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Trace determination of cadmium in water using anodic stripping voltammetry at a carbon paste electrode modified with coconut shell powder

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Abstract

Background: Increasing awareness on the environmental impact of heavy metals has increased a considerable interest in the determination of metals in natural water bodies. The present paper describes the development and electrochemical application of carbon paste electrode modified with fibrous part of coconut shell for the determination of cadmium in water samples.

Methods: Determination was carried out using anodic stripping voltammetry. It is a two-step process. First, the metal ions get accumulated at the electrode surface at open-circuit potential, followed by a potential scan for voltammetric determination of cadmium.

Results: Different parameters affecting the determination of Cd (II) were optimized and are as follows: HCl as stripping solvent, acetate buffer of pH 5 as accumulating solvent, and 15-min accumulation time. Triton X-100, cetyltrimethylammonium bromide, and sodium dodecyl sulfate were used as representative for neutral, cationic, and anionic surfactants, respectively, to see the effect of surface active macromolecules. Interference caused by other metal ions on the determination of cadmium was also studied.

Conclusions: The method shows the development of a sensor for the sensitive determination of cadmium with limit of detection at 105 μg L^{-1}. This technique does not use mercury and, therefore, has a positive environmental benefit.

Keywords: Plant-modified carbon paste electrode; *Cocos nucifera*; Cadmium; Stripping voltammetry

Background

Cadmium is classified as one of the priority pollutants which entered water streams through various industrial operations (Pan et al. 2012). It is ranked seventh by the Environmental Protection Agency in 'Top hazardous substances priority list'. Cadmium can easily be dissolved and transported by water (Li et al. 2009a, b). However, due to anthropogenic activities, its content can be elevated at the site of the action. High concentrations of cadmium ions can injure human health and pollute the environment. It is carcinogenic to human by damaging human immune and central nervous systems and causes diseases such as renal dysfunction and liver damage. Hence, the identification of cadmium-polluted sites is needed by society (Eshaghi et al. 2011).

Different analytical methods for the determination of Cd (II) ions have been reviewed several times (Sneddon and Vincent 2008; Ferreira et al. 2007; Pyrzynska 2007; Davis et al. 2006). Among them is stripping voltammetric determination of cadmium using mercury-based electrodes which is one of the very sensitive analytical methods available. But, due to different issues related to its harmful effects and disposal, it is strongly recommended to replace mercury with another electrode material. Recently, modified carbon paste electrodes can be a better substitute of mercury-based electrodes due to its simplicity of preparation, the versatility of chemical modification, rapid renewal of the electrode surface, and sensitivity equivalent to that of mercury-based electrodes (Roa et al. 2003; Sar et al. 2008; Heitzmann et al. 2005; Lu et al. 2011; Li et al. 2009a, b; Bagheri et al. 2012). Thus, modified carbon paste electrodes (MCPEs) and related sensors using different types of modifiers (chemicals, enzymes, and extracts) have been developed (Chow and Gooding 2006; Heitzmann et al. 2005; Ensafi et al. 2010; Portaccio et al. 2010).

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In comparison to the conventionally used MCPEs, plant-modified carbon paste electrodes represent a green approach in the environmental perspectives. The use of plant agricultural wastes as a modifier in carbon paste electrodes is due to the high metal hyper-accumulating properties in certain plants (Rajawat et al. 2013b; Mojica et al. 2006, 2007). They possess an electrochemically or chemically active moiety. These moieties could be any of the following: redox or ligand sites and ion-exchange sites, which possess certain functionalities or donor groups (Rajawat and Satsangee 2011).

Coconut shell has been widely used as an agricultural waste material for the sorption of Cd (II) from aqueous solution (Pino et al. 2006; Okafor et al. 2012). In continuation of our previous research work on modified carbon paste electrodes (Rajawat et al. 2012, 2013a) and keeping the above views in mind, the powder of coconut shell (*Cocos nucifera*) was used to modify the carbon paste electrode with the main goal of using it as a modifier material for the development of a sensor for the determination of cadmium.

