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Estimation of Sulphite Using Sodium Nitroprusside By A Newer Photochemical Method

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ABSTRACT

In these present investigation, photochemical ligand exchange reaction of sulphite and sodium nitroprusside has been studied. There are several different methods for determination of sulphite but as compare to other these new method is an economical, quicker and appropriate quantitative method. SNP (sodium nitroprusside) is one of the common photo labile complex which undergoes photochemical ligand exchange reaction with organic as well as inorganic ions swiftly. To estimate sulphur, nitrogen containing anions and electron opulent organic molecules, some recent determinations have been made to develop such reaction. The progress of the reaction is observed by spectrophotometric analysis. The outcome of reaction parameters i.e.effects of pH, concentration of SNP (sodium nitroprusside), intensity of light, ligand concentration, etc. was studied. The determinations were made to minimize the percentage error and some optimum conditions were achieved. Such reaction can be used for the determination of sulphite in the range of millimoles to micromoles; hereafter it is significant to know whether such assessments can be done effectively and that too with the desired precision.

KEYWORDS: Sulphite, sodium nitroprusside, photochemical reaction, % error

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INTRODUCTION

Photosensitized reactions are commonly used in variousbiological and technical field. Inorganic chemist may seek an improved understanding through the example that photochemistry gives the relationship between reactivity and electronic and molecular structure[1].Wayne and Burrows *et al.*[2]have reported photosensitized reactions like copolymerization. In photochemistry, the excited states of atoms play an important role.

Nemono*et al*.[3] have usedPt/TiO_2 nanoparticles for the study of Photocatalyticdecomposition of NH_3 to N_2 and H_2 in aqueous solution.Photodegradation and adsorptionof 1,4-dioxaneon titanium dioxidehave been carried outby Yamazaki *et al*.[4]. Kiselev*et al*.[5]have useddry and wet rutile titanium dioxide for the photodegradation and adsorption of di-isopropyl fluorophosphate and dimethyl phosphonate.Fe promoted photodegradation of microcystin- LR usingTiO_2have been studied by Yuan *et al*.[6].

TiO_2/Pt nanoparticles has been studied by physiochemical and photocatalytic characterization by Liu *et al*.[7]. TiO_3-TiO_2 composite materialhas been used for photocatalytic bleaching of methylene blue by Udea[8] and Otsuka. Maldonado *et al*.[9]have reported the comparative study ofFenton plus photo-Fenton and titanium dioxide. Liu *et al*.[10] have investigated treatment of waste water by photodegradation using U.V./TiO_{2}.Theeffect of inorganic anions has been observed by Zhu *et al*.[11]on photocatalytic oxidation of aqueous ammonia and nitrite by using titanium dioxide.

Tsuji*et al.*[12]have studied Pb-doped ZnS overH_2 evolution from an aqueous K_2SO_3 solution under visible-light irradiation (λ>420 nm) to check the photocatalytic activity. Wetchakun*et al.*[13]reported that 2.0 % Fe-doped ZnS sample possess the maximum degradation activity because of the presence of Fe in ideal amount and the rise of surface area and light in UV region. Fe loaded ZnS nanoparticles ($Zn_{1-x}Fe_xS$; where x = 0.00, 0.03, 0.05 and 0.10) synthesized via chemical precipitation method by Chauhan *et al.*[14]. They found that the Fe loaded ZnS bleaches Methylene Blue considerable faster as compared tounloaded ZnS upon its exposure to the visible light thanUV light.

Kaur*et al.*[15]have preparedtransition metal doped ZnO nanoparticles and ZnO($Zn_{0.99}M_{0.01}O$; M=Mn, Cu, Ni and Co) by thermally decomposition of their oxalate precursor. A series of W-doped ZnOnanoparticles with dissimilartungsten content has been synthesized viasol–gel method by Moafi*et al.*[16]. The photocatalytic behaviour ofW-doped ZnOandundopedZnO was investigated by photodegradation of methylene blue in aqueous solution.

Anovel Al-doped ZnO (AZO) photocatalysts has been synthesized by Omidi*et al.*[17] with dissimilaraluminum concentration (0.5–6.0 mol%) via a facile combustion process followed by calcination process at 500 °C for 3 hour. The photocatalytic behaviour of the samples were investigated by photodegradation of methyl orange under visible and sunlight radiation.Huang *et al.*[18]have synthesized Bi_2S_3 nanostructures to study their photocatalytic activities in organic dyesdegradation.

