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Short Communication

Development of new spectrophotometric determination of titanium in homeopathic pharmacy using Ponceau S as a reagent

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Spectrophotometric method has been developed for the determination of titanium(III). The linear calibration curve was obtained with 2-10 g/ml titanium. The titanium was determined in pharmaceutical preparation with coefficient of variation 3.0%. The concentration of titanium was calculated from the standard calibration curve and was found 111.25 µg/ml with coefficient of variation 3.0%. The obtained results agreed with the results obtained from hydrogen peroxide (H_2O_2) method.

Key words: Determination, medicine, Ponceau S, spectrophotometer, titanium.

INTRODUCTION

The paint, pigment, paper and pulp industries discharge a very high amount of titanium. Titanium is a metal that has been used in various and varied medical applications for about 40 years. Although many biomaterials have come and gone during this period, titanium is one of the few that has seen its uses and reputation enhanced over the years.There are various methods for the determination of titanium utilizing a number of ligands such as pyridylazoresorcinol (Zhou, 1990a), 5-bromopyridylazodiethylaminophenol (Zhou, 1990b), thiazolylazoresorcinol with mordant red 19 (Zhou, 1994), solochrome violet relay services (RS) (Wang and Mahmud, 1986), pyrocatechol violet (Vukomanovic and van Loon, 1994), Beryllon (III) and cupferron (Xin et al., 1995). Ponceau S has been used for the staining of paper electrophoresis strips for the determination of serum albumin using cellulose acetate electrophoresis clearing and preserving cellulose acetate electro-pherograms stained with Ponceau S (Meulemans, 1960; WuEsTm, 1965). It is used for the electrophoresis of the high-tyrosine proteins of keratins on cellulose acetate (Blagrove et al., 1975). Kohn and Pamel (1978) described a method of immunofixation using cellulose acetate immunofixation technique. This is followed by washing and staining by Ponceau S. Heick et al. (1980) has compared the protein of urine and cerebral spinal fluid by using Ponceau S dye. El-Bahi et al. (1992) used Ponceau S stained nitrocellulose strips for comparison of detected stable diagnostic antigen from bile and feces of Fasciola hepatica infected cattle The present work examines the potentials of the reagent Ponceau S for the spectrophotometric determination of titanium in pharma-ceutical preparation.

MATERIALS AND METHODS

Ponceau S (0.004 M)

Ponceau S (0.306) (BDH) was dissolved in 100 ml of distilled water. Solution was further diluted 10 times with distilled water before spectrophotometeric studies.

Titanium (III) solution (1000 µg//ml)

The solution (III) chloride (1.8 ml) (15% Ti Cl3 solution, E. Merck) was added to 20 l of 37% hydrochloric acid (Merck) and diluted to 100 ml with distilled water.

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Abbreviations: H_2O_2, Hydrogen peroxide; **RS,** relay services; **HCL,** hydrochloric acid; **$KBrO_3$,** potassium bromate.

Apparatus

Absorbance measurements were recorded on a Hitachi 220 UV/Vis spectrophotometer equipped with 150 W deuterium lamp and 1 cm quartz cells. All pH measurements were made with an Orion 420 A digital pH meter with a combined glass-calomel electrode. A laboratory temperature of 30 ± 2°C was used through out study. All solutions were previously brought to this temperature.

Chemicals

GR grade chemicals of sodium acetate, acetic acid, sodium bicarbonate, sodium carbonate, boric acid, borax, ammonium chloride, ammonia, chloroform, acetonitrile, hydrochloric acid (HCl) (37%) (E. Merck), were used. Freshly prepared double distilled water for all glass was used through out the work. The buffer solutions in the pH range 1-10 at unit interval were prepared potassium chloride (1M), HCl (0.1M); pH 1-2, sodium acetate (1M)-acetic acid (1M); pH 3-6,ammonium acetate(1M)- acetic acid (1M); pH 7, boric acid(1M)- borax(1M); pH 8-9 and ammonium chloride (1M)-ammonia pH 10.

All glasses were previously soaked in 10% nitric acid for 24 h and rinsed with double distilled water before the use.

Analytical procedure for the determination of titanium (III)

To volumetric flask (10 ml) was added 1.4 ml of Ponceau S solution (0.4m M), 0.4 to 2.0 ml of solution containing 20-100 µg of titanium (III) and the volume was adjusted to 10 ml with 3 M HCl. The decrease in absorbance was measured at the wavelength of 520 nm. The blank solution was prepared following the same procedure except addition of titanium (III).

Effect of pH and acid concentration

To different volumetric flasks (10 ml) were added Ponceau S (1.4 ml) (0.4 mM), titanium (III) (1.0 ml) (50 ppm), different buffer solutions within the pH range 1- 10, at unit interval (2 ml) and volume was adjusted to the mark with water. Ponceau S solution (1.4 ml) (0.4 mM) and titanium (III) (1 ml) (50 ppm) were transferred to volumetric flask (10 ml) and added HCl (5M) 0.2-2.2 ml with an interval of 0.2 ml. The volume was adjusted to 10 ml with water. Ponceau S solution (1.4 ml) (0.4 mM) and titanium (III) (1ml) (50 ppm) were transferred to volumetric flask (10 ml) and added HCl (37%) 2-6 ml with an interval of 1.0 ml. The volume was adjusted to 10 ml with water. The absorbance of each of the solution was measured against reagent blank containing same acid concentrations and pH.

