

**Performance analysis of three distinct  $\text{Ni}_x\text{V}_2\text{O}_y$  single-phase nano self-assemblies for asymmetric supercapacitor fabrication and effective detection of low-concentration hazardous herbicide**

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## **Experimental Section**

### **Electrochemical Measurements for supercapacitor applications**

The electrochemical characteristics of the single-phase nickel vanadate composites  $\text{NiV}_2\text{O}_6$ ,  $\text{Ni}_2\text{V}_2\text{O}_7$ , and  $\text{Ni}_3\text{V}_2\text{O}_8$  were analyzed using Origa Flex- OGF 500 electrochemical workstation with a three-electrode system. The as-prepared nickel vanadate composites, Ag/AgCl, and platinum wire were used in the three-electrode system as the working, reference, and counter electrodes, respectively. The 3M KOH was used as an electrolyte. The working electrode was prepared by mixing active material (85%), activated carbon (10%), and PVDF (5%) in methyl-2-pyrrolidinone (NMP) solvent. The obtained homogeneous mixture was uniformly spread on Ni foam with an area of about  $1 \times 1 \text{ cm}^2$ . The coated Ni foam was heated to  $70^\circ\text{C}$  in a hot air oven and maintained for 12 hours.

### **Preparation of $\text{Ni}_x\text{V}_2\text{O}_y$ modified GCE for the detection of bifenoX**

The required stock solution of bifenoX ( $2020 \mu\text{M}$ ) was prepared using methanol. The Britton Robinson buffer (B-R) was used as the electrolyte. The B-R buffer solution of pH 2.5 was prepared as follows: The B-R buffer solution was prepared by adding 15 ml of 0.2 M sodium hydroxide solution into 100 ml of a mixed acid, containing 0.04 M of each boric, orthophosphoric and acetic acids. The glassy carbon electrode (GCE) was then manually polished with fine emery paper, completely washed in 10% NaOH solution for 2 minutes each, rinsed with Millipore water, and dried in the open air. The polished GCE was degreased using trichloroethylene. The GCE was then modified with 7  $\mu\text{l}$  of  $\text{Ni}_x\text{V}_2\text{O}_y$  solutions by drop-casting. A homogeneous layer was gradually attained when GCE was left to dry. The modified electrodes were prepared using three  $\text{Ni}_x\text{V}_2\text{O}_y$  phases, namely  $\text{NiV}_2\text{O}_6$ ,  $\text{Ni}_2\text{V}_2\text{O}_7$ , and  $\text{Ni}_3\text{V}_2\text{O}_8$ , for bifenoX detection. The experiments were repeated thrice to verify the reliability of

electrochemical measurements. The electrochemical measurements were carried out using cyclic voltammetry and square wave voltammetry.