Gao *et al.*[19]have prepared hierarchical BiOCl microspheres. BiOCl showedvery high photodegradation rate overCarbamazpiene. Pala *et al.*[20] studied photocatalyic degradation of cyanide in waste water using new generated nanothin film photocatalyst($K_2La_2Ti_3O_{10}$,KLTO). Marchena*et al.*[21] have reported ZSM-5and ammonium-Y encapsulatedtungstophosphoric acid (TPA). The encapsulation of TPA on NH_4Y and NH_4ZSM5 zeolites is an efficient method to prepare catalysts with good photocatalytic activity overdegradation of 4-chlorophenol. Photocatalytic degradation ofp-chlorophenol have been studied by Nguyen and Juang[22]over H_2O_2/TiO_2 hybrid in aqueous medium under irradiation.

MATERIALS AND METHODS

Experimental

Stock solutionshave been prepared by dissolving 0.126 g of sulphite (0.01M) and 0.297 g of sodium nitroprusside (0.01M) separately in 100 mL doubly distilled water.20 mL stock solution ofsodium nitroprusside was diluted to 100 mL to form M/500 concentration. Further it was divided into five equal sets so that each set contain 20 mL solution. In each beaker, 0.4 mL, 0.8 mL, 1.2mL, 1.6mL and 2.0 mL solution of sulphite(M/100) was added. Further, all the sets were exposed to a 200-watttungsten (W)-lamp for ¼ hour. As a result, the colour of solution was changed to peacock bluefrom light red.

5.0 mL sample solution was taken out from each setand change in optical density has been checked spectrophotometrically (λ_{max}= 620 nm).

A graph of optical density to known concentration of sulphiteion has been plotted i.e.[8.23 x 10^{-4} M, 7.42 x 10^{-4} M, 5.66 x 10^{-4} M, 3.84 x 10^{-4}M, 2.91 x 10^{-4}M, 1.96 x10^{-4}M].

1.0 mL sulphitesolution of known concentration was mixed with 20 mL of sodium nitroprusside (M/500). As prepared reaction mixture was exposed to W-lamp under idealenvironments. The optical density was checked spectrophotometric analysis and calibration curve has been made in order to check the sample concentration. The percentage error was find outfrom determined concentration for sulphite solution.

RESULT AND DISCUSSION

Effect of pH

The photochemical reaction of sulphite ion with sodium nitroprusside may be influenced by the change in value of pH.Hence, the effect of pH was examinedon determination of sulphite at various pH range. The results are given in Table 1.

Table 1 Effect of pH*

S. No	pH	Error (%)
1	1.6	2.8
2	1.8	2.0
3	2.1	1.4
4	2.5	2.2
5	3.0	2.8
6	3.5	3.2
7	4.0	4.0
8	5.0	4.4
9	6.0	4.6
10	7.0	4.8

*[SNP] = 1.04 x 10^{-2} M, Light Intensity = 12.0 $mWcm^{-2}$, [Sulphite] = 9.52 x 10^{-3} M, λ_{max}= 620 nm

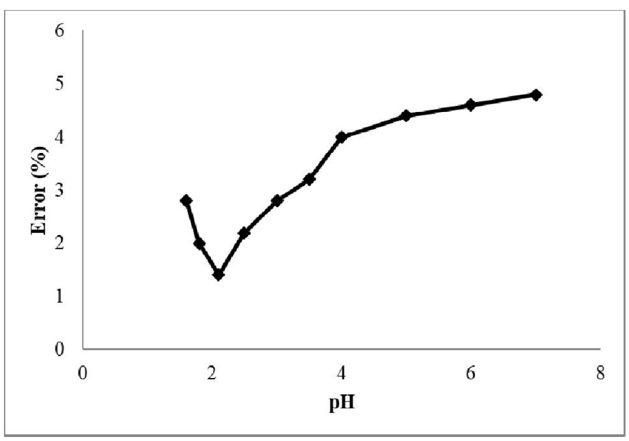

Figure 1- Effect of pH

As per the data, the pH found for least error is 2.1 i.e. 1.4 which is in acceptable range and the pK_{a1} and pK_{a2} for sulphuric acid are 1.89 and 7.21 respectively. At pH = 2.1 the sulphurous acid exists as HSO_3^- ion rather than SO_3^{2-} ion. Hence HSO_3^- ion enters in the coordination sphere of sodium nitroprusside to form complex rather than SO_3^{2-} ion. Above pH = 2.1 again the percentage error increases because ofdecreasing concentration of HSO_3^- as compared to SO_3^- ion.

