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Ferrite-based Nanoparticles: Synthesis, Characterization, and Non-enzymatic Electrochemical Sensing Applications

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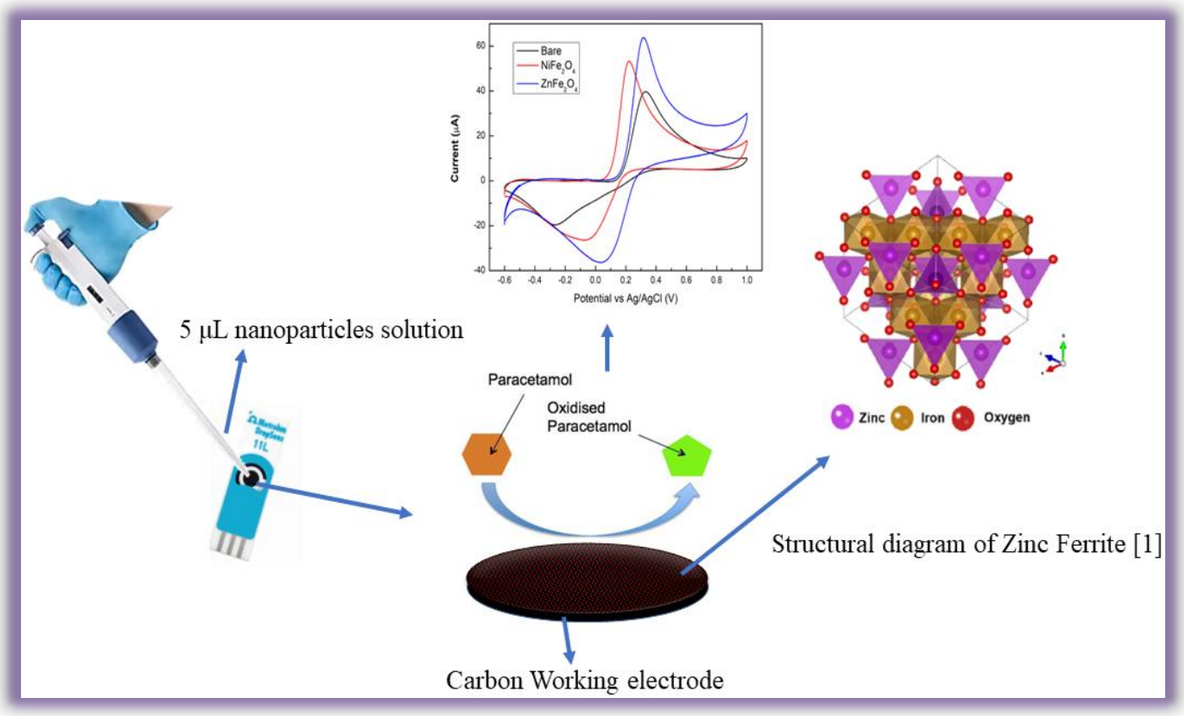
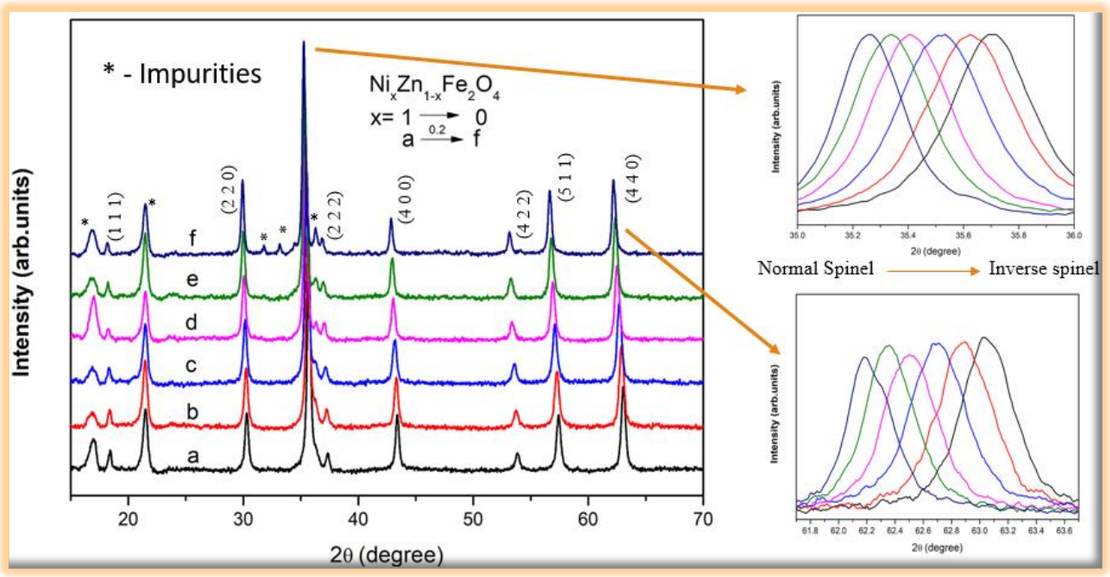
Graphical abstract



