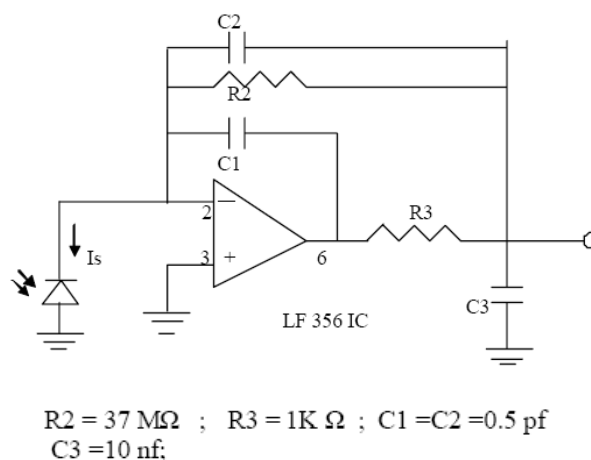


Fig. 2. Feedback PD amplifier.

2.6. Data Acquisition Card

In the present study, Data acquisition card (ALS – PL -02) is used [6]. It has ADC/DAC digital input /out puts compatible to XT/AT PC. This card provides 12 bit A/D conversion facility, jumper selectable 8 differential / 16 single ended channel, 12 bit D/A conversion facility (2 channels) and 48 programmable parallel input lines. All these features are required for designing a PC based fluoride ion concentration measurement system. In this scenario DIOT card is used in the present work. Fig. 3 shows the photograph of the DIOT card used in the present work.



Fig. 3. Photograph of the DIOT card.

2.7. Personal Computer

The output voltage coming from the feedback PD amplifier was measured by using PC through the Data acquisition card. The technique developed in the present study need extra plug in cards such as DIOT card [7]. Interfacing of the external hardware with PC was accomplished by DIOT card.

3. Method

The instrument requires a high brightness LED, regulated 12V power supply together with a series combination of a 510 Ω resistor and a 10 k Ω potentiometer was used as current source for the LED [8]. The current can be adjusted in the 1-20 mA range, assuming a $V_f=1.8$ V. A digital multimeter was included to adjust the current accurately. The 510 Ω resistor sets the maximum current available and must always be included to avoid permanent damage to the LED due to an excessive current passing through it.

For a flexible operation, the emitted light was conducted through a fibre optic. A Polymeric type fibre with a 1 mm diameter was selected because of its low cost and low loss at the working wavelengths. The fibre is covered with a black polyethylene cladding. A homemade plastic coupling was use to join the fibre to the LED. The detector used was a light to voltage optical sensor this is a very small combining a photodiode and a transimpedance amplifier with a 16 M Ω integrated feedback resistor on a single IC. In appearance it is similar to a conventional low power transistor also having three leads used as ground, power supply and output.

The voltage signal produced by the incident light on the photo diode was read by the computer through the interfacing network. Soft ware is run to get the results. Thus intensity data are the primary data obtained. To transform these data into absorbance, a logarithmic transformation is needed, taking into account the well known relation of the primary data to the final absorbance values are obtained from the equation (1).

$$A = \log \{(E_0 - E_\infty) \div (E - E_\infty)\}, \quad (1)$$

where

E = is the potential measured with the sample placed into cell holder;

E_0 = is the potential measured with the reference solution;

E_∞ = is the potential in the absence of light.

4. Preparation of Tooth Pastes Solution and Determination of Fluoride Ion Concentration

0.5690 gm of tooth paste sample was weighed and dissolved in few CC'S of water in a beaker 5 to 6 drops of con HNO_3 or sulphuric acid is added to the solution. The mixture is boiled thoroughly for few minutes and cooled. The solution is carefully transferred into 250 ml standard flask. The contents in the breaker are washed and once again transferred & removed. The solution in the 250 ml standard flask is made up to the mark with double distilled water. Stopper is placed and the solution is shaken thoroughly to get a solution of uniform concentration. This solution is taken as reference standard solution of 0.1 M concentration [9]. Solutions of various concentrations are prepared by applying the principle of dilution. These solutions are taken in cuvettes. The voltage signal produced by the incident light on the photodiode was read by the computer, Thus intensity data are the primary data obtained. To transfer these data into absorbance a logarithmic transformation is needed.

