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Research Article

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A new optical analytical method for quantitative determination of lead on paper platform and its application for removal from aqueous sample

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ABSTRACT

A new optical analytical method, "digital RGB Analysis" is proposed instead of the conventional optical method, "spectrophotometry". MATLAB image processing tool can transform the color information into digital RGB values that can be treated as analytical information. The tool can simulate the color variations by optimization of visual color sensor with computer assistance and thus make colorimetry (semi quantitative analysis) as an accurate determination method. The Paper optode has been prepared by immobilizing potassium iodide (KI) and starch on thin layer chromatographic (TLC) strip. The reaction is based on indirect determination of lead by determining iodine liberated which results into blue colored spot; followed by scanner based detection. The obtained pattern was analyzed using image processing tool of MATLAB software to determine lead. All parameters affecting intensity on optode have been optimized. The proposed sensor was linear in the range 0.024-12µg mL^{-1} {24 µL of 1-500 µg mL^{-1} (R^2=0.998)} for G and B values. The minimum detection limit was found 3ng mL^{-1}(6µL of 5 µg mL^{-1}).The proportionality in intensity of the spot color on the optodes loaded with varying amounts of lead suggests its potential applications for screening of lead. The paptode can be used for health check and pollutant check at home. Thus the paper optode has great potential for this purpose.

Keywords: MATLAB; paper optode; thin layer chromatography, RGB analysis.

INTRODUCTION

Determination of heavy metals such as lead is very important because it show toxic effect on living systems. Lead is used in lead acid batteries, bullets, shots, solder metal component, as fusible alloy component, paints, additive in gasoline, as electrodes and in stained glasses. The manifestations of lead exposure include dullness, restlessness, irritability, poor attention span, headache, tremor, hallucination and loss of memory, which can progress to delirium, mania, convulsions, paralysis, coma and death [1]. Lead is also known to be able to cross the placental barrier and, thus, may cause possible damage to the fetus and newborn [2-4]. According to the WHO, the maximum permissible limit (MPL) is 0.05 mgL^{-1} of lead in drinking water [5].

The usual methods for the determination of lead in solution involve potentiometry, spectrophotometry, atomic absorption spectrometry [6-8], flame atomic absorption spectroscopy [9-11], graphite furnace atomic absorption spectroscopy [12], inductively coupled plasma–atomic emission spectroscopy [13] inductively coupled plasma–mass spectrometry [14] adsorptive stripping voltametry [15], voltametry [16], solid phase extraction [17] and anodic stripping voltametry [18].

In present paper we describe a new device as electronic tongue that we called "paptode" or paper optode. The paptode is simply a paper or other ordinary porous material such as clay or cotton or Whatman filter paper or ordinary filter paper or even TLC can be used as a substrate support for reagent.

The paptode are advantageous over conventional optodes in following ways:
(a) The immobilization of reagent is very easy in case of paptode
(b) In this method, we can introduce several reagents on a single strip. Therefore, by using a series of non-specific (or even low-selective) reagents, we can estimate the quantity and quality of multiple analyte in the solution by applying of mathematical methods, exactly similar to the analysis of tongue's signals in our brain.

(c) Since diffusion in a porous material is higher than in polymeric membrane the response time in paptodes is shorter than typical optodes.

(d) The uniformity pattern is not important in paptodes but is of paramount importance for optodes especially in obtaining reproducibility and repeatability of the system.

Amlathe and Gupta [19-20] developed indicator tubes and test strips for semi quantitative determination of hydrazine, later on utilizing the concept paptodes for quantitative determination of metal ions Fe, As, Hg, Zn, Se [21-26] have also been developed. The DCA analysis has also been reported for lithium [27]. The paptodes for Pb (II) has not been yet reported as revealed in literature.

In the proposed method, we have developed paptode for determination and removal of lead utilizing MATLAB assisted digital RGB analysis.

EXPERIMENTAL SECTION

2.1 Apparatus and software:

JEOL JSM -6390 A SEM Made in Japan, JEOL JFC-1600 Sputter Coater unit. The scanner (HP-SCANJET G2410) was used for scanning the strips. Resolution of scanner was regulated at 300 dpi. For analyzing color values in **RGB** (red, green, blue) system, the MATLAB software, which was written in Visual Basic 6 Media was used .A MICROLITE micro pipette was used for injecting samples on strips.