Methods

Chemicals and reagents

For DPASV study, first, accumulation was done under open-circuit potential by placing the electrode in a metal solution with stirring for a certain time, rinsed with deionized distilled water followed by medium exchange for stripping analysis.

All chemicals were of analytical reagent grade. A 1,000-ppm stock solution of Cd (II) was prepared by dissolving an appropriate amount of cadmium nitrate (Merck & Co., Inc., Whitehouse Station, NJ, USA). The working solution was prepared daily by the dilutions from the stock solution. Graphite powder (<20 μm) and mineral oil Nujol (light, density 0.838) were obtained from Aldrich (Wyoming, IL, USA). Triply distilled water (ELGA, Millipore Co., Billerica, MA, USA) was used throughout the experiment.

Procedure

All quantitative measurements were carried out in anodic stripping voltammetry using differential pulse (DP) to achieve the sensitivity required for trace analysis. Each DPASV run was made up of two steps: accumulation under open circuit where the modified electrode is immersed in metal solution for a certain time. The electrode was then rinsed with deionized distilled water, followed by medium ex-change for stripping analysis. All measurements were carried out at room temperature (24 ± 1°C). Finally, the calibration curves were plotted and the influence of various substances as potential interference compounds on the determination of Cd(II) ions was studied under the optimum conditions.

Preparation of coconut shell powder-modified carbon paste electrode

Coconut was purchased from the local market of Agra. The coconut shell between the outer layer and inner layer (i.e., mesocarp) was separated. It was properly washed with water and dried in an oven at 50°C. The dried material was grounded and passed through the sieve. Fraction of the particles with size less than 150 μm was selected for electrode preparation. Unmodified carbon paste electrode was prepared by mixing the graphite powder with the mineral oil (80:20 *w*/*w* ratio) using mortar and pestle. MCPEs of different proportions (5%, 10%, 15%, 20%, 25%, and 30% *w*/*w*) were prepared by substituting the corresponding amount of graphite powder with coconut shell powder. The mixture is thoroughly hand-mixed in a mortar and pestle. The paste was pressed in a glass tube with an inner diameter of 3 mm and a depth of 4 cm to form a target electrode surface. A copper wire was inserted from the backside for electrical contact. A smooth and fresh electrode surface was obtained by pushing the electrode material from the backside, removing a small amount of paste from the electrode tip, and polishing the electrode surface on a photo paper.

Results and discussion

Characterization of the coconut shell powder-modified carbon paste electrode

Morphological characterization

Scanning electron microscope images of (a) unmodified and (b) coconut shell powder-modified carbon paste electrodes are shown in Figure 1. The comparison of the scanning electron microscope (SEM) images shows a difference in the morphology which was observed after the modification with coconut shell powder. The unmodified CPE surface is homogenous and fine pores are uniformly distributed, whereas after the modification, the pore size was increased and the distribution of the pores on the surface become uneven.

Increase in the roughness value for coconut shell powder-modified CPE (CS-MCPE) was justified with the atomic force microscope (AFM) results. 3-D atomic force microscope images of unmodified CPE (a) and CS-MCPE (b) are shown in Figure 2. Increase in the average surface roughness of the prepared electrode was observed after modification with coconut shell powder from 84.55 to 183.96 nm, respectively, for unmodified and modified CPE. It justifies the results obtained from SEM.

Electrochemical characterization

All the electrochemical experiments were performed using a μAutolab Type III potentiostat (Eco Chemie, Utrecht, Netherlands) controlled by a PC using the NOVA 1.8 software. A three-electrode system containing the modified carbon paste electrode as working, an Ag/AgCl (3.0 mol L^{-1} KCl) as reference, and a platinum wire as auxiliary electrodes was used.

