

# Synthesis and Characterization of Electrospun 2D MoS<sub>2</sub> Composite Carbon Nanofibers for Determination of Vanillin

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## Abstract

Vanillin (Van), the main component is 3-methoxy-4-hydroxy-benzaldehyde. Vanillin is not only used in the food industry, but also widely applied in the field of medicine. A glassy carbon electrode (GCE) modified electrospun 2D molybdenum disulfide (MoS<sub>2</sub>) nanoparticles decorated carbon nanofibers (MoS<sub>2</sub>-CNF) were first applied for determining the amount of Van. The synthesized MoS<sub>2</sub>-CNF were characterized by scanning electron microscopy (SEM), Energy Dispersive Spectrometer(EDS), Electrochemical impedance technique(EIS). The electrochemical detection of Van was successfully conducted in 0.1 M phosphate solution (pH 10) and showed a good linear response in the range of 0.3 to 135  $\mu$ M. The proposed electrode showed a good amperometric response signal toward Van and result in the determination of real sample.

**Key words:** Electrospinning, molybdenum disulfide, carbon nanofibers, Vanillin

## Introduction

Vanillin (Van), which is an important flavorant with full milk aroma, and is mainly exists in the seeds of vanilla. Vanillin can give people a positive and pleasant emotional experience, with anti-epilepsy and anti-anxiety effects, however, overtaking Van can lead to some undesirable consequences to their consumers. Specifically, the high dose of Van can cause potential damage to human liver and kidney<sup>1</sup>. Therefore, the detection of Van is significant in many field in many field as food industry, medical intermediate. Molybdenum disulfide (MoS<sub>2</sub>) is an atomically transition-metal dichalcogenide, which has attracted widely attention, due to its thermoelectric, optical, and mechanical properties in hydrogen evolution catalyst, and for optoelectronic materials<sup>2</sup>. Electrospinning is a versatile technique to construct well-aligned nanofibers with average diameters in the sub-micrometer to nanometer range. Compared to other methods of preparing CNF, electrospinning is more simpler and lower in cost<sup>3</sup>. Electrochemical techniques have the advantages of simplicity, rapidity, high sensitivity and low cost for the analysis of compounds comparing to other detecting

methods, such as ultraviolet spectrophotometry, liquid chromatography, fluorescence spectroscopy<sup>4</sup>.

In this work, molybdenum disulfide loaded carbon nanofibers (MoS<sub>2</sub>-CNF) were synthesized by electrospinning polymer precursor followed with successive annealing process. Extensive characterizations of MoS<sub>2</sub>-CNF were studied by scanning electron microscopy (SEM), Energy Dispersive Spectrometer(EDS). In order to contract intrinsic properties of electrospun MoS<sub>2</sub>-CNF for electrochemical sensing application, different thermal treatment conditions were chosen in annealing process. The obtained MoS<sub>2</sub>-CNF were dropped on a glassy carbon electron (designate as MoS<sub>2</sub>-CNF/GCE) and applied in sensitive detection of Van by cyclic voltammetry (CV) and current-time (I-T). The proposed electrode showed a low detection limit, high sensitivity and high stability for detection of Van.

## Characterization of MoS<sub>2</sub>-CNF

Fig.1 A is the morphology and microstructure of the prepared MoS<sub>2</sub>-CNF investigated by SEM. As we can see from Fig.1 A the diameter about 200 nm, MoS<sub>2</sub>-CNF nanofibers have rough surface, which had s higher surface area to

volume ratio. As shown in Fig.1 B, in contrast, we synthesized a composite material that isoconcentration MoS<sub>2</sub> nanosheets hosted in PAN, however, due to the poor electrical conductivity of MoS<sub>2</sub>, it causes the low electrochemical response to van. Fig.1 C showed the EDS spectrum of MoS<sub>2</sub>-CNF. We can see from Fig.1 C, which mainly includes four elements (C, O, Mo, S). As can be seen in Fig.1 D, the diameter of high frequency semicircles of MoS<sub>2</sub>-CNF/GCE is significantly decreased compared to bare GCE and CNF/GCE, indicating that MoS<sub>2</sub>-CNF has the better electrical conductivity. The lower R<sub>ct</sub> value a material possesses, the higher electron-transfer ability is acquired.

