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Article

Synthesis, Characterization, Electrochemical, and Adsorption Studies of Novel Ternary Single-Walled Carbon Nanotubes/Hydroxyapatite/Polypyrrole Nanocomposite

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ABSTRACT

The world today faces new and important challenges, among them energy crisis and the improvement of electrochemical properties of materials are the prominent ones. The way to improve the mechanical and electrochemical properties for (carbon nanotubes [CNTs]) is to incorporate some reinforcement materials such as hydroxyapatite (HAP) and polypyrrole (PPy). The synthesis of nanocomposites based on CNTs and HAP coating has been applied with PPy by *in situ* polymerization method. CNTs provide strength and toughness to delicate HAP (HAP and PPy) thereby acting as brilliant reinforcement material. First, a mixed acid solution was treated with CNTs to obtain functionalized CNTs. Then, in the next step, HAP is dispersed in CNTs after which PPy is coated on the resultant mixture, leading to the formation of PPy/single-walled CNTs (SWCNT)/HAP composite. The nanocomposite has been synthesized by varying the percentage of f-SWCNT from 0.25% to 0.75%. The sol–gel method was employed for preparing the nanocomposite, in which it described by the *in situ* preparation of SWCNT/HAP/PPy nanocomposite. The nanocomposite formation was confirmed by "scanning electron microscopy, Fourier-transform infrared spectroscopy, and thermogravimetric analysis." The design and fabrication of structural nanomaterials are essential for many applications. The present work describes the electrochemical properties and removal of heavy metals from wastewater.

Key words: Hydroxyapatite, Nanocomposite, Polypyrrole, Single-walled carbon nanotubes

1. INTRODUCTION

Hydroxyapatite (HAP) is a natural mineral that is the hydroxyl end member of the complex apatite group with the formula $Ca_{10}(PO_4)_6(OH)_2$. Human bones contain enamel (96%) and dentine (4%) calcium HAP [1,2]. HAP is present in human bone tissue and teeth. It is from the phosphate calcium class and is based on biomaterials [3]. In the biomedical field, a vital role is played by it, as it has stellar biocompatibility with the tissues of humans. It has distinct bioactive properties and resembles closely to the mineral component present in the tissues of humans. Its calcium to phosphate ratio is 1.67 which is identical to that of the natural bone [1]. Therefore, it is known as one of the most promising orthopedic biomaterials to prepare nanocomposite and improve the properties such as chemical and biomedical properties [2]. Thus, materials like carbon nanotubes (CNTs) are needed to be reinforced in its structure for improving the chemical, biomedical, as well as corrosion resistant properties. Recently, CNTs have attracted significant attention in the preparation of nanocomposites with (HAP) [3] (Figure 1).

CNTs are widely multi-use materials which display massive potential for several applications [1], Single-walled CNTs (SWCNTs), covalently functionalized with the carboxylic group, were used as f-SWCNTs have excellent solubility in water and some organic solvents [4]. SWCNTs play primary role in nanotechnology and are currently between the most intensively investigated materials [5]. They possess small dimensions and have a high aspect ratio (length to diameter). They are also known to exhibit some mechanical, [24] electrochemical, and bioactive properties, according to several published reports recently. CNTs are able to be reinforced with materials such as HAP and PPy [6]. CNTs have exceptional physical and chemical properties such as good chemical stability, small density, thermal and electrical conductivity, as well as high tensile strength (60 GPa) [7] [25]. There are several studies on CNTs for the synthesis of the nanocomposite, which explains as a key factor in improving some the structural properties, for example, wear resistance, fracture toughness, and strength. Further, the biocompatibility of CNTs in some application has been studied to a very less extent [8-13]. Herein, novel CNT-reinforced with HAP nanocomposites were fabricated by the *in situ* chemical deposition of HAP on homogeneously dispersed CNTs.