Electrochemical characterization of the prepared electrodes was carried out using cyclic voltammetry. Cyclic voltammetry study for the CS-MCPE in $K_3[Fe(CN)_6]$ solution with 0.1 M KCl as supporting electrolyte was

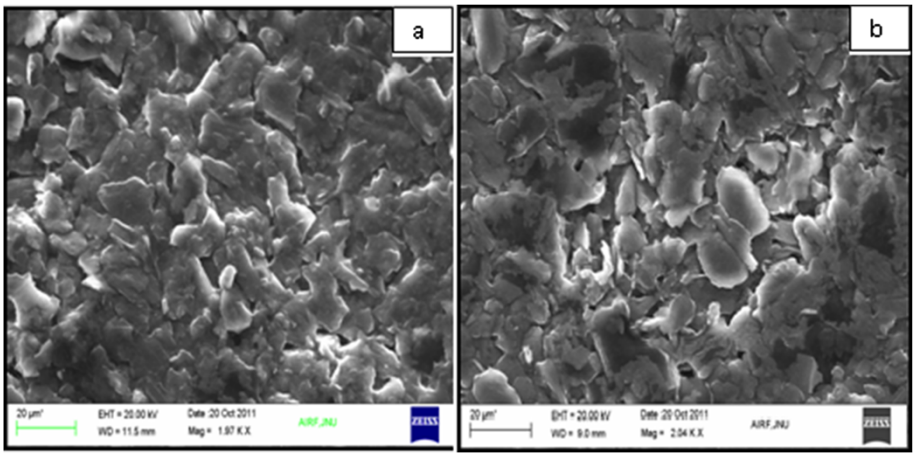

Figure 1 SEM images of the carbon paste electrode. (a) Unmodified carbon paste electrode and **(b)** coconut shell powder-modified carbon paste electrode.

carried out to determine the effect of modification on charge transfer activity, and the results are given in Figure 3. Well-defined cyclic voltammogram (CV) was observed for the unmodified CPE (Figure 3, curve a). With modified CPE, the redox peak currents decreased, and the peak potential difference increased remarkably (Figure 3, curve b). Because the coconut shell powder acts as an inert electron and a mass transfer blocking layer, it hinders the diffusion of ferricyanide toward the electrode surface. Based on the CV results, we can conclude that the coconut shell powder was successfully embedded on the surface of the modified electrode.

Electrochemical impedance study

The FRA module with the same instrumental setup was used for the electrochemical impedance study (EIS). For EIS, the AC amplitude of 10 mV at a frequency of 1 to 100,000 Hz was applied. Figure 4 shows the Nyquist plots of the electrochemical impedance study for the unmodified (Figure 4 curve a) and modified (Figure 4 curve b) CPE in 10 mM $K_3[Fe(CN)_6]$ solution. The results of the EIS studies were analyzed by fitting the appropriate equivalent circuit. The circuit which most fitted to the unmodified CPE is given in Figure 4b. Here, R_s is the solution resistance, Q is a constant phase element, and R_p is charge transfer resistance. The same circuit was used to analyze the EIS results of the CS-MCPE. The charge transfer resistance value observed for CS-MCPE is high in comparison to the unmodified CPE. The electrochemical impedance spectroscopy results are in accordance with the cyclic voltammetry results.

Electrochemical studies for metal determination

The ability of the CS-MCPE for the determination of Cd (II) was evaluated by the accumulation of Cd (II) at open-circuit potential and its electrochemical stripping in electrolyte solution using cyclic voltammetry. The CV curves recorded in 0.1 M hydrochloric acid did not show any peak without accumulation (Figure 5 curve a), whereas after accumulation for 10 min, well-defined oxidation peak appears (Figure 5 curve b), corresponding to the

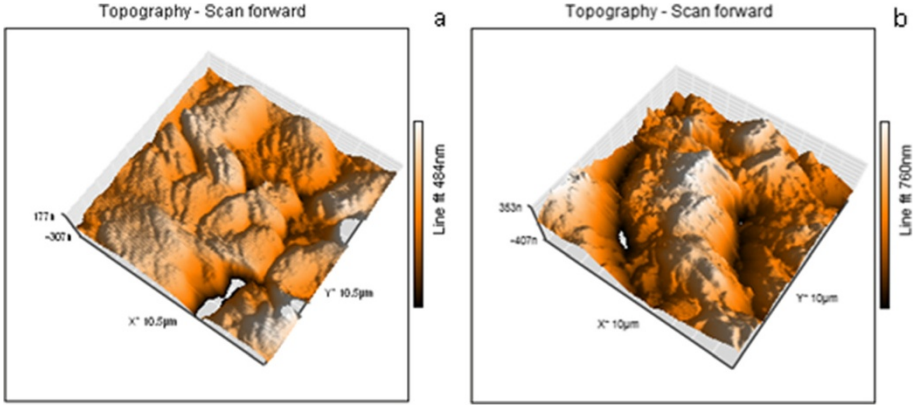

Figure 2 AFM images of the carbon paste electrode. (a) Unmodified carbon paste electrode and **(b)** coconut shell powder-modified carbon paste electrode.

