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Green Synthesis and Characterization of Sodium Banana Peel Xanthate Carbon Dot (SBPX C-Dot) and Preparation and Utility of Carbon Composite Paste Electrode for Selective Potentiometric Sensing of Hg (II) Ions

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Keywords: sodium banana peel xanthate C-Dot (SBPX C-Dot), carbon composite past electrode (CCPE), potentiometric sensor.

ABSTRACT. A green approach has been used for the synthesis of fluorescent sodium banana peel xanthate Carbon Dot (SBPX C-dot) with the use of yellow banana peels as carbon source. The Carbon Dot were synthesized by hydrothermal treatments. pigments and other low molecular weight hydrocarbons, most of the above materials on reaction with carbon disulphide Banana peel is mostly composed of cellulose (8.4 nmol L⁻¹), pectin (10-21%), hemicellulose (6.4-9.4 %), lignin (6-12%). Banana peel was first treated with 10% NaOH for a day to hydrolyze or digest or disintegrate into the low molecular weight and bigger molecular weight compounds, with a lot of hydroxyl functional groups, which make the Banana peel a potential substrate for the synthesis of Banana Peel Xanthate C-Dot (SBPX C-Dot). The Banana Peel Xanthate C-Dot (SBPX C-Dot) are analyzed and identified by FT-IR, Raman Spectroscopy and UV-Visible spectroscopy. Effect of pH on UV-Visible and Fluorescence Spectroscopy were carried out in the pH range (pH=1-10). Field Emission Scanning Electron Microscopy was used to study the surface morphology. The utility of (SBPX C-Dot) with Carbon Paste to form a Composite Electrode (CCPE) for potentiometric sensing of Hg (II) ion was accomplished in aqueous acetate and chloride medium at different pHs (pH=1-10). Reasonable and selective sensing was observed with CCPE for Hg (II) ion is observed.

Introduction. Xanthates are one of the important organosulphur compounds used in mining and rubber industry. They are the derivative of xanthic acid. They are also known as xanthogenates, carbon dithioates and salts of xanthic (dithiocarbonic) acids, These organosulfur compounds are important in two areas, the production of cellophane and related polymers from cellulose and secondly in mining for the extraction of certain ores. Different xanthates have different strengths. The interaction of typical collector xanthate with pyrite, the most abundant sulfide minerals is electrochemical in nature [1]. The strength of xanthate as a collector is based on the alcohol chain attached to the xanthate molecule with ethyl being the weakest and amyl being the strongest. They are also versatile intermediates in organic synthesis. They also have wide ranging properties such as optical, electrical and magnetic characteristics. Their thin films show different properties such as an antibacterial agent, magnetic and semiconductor material, which allowed them to be used for data storage, solar cell production and water purification [2]. The xanthates are widely used as chelating reagents in analytical chemistry. Although many analytical methods such as titrimetric, polarography and photometry are available for the determination of xanthate, the hydroxyl group was chemically modified by introducing sulfur groups with the carbon disulfide treatment in alkaline medium. The carbon disulphide is a type of compound called hetero-allene, which by its symmetric nature and possession of more bonding character, is a good complexing agent [3].

EXPERIMENTAL METHOD

Preparation of Sodium Banana Peel Xanthate. The banana peel collected from the market was cut into small pieces stoichiometric amount and was treated (10% NaOH) solution to hydrolyze banana peel was used as a base for further reaction as such to prepare banana peel xanthate carbon disulfide (0.76 g) was slowly drop by drop added to the above cooled solution. The reaction mixture was stirred for 12 hrs and was used as such for further work.

Synthesis of Sodium Banana Peel Xanthate C-Dot. The sodium banana peel xanthate [4] obtained above was transferred to an autoclave and kept in a muffle furnace at 180°C [hydrothermal condition] for 12 hrs to prepare banana peel xanthate carbon dot and the resultant banana peel xanthate carbon dot was collected and stored in an airtight container. The sodium banana peel xanthate C-dot as prepared was found to be aqueous soluble and fluorescent nature.

Preparation of Composite Carbon Paste Electrode (CCPE). The composite carbon paste electrodes was prepared by mixing the xanthate C-Dot as electro active material with a binder (paraffin wax) and the blend was mixed until a homogenous paste besides graphite powder [5]. The electrode was prepared as a paste in the ratio of xanthate C-Dot: paraffin wax and graphite (5:1:1-w:w:w). The composite paste was heated in a water bath and the composite was then packed in a disposable glass tube (3 mm i.d.) and a mild pressure was applied and then the electrode was removed from the tube by manual means. Electrical contact to the composite carbon paste electrode was made with a crocodile clip copper wire. The electrode surface was polished every time for a fresh surface using an emery paper of 600 grit.