2.2 Chemicals and Reagents: All reagents used were analytical grade chemicals. Double distilled water is used throughout the experiment.

Lead solution:

500 µg mL^{-1} stock solution of lead was prepared by dissolving 57.5 mg of PbO_2 in 5% HCl and then made up to 100 mL by double distilled water. The stock is stable for one week, if kept at room temperature and for 15 days, if kept in the refrigerator. Required dilutions were prepared accordingly.

Potassium iodide (KI):

10 % KI solution was prepared by dissolving 10 g salt in 100 mL of double distilled water.

Starch:

4% starch was prepared by dissolving 4g starch in 100 mL of double distilled water which was found stable for 3 days. It was prepared daily for flawless analysis.

2.3 Procedure:

Preparation of Paper Optode:

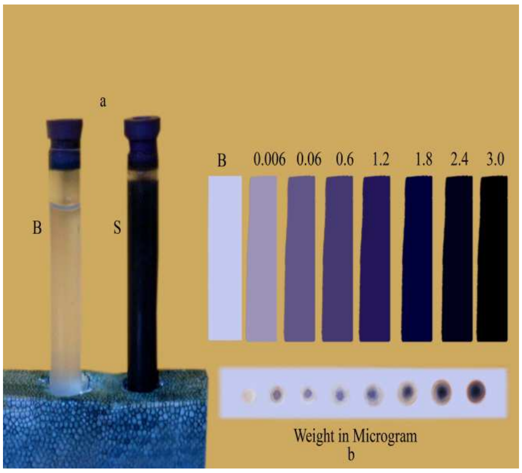
To construct the paptode for lead, strips of TLC (standard Indian make) were immersed in 10% solution of KI for few seconds and then dried in a temperature controlled oven (to speed up drying) followed by immersing in 4% solution of starch and then dried again in an oven at the same temperature but not higher than 70^0C to avoid brittleness of strips. The prepared paptodes were stored in cool and dry place and can be used for 25 days.

Mode of Operation:
Aliquots of 24µL of lead solutions were injected on these strips. Light purple to deep indigo blue or violet (depending upon the concentration of lead) [28] colored spot is formed as soon as the drop of analyte comes into the contact**.** The spots turn brown on drying but turn blue again on being moistened. Intensity of colored spot developed on paptode was found to be proportional to the concentration of the analyte. The spots were not perfectly homogenous; therefore, we have selected the central area of spot which was found to have maximum intensity for analyzing the color. The spots were scanned using a flatbed scanner and the obtained images have been transferred to computer for MATLAB assisted digital RGB analysis and the intensity of color-spots was determined.

The **RGB** color model is an additive color model in which red, green and blue light are added in various ways to produce a broad array of colors. Any color can be analyzed to obtain its corresponding **R**, **G** and **B** value. Effective intensity for any color values of color spots was calculated by following formulae:

$A_r = -\mathrm{Log}\,(R_s/R_b)$ _______ (1);
$A_g = -\mathrm{Log}\,(G_s/G_b)$ ________(2);
$A_b = -\mathrm{Log}\,(B_s/B_b)$ ________(3);

Where, A_r, A_g, A_b are effective intensities of red, green and blue color respectively ,R_s, G_s, B_s and R_b, G_b, B_b refer to R, G and B values of sample and blank respectively. The calibration curve is obtained by plotting effective intensities of R, G and B values vs. analyte concentration.



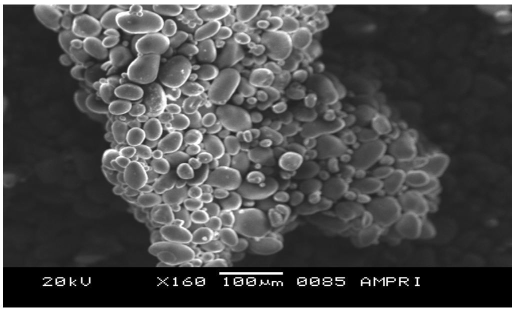
Photograph showing-color variation before and after adding lead sample
Where; B- blank, S- sample, a- color development in solution, b- color development on injecting various concentration of analyte