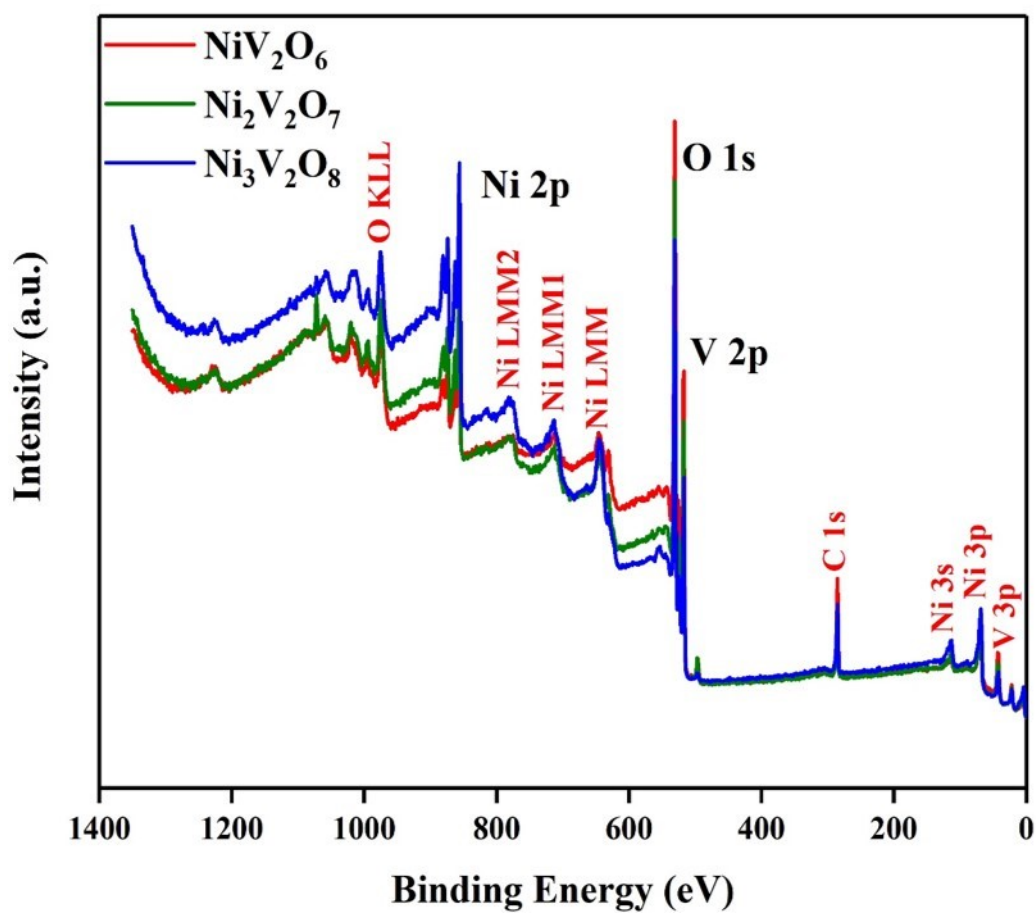


Figure 1. XPS Survey spectra of  $\text{NiV}_2\text{O}_6$ ,  $\text{Ni}_2\text{V}_2\text{O}_7$  and  $\text{Ni}_3\text{V}_2\text{O}_8$