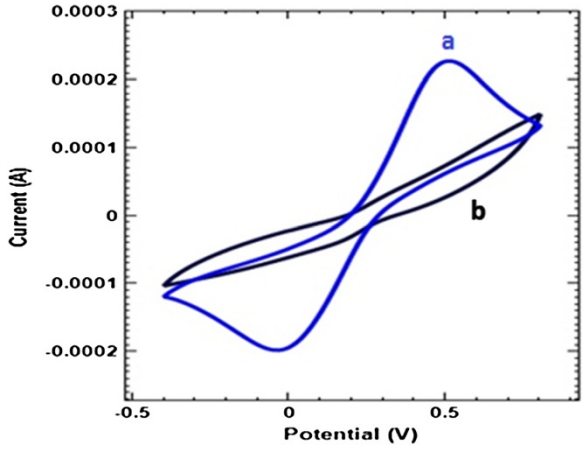

Figure 3 Cyclic voltammograms of the carbon paste electrode. **(a)** Unmodified CPE and **(b)** coconut shell powder-modified carbon paste electrode in potassium ferricyanide solution with 0.1 M KCl at the scan rate of 100 mV/S.

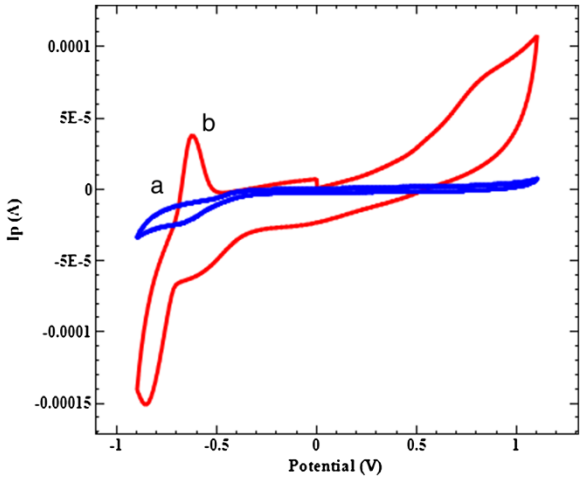

Figure 5 Cyclic voltammograms of coconut shell powder-modified carbon paste electrode in 0.1 M HCl. **(a)** Without accumulation and **(b)** after 10-min accumulation of Cd (II) at a scan rate of 50 mV/S.

oxidation of the accumulated cadmium which proves that cadmium metal gets accumulated at the electrode surface.

Based on the cyclic voltammetric results, differential pulse anodic stripping voltammetry (DPASV) was used. The DPASV result show a well-defined and symmetric anodic stripping peak with peak potential at −713 mV after the accumulation (Figure 6 curve b), whereas a flat curve was attained without accumulation (Figure 6 curve a). So, the peak was because of the oxidation of Cd (II) in the stripping step. Therefore, differential pulse anodic stripping voltammetry was employed in the present work for the determination of cadmium. All measurements were carried out at room temperature (24°C ± 1°C). During the experiment, the electrodes were stored in 0.1 M HCl solution.