RESULTS AND DISCUSSION

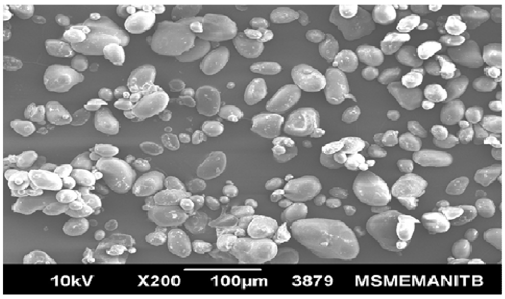
3.1 Chemical reaction:

Lead (IV) oxide forms lead (IV) chloride on being dissolved in cold and concentrated HCl. lead (IV) chloride reacts with KI to form lead (IV) iodide which being unstable breaks down to lead (II) iodide and liberate iodine which then subsequently binds with starch to form complex to produce a blue colored product. Lead iodide volatizes off, this has been confirmed by electron diffraction studies as no lead or lead compound peak was found. Thus even the strip contains both the reagent but no color production takes place until lead sample is injected. It is recommended to use a very little amount of reagent otherwise strips may turn brown due to environmental oxidation.
$PbO_2 + 4HCl \rightarrow PbCl_4 + 2\,H_2O$
$PbCl_4 + 2KI \rightarrow PbI_4 + 2KCl$
$PbI_4 \rightarrow PbI_2 + I_2$
(Volatizes off)
I_2 + Starch→I_2-starch complex
(Blue violet)

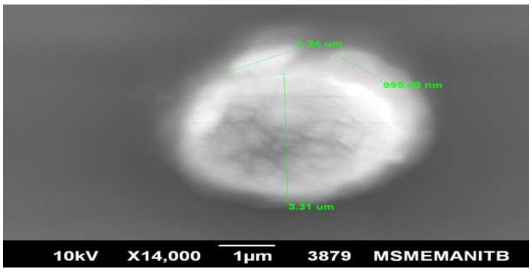
The disposable sensors so prepared were investigated under scanning electron microscope (SEM) after gold coating. SEM images of compound reveals that it is produced in bunches of oval / spherical particles (Micrograph-M: 1, M: 2, M: 3 and M: 4).



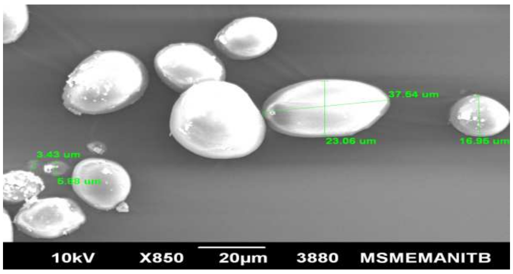
M: 1 Dye particle bunch



M: 2 Scattered dye particles



M: 3. Dye particle with nm diameter



M: 4 Dye particles with µm diameter

3.2 Optimization of conditions:

3.2.1 Injection volume:

The influence of volume of analyte which must be injected onto strip was investigated. The optimum sample volume was obtained to be 24µL.Increased volume injection leads to more diffusion of spots and thus consequently decreases the intensity of color.