Conductive polymers (CPs) have attracted huge attention in the past few decades, because they simultaneously display superior chemical and physical properties such as resistance to corrosion, possess low density, and chemical diversity, high flexibility as well as its ease of controlling shape and morphology, thus playing a major role for

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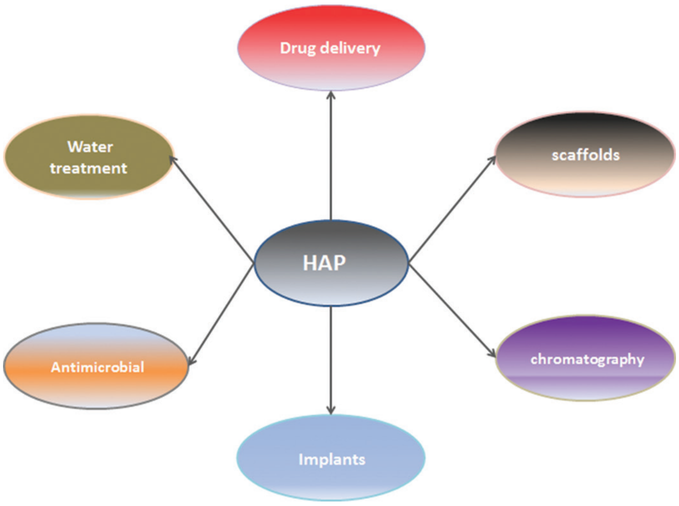

Figure 1: Applications of hydroxyapatite.

tunable conductivity over their usual inorganic counterparts [14]. Polypyrrole (PPy) is the most common conducting polymer (CP) among other conducting polymer (CP) plastics, due to useful properties such as environmental stability, easy manufacturing, excellent thermal stability, very good electrical conductivity and optical property high specific capacitance, and low cost [17,18,19]. On the other hand, the *in situ* polymerization of pyrrole reduces the rate of polymerization of pyrrole monomers and also ensures that the polymer is attached evenly to the SWCNTs surface [15].

In the present work, the synthesis of SWCNTs reinforced mesoporous HAP with PPy coating is successfully performed using a sol–gel method. The synthesis of successive generations of PPy and HAP linked to the surface of SWCNTs. The synthesis was confirmed using several characterization techniques by "scanning electron microscopy (SEM), Fourier-transform infrared spectroscopy (FTIR), and thermogravimetric analysis (TGA)."

The contamination of heavy metals poses a severe threat to our environment as they accumulate in the human body as well as other living organisms. There are various chemical as well as physical methods such as precipitation, ion exchange, solvent extraction, filtration, reverse osmosis, sedim entation, and cation surfactants which have been developed to remove high concentrations of these hazardous metals from wastewater [22]. Thus, it becomes an absolute necessity to develop novel materials for effectively removing heavy metals and dyes simultaneously from wastewater [26]. In this context, we have synthesized a novel ternary nanocomposite SWCNT/HAP/PPy and studied its adsorption capacity in terms of heavy metal removal notably Cu (II) and Cd (II).We have also reported applications such as the electrochemical behavior by studying the scan rate on anodic and cathodic peak currents as well as potentials in the electrochemical application.

2. EXPERIMENTAL

2.1. Chemical Reagents and Materials

The reagents used for the synthesis of nanocomposite were pyrrole, ethanol, and single-walled CNTs which were purchased from Sigma Aldrich and Platonic Nanotech respectively. HAP $(Ca_{10}(PO_4)_6(OH)_2$ was purchased from Fisher Scientific and ferric chloride was procured from Finar chemicals, respectively. Nitric acid and sulfuric acid were procured from SRL chemicals. Double-distilled water (DDW) was employed for carrying out all the experimentation.

2.2. Acid Functionalization of SWCNT

The first step was addition of 1.0 g of SWCNT to 200 mL of $(HNO_3{:}H_2SO_4)$ in the ratio of 1:3 yield- ing f-SWCNT. As a result of this acid functionalization, the character at the ends of the SWCNTs is transformed from hydrophobic to hydrophilic on addition of the COOH group.

2.3. Chemical Polymerization of Pyrrole into SWCNT/HAP: Conversion of Binary to Ternary SWCNT/HAP/PPy Nanocomposite

Various quantities of the as prepared SWCNT/HAP, namely, 0.25 g and 0.75 g were taken. 0.1 M ferric chloride solution dissolved in 100 ml of DDW was added to each variable amounts. After this, pyrrole (0.987 g) was added to the above mixture and the resultant solution was sonicated for 4 h. The resulting ternary solution was centrifuged at 3600 rpm and washed with DDW and ethanol subsequently. The solution was then kept in air oven at 60–65□. Finally, the product obtained was named SWCNT/HAP/PPy.

