

CHAPTER 8

Conclusions and Future Scope of Work

8.1 Comparative Summary of Results for the Three Developed Chemical Methodologies:

We have successfully prepared nanostructured α -Fe₂O₃ and series of Ni-Zn ferrite powders by using three chemical routes. The important results have been summarized in Table 8.1 and Table 8.2.

Table 8.1 Comparative summary of results for nanostructured α -Fe₂O₃ synthesized by PVA precursor based method (Method 1), Oxalate precursor based method (Method 2) and EDTA precursor based method (Method 3).

Characterization Technique	Method	Results
Thermal Analysis	Method 1	Decomposition temperature of precursors was almost same (~450 ⁰ C) for all the three precursors in air atmosphere.
	Method 2	
	Method 3	
X-Ray Analysis	Method 1	Formation of single phase α -Fe ₂ O ₃ nanopowder occurred due to calcination of precursors at 450 ⁰ C for two and a half hours in air.
	Method 2	
	Method 3	
TEM Study	Method 1	Average particle size (\pm 5 nm)
		~ 30 nm

	Method 2	~ 25 nm
	Method 3	~ 35 nm
SEM Study	Method 1	Microstructure of synthesized nanoparticles. Particles were elongated in shape. There was presence of agglomeration and porosity in the nanopowders.
	Method 2	
	Method 3	
Room temperature DC electrical resistivity measurement	Method 1	$\sim 10^5 \Omega \text{ cm}$
	Method 2	$\sim 10^7 \Omega \text{ cm}$
	Method 3	$\sim 10^8 \Omega \text{ cm}$

Table 8.2 Comparative summary of results for nanostructured $\text{Ni}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$ ($0 < x < 1$) synthesized by PVA precursor based method (Method 1), Oxalate precursor based method (Method 2) and EDTA precursor based method (Method 3).

Characterization Technique	Method	Results
Thermal Analysis	Method 1	Major decomposition of the precursor occurred at $\sim 550^\circ\text{C}$ in air atmosphere.
	Method 2	Major decomposition of the precursor occurred at $\sim 550^\circ\text{C}$ in air atmosphere.
	Method 3	Complete decomposition of the precursor occurred at $\sim 450^\circ\text{C}$ in air atmosphere.
X-Ray Analysis	Method 1	Formation of single phase Ni-Zn ferrite nanopowder occurred by calcination of precursor at 950°C for two and a half hours in air.

	Method 2	Formation of single phase Ni-Zn ferrite nanopowders occurred by calcination of precursor at 850 ⁰ C for two and a half hours in air.	
	Method 3	Formation of single phase Ni-Zn ferrite nanopowder occurred by calcination of precursor at 450 ⁰ C for two and a half hours in air.	
TEM Study		Average particle size (± 5 nm)	
	Method 1	~ 50 nm	
	Method 2	~ 35 nm	
	Method 3	~ 35 nm	
SEM Study	Method 1	Microstructure study of the as synthesized nanopowders. Particles were round in shape. There was presence of agglomeration and porosity in the nanopowders.	
	Method 2		
	Method 3		
Room temperature DC electrical resistivity	Method 1	~10 ⁶ - 10 ⁷ Ω cm	
	Method 2	~10 ⁷ Ω cm	
	Method 3	~10 ⁵ - 10 ⁷ Ω cm	
VSM measurement		Saturation magnetization M_s (emu/g)	Value of M_s were found to increase with decreasing value of x for the composition $Ni_{1-x}Zn_xFe_2O_4$ ($0 < x < 1$)
	Method 1	49.3 to 61.1	
	Method 2	34.7 to 49.4	
	Method 3	32.4 to 41.9	

8.2 Conclusions:

Based on the summary of results obtained for nanostructured α -Fe₂O₃ and series of Ni-Zn ferrite powders, the following conclusions have been drawn.

1. Three aqueous solution based chemical methods have been developed for synthesis of nanosized, single phase α -Fe₂O₃ and Ni_{1-x}Zn_xFe₂O₄ (0 < x < 1) powders.
2. Advantages offered by these developed aqueous solution based methods that make them attractive are as follows:
 - (i) metal alkoxides or complex metal compounds, which are expensive, difficult to handle, synthesize, and sometimes toxic were not used in the developed methods.
 - (ii) strong acid, base or organic solvents were not used in any of the methods.
 - (iii) use of simple and cheap metal nitrates as starting materials and water as solvent helps in reducing the processing cost as compared to other reported wet chemical methods.
 - (iv) moreover, any elaborate experimental setup is not required for the synthesis of nanopowders by these methods
 - (v) Unlike other reported methods, Zn loss was not observed during high temperature sintering of pellets, prepared by the synthesized nanopowders. This ensures the maintenance of stoichiometry of the final product, which govern the electrical and magnetic properties of the materials.
3. The particle size, electrical and magnetic properties of the nanopowders vary with the chemical methodology that is used for their synthesis. The developed chemical methods can therefore be used to synthesize nanopowders with desirable characteristics.

8.3 Limitations of the developed chemical methods:

Some of the limitations of the developed methods are:

- (i) These methods are not suitable for synthesizing metallic nanoparticles or nanopowders that contain metal ions in lower/unstable oxidation state.
- (ii) These methods are not appropriate to synthesize metal nitrides, carbides.

Future Scope of Work:

1. These methods can be extended to synthesize other nanostructured oxide and multicomponent oxide systems such as TiO_2 , ZrO_2 , BaTiO_3 and $\text{BaFe}_{12}\text{O}_{19}$.
2. The porosity of the as-synthesized nanopowders can be explored for humidity sensor applications.
3. Detailed electrical, magnetic and microwave absorption studies of Ni-Zn ferrite nanopowders should be performed to assess the potential of these as-synthesized nanopowders for applications such as cores, inductors, EM absorbers, sensors and bio-medical applications.