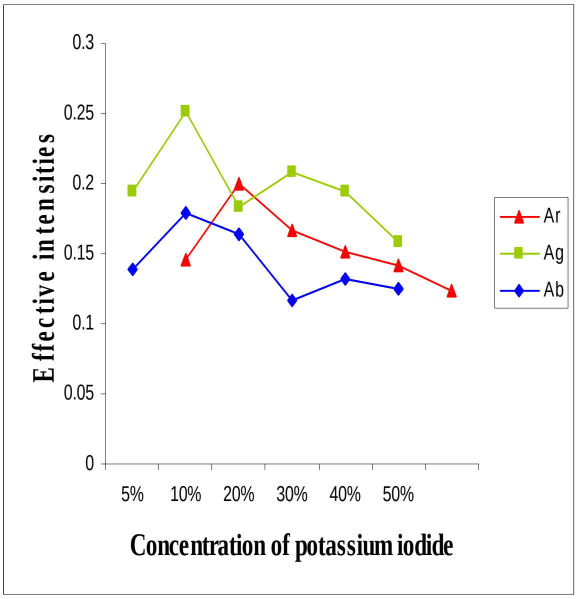


Figure: 1: Optimization of KI

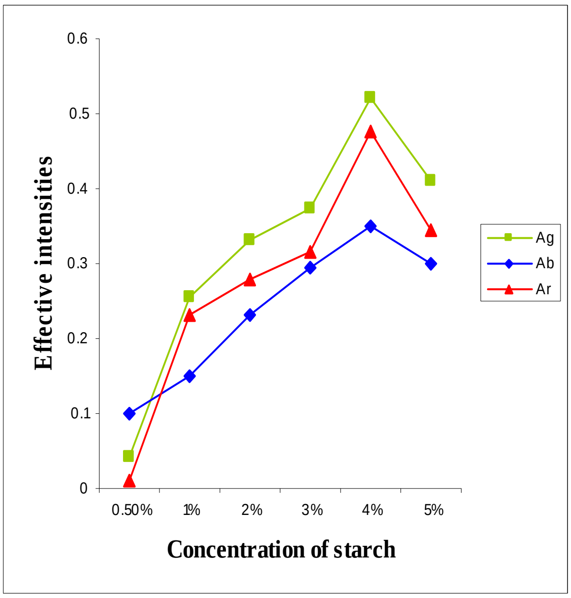


Figure: 2 Optimization of Starch

3.2.2 Effect of reagent concentration:

In order to study the effect of potassium iodide and starch concentration, solutions with various concentrations of reagents were prepared and immobilized on TLC strips and then allowed to dry. After drying 24µL of a standard solution containing 100µg mL^{-1} of lead was injected on each strip. After drying, the strips were scanned and their colors were analyzed by the software, and the effective intensities of R, G and B values were plotted vs. concentrations of KI and starch. Figure 1 and 2 shows the effect of KI and starch concentration on spot intensities respectively. Figure 1 show, at 10% of KI maximum color intensity was observed and figure 2 show at 4% of starch maximum color intensity was observed. Therefore, 10% potassium iodide and 4% starch were selected as optimum.

3.3 Drying Methods:

Different methods of drying such as drying at room temperature, oven and hot air were used for drying the strips after injection of reagents onto strips but no considerable change in signals was observed. However, an oven is recommended for increasing the rate of drying. The open air drying is not recommended to avoid environmental oxidation.