2.4. Electrochemical Measurements

A cell consisting of three electrodes was used to perform all the electrochemical studies. The platinum wire acted as the counter electrode, platinum solid was employed as the working electrode, and the Ag/AgCl (3 M, KCl) was used as the reference electrode. The potential was swapped from −0.1 to +0.6 with 5 s before starting the measurements. The scan rate was studied at 10 mVs^{-1}, 25 mVs^{-1}, 50 mVs^{-1}, and 100 mVs^{-1} values. The CV analysis was accomplished in 1 M KCl solution by the frequency response analyzer.

2.5. Adsorption Experiment

For studying the adsorption phenomenon on the novel ternary nanocomposite SWCNT/HAP/PPy with respect to Cd^{2+} and Cu^{2+} ions from aqueous solution, the technique employed was the batch adsorption [23]. All the experiments were conducted using a fixed amount of the ternary nanocomposite (20 mg) in 20 mL of varying concentrations of metal salt solutions of Cd^{2+} and Cu^{2+} with varying concentrations (1, 1.5, 2.5, 3, and 3.5 ppm). The solutions were agitated on a magnetic solution for 20 h at neutral pH and ambient temperatures. After the adsorption process was complete, the solution was filtered and the concentrations of the filtrates remaining were determined using atomic absorption spectroscopy technique.

2.6. Characterization

To study the structural aspects of SWCNT and SWCNT/HAP/PPy, the composite was characterized using SEM using JEOL, JSM, and 6510-LV (Japan). The FT-IR spectra of HAP, SWCNT, and SWCNT/HAP/PPy ternary nanocomposite were recorded on a Perkin Elmer 1750 FT-IR spectrophotometer on Kbr pellets. TGA of HAP,SWCNT, PPy, and SWCNT/HAP/PPy ternary nanocomposite were performed employing the Perkin-Elmer (Pyris Diamond) instrument, which heated samples from 20°C to 900°C at the rate of 10°C min^{-1} in nitrogen atmosphere at the flow rate of 180 mL/min.

3. RESULTS AND DISCUSSION

3.1. FTIR Spectroscopic Analysis

The FTIR spectra of the pristine SWCNT and SWCNT reinforced with HAP with PPy in two concentrations, that is, 0.25% and 0.75% SWCNT/HAP/PPy, respectively, are shown in Figure 2 HAP exhibits FTIR peaks at 563, 602, 1044, 1087, and 1413 (attributed to the PO_4^{3-} group). The bands appearing at 602 cm^{-1} and 563 cm^{-1} appearing from $[PO_4]^{3-}$ also indicate that the HAP is well crystallized. The band appearing at 960 cm^{-1}, 1044 cm^{-1}, and 1087 cm^{-1} are due to the $[PO_4]^{3-}$

mode of phosphate [16]. The band at 1641 and 3442 cm^{-1} correspond to the OH^- group [17]. We observed the peak of f-SWCNTs at 3300 O-H group. As a result of the formation of C–N coordinate-covalent bonds between the conductive polymer chain and the SWCNT fragments, there is an enhancement of the spectra [18]. In the FTIR spectra, the bands of phosphates and the carbonate were observed for 0.25% and 0.75% SWCNT/HAP/PPy correspondingly. The characteristic peak for SWCNTs was observed at 2880 cm^{-1} in the spectra of CNTs/HAP.

3.2. TGA and Differential Thermal Gravimetry Analysis (DTG)

DTG curves analysis of f-SWCNT, SWCNT/HAP, and two different concentrations of SWCNT/HAP/PPy nanocomposites is shown in Figure 3. TGA curves are used to examine the thermal stability for nanocomposite [20]. TGA is generally used to determine the percentage of polymer that is bound to the carbon nanotube [20]. The schematic diagram TGA is shown in Figure 4. In the curve for f-SWCNT, the starting point for weight loss was at 380°C and the percentage loss weight was observed at 35%. While the f-SWCNT/HAP showed weight loss at 320°C, the degradation at 390ºC could be attributed to the loss of the pure f-SWCNT. Meanwhile, the SWCNT/HAP/PPy showed weight loss at 430°C which was 50°C higher than that displayed by pure f-SWCNT/HAP. The rise in the degradation temperature was indicative of the incorporation of F-SWCNT into the HAP/PPy which exerted a thermally stabilizing effect in the composite [21].

