Construction and Application of Electrolytic Cell for Iodine Determination

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ABSTRACT

Iodine is very important element and has a key role in different fields of life so its analysis is of great significance. For the determination of iodine usually iodometric methods are used, but the range of this method is up to molar level. In the present work a simple, easy and economically feasible method is electroanalytical method is developed. An electrolytic cell for the determination of iodine was constructed which was based on redox reaction. The determination of iodine was done by using KI electrolysis mechanism. NaCl (10 ppm) was taken as a standard and solutions of different iodine concentration were added at each time and current (μA) were recorded, with the help of which standard curve was prepared. It was applied to various unknown standard samples and appropriate results were obtained. Similarly to check the practical applicability of the method it was successfully applied to different table salts (Swaad and Raaz). The range of this method is up to micro level (upto 10 ppm) and also had a considerable error rang but that will be overcome by further modification of this method.

Key words: Iodine, determination, electrolytic cell

1. Introduction

Iodine is an element with atomic number 53. The name is derived from Greek word *ioeides* meaning violet or purple, due to color of elemental iodine vapor. Iodine and its compounds are primarily used in nutrition and industrially in the production of acetic acid and certain polymers. Iodine's relatively high atomic number, low toxicity and ease of attachment to organic compounds have made it a part of many x-ray contrast materials in modern medicines. Iodine is found on earth mainly as the highly

water soluble iodide ion, which concentrates it in oceans and brine pools, like the other halogens, free iodine occurs mainly as a diatomic molecules I_2. In the universe and on earth, iodine's high atomic number makes it a relatively rare element.

Demin *et al* investigated the behavior of Cu (II), Cd(II), Pb(II), and I^- in aqueous solution of NaCl by using stripping voltammetry. For sequential stripping determination of Cu (II), Cd (II), Pd (II) and iodide a new method was proposed in which indicator electrode which was made from carbon glass ceramic modified with carbon glass ceramic modified with mercury.

Kaverin *et al* studied an acid solution of KI (0.2M) and investigated that molecular iodine generation at the Pt anode. The calculated efficiency of iodine current was in a wide spread range of anode current densities (3-100 mA/cm^2) [2].

Fierro *et al* performed the electrochemical detection of iodide and iodine via cyclic voltammetry on boron doped diamond (BDD) electrodes individualy and simultaneously in a 1M $NaClO_4$ (pH 8) solution, representative of typical environmental water conditions [3].

Campos measured the dissolved total reducible iodine in natural waters by using cathodic stripping voltammetry. At pH $\leq$ 2.7 iodate is quickly reduced to iodide by ascorbic acid (0.5 mM), and total iodine is measured as iodide [4].

Jow *et al* using a direct electrochemical reduction method for the determination of the iodine adsorption number (I_N) of carbon black (CB). The obtained results by the electrochemical method are close to that obtained by traditional titration methods [5].

Tian and Nicolas done iodine analysis upon seawater samples by differential pulse polarography and cathodic stripping square wave voltammetry methods, respectively and directly determined dissolved iodate and iodide [6].

Langenauer and Krahenbuhl determined fluorine and iodine in some biological reference materials. For iodine radiochemical neutron activation analysis was used and for fluorine ion-selective potentiometry. From the matrix elements the two elements were separated in the presence of vanadium (V) oxide by pyrohydrolysis [7].

Nguyen *et al* done for urinary iodine analysis a sensitive and specific ion-pair reversed-phase high performance liquid chromatography (HPLC) method. In this method a silver working electrode is used and based on pulsed amperometric detection (HPLC–PAD), which improves peak shape, electrode stability as well as linearity and reproducibility [8].

All the reported methods reported reproducible and good results for determination of iodine. However, those all are based on using expensive instruments and costly methods. While in our present work we attempted to construct and utilize simple electrolytic cell for easy and economical determination of iodine upto ppm level.

2. Experimental

2.1. Materials and Methods

All experimental work was performed at Analytical Chemistry lab, Abdul Wali Khan University Mardan, Pakistan. All the chemicals: Potassium iodide (KI), Sodium Chloride NaCl and distilled water were of analytical grade. Battery 6 V, alligator clips and multimeter were purchased from the local market. Two graphite electrodes were collected from 1.5 V Dry cells.

2.2. Collection of Table salts

The *table salts* (RAAZ and SWAAD salt) were collected from the utility store in Manga, District Mardan, KP, Pakistan.

2.3. Preparation of solutions for calibration graph

0.1 g of NaCl was taken and dissolved in 100 ml of distilled water to prepare 1000 ppm solution. 10 ppm solution was prepared from it by using dilution formula $C_1V_1 = C_2V_2$. Then 0.1 g of KI was taken and dissolved in 100 ml of distilled water to prepare 1000 ppm solution. 1, 2, 3, and 4 ppm solutions of KI from 1000 ppm solution were prepared by dilution.

2.5. Preparationof salt solutions (Swaad and Raaz salt)

0.1 g of Swaad Salt was taken and dissolved in 100 ml of distilled water to prepare 1000 ppm solution. Then prepare 10 ppm solution from it by using dilution formula $C_1V_1 = C_2 V_2$. Similarly solutions from Raaz salt were prepared by the same method.

2.6. Electrochemical study of solutions prepared for calibration graph

The prepared 1, 2, 3, and 4 ppm solutions of KI were added to 10 ppm solution of NaCl one by one respectively in an electrolytic cell containing two graphite electrodes connected to a multimeter through which current (µA) was recorded and a 6 V battery.