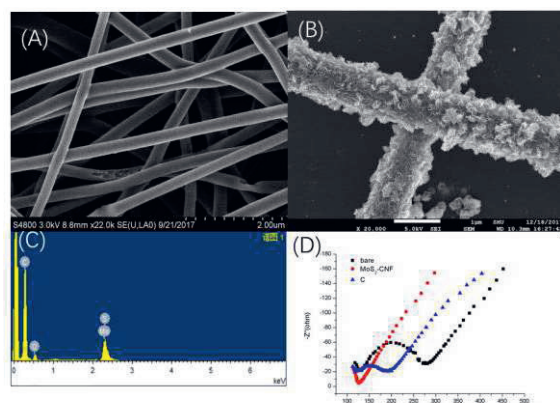


Fig.1 A SEM image of MoS<sub>2</sub>-CNF; B SEM image of MoS<sub>2</sub> Nanosheets Hosted in PAN; C EDS of MoS<sub>2</sub>-CNF; D EIS pattern of bare GCE, CNF/GCE and MoS<sub>2</sub>-CNF /GCE

### Experimental result

In order to explore the electrocatalytic of Van on MoS<sub>2</sub>-CNF/GCE, a I-T comparison was conducted on bare GCE, CNF/GCE and MoS<sub>2</sub>-CNF/GCE respectively in 0.1 M phosphate solution (pH 10) containing 10 μM Van. As Fig.2 showed that MoS<sub>2</sub>-CNF/GCE has obvious electrocatalytic effect on vanillin. The bare electrode current response of vanillin was negligible, but compared with CNF/GCE, MoS<sub>2</sub>-CNF/GCE was 5.21 times of CNF/GCE.

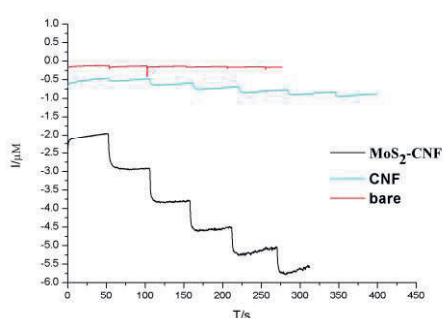


Fig.2 I-T comparison was conducted on bare GCE, CNF/GCE and MoS<sub>2</sub>-CNF/GCE

Fig.3 A showed the I-T curve of detection of vanillin with the vanillin concentration ranging from 0.3 to 135 μM under constant stirring condition. From Fig.3 B we can clearly see that the oxidation current of vanillin increased with the concentration of vanillin increasing, the modified electrode showed a good linear response in the range of 0.3 to 135 μM. The linear regression was  $I_p = 1.1459 + 0.03617C$  (μM) (R = 0.996)

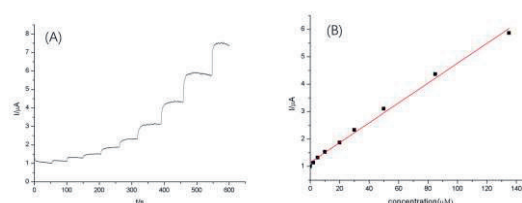


Fig.3 A I-T of Van in the concentration range from 0.3 μM to 135 μM on MoS<sub>2</sub>-CNF/GCE; B the linear regression of Van on MoS<sub>2</sub>-CNF/GCE.

### Analysis of real sample

Tab.1: Results of real sample analysis at MoS<sub>2</sub>-CNF/GCE (n = 3)

No.	Detected(μM)	Added(μM)	Found(μM)	Recovery
1	2.04	10	9.77	97.7%

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