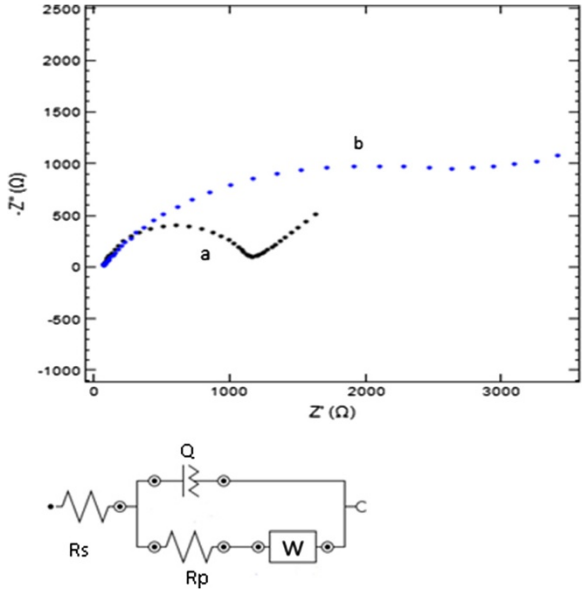

Figure 4 Electrochemical impedance spectra of the carbon paste electrode. **(a)** Unmodified (curve a) and coconut shell powder-modified (curve b) CPE in potassium ferricyanide solution. **(b)** The circuit which most fitted to the unmodified CPE.

FTIR studies

The accumulation of metals at the electrode surface is thought to be mainly by electrostatic attraction between different functional groups on the coconut shell powder embedded at the electrode surface and cationic metal ions. Ionization of these groups in aqueous solution enables them to participate in cation binding. The binding of the metal ions at the electrode surface was studied using FTIR. An FTIR spectrum of the coconut shell powder before and after its treatment with metal solution

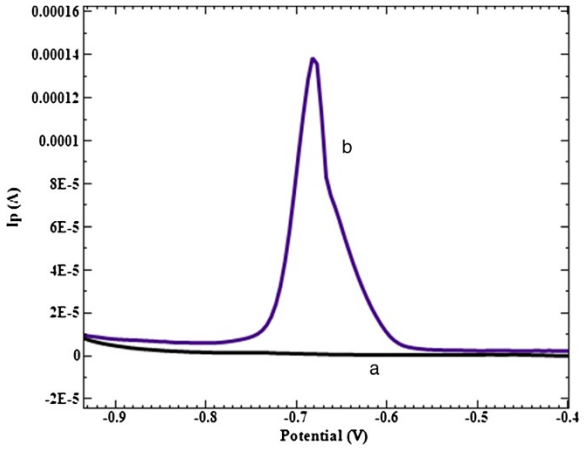

Figure 6 Differential pulse anodic stripping voltammograms for determination of Cd (II). **(a)** Before accumulation and **(b)** after 10-min accumulation; stripping solution is 0.1 M HCl.

is shown in Figure 7. The FTIR spectroscope analysis of the finely powdered and dried coconut shell powder (native) indicates broad absorption bands at 3,425.5, 1,625.9, 1,448.5, and 1,261.4 cm^{-1} (Figure 7 spectrum a) representing –OH stretching, C = O stretching, OH bending, and C-O stretching vibrations. After treatment with Cd (II), the adsorption bands shifted to 3,431.3, 1,631.7, 1,446.6, and 1,257.7 cm^{-1}, respectively (Figure 7 spectrum b). Slight shift in the adsorption bands indicated that the –OH, C-O, and C = O groups are involved in the Cd (II) binding.

Mechanism of accumulation

Based on our experimental findings and pertinent information available on the relevant topic, a mechanism for metal binding at the modified electrode surface is proposed. Coconut shell powder contains oxygen-containing functional groups in lignins and cellulose. These groups may constitute a physiologically active group to interact with the Cd (II) ions. The mechanism of the accumulation at the modified electrode is as follows:

$$(Cd^{2+})_{solution} + (MCPE)_{surface} \longrightarrow (Cd^{2+}\text{- MCPE})_{adsorb} \quad \text{(Accumulation step)}$$

$$(Cd^{2+}\text{- MCPE})_{adsorb} + ne^- \longrightarrow (Cd^{0}, MCPE)_{adsorb} \quad \text{(Reduction step)}$$

$$(Cd^{0}\text{- MCPE})_{adsorb} - ne^- \longrightarrow (Cd^{2+})_{solution} + (MCPE)_{surface} \quad \text{(Stripping step)}$$

Optimization parameters

Different parameters affecting the voltammetric determination of Cd (II) such as amount of modifier, accumulation media, accumulation time, and stripping media were investigated.