2.7. Electrochemical studies

10 ppm solution of RAAZ and SWAAD Salt individually were taken in the electrolytic cell and current (µA) were recorded.

3. Results and Discussion

3.1. Method used for Determination of Iodine

Iodine is an important element, having lot of application in different fields of life, specially used as daily intake as a table salt. Due to its importance different methods are used for determination its determination. In this present work an Electrolytic method for determination of iodine was successfully developed. The method is based on Redox reaction.

Table 1: Investigation of the current (µA) as a function of KI concentration (ppm) added to 10 ppm solution of NaCl present in the electrolytic cell container.

S. No.	KI (ppm) added to NaCl (10 ppm)	Current (µA)
1	0	724
2	1	754
3	2	776
4	3	810
5	4	843

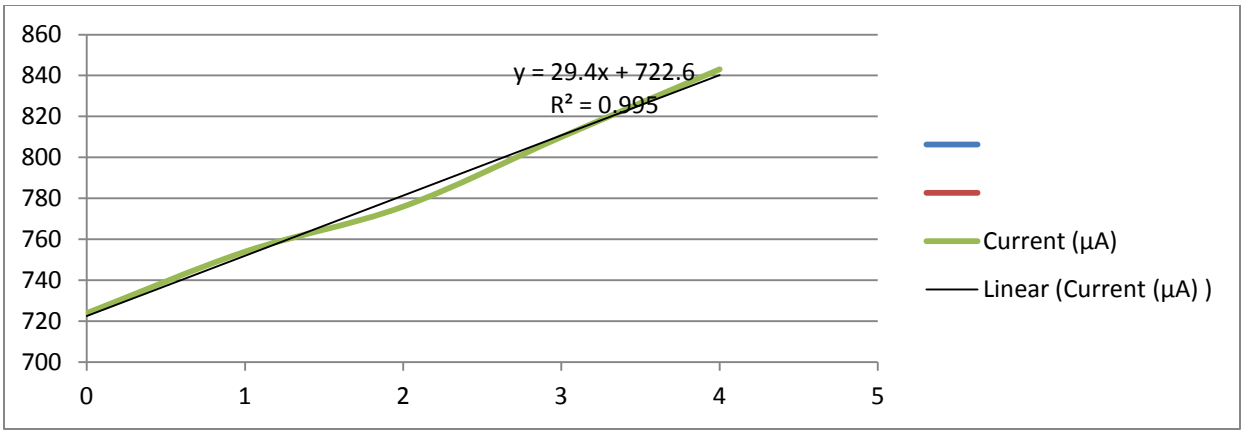

Fig 1. Relationship between current and NaCl(10 ppm) solution having different concentration of KI in an electrolytic cell.

3.2. Investigation of Potassium Iodide Concentration as a Function of Current

For the determination of potassium iodide concentration, a standard curve was prepared by using KI as a standard and NaCl as supporting electrolyte as shown in Table 1. The KI standard solutions of different concentrations were prepared and its produced current in electrolytic cell was recorded. Figure 1 indicates that the straight line curve was obtained by plotting the current against concentration which means the current increases as the concentration of KI increases in the cell. In the same way the method was applied to different samples of KI of unknown concentration. It was found that as the concentration of KI solution decreased, the percent error decreased which means at low concentration the results are good (data not shown).

3.3. Difference Between the Actual and observed concentration of Potassium Iodide after Applying Standard Curve

The method gave good results for the KI sample with unknown concentration however, small positive and negative errors were also observed (data not shown).

3.4. Difference Between the Actual and Observed Concentration of Iodine in Swaad table salt

For the determination of iodine in Swaad table salt, solution of 10 ppm was prepared and the current (µA) was recorded. Table 2 shows that after applying the standard curve to its respective current (µA) of the solution,

an increase in concentration in other words a positive error was observed compared to the amount written on the packing. This increase in observed concentration was about 0.184 % in 10 ppm solution of Swaad table salt, which we think might be due to some determinate error like instrumental errors or some indeterminate errors.

3.5. Difference Between the Actual and Observed Concentration of Iodine in Raaz table salt

Similarly for the determination of iodine in Raaz table salt, solution of 10 ppm was prepared and the current (µA) was recorded. Table 3 shows that after applying standard curve to its respective current (µA) of the solution, an increase value of concentration was observed compared to actual concentration of the salt solution or the amount written on the packing. This positive error in concentration was about 0.047 % in 10 ppm solution of Raaz table salt, which might be due to errors as discussed for the Swaad table salt.

Table 2. Concentration of iodine in Swaad Salt calculated from standard curve.

Current (µA)	y = 29.4x + 722.6 (Exp)	Actual	% Error
740	0.591837 ppm	0.5 ppm	0.183673

Table 3. Concentration of iodine in Raaz Salt calculated from standard curve.

Current (µA)	y = 29.4x + 722.6 (Exp)	Actual	% Error
746	0.795918	0.76	0.047261

4. Conclusions

Iodine is one of the essential elements for human body and plays a key role in several fields. There are various methods which are successfully used for the determination of iodine on commercial level but the electrochemical techniques which are based on redox reaction are still one of the most popular. In the present study an electrolytic KI cell was constructed and was used for iodine determination. The aim of our study

was to check the feasibility of using our electrochemical method for the determination of iodine. We applied our method to different table salts successfully. We presented relatively new approach for the determination of iodine in table salts, and got good results. However, this is only initial work, and still need improvements. Our method is easy, simple and economically feasible. In future if this method is improved it is possible to get good results and it can be used for determination of various samples having electrolytic constituents.

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