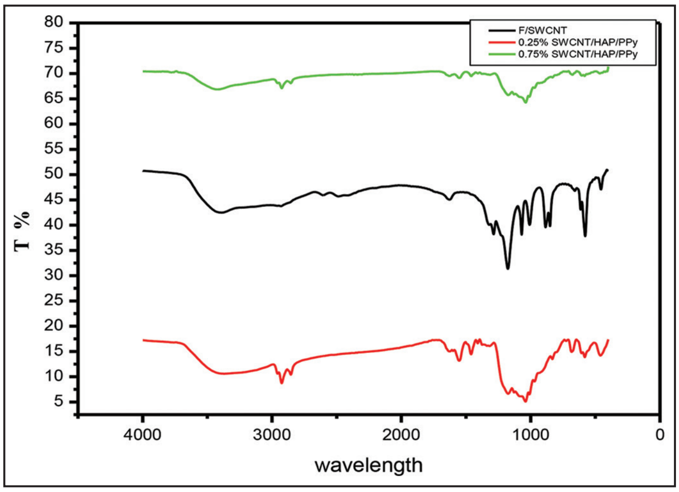

Figure 2: Fourier-transform infrared spectroscopy spectra of composite powders 0.25 wt. % single-walled carbon nanotubes/hydroxyapatite/polypyrrole (SWCNT/HAP/PPy) and 0.75 wt. % SWCNT/HAP/PPy nanocomposite.

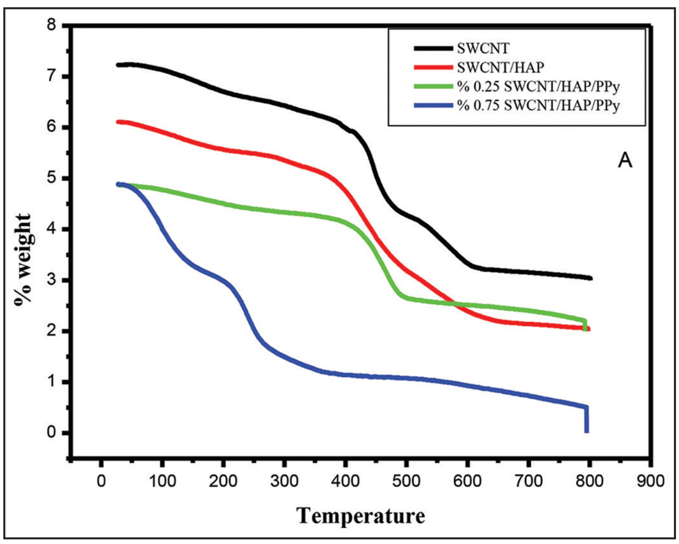

Figure 3: Differential thermal gravimetry analysis curves of single-walled carbon nanotubes (SWCNT), SWCNT/hydroxyapatite (HAP), 0.25% SWCNT/HAP/polypyrrole (PPy), and 0.75% SWCNT/HAP/PPy.

3.3. SEM

The surface morphology of PPy, HAP/PPy, and SWCNT/HAP/PPy nanocomposite was revealed by SEM micrographs, as shown in Figures 5a and b and 6a and b, respectively. The morphology of pristine PPy (Figure 3a) shows that various globular nanoparticles were agglomerated. In the case of SWCNT/HAP/PPy nanocomposite, there was a slight variation in morphology as some globular nanoparticles became larger in size (Figure 6b). No free HAP nanoparticles and SWCNT were present which confirmed that pyrrole was successfully polymerized on surface of SWCNT and HAP.