RESULT AND DISCUSSION

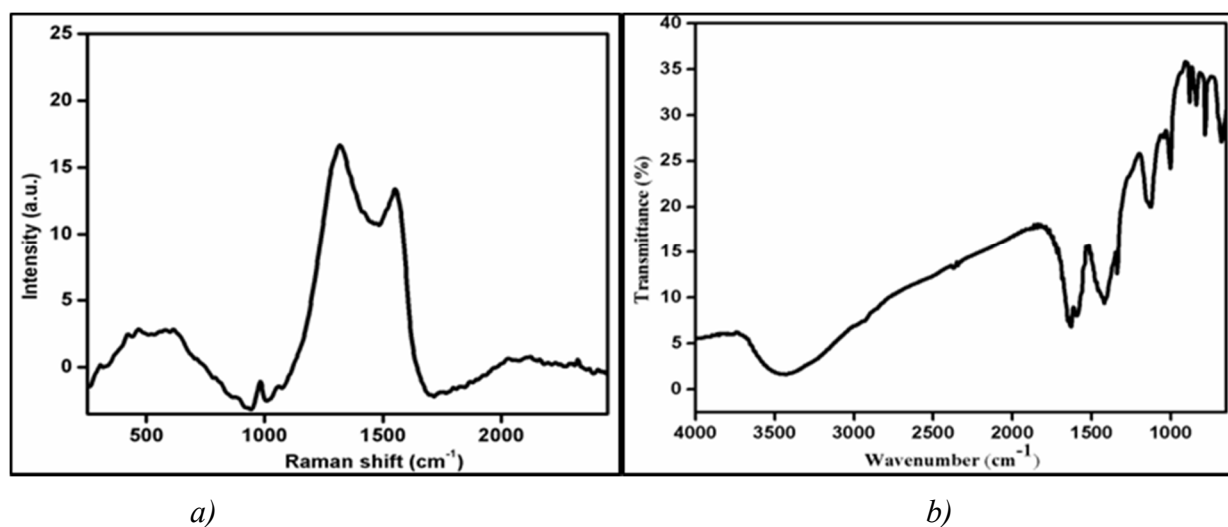


Fig. 1. FT-IR & Raman Spectroscopy of Sodium banana peel xanthate C-Dot.

The FT-IR spectrum for the Sodium Banana Peel Xanthate C-Dot was shown in the Fig. 1, a which indicates the appearance of the various absorption bands and the peaks at 3441 cm⁻¹ was very broad and strong and can be assigned to the Stretching vibration of hydroxyl (-OH) group either from water or from adsorbed moisture or both. A prominent and very sharp peak observed at 1625 cm⁻¹ which was concluded to be due to the Stretching vibrations of C-O-C group, the other bands to 1409 and 1012 cm⁻¹ allotted respectively to the Stretching vibrations of connections C-O-C-S and (C=S). The peak at 677 cm⁻¹ is due to the Stretching vibration of C-S the infra red spectrum. Hence these FT-IR peak confirmed the presence of Sodium Banana Peel Xanthate C-Dot peaks.

The Raman spectra of Sodium Banana Peel Xanthate C-Dot are shown in the Fig. 1, b. It was assigned that the bands by analogy to the data as follows: 977 cm⁻¹ for Stretching vibration of V (C=S), 1057 cm⁻¹ for Stretching vibration of Vs (COC single bond), 1316 cm⁻¹ for Stretching vibration of Vas

(SCS single bond) and 1559 cm^{-1} for Wagging vibration of (CH_3), and 2714 cm^{-1} for Stretching vibration of (CH_2). The functional group conformation of Raman spectrum of the bands which leads to the assumption that the Sodium Banana Peel Xanthate C-Dot is the main / major products present in the Sodium Banana Peel Xanthate C-Dots.

