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Synthesis of Stannic oxide and its Application as Adsorbent to Study Adsorption of Copper (II) and its Separation from other Elements

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ABSTRACT

A method has been developed for the quantitative adsorption, of Cu (II) and its separation from other elements. Spectrophotometric determination of Cu(II) has been carried by employing tetraethylenepentamine (TEPA) as a spectrophotometric reagent. Stannic oxide has been prepared by co-precipitation method which is a nanomaterial. Stannic oxide is found to be stable towards acids, bases and most of the common chemical reagents. The effects of different parameters such as pH, time of contact, amount of stannic oxide in the adsorption of 50.0µg of Cu (II) has been studied. It has been observed that 250 mg of stannic oxide is sufficient for maximum adsorption ($94.34 \pm 1.34\%$) of 50.0µg Cu (II) at pH 5.0 and contact time of 5.0 minutes. Under the optimum conditions of adsorption, the effect of various anions and cations in the adsorption of Cu (II) also has been studied. Interfering cations has been masked by using suitable masking agents so as to make the process more selective. Distribution co-efficient values have been determined for various cations under experimental conditions which enable to predict affinity of various cations for the adsorption of stannic oxide.

Keywords: Adsorption, Two Component Adsorbent, Copper (II), Tetraethylenepentamine (TEPA).

1. INTRODUCTION

Adsorption is a process in which atoms or molecules move from a bulk phase onto a solid or liquid surface. At the molecular level, adsorption is due to attractive interactions between a surface and the species being adsorbed.

Stannic oxide is two component ion exchange material which is thermally and chemically stable¹⁻². It shows physisorption phenomenon towards many transition elements. Stannic oxide is regarded as quite stable, amorphous and hydrated to a variable extent³. Stannic oxide has been used for separation of metal ions, anions, pesticide⁴ and phenolic compounds previously. Microgram quantity of Cu(II) in the urine sample using diffused reflectance spectroscopy has been determined by adsorption on KU-2 cation exchanger⁵. A new method for determination of copper in liquid edible oils by flame atomic absorption spectrometry and spectrophotometrically using [N,N' Bis(salicylidene)-2,2' dimethyl-1,3-propanediaminato] has been developed⁶. In this investigation, the method has been developed for separation of Cu (II) by employing stannic oxide as an adsorbent and spectrophotometric determination by using TEPA as a color developing reagent⁷⁻⁸. The stable Cu (II)-TEPA complex obeys Beer-Lambert's law in the range 0 to 800µg and absorbs in visible range⁹.

The method does not require advance instrumentation, expensive chemicals and can be easily carried out within an hour.

2. EXPERIMENTAL

2.1 Chemicals and Reagents

All the chemicals used were of AR grade. For all dilutions double distilled deionized water was used. 10 mg/ml metal ion solution of Cu (II) was prepared by dissolving AR grade copper chloride (CuCl₂) salt and was standardized gravimetrically by the methods given by the Vogel¹⁰. All the other metal ion solutions (10mg/ml) were prepared by dissolving their appropriate salts in double distilled deionized water. The strength of the solutions was determined by the usual method, 2% TEPA solution was prepared by diluting 2 grams of TEPA to 100 cm³ by using double distilled deionized water.

3. PROCEDURE

The stannic oxide used in the present investigation was synthesized by the method of a co-precipitation 1.10 cm³ solution containing 50.0µg of Cu (II) whose pH adjusted at 5.0 was added to 250 mg of stannic oxide in a cone capacity of 25 cm³. The mixture was equilibrated, centrifuged using high-speed centrifuge machine and the supernatant liquid was collected in 25 cm³ standard measuring flask. Acetate buffer was used to maintain pH of the solution. Colour was developed by adding 1 cm³ of 2% TEPA reagent. Absorbance was measured on Equip-Tronics made UV-Visible digital spectrophotometer EQ-825 at 650nm against blank. Percentage adsorption was calculated by using the standard formula.

4. RESULTS AND DISCUSSION

4.1 The average crystallite size of the stannic oxide

The stannic oxide synthesized by the method of co-precipitation was subjected to XRD. (Fig. 1). The average crystallite size of the sample was calculated using Scherer equation and was found to be 24.196 nm.