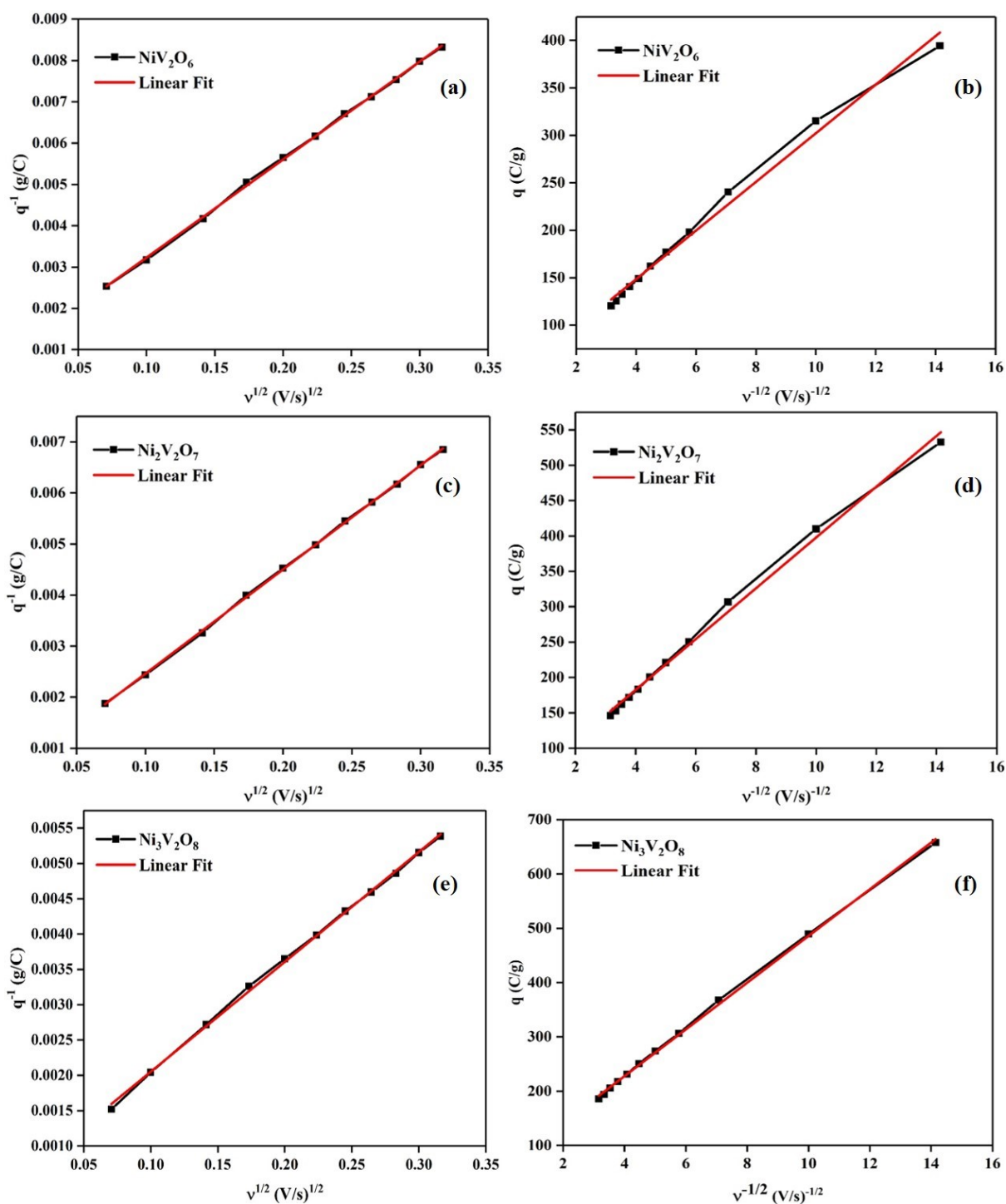
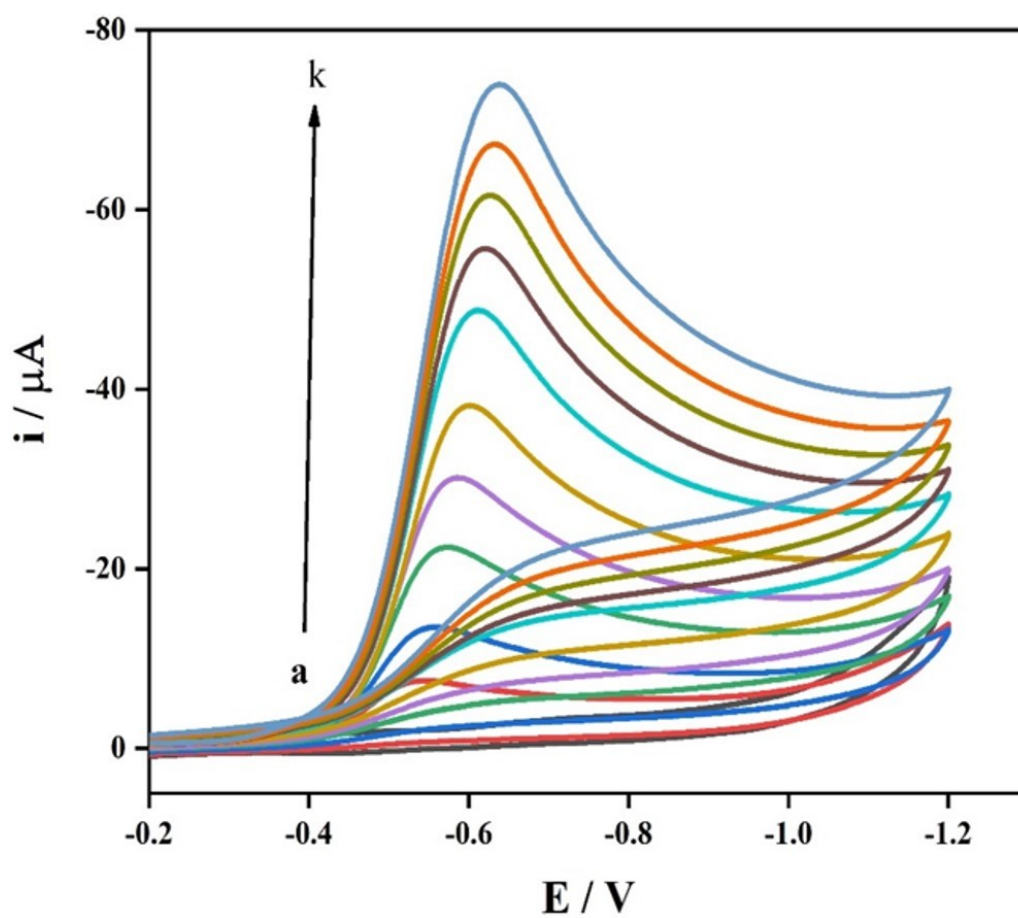
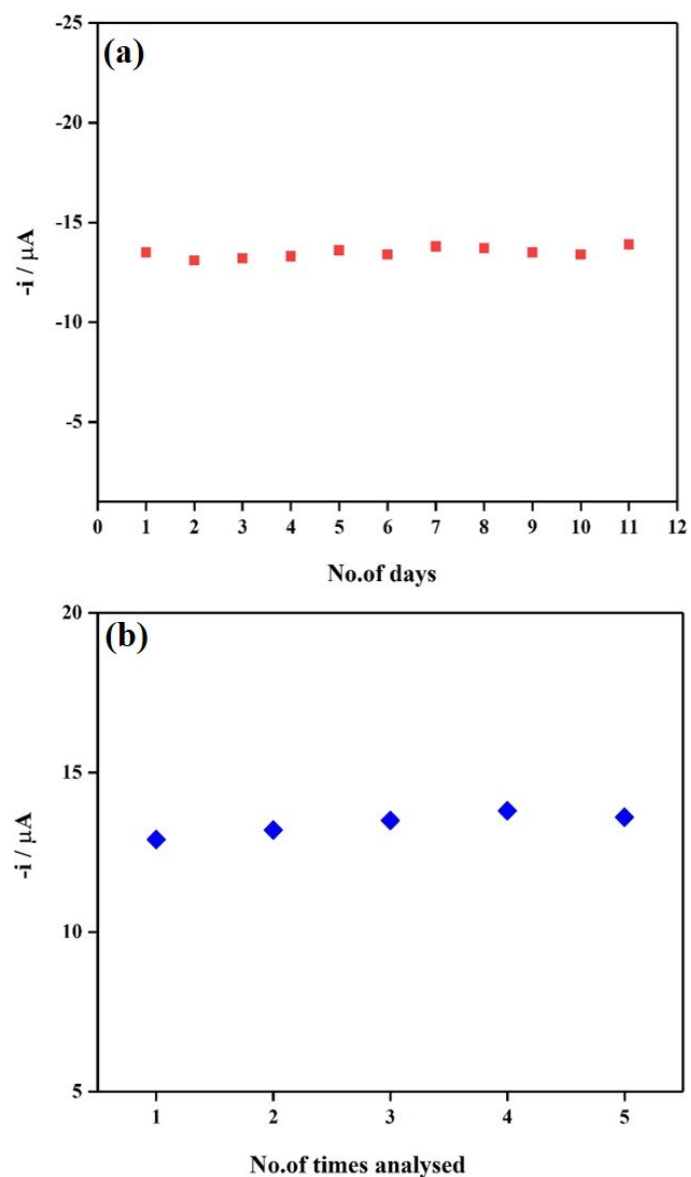


Figure S2. Linear fit ( $C$  vs  $v^{1/2}$ ) of  $\text{NiV}_2\text{O}_6$ ,  $\text{Ni}_2\text{V}_2\text{O}_7$  and  $\text{Ni}_3\text{V}_2\text{O}_8$  (a, c, e), Linear fit ( $C^{-1}$  vs  $v^{-1/2}$ ) of  $\text{NiV}_2\text{O}_6$ ,  $\text{Ni}_2\text{V}_2\text{O}_7$  and  $\text{Ni}_3\text{V}_2\text{O}_8$  (b, d, f).



**Figure S3.** Cyclic Voltammograms at different concentrations i) 10, ii) 20, iii) 30, iv) 40, v) 50, vi) 60, vii) 70, viii) 80, ix) 90, x) 100 and xi) 110 nM bifenox at scan rate  $100 \text{ mVs}^{-1}$



**Figure S4. Stability and Reproducibility study for the reduction of 20 nM bifenox in 2.5 pH B-R buffer solution. Sweep rate: 100 mVs<sup>-1</sup>**