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Silver tungstate nanoparticles: Characterization and Electrochemical Sensing property

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Abstract: Silver tungstate (Ag_2WO_4) nanoparticles were synthesized by a facile and cost effective co-precipitation method. The synthesized nanoparticles were characterized by XRD, FT-IR spectroscopy, and SEM analysis. The XRD pattern reveals that the formation of Ag_2WO_4. In addition, by using the XRD data lattice parameter values were also calculated. The FT-IR and Raman analysis confirms the presence of Ag-O and W-O bonds in Ag_2WO_4 nanoparticles. The morphology of the as-synthesized Ag_2WO_4 was analyzed by SEM. The electrochemical sensing behavior of as synthesized Ag_2WO_4 nanoparticles towards catechol was investigated using cyclic voltammetry.

1. Introduction

Transition metal tungstates are important members of inorganic materials which are widely applied in many fields. In recent decade, a number of research works on the synthesis and characteristic investigating of nano and micro-sized transition metal tungstates. Metal tungstates are represented in general formula that MWO_4 ($M=Cu^{2+}$, Mn^{2+},$2Ag^{+}$…). Some of the reported metal tungstates are $MnWO_4$ [1], $FeWO_4$ [2], etc. These metal tungstates are applied potentially in various fields like optical fibers [3], photoluminescence, photocatalyst [4], and so on. Among metal tungstates Silver tungstates are applied in various field like photocatalyst, electrocatalyst, etc. There is a report towards the electrochemical detection of nitro phenol. Other than that to the best of our knowledge, the reports on the electrocatalytic properties of Ag_2WO_4, are obtained rarely. In this work, Ag_2WO_4 with rod shape was prepared via a co-precipitation method. The as-synthesized samples were modified directly on a glassy carbon electrode (GCE), and their electrocatalytic performances for catechol, in a KCl solution were investigated. The results show that Ag_2WO_4haselectrocatalytic activity towards the detection of catechol.

2. Experimental

2.2. Reagent

Silver acetate and sodium tungstate were purchased from Qualigens and used as received. Catechol was purchased from CDH, India. Other chemicals used were of analytical reagent grade. Double distilled water was used thought the experiment. All chemicals were used without further purification.

2.2. Synthesis of Ag_2WO_4nanoparticles

Ag_2WO_4 nanoparticles were prepared via co-precipitation reaction in aqueous media by addition of Ag^+ solution, in tungstate solution under vigorous stirring. When the mixing process was completed, the formed Ag_2WO_4suspension was filtered and washed with distilled water and ethanol for three times and then dried in oven at 90 °C for 2 h. In order to form crystalline Ag_2WO_4particles, the prepared samples were annealed at 600°C.

2.3. Instrumentation

FTIR spectroscopy was studied using Schimadzu FTIR 8300 series instrument. The structure was analyzed by a Rich Siefert 3000 diffractometer with Cu-Kα1 radiation (λ = 1.5418 Å). The morphology was analyzed by HITACHI SU6600 (SEM) Scanning Electron Microscopy. The electrochemical experiments were performed on a CHI 1103A electrochemical instrument using the modified electrode and bare GCE as working electrode, a platinum wire was the counter electrode, and saturated calomel electrode (SCE) was the reference electrode.

2.4. Preparation of Ag_2WO_4 coated GCE

The Ag_2WO_4 suspension was prepared by dispersing a 5 mgof Ag_2WO_4 in 10 mL of ethanol during 20 min of ultrasonic agitation. Prior to modification, the GCE was mechanically polished with alumina paste of different grades to mirror finish, rinsed, and sonicated in distilled water for 2 min. Finally, the GCE was coated with 10 µL of the suspension and dried in air.

3. Result and discussion

3.1. XRD,FT-IR and Morphological Analysis

The XRD pattern of the as-synthesized Ag_2WO_4sample is shown in Fig:1a. All the diffraction peaks indexed to the orthorhombic structure of Ag_2WO_4 is orthorhombic with space group pn2n.Which are well match with reported value of JCPDS No 00-034-0061.The FT-IR spectrum of as synthesized Ag_2WO_4 nanoparticle is shown in Fig:1b. FT-IR spectrum of as synthesized Ag_2WO_4 shows strong broad band at 3436 and 1632 cm^{-1} is indicating the vibration of H-O bonds on the surface water molecule, δ H_2O. The intense absorption bands at 833 cm^{-1} for Ag_2WO_4 are ascribed to bonds between (←O←W←O←) of anti-symmetric stretching vibrations within distorted [WO_6] clusters.[6] Morphology analysis was carried out for the Ag_2WO_4 nanoparticle by using SEM analysis. It shows that the Ag_2WO_4 are rod like morphology with nm meter range particles.