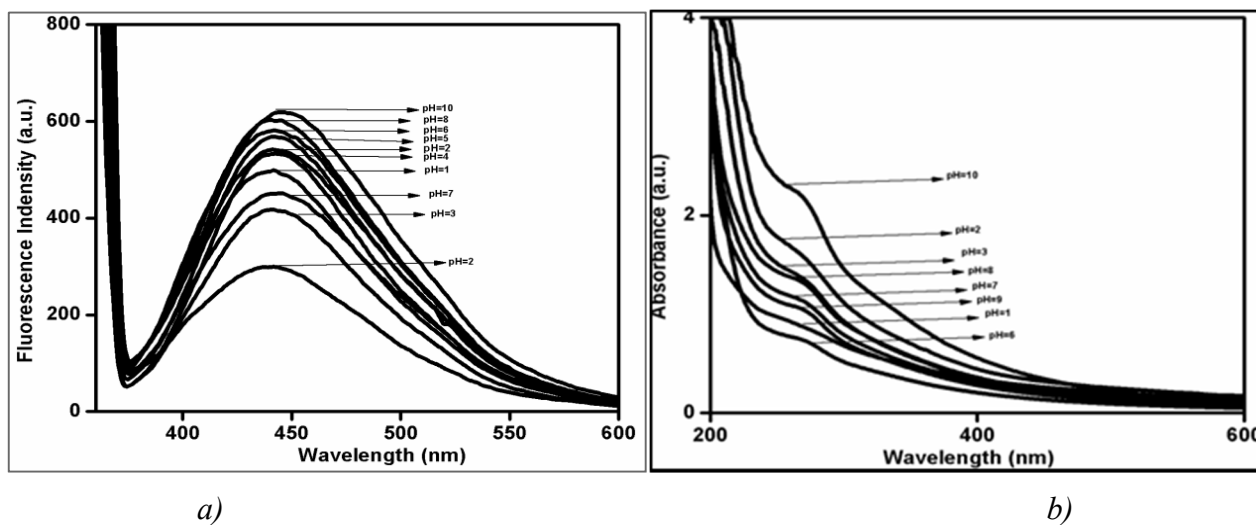


Fig. 2. UV-Vis & PL Spectroscopy of Sodium banana peel xanthate C-Dot.

The Ultra Violet-Visible (UV-Vis) spectroscopy of sodium banana peel xanthate C-Dot in Effect of pH solutions (pH=1-10) shows the Fig. 2, a absorption peak increases and decreases in effect of pH solutions. Shows the absorption peak at 226 nm. This shows the presence of $\pi -\pi^*$ transition. This transition occurs from the C=S bond, this bond is a part of the xanthate. The Ultra Violet-Visible (UV-Vis) spectroscopy of sodium banana peel xanthate C-Dots is concentration 10mg/10ml (pH=1-10) shows peak in ultraviolet region in aqua’s solvents used in soluble of sodium banana peel xanthate C-Dot.

The photoluminescence spectroscopy of sodium banana peel xanthate C-Dotin solution, are strongly affected by changes in pH Shows the Fig. 5 with the spectra recorded at low pH being weaker than those at high pH. Effect of pH solutions (pH=1-10) shows the intensity peak increases and decreases in effect of pH solutionsn intensity peak at 447 nm. This shows the presence of $\pi -\pi^*$ transition. Inwhich transition occurs from the C=S bond, The photoluminescence spectroscopy of sodium banana peel xanthate C-Dot is the concentration 10mg/10ml (pH=1-10) in aqua’s solvents used in soluble of sodium banana peel xanthate C-Dot.

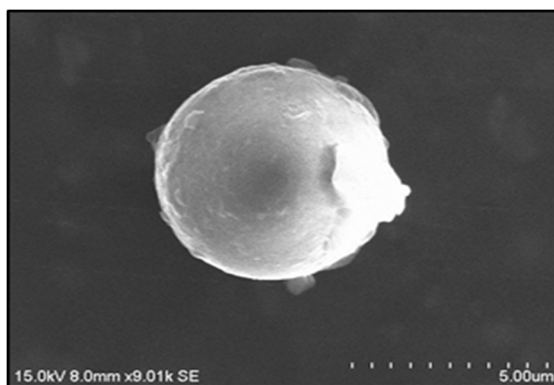


Fig. 5. FESEM & EDX micrascopy image of Sodium banana peel xanthate C-Dots

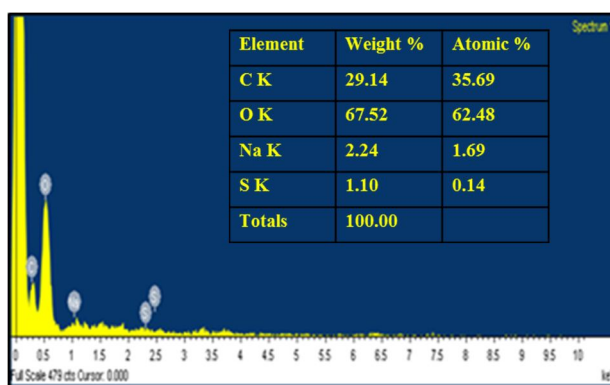


Fig. 6. FESEM & EDX microscopy image of Sodium banana peel xanthate C-Dots.