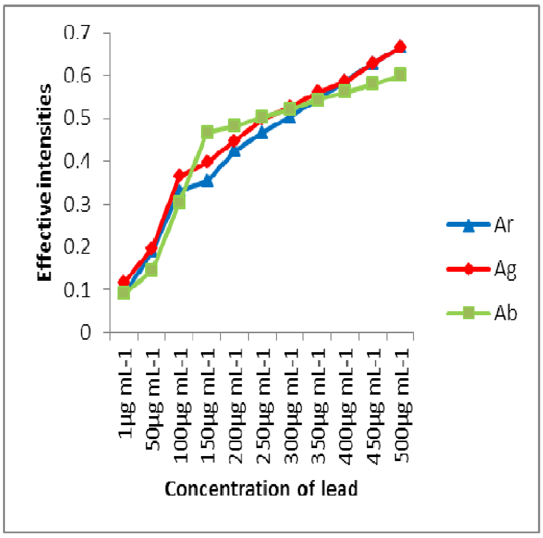


Figure: 3.1: Calibration for 1-500µg mL^{-1}

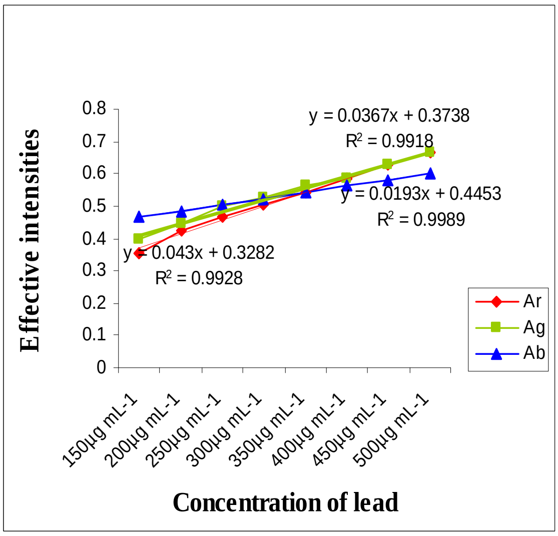


Figure: 3.2: Calibration for 150-500µg mL^{-1}

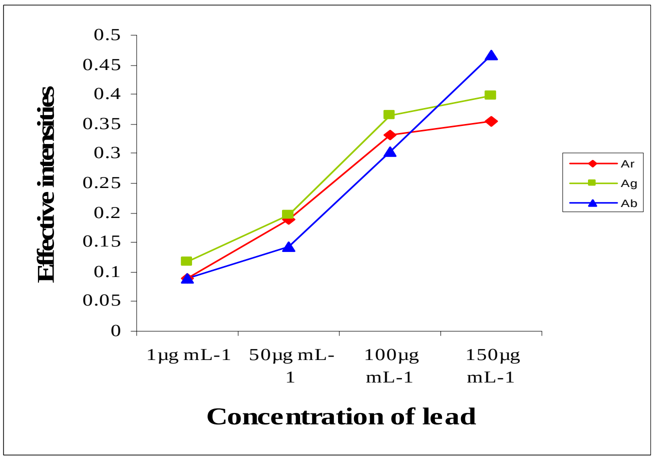


Figure: 3.3: Calibration for 1-150µg ml^{-1}

3.4 Calibration Curves:

Calibration curve was found linear for 150-500 µg mL^{-1} of lead for all three values (R, G and B). From figure 3.1 it is clear that rather the R, G and B all values increases with increase in concentration from1-500 µg mL^{-1} but the curve is linear in the range of 150-500 µg mL^{-1} (figure 3.2). It is clear from figure 3.3 that below 150 µg mL^{-1} of lead R, G and B values follow a different pattern of linearity. In the lower concentration range R values are linear from 1-100 µg mL^{-1} and then 100-150 µg mL^{-1}, G values are linear in the range 1-50, 50-100 and then100-150 µg mL^{-1} while B values are linear in the range 1-50 and then from 50-150µg mL^{-1}. At different concentrations of lead different shades are obtained and hence the RGB follows different order that is why linearity range varies.

3.5 Reproducibility of the system:

Reproducibility of sensor has been investigated at six different sensors under optimum conditions for various concentrations of lead. The results are reported in table1and concludes that the proposed method is reproducible.

Table: 1: Average, SD (standard deviation), RSD (relative standard deviation) Of seven repetitive analyses

S. No.	µg mL $^{-1}$of lead solution	Avg. A_r	SD A_r	RSD A_r	Avg. A_g	SD A_g	RSD A_g	Avg. A_b	SD A_b	RSD A_b
1.	500	0.685	0.013	1.94	0.6689	0.013	1.94	0.6214	0.018	2.96
2.	450	0.608	0.018	2.95	0.6107	0.0096	1.58	0.5731	0.0095	1.66
3.	350	0.539	0.011	2.04	0.5358	0.020	3.74	0.5137	0.014	2.75
4.	50	0.172	0.01	5.83	0.1914	0.012	6.46	0.1454	0.015	10.45

Where; A_r = -log of R value of sample/R value of blank; A_g = -log of G value of sample/G value of blank and A_b = -log of B value of sample/B value of blank

3.6 Effect of Time:

3.6.1 Response Time:

The response time of the system was evaluated under optimum conditions for 100µg mL^{-1}. It is the time required to achieve maximum color intensity. In the proposed method spot of maximum color intensity develops instantly.

3.6.2 Stability of spots:

To study the stability of color spots, 100 µg mL^{-1}of lead was injected under optimum conditions on the sensor. Scanning of the sensor was done in the time period of 5, 10, 30, 60,120, 180 and 240 minute and then after 24 hr and 48 hr. The developed spot remain stable for 30 minute.

3.6.3 Stability of paper optode:

To study the stability of sensor, after immobilizing reagents on the Whatman paper strip, it was used periodically each day. No significant change was observed within15 days, after 16th day spot intensity reduces to nearly half. After 25th day no spot develops. Therefore, the prepared sensor can be used at best for 15 days and on a stretch for 25 days. The sensors were stored in dark and in an air tight container to avoid environmental oxidation.

3.6.4 Detection Limit (DL):

For each RGB factor there is one DL [21].Theoretical DL$_s$ of the method were 0.024 µg mL^{-1}(24µL of 1 µg mL^{-1}) for R, B and 0.012 (12 µL of 1 µg mL^{-1}) for G values respectively. All three values can be considered but G values are more sensitive and follow the standard linearity pattern as shown in figure 3.2 by trend line therefore, values are recommended for determination. Practical DL determined was 3ng mL^{-1}(6µL of 0.5 µg mL^{-1}). The practical DL is the lowest concentration, which gives color on strip no matter falls in linearity range or not.