4. APPLICATIONS

4.1. Electrochemical investigation

The cyclic voltammetry represents (CV) for F-SWCNT, 0.25% wt SWCNT/HAP/PPy, and 0.50 wt % SWCNT/HAP/PPy nanocomposite. As evident from (Figure 7), 0.50 wt % SWCNT/HAP/PPy nanocomposite enclosed the largest CV-integrated area. Comparing f-SWCNT and 0.25 wt % SWCNT/HAP/PPy indicate excellent

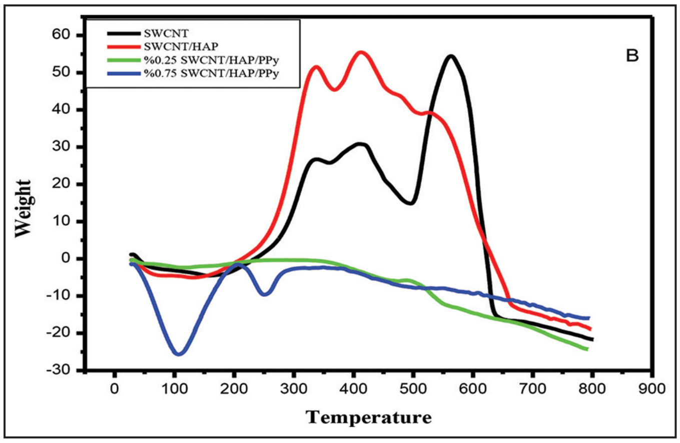

Figure 4: Thermogravimetric analysis curves of single-walled carbon nanotubes (SWCNT) and SWCNT/hydroxyapatite/polypyrrole (HAP/PPy) and the effect of the concentration of SWCNT/HAP on 0.25% SWCNT/HAP/PPy and 0.75% SWCNT/HAP/PPy.

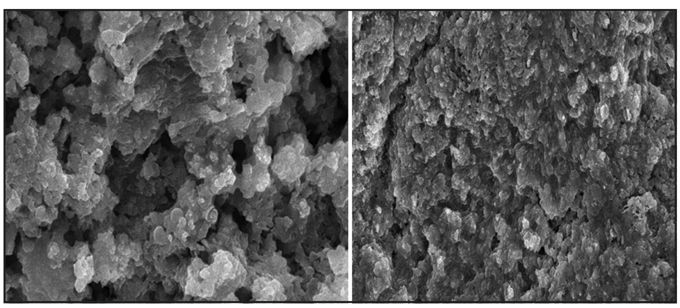

Figure 5: Scanning electron microscopy images of (a) polypyrrole (PPy) and (b) hydroxyapatite (HAP)/PPy at 1 μm and 10 μm, respectively.

capacitive performance. The improvement and development behavior can be a result of the synergetic effect of highly pseudo capacitive HAP PPy [15]. Effect of scan rate on super capacitance behavior of SWCNT/HAP/PPy [4] was studied. To understand the electrochemical behavior of the nanocomposite, we studied the effect of scan rates (Figure 8) and observed the cathodic peak current (Ipc) and the anodic peak current (Ipa) (Figure 9).

4.2. Adsorption Studies of Cd $^{2+}$ and Cu $^{2+}$ Ions

On mixing metal salt solutions of Cd^{2+} and Cu^{2+} of varying concentrations (1, 1.5, 2.5, 3, and 3.5 ppm each) with the required amount of ternary nanocomposite SWCNT/HAP/PPy, the nanocomposite was found to obey the equilibrium process:

Metal ions solution + Adsorbent ⇄ metal ions adsorption on adsorbent surface:

The following equation was employed for calculating the value of adsorption efficiency (%) and adsorption capacity, (qe and mg g^{-1}) [23-27]:

$$(\%)\,Removal = \frac{C_i - C_f}{C_i} \times 100$$

$$q_{e,} = \frac{(C_i - C_e)V}{m}$$

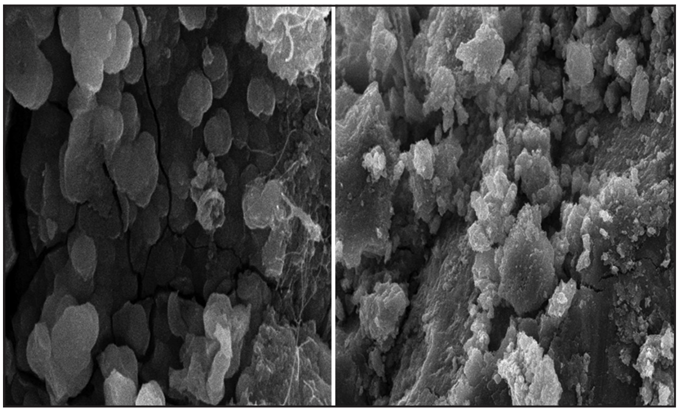

Figure 6: Scanning electron microscopy images of (a) single-walled carbon nanotubes/hydroxyapatite/polypyrrole (SWCNT/HAP/PPy) and 6(b) SWCNT/HAP/PPy at 1 µm and 10 µm.