The experimental results are plotted as absorbance versus volume. Absorbance data were calculated from the voltage measurements, and according to equation (1). A Good straight line is obtained in the tested interval indicating the method can be successfully follows the Beer's law, in spite of the fact that the deviation are expected when the radiation used is not monochromatic. The percentage of the fluoride is evaluated according to the graph. The same procedure is applied for all tooth pastes.

4.1. Preparation of Sodium Fluoride

A stock solution of sodium fluoride is prepared by dissolving 4.199 gm of sodium of fluoride in one litre distilled water to get a solution of 0.1 molar concentrations. The molecular weight of sodium fluoride is 41.99 gm. 41.99 gm of sodium fluoride in 1000 ml is equal to 1 molar. 1000 ml of 1 molar contains 19 gm of fluoride in 1 liter. 0.1 molar contains 1.9 gm of fluoride in 1 liter. A stock solution 0.1 molar concentration is prepared by dissolving 1.05 gm of sodium fluoride in 250 ml standard flask by double distilled water. The solutions of various concentrations are prepared by applying the principle of dilution ($m_1v_1 = m_2 v_2$).

4.2. Statistical Analysis

The software is developed in "C" language in getting the voltage values for different concentrations of different tooth pastes. The absorbance values are observed in PC. The flow chart of 'C' program developed in the present work is given in Fig. 4.

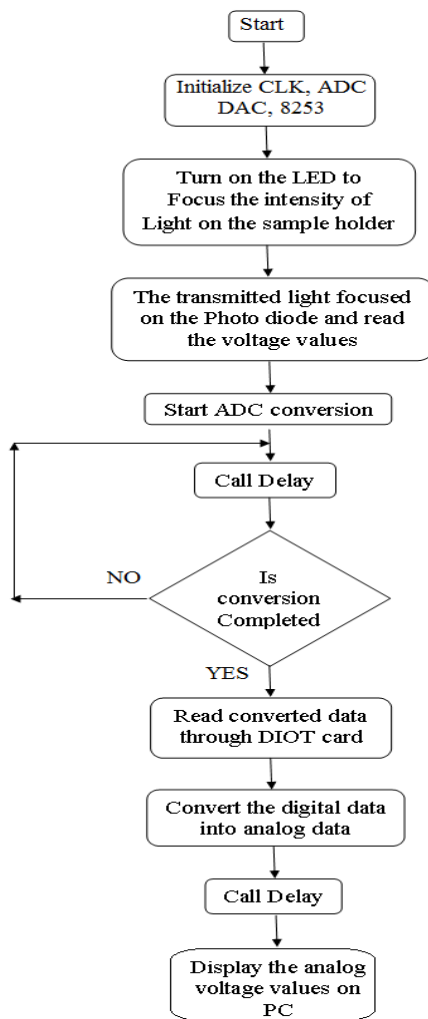


Fig. 4. Flow Chart.

5. Results

Experimental results are shown in tables 1-7 and figures 5-10.

Table 1. Pepsodent.

Estimation:
 0.5690 -100 ml - 0.1 M
 1.4225 -250 ml - 0.1 M
 $E_0 = -104.1 \text{ mV}$

S.no	Volume	Voltage (e)	Voltage (e_{∞})	Absorbance
1.	10	-99.9	1.5	0.0176
2.	20	-95.8	1.5	0.0352
3.	30	-91.5	1.5	0.0528
4.	40	-87.7	1.5	0.0704
5.	50	-83.6	1.5	0.088
6.	60	-79.9	1.5	0.1056