EFFECT OF CONCENTRATION OF SULPHITE

The effect of sulphite ion concentration has been checked by taking various concentration of sulphite ion by keeping other parameters fixed. The results are given in Table 2.

Table 2 Effect of concentration of sulphite*

S. No	[Sulphite] x 10^{-3} M	Error (%)
1	7.14	3.2
2	7.50	2.9
3	7.93	2.5
4	8.73	1.9
5	9.02	1.6
6	9.52	1.4
7	10.10	2.0
8	10.31	2.8
9	10.71	3.6
10	11.50	4.0

*Light Intensity = 12.0 $mWcm^{-2}$, λ_{max} = 620 nm

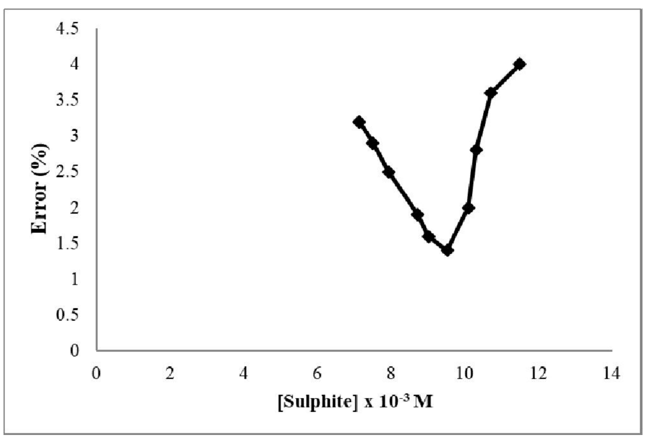

Figure 2- Effect of concentration of sulphite

The minimum error found in the sulphiteion determination is at 9.52 x 10^{-3}M concentration of sulphite ion i.e. 1.4% which is in acceptable range.

The complex formation tendency increases upon increasing the concentration of sulphite ion. Therefore, the percentage error for determination of sulphitewas found minimum. Upon increase theconcentration above 9.52 x 10^{-3}M, the movement of sulphite ions inhibitsdue toits higher concentration. It results in the increase of percentage error.

EFFECT OF CONCENTRATION OF SODIUM NITROPRUSSIDE

The effect of concentration of sodium nitroprussidein the determination of sulphite ion has been examined. Various concentration of sodium nitroprusside has been taken by keeping other parameters fixed. The results are given in Table 3.

Table 3 Effect of concentration of sodium nitroprusside*

S. No	[SNP] x 10^{-2} M	Error (%)
1	0.88	3.8
2	0.91	3.6
3	0.94	3.0
4	0.98	2.6
5	1.01	2.0
6	1.04	1.4
7	1.07	2.2
8	1.09	2.8
9	1.11	3.3
10	1.12	4.0
11	1.14	4.2

*[Sulphite] = 9.52 x 10^{-3} M, pH = 2.1, Light Intensity = 12.0 mWcm^{-2}, λ_{max} = 620 nm

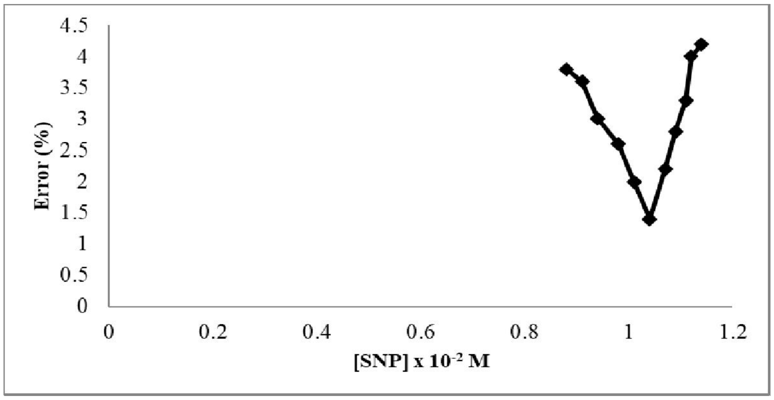

Figure 3 Effect of concentration of sodium nitroprusside

The minimum error in the sulphiteion determination is found at 1.04 x 10^{-2} M sodium nitroprusside concentration i.e. only 1.4% which is in acceptable range.