The surface morphology and size of the sodium banana peel xanthate C-Dot shows the Fig. 5 were observed by the Field Emission Scanning Electron Microscopy (FESEM) analysis. The sodium banana peel xanthate C-Dot naturally presents in the surfactant in the banana peel. FESEM morphology of the image of sodium banana peel xanthate C-Dot which is clear spherical in shape. The partial size of sodium banana peel xanthate C-Dot is 135 nm. shows the Fig. 6 EDX was also performed on the sodium banana peel xanthate C-Dot. the elements for C, O, S and Na were observed.

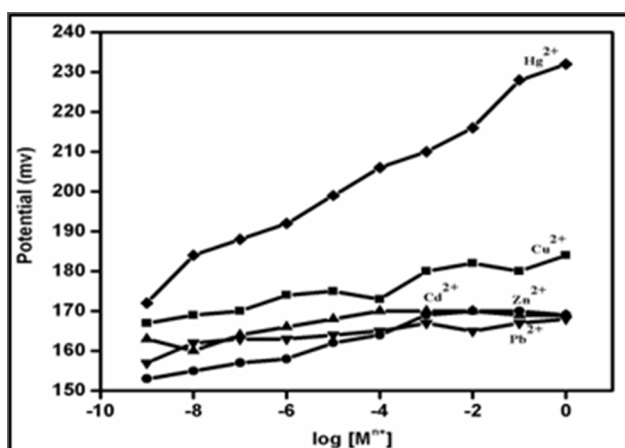


Fig. 7. Potentiometric Sensing of Sodium banana peel xanthate C-Dot in different metal ions solutions.

The sensitivity of a potentiometric CCPE depends on the carbon composite paste electrode. The influence of C-Dot in the composite carbon paste was studied. The working electrode is (CCPE) and reference electrode (SCE) were dipped in 10 ml of electrolyte solution (0.0001 M KCl) added and 5ml of DD water taken into beaker.

The metal ion influence on the potentiometric responses were tested with for different metal ions at different pHs. The potentiometric behavior was noted and individual behavior was noted in a graph. Out of the metal ions tested (Cu^{2+} , Cd^{2+} , Hg^{2+} , Zn^{2+} , Pb^{2+}) Hg^{2+} show a very promising results and was shown in the Fig. 7 The Fig. exhibits good response and linearity. This sensor revealed a great enhancement in selectivity for mercury ions in comparison with the previously reported mercury sensors [6]. The developed electrode exhibits higher selectivity for mercury ions compared with other metal ions it was successfully used as an indicator electrode (CCPE) in potentiometric sensor of Hg^{2+} against. It is obvious that the slope ($R^2= 0.9948$) of the linear part is near to the expected according to the Nernst equation. However, the electrode does not exhibit Nernstian responses for other cations

and even the potential responses have no significant change with the increase of the concentration of Hg^{2+} (0.0001 M) sodium banana peel xanthate C-Dot was selected as the carrier for the construction of the mercury ion selective electrode. It is well known that selectivity, sensitivity and linear range of the ion selective electrode are influenced strongly by the carrier composite electrode. Thus, it is indispensable to investigate the influence of carrier amounts on the potential responses of the developed CCPE. The experiment was repeated for concordant potential values.