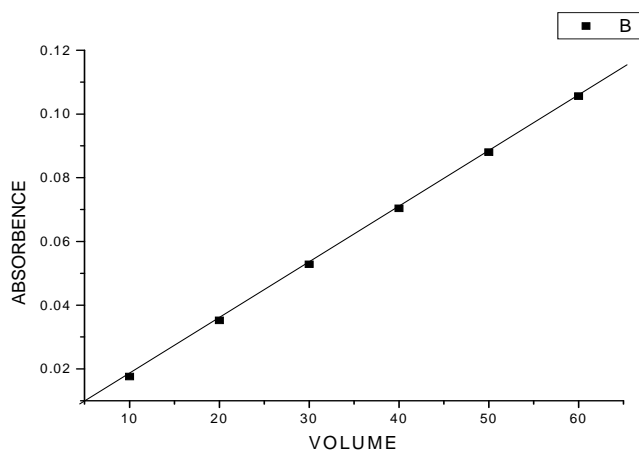


Fig. 5. Graph of Pepsodent tooth paste.

Table 2. Dabur Red.

Estimation:
 0.5690 gm - 100 ml -0.1 M
 1.4225 gm – 100 ml - 0.1 M
 $E_0 = -76.4$ mV

S.no	Volume	Voltage (e)	Voltage (e_{∞})	Absorbance
1.	10	-72.3	1.5	0.02348
2.	20	-68.0	1.5	0.04696
3.	30	-64.4	1.5	0.07044
4.	40	-61.0	1.5	0.09392
5.	50	-57.9	1.5	0.1174
6.	60	-54.9	1.5	0.14088

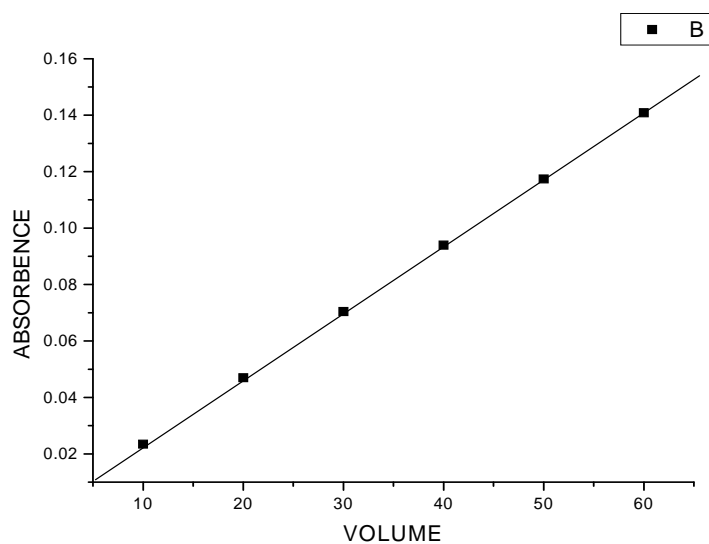


Fig. 6. Graph of Dabur Red tooth paste.

Table 3. Colgate.

Estimation:
 0.5690 gm - 100 ml - 0.1M
 1.4225 gm - 100 ml - 0.1 M
 $E_0 = -104.1$ mV

S.no	Volume	Voltage (e)	Voltage (e_{ox})	Absorbance
1.	10	-97.9	1.5	0.02627
2.	20	-92	1.5	0.05285
3.	30	-86.6	1.5	0.0786
4.	40	-81.3	1.5	0.1056
5.	50	-76.5	1.5	0.1315
6.	60	-72	1.5	0.1573