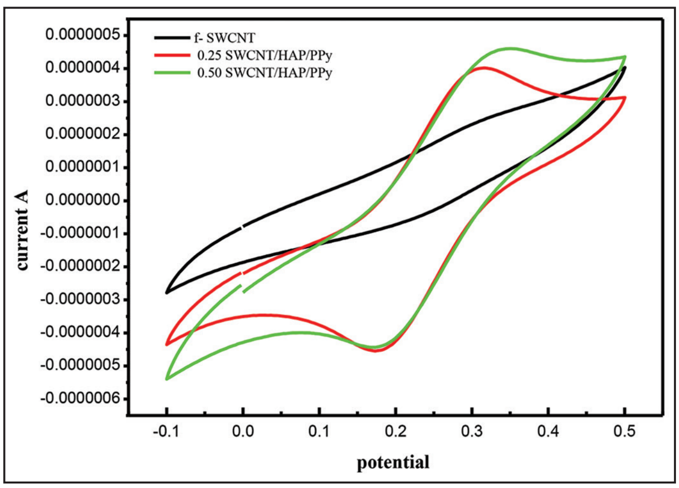

Figure 7: Cyclic voltammetric graphs for pure f-SWCNT, 0.25% single-walled carbon nanotubes/hydroxyapatite/polypyrrole (SWCNT/HAP/PPy), 0.50% SWCNT/HAP/PPy, and measured at scan rate 10 mVs^{-1}.

Where C_i, C_f, and C_e are the initial, final, and equilibrium metal concentrations (in mg L^{-1}), respectively, V is the volume of metal solution (in L), and m is the mass of the adsorbent in (g). By this adsorption process, the metal salts concentration (from 1 to 3.5 ppm each) with fixed adsorbent mass (25 mg) can be analyzed from q_e adsorption capacity, (mg g^{-1}) versus concentration (mg L^{-1}) curves. For the metal ions, it decreases from 80.0% to 55% (having maxima at 2.5 ppm) and 97% to 62.1% (having maxima at 2.5 ppm) for Cd^{2+} and Cu^{2+} ions, respectively (Figures 10 and 11). These observed trends

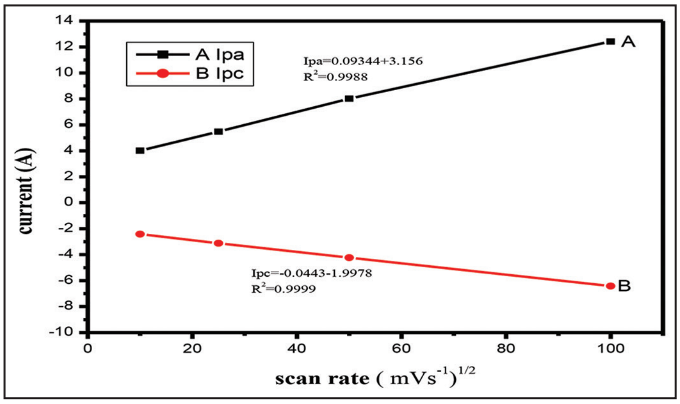

Figure 8: Effect of scan rate on the super capacitance behavior of the designed nanocomposite 0.50 wt.% single-walled carbon nanotubes/hydroxyapatite/polypyrrole.