The potentiometric Sensing of SBPX C-Dot with mercury chloride & mercury acetate solution carried out electrochemical reaction by taking the 10 ml of buffer solution added and 5 ml of DD water taken into beaker. Then indicator electrodes (CCPE) and reference electrode (calomel electrode) are immersed in that beaker solution. Electrodes are connected to potentiometer and sensing against 0.0001M of mercury Chloride (HgCl_2) & 0.0001M of mercury acetate ($\text{Hg}(\text{CH}_3\text{COO})_2$) solution in the pH range 1.0-10. Electrode (Fig. 8 (a, b) the effect of pH on the response of the electrode was respectively investigated in the two Hg^{2+} solutions (namely HgCl_2 and $\text{Hg}(\text{CH}_3\text{COO})_2$) solutions and different pHs. As shown in the Fig. 8 (a) (HgCl_2), the potential does not change apparently at pH range 1.0, 3.0, 4.0, 6.0 and 10, which can be used as the working pH range of the proposed electrode. PH 8.0 and 9.0 is potential slightly change. In addition, pH 2.0, 5.0 and 7.0 is potential is good compare with other PH solutions. The show in the Fig. 8 (b) ($\text{Hg}(\text{CH}_3\text{COO})_2$) shows that the potential does not change apparently at pH range 1.0, 2.0, 4.0 and 9.0 which can be used as the working pH range of the proposed electrode. PH 3.0, 5.0 and 10 is potential slightly change. Moreover, pH 6.0, 7.0 and 8.0 is potential is good compare with other PH solutions. While, outside this range, the potential changed significantly. The potential was increases and decreases in effect of pH solutions. SBPX C-Dot potential noted.

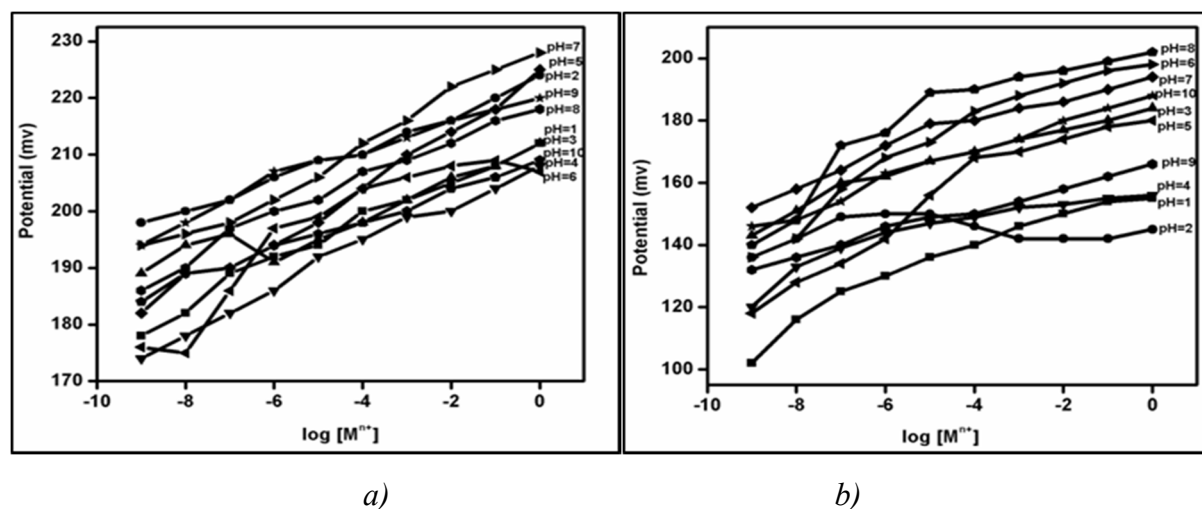


Fig. 8. Potentiometric Sensing of Sodium Banana Peel Xanthate C-Dot at Effect of pH (pH=1-10) solutions (a). HgCl_2 , (b). $\text{Hg}(\text{CH}_3\text{COO})_2$

Summary. Sodium banana peel xanthate C-Dot (SBPX C-Dot) were synthesized using hydrothermal method and characterize by UV-Visible and Raman, FT-IR studies. The Sodium banana peels xanthate C-Dot as florescent nature and study with photo luminance spectroscopy (PL). The C-Dot materials newly prepared by Composite Carbon paste electrode. Using the potentiometric metal ion sensing were d^{10} and d^9 metals to know their behavior in presence of buffer. The xanthate C-Dot e.m.f of the cell is the difference in the potentials (voltages) of cathode and anode contact with suitable electrolytes at the electrode. The potential depend on the concentration of the electrolyte and chemical of the electrode. different metal ions added in the (Cd^{2+} , Cu^{2+} , Zn^{2+} , Pb^{2+}) is not behavior and only metal of (Hg^{2+}) is very good sensing the CCPE can be employed with successful results for the potentiometric sensors.

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