X-ray diffraction spectra of Ni-Zn mixed ferrites and zoomed part of the phases (3 1 1) and (4 4 0) clearly show the transition from spinel to inverse spinel.



Materials and methods

Materials
- $Zn(NO_3)_2.6H_2O$
- $Ni(NO_3)_2.6H_2O$
- $Fe(NO_3)_3.9H_2O$
- CH_4N_2O (Urea)
- DI Water, Methanol
- Paracetamol, PBS buffer

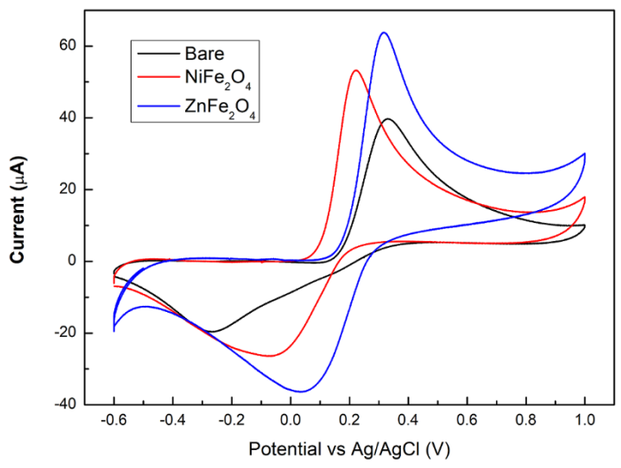
Electrodes modification
- Methanol as solvent
- 3:1 material to solvent
- Carbon working electrode
- 5 μL solution
- Drop casting
- Overnight drying

Synthesis
- Autocombustion 600ºC [2]
- Annealed at 600ºC (2h)
- Cooled and grounded

Electrolytic solution
- Paracetamol
- 0.1M PBS buffer
- pH 6.9

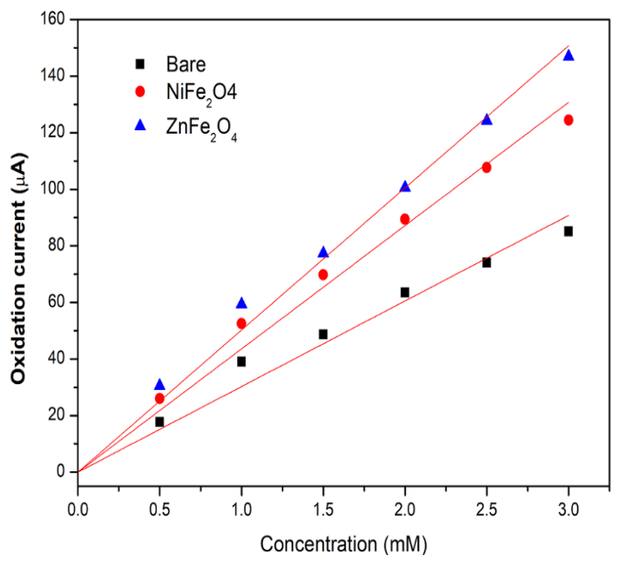
Electrochemical measurements

Cyclic voltammograms of 1mM paracetamol in 0.1M PBS pH 6.9 with different electrodes and their corresponding oxidation currents and potentials.



Electrode	Oxidation Potential (mV ± SEM)	Oxidation Current (µA ± SEM)
Bare	326.80 ± 0.73	39.11 ± 0.16
$NiFe_2O_4$	246.6 ± 3.2	51.53 ± 0.80
$ZnFe_2O_4$	307.0 ± 6.0	59.17 ± 0.63