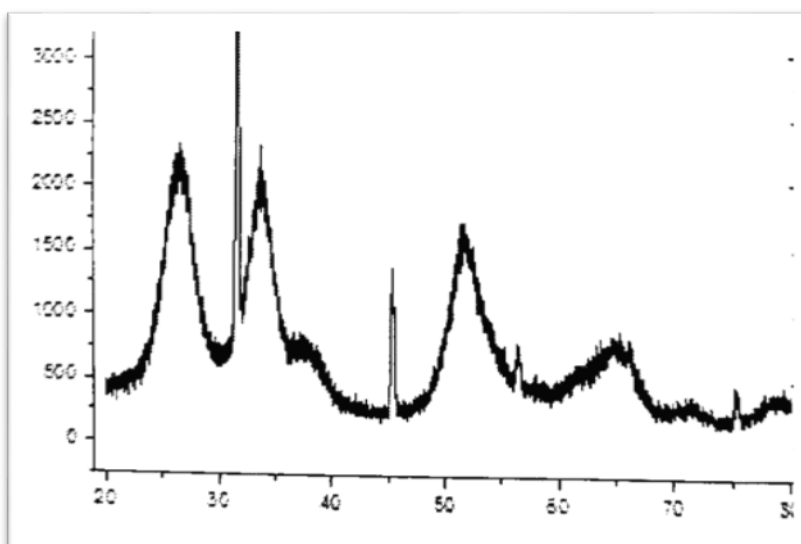


Fig. 1: XRD of Stannic oxide

4.2 Absorption spectrum of Cu(II)-TEPA complex

The absorption spectrum for Cu(II)-TEPA complex was obtained against double distilled deionized water as blank over the range of 400nm- 800nm. It is seen that Cu(II)-TEPA complex has its absorption maximum at 650 nm as shown in Fig. 2.

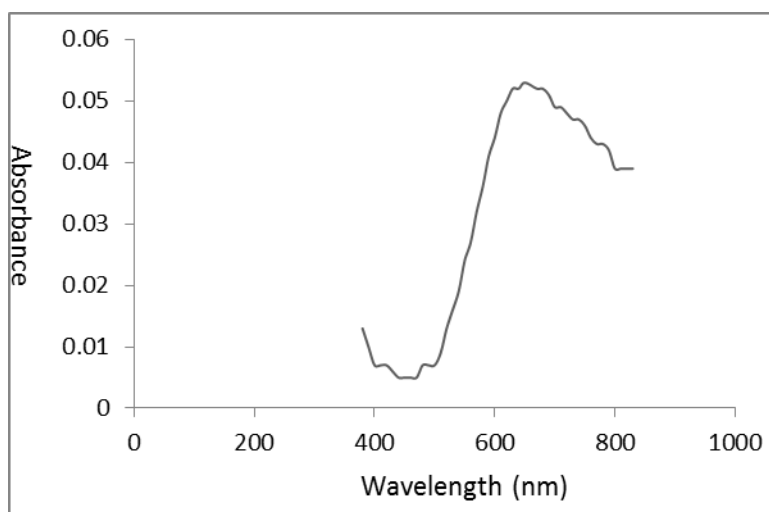


Fig. 2: Absorption Spectrum of Cu (II)-TEPA Complex

4.3 Effect of pH

The effect of pH in the adsorption of 50.0µg Cu (II) on 250 mg of stannic oxide was studied. It was observed that the adsorption of Cu (II) in the pH range was increased from 2.0 to 5.5 and remained constant after pH 5.0. From the Fig. 3, it is clear that the percentage adsorption of 50.0 µg of Cu (II) on 250 mg of stannic oxide is maximum at pH 5.0. Hence all further adsorption studies were carried at pH 5.0.