Analysis of test solutions

The accuracy of method was checked by analysis of unknown concentrations. Test solutions (1- 3 ml) were transferred to volumetric flask (10 ml) containing 1.4 m Ponceau S solution (0.4mM) and analytical procedure was followed. The amount of titanium in test solution was calculated from calibration curve.

Sample analysis for titanium

Gin Seng (B. M. Homeopathic pharmacy, Lahore, Pak) (0.5 ml) was transferred to a crucible and was gently heated on a burner, followed by in muffle furnace (Phoenax Furnaces Ltd, Shefflied, England), at 550°C for 6 h. The white residue was dissolved in HCl (3M) and volume was adjusted to 50 ml with 3 M HCl solution. To 10 ml volumetric flask, a solution 1.4 ml mM Ponceau S was transferred and 5 ml of sample solution was added and the volume was adjusted with 3M HCl solution. The absorbance was recorded at 520 nm as described in analytical procedure.

Spectrophotometric determination of titanium (III) using hydrogen peroxide

A sample solution (1.0 ml) was transferred to a conical flask (25 ml) and the analytical procedure was followed (Stewart et al., 1974)

RESULTS AND DISCUSSION

General characteristics of the reaction

Ponceau S is a red coloured dye. It absorbs at 520 nm with molar absorpitivity of 32500 $L.mol^{-1}.cm^{-1}$. Initially it was examined for possible catalytic determination of metal ions by its decomposition with hydrogen peroxide (H_2O_2) or potassium bromate ($KBrO_3$) but when titanium (III) in acid solution was added a decrease in intensity of the colored dye was observed at 520 nm. After preliminary optimization, it was found that there was no effect of H_2O_2 or $KBrO_3$ but was due to the complex formation with Ti (III). As titanium(III) is easily hydrolysed in aqueous solution, therefore, the final concentration of hydrochloric acid was examined at 3 M.

Effect of variables

The final concentration of Ponceau S solution was varied from 0.01 mM to 0.056 mM. It was observed that when reagent in HCl was added, the 0.016 mM solution turned yellow immediately with the addition of titanium(III), afterwards, there was a regular increase in the absorbance upto 0.056 mM. The sensitivity of spectrophotometer was poor above the concentration. Thus, the final concentration of Ponceau S (0.056 mM) was selected for further study and 0.4 mM, 1.4 ml was added in 10 ml.

Effect of hydrochloric acid concentration

The effect of hydrogen ion concentration on the change in absorbance of Ponceau S with titanium (III) was investigated. It was observed that change in absorbance occurred in acidic media only. It was therefore the change in absorbance with HCl concentration was further examined. The concentration of HCl was varied from 0.5 M to 4.0 M in the final volume, and it was considered that the solution which gave maximum difference between the blank (B) and the analyte (A) was considered as optimal. It was observed that a similar absorbance was obtained using 0.5-4 M concentration of HCl. As titanium(III) solution is readily hydrolysed in aqueous, therefore, 3 M HCl was maintained in the final volume and was selected

for the prevention of Ti(III) solution.

Effect of titanium (III) concentration

The change in absorbance of Ponceau S with a concentration of titanium (III) was examined. A linear calibration curve was obtained by plotting (B-A) against amount of titanium (III) within 2-10 µg/ml in the final volume with coefficient of correlation (r) = 0.982

Interference

The effect of different cations and anions on the determination of the 5 µg/ml of titanium (III) was investigated for Ca (II), Mg (II), K (I), Na (I), Al (III), Mn(II), Co (II), Cu (II), Zn (II), Cd (II), Cr (III), NO_3^-, NO_2^-, Cl^-, Br^-, SO_4^-, citrate, tartrate, BrO_4 and ascorbic acid, it was observed that their concentration when added 10 times the concentration of titanium (III), did not affect the determination of titanium (III). However, Mo (IV), Mo (VI), Fe (II), Fe (III), V (V) and V (VI) when added at the same concentration of titanium (III), interfered the determination of titanium and enhanced the decrease in the absorbances with increase in different difference between the blank (B) and analyte (A) absorbance (B-A) and interfered.

Sample analysis

The titanium contents in a pharmaceutical preparation (Gin Seng Tonic) was analysed by dry ashing, followed by acid digestion. The spectrophotometeric determination was carried out using analytical procedure. The concentration of titanium was calculated from the standard calibration curve and was found 111.25 µg/ml with coefficient of variation 3.0%. The sample was analysed spectrophotometrically (Stewart et al., 1974) for the contents of titanium (III) and was found 110.21 µg/ml with coefficient of variation 2.5%.

Conclusion

The proposed spectrophotometric method has an acceptable sensitivity and selectivity for the determination of titanium (III) though a few metal ion affected the determination. The method was applied successfully for the determination of titanium in pharmaceutical preparation. It can also be concluded that this study constitutes the basis of a use of Ponceau S for detecting other transition metals.

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