SEM – standard error mean



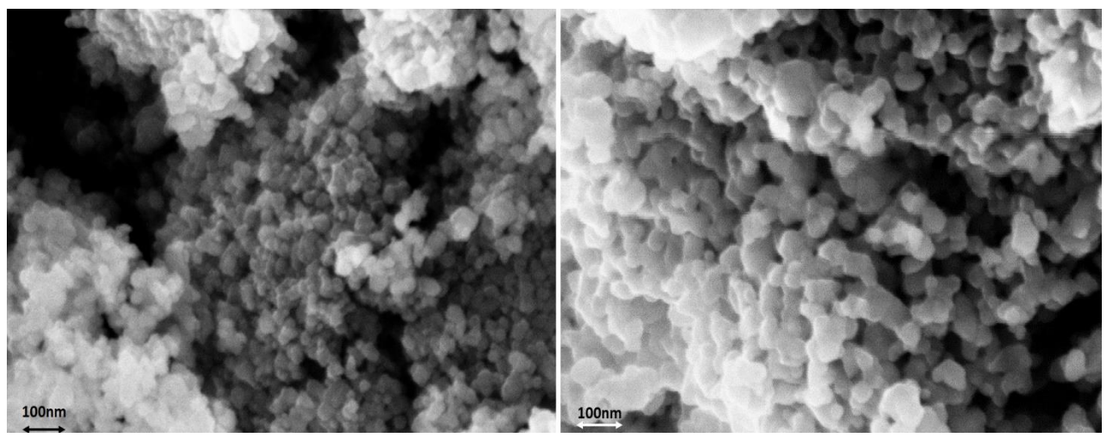
Electrode	Sensitivity (µA/mM ± SEM)	ΔE_p (mV ± SEM)
Bare	30.2 ± 1.0	594.4 ± 1.2
$NiFe_2O_4$	43.6 ± 1.1	290.6 ± 1.3
$ZnFe_2O_4$	50.26 ± 0.98	278.3 ± 2.7

ΔE_p – Peak to peak separation

Calibration curves of 3 different electrodes and the slopes indicate the sensitivity of respective electrochemical sensors.

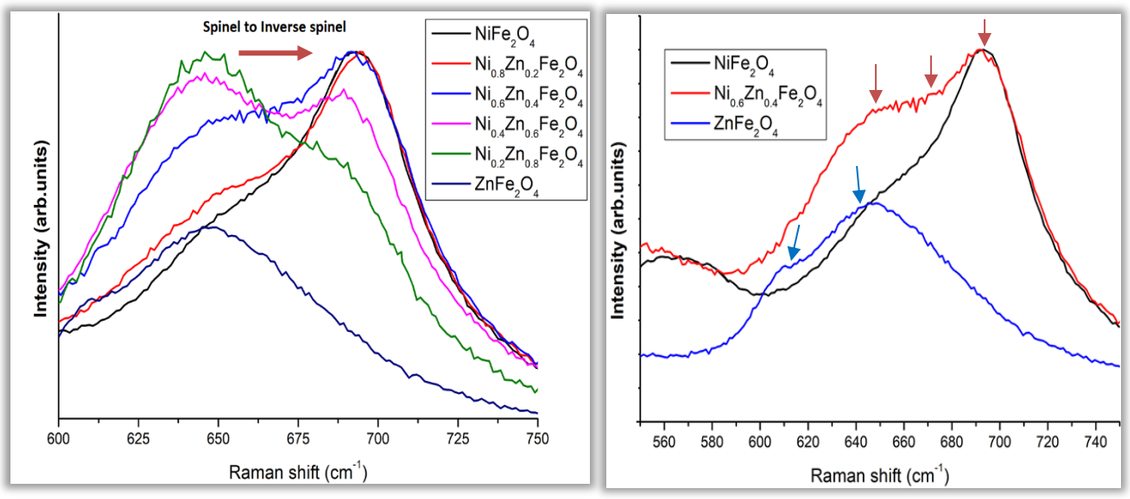
$NiFe_2O_4$ and $ZnFe_2O_4$ have lesser peak to peak separation compared to the bare electrode which gives an indication of faster reaction at the interface leading to higher kinetic rate constant.

Materials characterization



FE-SEM pictures of $NiFe_2O_4$ (left) and $ZnFe_2O_4$ (right).

Spherical shape particles with an average particle size of 30 – 40 nm



Microraman spectra of intensive A_{1g} band showing the transition from spinel to inverse spinel (left). The band shows a doublet and triplet-like shape due to pure and mixed ferrites [3, 4, 5]. The right figure shows three red arrows indicating 3 different molecular vibrations due to the presence Fe, Ni, and Zn whereas two blue arrows indicating only two molecular vibrations because of Fe and Zn.

Future work

Electrochemical measurements of the other mixed ferrite-based sensors.

Calculation of kinetic rate constant (k), electron transfer rate coefficient (α).

Chronoamperometric measurements to calculate the active surface area of the working electrodes.

Computational approach to calculate the kinetic rate constant.

References

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