3.7 Interference studies:

To study the selectivity of the proposed method, the effect of various species on the determination of 100µg mL^{-1} of lead was tested under optimum conditions without any masking. The tolerance limit was defined as the concentration of the added species that causes ± 5 % relative error. The results are indicated in Table 2.

3.8 Application of Paper Optode:

The developed paptode is dual in nature. These can be used as sensor to detect lead as minimum as 3ng mL^{-1} as well as filter to remove up to 93% of lead when a maximum of 500 µg mL^{-1} of lead (super saturation stage) is allowed to be filtered.

Table: 2: Tolerance limit of various interfering species on determination of lead

S. No.	Interfering species	Tolerance limit (mgL^{-1})
1	Mn^{++}	100
2	K^+, Cr^{+++}, Zn^{++}, Na^+, HCO_3^-, Cl^-, Cd^{++}	10000
3	As^{+++}	75
4	Hydrazine	100
5	Se^{+++}	200
6	Ba^{++}	5000
7	Fe^{++}, NH_4^+, SO_4^{--}	12500
10	B^{+++}	1000
11	Cu^{++}	12500
12	Hg^{++}	500
13	Al^{+++}	2500
14	Ca++	2000

3.8.1 In Analysis of real samples:

The paptode is applied as sensor to detect lead in cosmetic, food and biological sample. Using the calibration curve, the concentration of lead in Sindoor (minium) which is used by Indian married women in the parting of their hair has red lead as a component; kumkum (vermillion) which may also have red lead as a component, edible oil and urine samples were calculated. The analysis reveal that lead can be determined quantitatively in the region assessed (table 3).

Table: 3: Percent recovery of lead in environmental samples

S. No.	Sample	Lead originally found	Lead added	Total Lead found	% recovery
1	Sindoor -1	<1µg	10µg	10 µg	90.9
	-2	>1µg	20µg	20 µg	90.9
	-3	<1µg	50 µg	48 µg	94.1
2	Kumkum -1	Below 3ng (below detection limit)	10 µg	10 µg	90.9
	-2		20 µg	21 µg	100.5
	-3		50 µg	50 µg	98.03
3	Urine- - 1	Not found	10 µg	10 µg	100.0
	-2		20 µg	19 µg	95.0
	-3		50 µg	49 µg	98.0
4	Edible oil -1	1 µg	100 µg	110 µg	110.0
	-2	1 µg	200 µg	210 µg	105.0
	-3	1µg	500 µg	550 µg	110.0

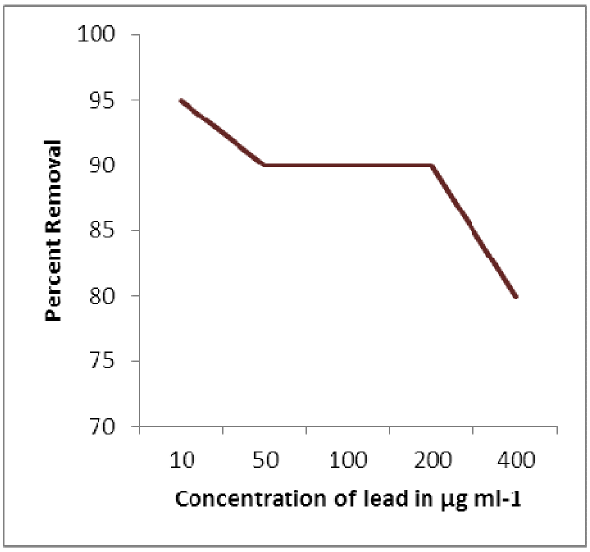
3.8.2 In Removal of Lead:

The paptode is applied as filter to remove lead from aqueous sample.