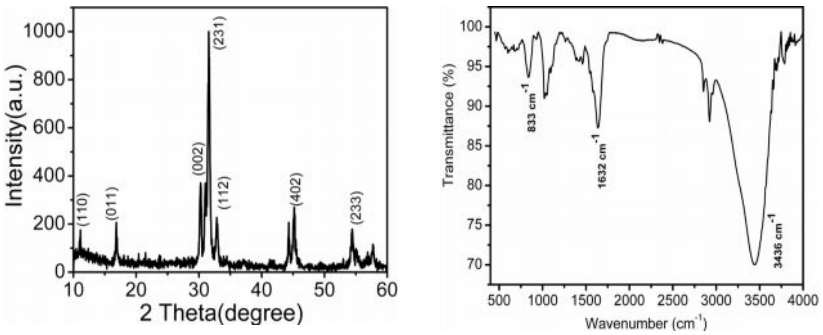


Fig: 1a, 1b, XRD pattern and FT-IR spectrum of Ag_2WO_4

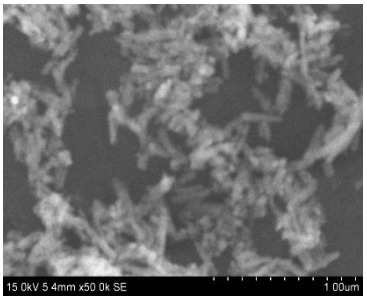
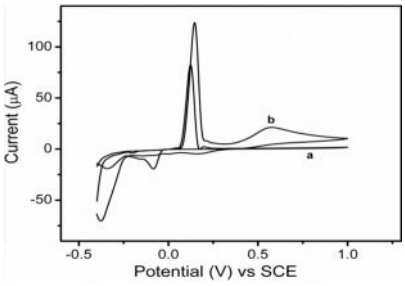


Fig: 2. SEM image of Ag_2WO_4

3.2. Electrocatalytic property

Fig.3a shows the electro oxidation of 1 mM catechol at bare and Ag_2WO_4 modified GCE (Ag_2WO_4/GCE) in 0.1 M KCl as the electrolyte. Bare GCE shows a broad oxidation peak at 0.51 V. However, the Ag_2WO_4/GCE shows an oxidation peak at +0.57 V with higher current response (21.8 μA). This oxidation potential is higher or comparable with other modified electrodes [7]. Hence it is clear that the oxidation potential for catechol at the Ag_2WO_4/GCE was shifted to positive direction with enhanced current response than the bare GCE, indicating the electrocatalytic ability of theAg_2WO_4 modified electrode.Fig.3b shows the effect of scan rate on the peak current for the Ag_2WO_4/GCE electrode was studied within the range from 10 mV s^{-1} to 500 mV s^{-1}. From the CV results, we can see that the peak currents enhanced and the oxidation peak potentials shifted in a more positive direction as the scan rate increased in KCl solution with 1mM catechol. This enhanced electrocatalytic ability is attributed to the larger available surface and surface hydroxyl group.



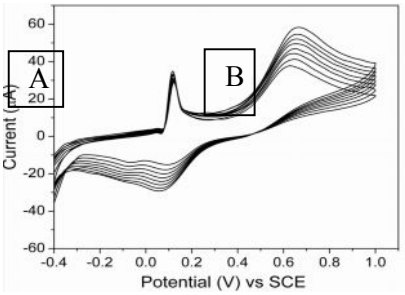


Fig: 3(A).Cyclic voltammograms of (a) bare and (b) Ag_2WO_4modified GCE in 1 mM catechol at the scan rate of 10 mVs^{-1},

Fig: 3(B):Cyclic voltammograms of Ag_2WO_4 modified GCE in 1 mM catechol at the scan rate of 30,50,70,90,100,150,200,250 mVs^{-1}.

3.3. Conclusion

Silver tungstate nanoparticles were synthesized by simple co-precipitation method. The structure of the Ag_2WO_4 nanoparticles was characterized by XRD and FTIR spectroscopy. The morphology of the Ag_2WO_4 was confirmed by SEM. The synthesized nanoparticles were employed to modify the GCE to evaluate the electrocatalytic performance towards biomolecules such as catechol. The results exhibit that the Ag_2WO_4 nanoparticles will have potential in the field of bio-sensor.

Acknowledgments

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4. References

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