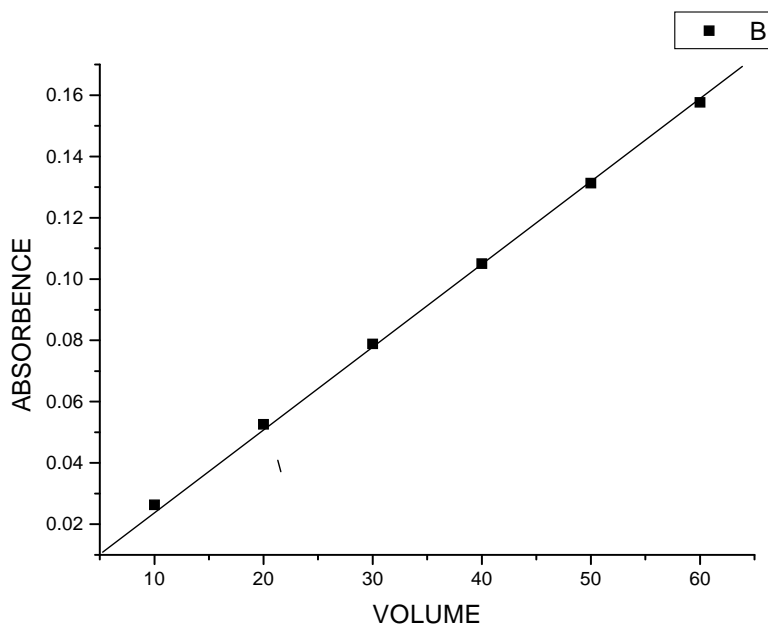


Fig. 7. Graph of Colgate tooth paste.

Table 4. Close Up.

Estimation:
 0.5690 gm -100 ml -0.1M
 1.4225 gm -100 ml -0.1M
 $E_0 = -76.4$ mV

S.no	Volume	Voltage (e)	Voltage (e_{ox})	Absorbance
1.	10	-71.6	1.5	0.0276
2.	20	-67.1	1.5	0.0552
3.	30	-62.9	1.5	0.08265
4.	40	-58.9	1.5	0.1105
5.	50	-55.2	1.5	0.1379
6.	60	-51.7	1.5	0.1656

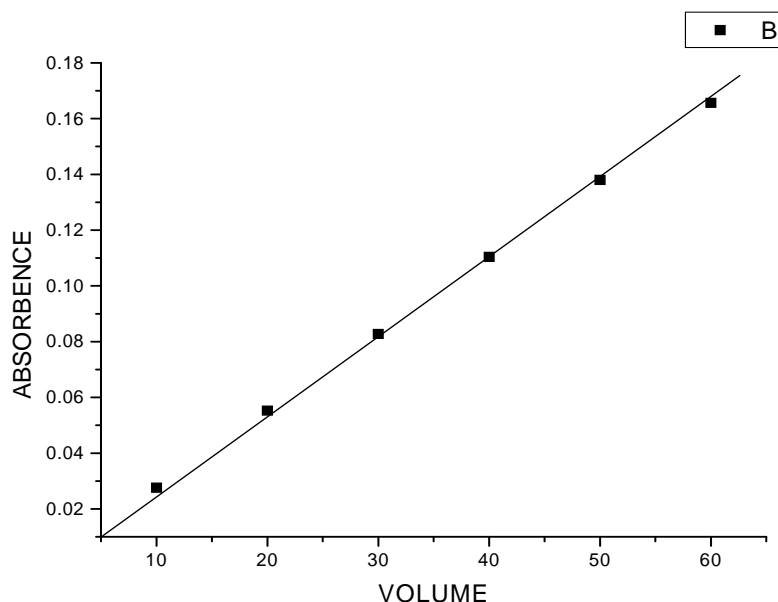


Fig. 8. Graph of Close Up Tooth paste.

Table 5. Anchor Gel.

Estimation:

0.5690 gm - 100 ml - 0.1M

1.4225 gm - 100 ml - 0.1 M

$E_0 = -104.1$ mV

S.no	Volume	Voltage (e)	Voltage (e_{ox})	Absorbance
1.	10	-94.1	1.5	0.0432
2.	20	-85.1	1.5	0.08614
3.	30	-76.8	1.5	0.1299
4.	40	-69.4	1.5	0.1730
5.	50	-62.7	1.5	0.2160
6.	60	-56.6	1.5	0.2594