Amount of modifier

The effect of the amount of coconut shell powder in the modified CPE on the peak current was investigated and shown in Figure 8. The results indicated that the peak current increases with the increase in the amount of coconut shell powder initially because the increase in the amount of the coconut shell powder results in the increase of binding sites, which facilitates the accumulation of cadmium at the electrode surface. However, at 20% of coconut shell powder relative to the mass of carbon powder, the highest peak currents were obtained. After 20% modifier, the continuous increase in the amount of the modifier causes a decrease of the peak current, because excessive coconut shell powder results in the decrease of conductivity of the modified electrode.

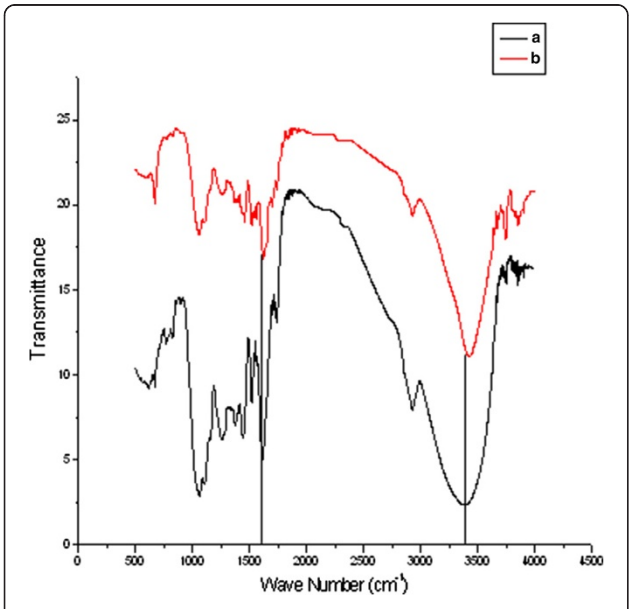

Figure 7 FTIR spectra of coconut shell powder before (a) and after treatment with Cd (II) (b).

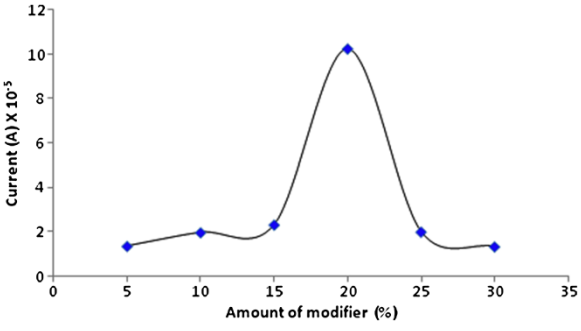

Figure 8 Effect of the amount of modifier on DPASV current response.

So, the best ratio of the modifier in the carbon paste composition is 20% (*w/w*) for the determination of Cd (II).

Accumulation media

The accumulation of cadmium was examined in supporting electrolytes such as acid solution, base solution, and different buffers like acetate buffer, phosphate buffer, and Britton-Robinson buffer. Voltammetric peaks were observed in most of these electrolytes; however, in acetate buffer solution, the anodic peak current was higher, and better defined peak shape was observed for Cd (II). A baseline for the determination of metals in acetate buffer is comparatively low, suggesting acetate buffer as the best accumulating medium.

The effect of the pH of accumulating media on the voltammetric response of the CS-MCPE was studied in a pH range between 3.0 to 6.0 in a solution containing 1 ppm Cd (II) with 1 mM sodium acetate buffer. As shown in Figure 9, anodic peaks current was increased as the pH is changed from 3.0 to 6.0, reaching a maximum at pH 5 and then decreases. The results show that maximum binding of these metal ions on the electrode surface occurs at pH 5. Since the ionization of functional groups depends on the accumulating solvents and its pH, at pH > pKa, most of these functional groups are mainly in ionized form and can exchange H^+ with metal ions in a solution. The concentration of acetate buffer was also varied from 0.1 to 100 mM. The maximum current value was obtained for 1 mM acetate buffer.