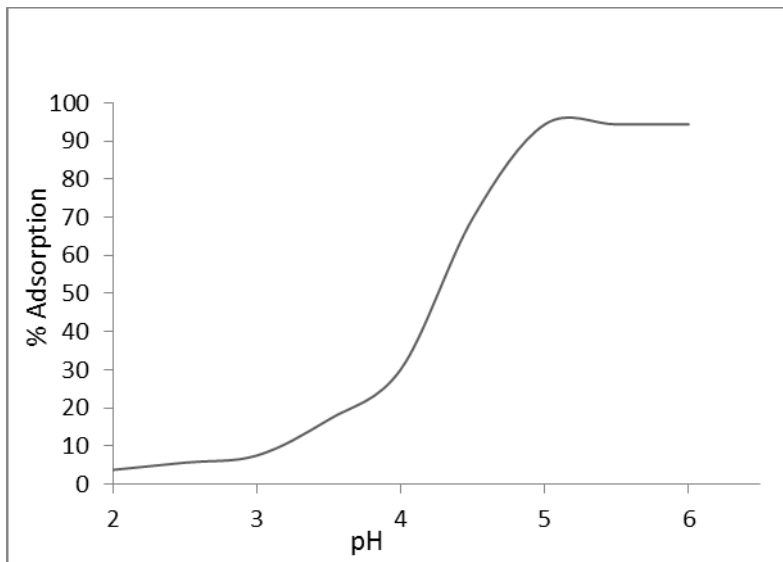


Fig.-3: Effect of pH

4.4 Effect of amount of adsorbent

The effect of the amount of stannic oxide on the adsorption of 50.0µg Cu (II) was studied at pH 5.0 as mentioned above. The amount of stannic oxide was varied from 50 mg to 250 mg. Fig.4 reveals that 250 mg of stannic oxide was adequate for the maximum adsorption of 50.0µg Cu (II) at pH 5.0.

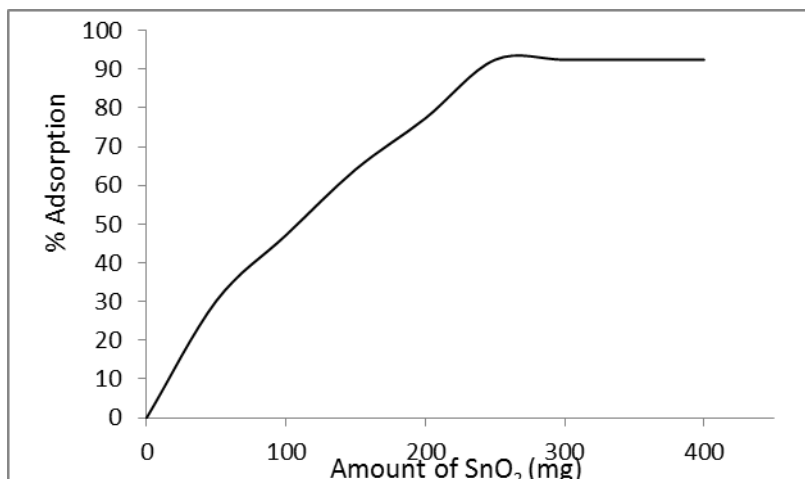


Fig. 4: Effect of Amount of Adsorbent

4.5 Effect of time of contact

The effect of time of contact in the adsorption of 50.0µg Cu (II) on 250 mg of stannic oxide was studied. From the fig.5, it is clear that 5.0 minutes are sufficient for maximum adsorption of 50.0µg of Cu (II) over 250 mg of stannic oxide at pH 5.0.

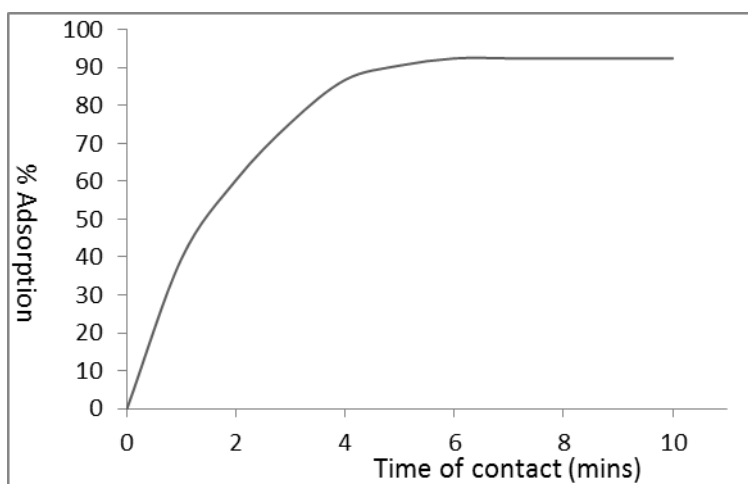


Fig. 5: Effect of Time of Contact

By studying various parameters in the adsorption of Cu (II) over stannic oxide, it is observed that 250 mg of stannic oxide is sufficient for maximum adsorption 50.0µg of Cu (II) at pH 5.0 and contact time of 5.0 minutes.

4.6 Reproducibility

Reproducibility of the method was evaluated by repeating the adsorption of Cu (II) at ideal conditions three times as mentioned above and was found to be $94.34 \pm 1.34\%$.