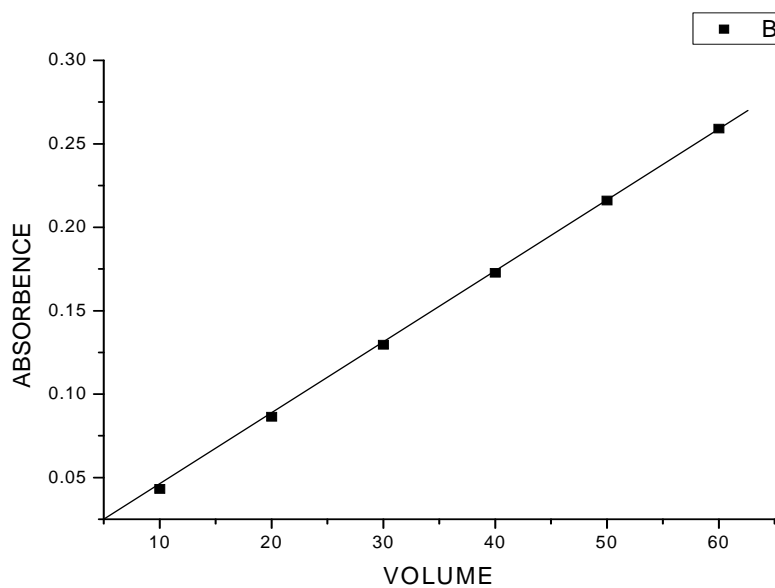


Fig. 9. Graph of the Anchor Gel tooth paste.

Table 6. Sodium Fluoride (NaF).

Estimation:

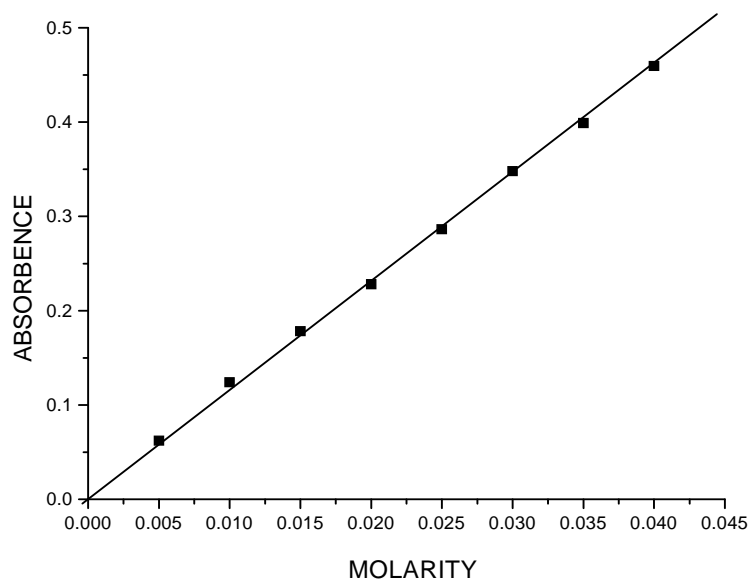
41.99 gm - 1000 ml - 1M

4.199 gm - 1000 ml - 0.1M

0.4199 gm - 100 ml - 0.1 M

 $E_0 = -209.6$ mV

S.no	Molarity	Voltage (e)	Voltage (e_{∞})	Absorbance
1.	0.005	-200.1	1.5	0.0199
2.	0.01	-191.2	1.5	0.0396
3.	0.015	-182.6	1.5	0.05943
4.	0.020	-174.3	1.5	0.0794
5.	0.025	-166.5	1.5	0.09917
6.	0.03	-158.9	1.5	0.1192
7.	0.035	-151.7	1.5	0.1392
8.	0.04	-144.9	1.5	0.1589
9.	0.045	-138.1	1.5	0.1796

**Fig. 10.** Graph of Sodium Fluoride.**Table 7.** Comparison Table.

S.No	Tooth paste	Fluoride Ion Concentration	
		Present study value (gm/L)	Indian standards (gm/L)
1	Pepsodent	0.01	0.01
2.	Dabur red	0.024	NF
3.	Colgate	0.027	0.01
4.	Close up	0.028	0.01
5.	Anchor gel	0.045	NF

NF: Non Fluoridated

6. Discussion

The decline of dental caries in industrialized countries can be attributed to the wide spread use of fluorides, mainly in the form of fluoride dentifrices. Approximately 500 million people of the world's population now use fluoride dentifrice making it by far the most important fluoride delivery system. Tooth brushing with fluoride dentifrice has become an important public health measure in preventing caries.