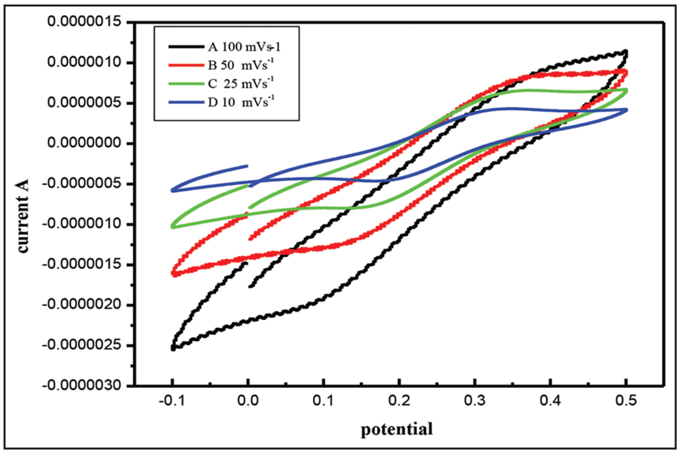

Figure 9: The cathodic peak current (Ipc) and the anodic peak current curves for the single-walled carbon nanotubes/hydroxyapatite/polypyrrole nanocomposite.

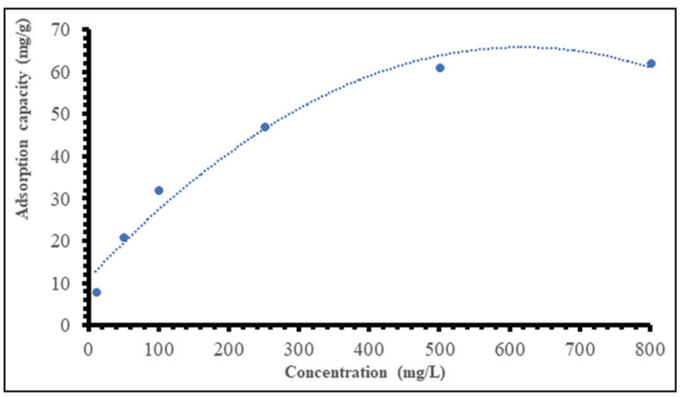

Figure 10: Adsorption capacity (qe) versus concentration curve for adsorption of Cd^{2+} ions.

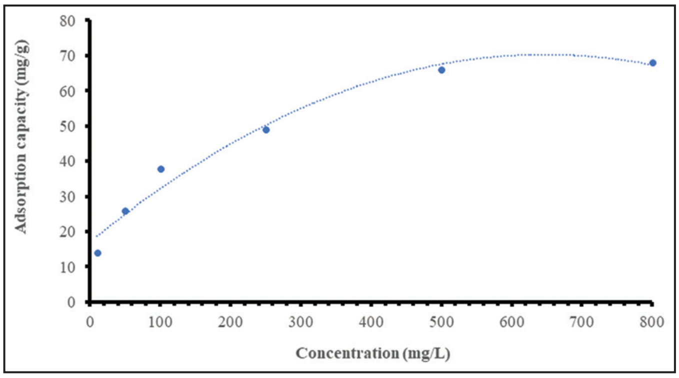

Figure 11: Adsorption capacity versus concentration curve for adsorption of Cu^{2+} ions.

might be due to the saturation of the adsorption sites present on the surface of the adsorbent material [21]. In the case of metal ions, the maximum value of q_e, was found at 2.5 ppm for both Cu^{2+} and Cd^{2+} ions.

5. CONCLUSION

The present study reports the facile synthesis of a novel ternary nanocomposite comprising SWCNT, HAP nanoparticles, and PPy. The *in situ* synthesis of ternary nanocomposite SWCNT/HAP/PPy was carried out in the presence of catalytic amount of $FeCl_3$. The formation of the nanocomposite is confirmed by analytical techniques such as FTIR, TGA, and SEM. The synthesized ternary composite was evaluated for its electrochemical performance by cyclic voltammetry and was found to exhibit excellent capacitive performance. The synthesized ternary nanocomposite was found to be very effective in the removal of heavy metal ions such as Cu^{2+} and Cd^{2+} ions from water. This study opens avenues for effective removal of other carcinogenic compounds and heavy metal ions by this nanocomposite and its derivatives. Assuredly, these kinds of nanocomposites are advantageous in wastewater treatment.

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8. CONFLICTS OF INTEREST

The authors declare that there are no competing interests regarding the publication of this paper.

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***Bibliographical Sketch**

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