4.7 Interference of various anions

The effect of different anions in the percentage adsorption of Cu (II) was investigated by taking the anions as a salt of Na⁺, K⁺ or NH₄⁺. It was observed that 50.0mg each of chloride, bromide, iodide, fluoride, acetate, bromate, tartarate, perchlorate, borate and EDTA did not interfere in the adsorption of Cu(II). 10 mg of dichromate, thiocyanate, bromate, cyanide, nitrate, nitrite ions did not interfere with the adsorption of Cu(II). The interference of carbonate, oxalate, and sulphate was removed by decomposing them prior to adsorption.

4.8 Interference of various cations and Distribution co-efficient (Kd) value

The interference of various cations (50.0 µg each) in the adsorption of Cu(II) on stannic oxide under the optimum conditions was also studied. From Table-1, it is observed that Sr(II), Zr(IV), Cd(II), Ce(IV), Ba(II), Mg(II), K(I), Be(II), Na(I), Co(II), La(III) and Mo(VI) were adsorbed up to 10%. Al(III), Ca(II), Hg(II), V(III), In(III), Tl(III), Cr (II), Mn(II) and Ag(I) were adsorbed in the range of 11% to 25%. Al(III) and In(III) were adsorbed in the range of 26% to 50% ; whereas, Tl(I), Ca(II), Mn(II), Ag(I), Fe(II), Fe(III), Zn(II) and Ni(II) were adsorbed above 50%. Interference of interfering cations were masked by using suitable masking agent so as to make the method more selective.

Table-1: Percentage Adsorption of Various Cations

Cu(II) ion concentration	: 50.0 µg
Amount of stannic oxide	: 250 mg
pH	: 5.0
Time of contact	: 5.0 minutes
Temperature	: $28 \pm 2^\circ\text{C}$

% Adsorption	Cations
< 10 %	Sr(II), Zr(IV), Cd(II), Ce(IV), Ba(II), Mg(II), K(I), Be(II), Na(I), Co(II), La(III), Mo(VI), Al(III)*, Ca(II)*, Hg(II)*, V(III)*, In(III)*, Tl(III)*, Cr (II)*, Mn(II)*, Ag(I)*, Fe(II)*, Fe(III)*, Zn(II)*, Ni(II)*
11-25%	Al(III), Ca(II), Hg(II), V(III), In(III), Tl(III), Cr (II), Mn(II), Ag(I)
26-50%	Al(III), In(III)
>50 %	Tl(I), Ca(II), Mn(II), Ag(I), Fe(II), Fe(III), Zn(II), Ni(II)

*: Interfering cations were masked by using suitable masking agent.

The K_d values have been calculated and used to determine the affinity sequence of various cations towards the adsorbent under ideal conditions. K_d values were calculated by using standard formula and as are in Table-2.

Table-2: Distribution Values for Different Elements

Cu(II) ion concentration	: 50.0 µg
Amount of stannic oxide	: 250 mg
pH	: 5.0
Time of contact	: 5.0 minutes
Temperature	: $28 \pm 2^\circ\text{C}$

Metal Ion	Kd-Value	Metal Ion	Kd-Value
Sr(II)	0.81	Hg(I)	6.19
Ba(II)	1.23	Fe(II)*	6.38
Zr(IV)	1.66	Ag(II)*	6.52
Mg(II)	2.1	Ni(II)*	6.86
Co(II)	2.55	Tl(I)	11.28
Cd(II)	2.73	Al(III)	12.63
Mo(II)	3.01	In(III)	17.14
La(III)	3.47	V(III)	26.66
Ce(II)	3.66	Cr(VI)	34.07
Be(II)	3.85	Mn(II)	46.95
K(I)	3.95	Ag(II)	55.23
Na(I)	4.24	Fe(III)	62.56
Zn(II)*	4.48	Fe(II)	126.66
Ca(II)	4.93	Zn(II)	170.52
Mn(II)*	5.51	Ni(II)	245.71
Fe(III)*	5.75		

5. CONCLUSION

The stannic oxide synthesized is crystallite material having average crystallite size 24.196 nm. From the above discussions it is clear that for the adsorption of 50.0 µg of Cu(II), 250 mg of stannic oxide is sufficient for maximum adsorption at pH 5.0 when equilibrated for 5.0 minutes. The interference of various cations can be masked by using suitable masking agent so as to make the method more selective. The method developed for the adsorption and determination of Cu(II) has been applied for the determination of Cu(II) from various synthetic copper complexes and alloys. The method is simple, selective and gives reproducible results.

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