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Supplementary Information

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Fabrication of Nickel Sulfide/Nickel Oxide Heterostructure for Efficient Electrochemical Oxidation of Methanol

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Brunauer-Emmet-Teller Analysis

BET analysis of NiS (Figure SI-1a) and NiO (Figure SI-1b) was carried out to study the Specific Surface Area (SSA) and porosity of the synthesized nanoparticles. The investigations were done using N₂ adsorption isotherm and Barret-Joyner-Halenda (BJH) analysis as shown in Fig. SI-1. The SSA calculated from the isotherm was investigated to be 27.120 m²/g and 31.465 m²/g for NiS and NiO, respectively. The pore volume and size were calculated from Barret-Joyner-Halenda (BJH) analysis. The average pore size for NiS and NiO are 2.6 nm and 2.7 nm, respectively (Table TS-1).



Figure SI-1 N_2 adsorption isotherm plots of (a) NiS and (b) NiO using BET analysis.

Both materials lie in the mesoporous range, which justifies their high catalytic activity for methanol oxidation. Increased pore size favors methanol exposure to the catalyst, prolonging the methanol residual time and improving conversion efficacy. The high surface area of mesoporous materials results in increased active sites and their mesoporous structure enhances the adsorption and diffusion of reactant molecules.

Sample	BET surface area (m²/g)	Total pore volume (cm ³ /g)	Total pore size (nm)
NiS	27.120	0.0818	2.6
NiO	31.465	0.0776	2.7

Table TS-1 BET surface area, BJH pore volume and pore size of pure NiS and NiO powder.

EDX of 0.5:0.5 NiS/NiO

The EDX of 0.5:0.5 NiS/NiO is shown in figure SI-2 below.



Figure SI-2 EDX of 0.5:0.5 NiS/NiO