The tendency of complex formation increases as the concentration of sodium nitroprusside increases. Upon increasing the sodium nitroprusside concentration above 1.04 x 10^{-2}M, it acts like internal filter and will not permit the desired light intensity to reach sodium nitroprusside molecule in the bulk of the solution. As a result,small number of sodium nitroprusside molecule will be excited to contribute in the formation of complex, which causes the rise in percentage error.

EFFECT OF LIGHT INTENSITY

The effect of light intensity in the sulphitedetermination has been checked by changing the distance between W-lamp and the exposed exterior part of the reaction mixture. The result isgiven in Table 4.

Table 4 Effect of light intensity

S. No	Light Intensity (mWcm^{-2})	Error (%)
1	6.0	5.6
2	7.0	5.0
3	8.0	4.8
4	9.0	3.8
5	10.0	2.6
6	11.0	2.0
7	12.0	1.4
8	13.0	1.4
9	14.0	1.4
10	15.0	1.4
11	16.0	1.4

[Sulphite] = 9.52 x 10^{-3}M, [SNP] = 1.04 x 10^{-2} M, pH = 2.1, λ_{max} = 620 nm

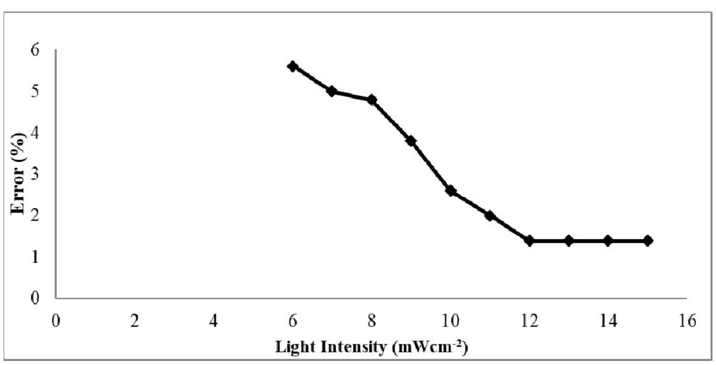

Figure 4 Effect of light intensity

In the determination of sulphite ion, the minimum error is found at 12.0 $mWcm^{-2}$ intensity of W-lamp, i.e. 1.4% which is inacceptablerange.

Upon increasing the light intensity, the number of photons striking per unit area per second was increased too. Therefore,formation of complex became easier upon increasing light intensity.Further, increase in light intensity above 12.0 $mWcm^{-2}$, the error remained constant.It indicates that the 12.0 $mWcm^{-2}$light intensity is enough for the maximum complex formation.

OPTIMUM CONDITIONS

The photochemical reactions of sulphite ion were carried out by takingsodium nitroprusside. It has beenfound that following condition is ideal for the determination of sulphite ion as it gives only 1.4%error, which is inacceptablerange.

(i) pH = 2.1

(ii) [Sulphite] = 9.52 x 10^{-3}M

(iii) [Sodium Nitroprusside] = 1.04 x 10^{-2}M

(iv) Light Intensity = 12.0 $mWcm^{-2}$

CONCLUSIONS

Determination of sulphitewasdone by performing photochemicalreaction using sodium nitroprusside. The reaction progress waschecked spectrophotometrically. The effects of variousreaction parameters i.e. pH, sulphite ions concentration, sodium nitroprusside concentration and light intensity were checked to improve the reaction conditions.Optimum reaction condition found for the determination of sulphite ion is 2.1 pH, 1.04 x 10^{-2}M concentration of sodium nitroprusside, 9.52 x 10^{-3}M concentration of sulphite ion and 12.0 $mWcm^{-2}$light Intensity.

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