The regular application of fluoride from Toothpaste confers protection primarily by exerting a topical effect on the erupted teeth. Fluoride interacts with the plaque /tooth system in four ways: (a) reduction of mineral solubility (b) inhibition of mineral dissolution (c) inhibition of acid production by Plaque bacteria and (d) the promotion of demineralization. However, the total fluoride contained in the dentifrice is not completely available. For the dentifrice to be effective in the prevention of caries, quantity as well as quality of fluoride is important.

The majority of dentifrices contain either fluoride in their composition as the fluoride ion (NaF) or as monofluorophosphate (MFP), which are considered active forms for controlling caries. Fluoride in dentifrice must be in the dentifrice in a soluble form to guarantee activity against caries. Depending on the formulation part of the fluoride may be inactive and this occurs mainly in the presence of calcium (Ca⁺⁺) and fluoride ions (F⁻). Although MFP is more stable in the presence of Ca⁺⁺, because fluoride is linked covalently to phosphate it undergoes hydrolysis over time and release F⁻. This reacts with Ca⁺⁺ to form insoluble fluoride that is inactive against caries.

Analyzing the five most Indian fluoridated dentifrices, I verified that the total fluoride concentrations in the samples on the individual dentifrices were reasonably close to the values provided by the manufactured companies. Amount of fluoride present in the different tooth pastes are tabulated in the tabular column. Over previous decades, the Government and dental professionals did not encourage the population to use fluoride products and their usages were strictly limited.

The acceptance of fluoridated dentifrices is changing in India and the effect of these changes can be observed in the increase of fluoridated dentifrices in the market share and the sudden increase in people consuming these products. However some (two) consumed dentifrices evaluated in this study did not specify the fluoride content on their packages. For the Indian consumers to make informed choices before purchase of a fluoride dentifrice, the packages should be clear in showing the fluoride concentration contained in the product.

The results of this study suggest that the dentifrice most consumed fluoridated dentifrices contain a sufficient concentration of soluble fluoride and may be used to prevent and control dental caries.

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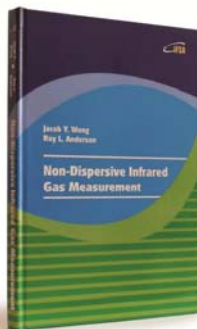
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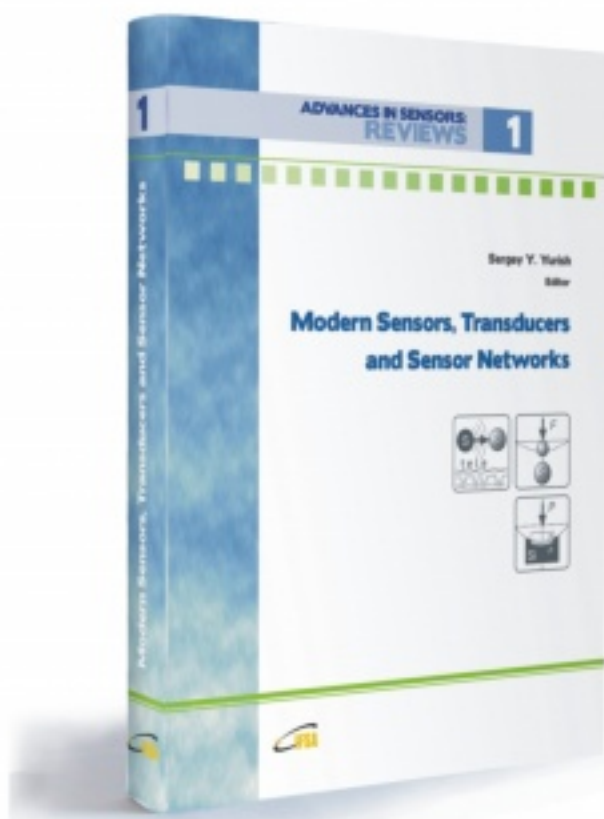
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