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Graphene Oxide Based Fluorescent Sensor For Tin Metal Ion

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Abstarct:

Graphene oxide is used as fluorescent probe for sensitive and selective detection of Sn^{+2} ion in aqueous medium (pH 4) based on controlled partial reduction approach. The formation of partially reduced graphene oxide under controlled partial reductions leads to the enhancement in fluorescence. Mechanism for partially reduced graphene oxide formation is further supported by Uv-visible spectroscopy, Thermogravimetric analysis (TGA). Graphene oxide shows selective ≈ 9 fold enhancement in fluorescence toward Sn^{+2} at concentration 40 $\mu g mL^{-1}$ over other metal ions such as Cd^{+2} , k^+ , Ca^{+2} , Fe^{+2} , Fe^{+3} , Hg^{+2} and Zn^{+2} with same concentration. The straight line calibration plot of enhancement in fluorescence of graphene oxide verses increasing amount of Sn^{+2} solution in the range of 4-40 $\mu g mL^{-1}$ is obtained with correlation coefficient 0.987. Further fluorimetric method is developed for detection of Sn^{+2} ion from environmental water sample with detection limit of detection (LOD) 4.7 $\mu g mL^{-1}$

Introduction:

Graphene oxide (GO) which is oxidized form of graphene is a two-dimensional, one atom thick carbon nanomaterial with exciting mechanical, electronic and thermal properties, which has wide applications in the field of biology, electronics and optics. Presence of oxygen containing functional groups, such as carboxylic acid, phenol hydroxyl and epoxide groups mainly at the basal plane or at edges, of GO results in the formation of mixture of sp² and sp³ hybridized carbon that open the optical band gap and exhibits fluorescence.

Tin is soft, silvery white a type of heavy metal. It combines with other chemicals to form water soluble compounds and enter as pollutant in natural water.⁵² Tin can enter in to the human body through canned foods and soft drinks.⁵³ At higher concentration Sn^{+2} can be readily taken up by human body causes damage to DNA, human embryonic kidney cell and diarrhea.^{52,54} GO shows selective enhancement in fluorescence towards Sn^{+2} ion, among other metal ions like Pb⁺², Cd⁺², K⁺, Hg⁺², Ca⁺², Fe⁺² and Mg⁺²..

EXPERIMENTAL SECTION:

Materials:

Graphite powder 100 micron (Sd fine, Mumbai), Potassium permanganate (Thomas Baker, Mumbai), Orthophosphoric acid, Buffer capsule pH 4 (Molychem, Mumbai) were used as received. Ultrapure water was produced by passing distilled water through a Millipore Unit (India) and was used in further experiments. Pb, Cdcl2, kcl, Mgcl2, cacl2, Feso4, Hgcl2 and Sncl2.

Preparation of Graphene oxide

Graphene oxide (GO) is synthesized by using improved method⁵⁵ with slight modifications. In typical experiment 1g of graphite powder and 6g of KMnO₄ was added in mixture of Cocn.H₂SO₄ and H3PO4. The reaction then heated at 55^oC for 12 h. The reaction mixture was cooled at room temperature, then poured into the mixture of ice (100ml) and 2 ml H₂O₂ (30%). The color of mixture is changed to bright yellow. The product is separated by centrifugation (3000 rpm for 1h), the supernatant layer was decanted. The product is mixed with double distilled water and shaked, then

this mixture is again centrifuged (3000 rpm for 1h) and supernatant layer is discarded again. Process washing and centrifugation is repeated for 3 more cycles. Finally the product is filtered through whatman filter paper, washed with 5% HCL and double distilled water. Final solid product is dried to room temperature and used for experimental work.

Preparation of GO solution and prGO1.

The produced GO of weight 0.005gm is added in 50 ml double distilled water of pH 4, the solution is sonicated for 1h to make dispersion of constant composition (0.01% (w/v)). This dispersion is further used as GO solution. The GO shows partial reduction in presence of Sn^{+2} among other metal ions. The addition of 4-40 µg/ml of sn^{+2} in GO solution shows increase in degree of reduction of GO which can be seen by naked eye. To support the mechanism of reduction, the partially reduced graphene oxide formed in presence of $20\mu g/ml Sn^{+2}$ is separated by using fridge drying technique and it is called as prGO throughout the discussion.

RESULTS AND DISCUSSION

UV-vis absorption spectra of pure GO and GO in Presence of Sn^{+2} metal ion solution in different concentration are shown in fig.1. Pure GO shows two absorption peaks 225 nm and 303 nm arises from the π - π * and n- π * transition respectively. The spectra changes upon addition of Sn⁺² metal ion solution.





To study thermal stability of GO and prGO thermogravimetric analysis (TGA) technique was employed as shown in Figure 2b, c. In the GO sample 78% weight loss was seen up to 200 ^oC, it is due to loss of oxygen containing functional group by pyrolysis and removal of steam.^{33,34} In contrast the prGO shows much increased thermal stability due to removal of the thermally labile oxygen containing functional group.³⁴ prGO sample shows 11% weight loss up 200 ^oC, this minor mass loss is attributed to the absence or removal of the oxygen containing functional groups due to partial reduction, further total 44% weight loss up to 700 ^oC was observed.



Fig. 2: TGA plot of GO and TGA plot of prGO

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Now to see the effect of metal ions on the optical property of GO, fluorescence spectra of aqueous dispersion of GO (0.002w/v %) is recorded at pH 4 in presence of different cations such as Sn⁺², Cd⁺², k⁺, Ca⁺², Fe⁺², Mg⁺², Hg⁺² and Zn⁺² each of 20µg/mL concentration. As shown in Figure 3a. and fluorescence was of GO is measured with increasing concentration of Sn⁺² from 20- 40 µg/ml at pH 4 and excitation wavelength is 320 nm.



Fig. 3: Fluorescence spectra of aqueous dispersion of GO (0.002 w/v %) shows selective enhancement towards Sn^{+2} than other ions such as Cd^{+2} , k^+ , Ca^{+2} , Fe^{+2} , Fe^{+3} , Hg^{+2} and Zn^{+2} each of 20µg/mL concentration. ($\lambda_{ex} = 320$ nm) & Fluorescence spectra of aqueous dispersion of GO in presence of different concentration of Sn^{+2} ion (A: 0 µg/ml to K: 40 µg/ml) at pH 4 and excitation wavelength is 320 nm.



Conlusion :

GO in acidic media (pH 4) used as fluorescent probe which shows partial reduction with addition of increasing amount of Sn^{+2} solution in the range of 4-40 µg mL⁻¹ the calibration plot obtained with correlation coefficient 0.987. Further this fluorimetric method is can developed for detection of Sn^{+2} ion from environmental water sample with detection limit of (LOD) 4.7 µg mL⁻¹.

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