Accumulation time

The influence of accumulation time on the stripping peak currents of 0.1, 1, and 10 ppm of Cd (II) in 1 mM sodium acetate buffer solution was investigated. An increase in the current response was observed with increasing preconcentration time initially, which indicates that cadmium was rapidly adsorbed on the modified electrode surface while further prolonged accumulation did not improve the peak height. In comparison to the three concentrations of metal ions selected for this experiment, the electrode surface gets saturated early for higher concentration compared to the low concentration. For further experiment, 1 ppm Cd (II) solution was used with accumulation time of 10 min.

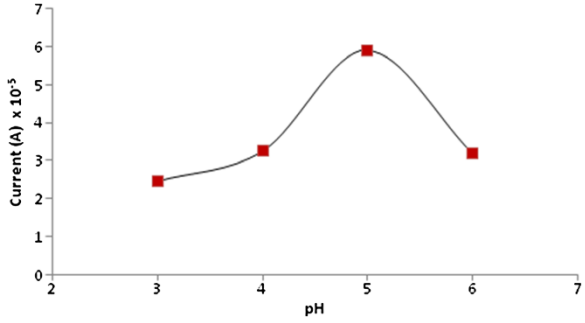

Figure 9 Effect of pH of accumulating solvent on the DPASV response of the modified carbon paste electrode.

Stripping media

The influence of the nature of stripping medium on the current response was investigated using hydrochloric acid, nitric acid, sulfuric acid, perchloric acid, phosphoric acid, sodium hydroxide, ammonium hydroxide, sodium chloride, and potassium chloride; the results are shown in Figure 10. The maximum current response was observed for hydrochloric acid solution as a stripping media, since in acidic solutions, its protons have the ability to displace the Cd (II) ions; in addition, the chloride ions of HCl promotes the stripping of metals. It is evident from the previous studies that the Cl^- ion is the best migrating ligand for Cd (II) (Rajawat et al. 2013a). The effect of the stripping solvent concentration was also studied by varying the concentration within the range 0.01 to 1 M. The optimum current response was observed at 0.1 M HCl.

Determination of metals in the presence of surfactants

The effect of the presence of the surfactants on the determination of Cd (II) was carried out using Triton X-100, SDS, and CTAB as representatives of non-ionic, anionic, and cationic surfactants, respectively. Typical voltammograms in the presence of increasing amounts of each surfactant were recorded, and the effect of surfactant on current value was presented in Figure 11. An increase in the current response was observed for the anionic surfactants, whereas for the cationic

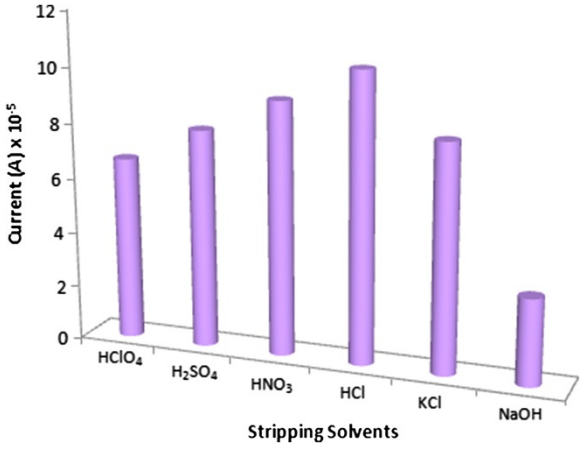

Figure 10 Effect of different stripping solvents on DPASV current response.

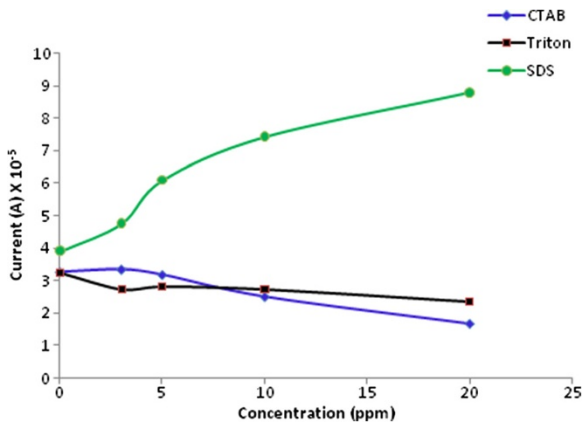

Figure 11 Effect of different surfactants on DPASV response of the CS-MCPE.