Heavy metal ions are highly toxic for animals and human beings. Presence of such toxic heavy metal in drinking water system is a potential health hazard. Heavy metal ions pose a series risk to the environment and endanger public health and the environment. Therefore, they should be removed from water and wastewaters before discharge. There is variety of techniques for removal of metal ions. Adsorption has been widely studied as an effective technique for removing toxic heavy metals such as Hg^{2+}, Cd^{2+}, and Pb^{2+} from aqueous solutions [29-35]. Removal of lead through bio waste or plant materials [36-41] and microorganism [42-43] has also been reported. However; these methods suffer from the complete elimination of heavy metals at very low concentrations and are expensive as these require advanced equipment and high energy. Thus, there is a hunger need for a cost effective method for removal of heavy metals from water. The proposed paptodes were also used as filter for removal of lead from aqueous sample. To apply these paptodes for removal; batch removal is employed for each concentration (100-500 µg mL^{-1}) of lead which is allowed to be filtered through paptode filter. The spot developed on paptode were scanned before and after adsorption to know the change in RGB values (figure: 4). It is also observed that increase in size of filter increases the % removal, studies are undergoing. Results of percent adsorption are reported in Table 4.The paptode filter is allowed to remain in contact of 100µg mL^{-1}lead solution for an hour and change in B value is observed at every 15 minute. It is found that best adsorption took place within 15 minute (Figure: 5). The analysis indicates that the proposed disposable paptodes are easy to be utilized, do not require any skilled labor or operation, removal of lead becomes instantaneous using these paptodes.

Table: 4: Percent Removal of Lead

S.no.	Conc. before adsorption	Conc. after adsorption	% Removal
1	500 µg mL^{-1}	100 µg mL^{-1}	80.0
2	200 µg mL^{-1}	20 µg mL^{-1}	90.0
3	100 µg mL^{-1}	10 µg mL^{-1}	90.0
4	50 µg mL^{-1}	5µg mL^{-1}	90.0
5	10 µg mL-1	0.5 µg mL-1	95.0



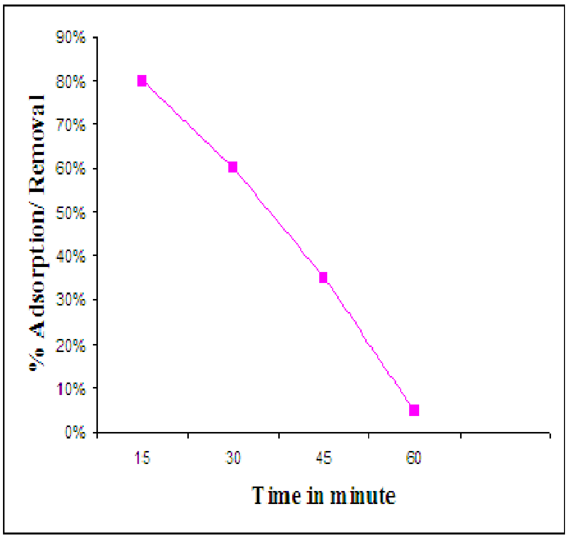


Figure: 4 Effect of concentration on removal **Figure: 5 Effect of Contact time on Removal**

CONCLUSION

The new optical analytical method, "digital RGB analysis" has remarkable advantages over the conventional optical method, "spectrophotometry" which is tedious, non selective, less sensitive and time consuming. The application of flatbed scanner enables the use of non-transparent supporting substrates in the proposed method while transparent supporting substrates is of prime importance in spectrophotometry. The proposed method utilizes the reflection properties of strips, thus turbid samples can be analyzed without any interference unlike conventional spectrophotometry. The developed method is found to be superior and produces quantitative results for determination of Pb (IV) over other existing methods (table 5). Unlike other spot test it produces quantitative results [44]. The method described in present paper does not need any expensive instrument; it is simple and rapid, sensitive, selective and free from interference of metal ions i. e. Cu^{2+}, Hg^{2+}. The developed paptode is also applicable for effective removal of lead due to wider range of linearity for determination of lead.

Table: 5: Comparison of proposed method with other methods

S. No.	Reagents	Method	Range of determination	Detection limit	Remarks
1	TritonX-114 [12]	Graphite Furnace Atomic Absorption Spectrometry	0.08-30 µg mL^{-1}	0.08 µg mL^{-1}	pH sensitive, Skilled operation is required, complex procedure
2	2-aminothiazole, polystyrene-divinylbenzene [15]	Solid Phase extraction	0.0-5.0 µg mL^{-1}	2.5 µg mL^{-1}	Skilled labor requirement, complex
3	Feather modified carbon paste electrode [16]	Anodic stripping voltametry	0.5-5.0 µg mL^{-1}	0.121 µg mL^{-1}	Less sensitive and complex
4	EM Quant®[45]	Spot test	NA	20µg mL^{-1}	Semi-quantitative
5	KI and starch (present method)	MATLAB Assisted Spectrophotometry	0.018-9 µg mL^{-1}	3ng mL^{-1}	Quantitative, Highly sensitive, removal is possible.

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