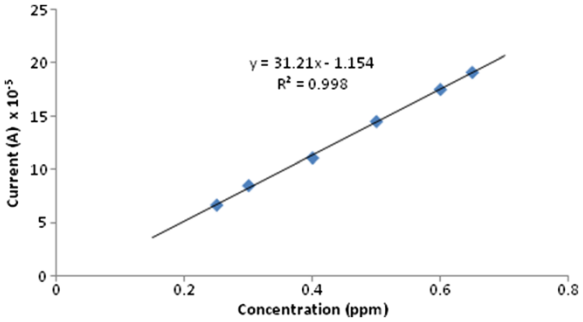

Figure 12 Standard addition curve for the determination of Cd(II) under the optimized conditions for 15 min preconcentration time.

surfactants, a decrease in the current response was observed. For neutral surfactant, not so much change was observed. No variation in the peak potential value was observed for Cd (II) determination on increasing amount of surfactant.

Interference study

The effect of the presence of other metal ions present in the solution on the anodic peak currents of Cd (II) was evaluated. A 10% change in the current response is assumed as a constant current response. No effect on the determination of Cd (II) was observed up to 250-fold of Pb (II), 5-fold of Cu (II), 25-fold Ni (II), and 30-fold Cr (VI). The results of interference study can be justified by hard-soft acid base theory. The main binding sites in these modifiers are COOH and OH groups, which are hard bases, whereas the metals Pb (II) and Cu (II) are intermediate; Cd (II), Hg (I), and Zn (II) are soft acids. According to hard-soft acid base theory, hard acid tends to form complexes with hard bases, and soft acids tend to form complexes with soft bases. Since copper is strongly bonded on electrode surface, so it is the most interfering metal.

Analytical characteristics

Analytical performance of the CS-MCPE was evaluated with standard addition of Cd (II) under the optimum conditions determined above, and the corresponding results are shown in Figure 12. A linear relationship was observed between the analytical signal and concentration ranging from 200 to 650 μgL^{-1}. In this region, the resulting equation is $y = 31.21x - 1.154$, with a correlation coefficient 0.998.

The detection limit is evaluated to be about 105 ppb ($S/N = 3$) after a 15-min accumulation. The limit of detection of the prepared electrodes was compared with the previously prepared electrodes; it is comparatively better with some of the previously reported electrodes (Beltagi et al. 2011; Roa et al. 2003). The stability of the prepared electrode was determined using the DPASV for the same modified electrode with an interval of 2 weeks over 6 months, and it was found 6 months, assuming 5% change in the current response as a constant current response.

Conclusion

The present paper demonstrates a simple, ecofriendly, and sensitive electrochemical method for the determination of cadmium based on the coconut shell powder-modified carbon paste electrode. Cyclic voltammetry and electrochemical impedance spectroscopy study results confirm the incorporation of coconut shell powder at the electrode surface. Open-circuit accumulation, followed by anodic stripping voltammetry, was used for the determination of cadmium. An enhancement in the current response was observed in the presence of anionic surfactants. Different factors affecting the sensitivity of the prepared electrode were optimized. The optimized conditions for the determination of Cd (II) using CS-MCPE are acetate buffer of pH = 5 as accumulating solvent, 15 min accumulation time and hydrochloric acid as the stripping solvent. Despite some mutual interference effects, cadmium ions can be reliably determined with low detection limits using the standard addition procedure. The utilization of plant-based electrode in place of mercury-based electrodes is an attempt to perform environment friendly electrochemical determination of cadmium.

Abbreviations

CPE: carbon paste electrode; CS-MCPE: coconut shell powder-modified carbon paste electrode; CTAB: cetyltrimethylammonium bromide; CV: cyclic voltammogram; DPASV: differential pulse anodic stripping voltammetry; FTIR: Fourier transform infrared; MCPE: modified carbon paste electrode; SDS: sodium dodecyl sulfate; SEM: scanning electron microscope.

Competing interests
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