“MICROWAVE SYNTHESIS AND CHARACTERIZATION OF FERRITES”

PROJECT REFERENCE NO. : 37S0352

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Keywords: Synthesis, Microwave, Ferrites, Nitrates

Introduction:

One of the most important problems of modern materials science is the development of new methods for preparing functional materials, which allow one to decrease power inputs and synthesis time. Microwave treatment is a promising method that satisfies these demands. Microwave heating has some advantages over common methods for heating solids and liquids. Among these advantages, noteworthy is the high rate and low thermal lag, the absence of the contact between a heated material and a heater, the uniformity of bulk heating, the possibility to selectively heating components in mixtures, and the high efficiency. The absorption of microwave radiation by substances occurs through two basic mechanisms associated with rotation of dipoles and ionic currents. Therefore, the substances with a high permittivity or ionic conductivity absorb microwaves most efficiently. Materials with a low dielectric loss factor and insulators can be heated after adding compounds that absorb microwaves, for example, magnetite, silicon carbide, or graphite. In salt systems, water of crystallization may act as such an “additive”.

Over the past few years, microwave treatment has been used for preparing complex oxides (CuFe₂O₄, YFe₃O₁₂, La₀.7Ba₀.3MnO₃). These works have shown that this treatment is a promising
method for synthesis of functional materials. Note, nevertheless, that the authors of the majority of the above works used oxides as starting reagents. This severely constrains the scope of objects that can be synthesized with the use of microwaves, because many oxides interact poorly with microwave radiation at room temperature. In this work, we have proposed a method for synthesis of ferrites with structures of the types (spinels MFe₂O₄, M = Co, Ni, Cu; and orthoferrite LiFeO₂ with a NaCl structure) based on a microwave treatment of metal salt mixtures.

**Objectives:**

The project principally involves microwave-assisted synthesis of lithium, copper, and nickel ferrites and their characterization.

The specific objectives are:

- To take stoichiometric quantities of metal nitrates with iron nitrate and synthesize ferrites by microwave heating
- To take stoichiometric quantities of metal nitrates with iron nitrate and synthesize ferrites (check samples) by conventional heating
- To analyse the XRD patterns of ferrites synthesized by microwave and conventional heating and conclude

**Methodology:**

Ferrite is a ceramic material formed by reacting metal oxides into a magnetic material. Ferrites are, like most other ceramics, hard and brittle. In terms of their magnetic properties, the different ferrites are often classified as "soft" or "hard", which refers to their low or high magnetic coercivity.

Analytical grade Fe(NO₃)₃·9H₂O, Ni(NO₃)₂·6H₂O, and Cu(NO₃)₂·6H₂O and reagent grade Li₂CO₃ were used as starting reagents. The salt mixtures were prepared taking into account the stoichiometry of reactions of various M-nitrates with Fe(NO₃)₃·9H₂O. The salt mixtures were ground and mixed well in an agate mortar, placed in re-crystallized alumina crucibles, and heated in a microwave furnace to 650°C at the rate of 15°C / min, soaked at 650°C for 5 min and cooled to room temperature. To obtain check samples, the salt mixtures were kept for 10 min in a muffle furnace preheated to 650°C and then cooled to room temperature. The phase compositions of the
samples were determined by X-ray powder diffraction and the XRD patterns were analyzed with the use of the JCPDS Database. The microwave furnace and muffle furnace used are shown below:

![Microwave furnace](image1.png) ![Muffle furnace](image2.png)

**Results and Conclusions:**

The ferrite samples synthesized by microwave heating and by conventional heating is shown below:

![Ferrites – Microwave synthesized](image3.png) ![Ferrites – Conventionally synthesized](image4.png)
The X-ray diffraction patterns of the microwave synthesized and conventionally synthesized ferrites are shown below:
It has been observed (using JCPDS indexing) that microwave ferritization is better than ferritization by conventional heating in the case of copper and nickel ferrites, in which both the starting materials used were nitrates. This is due to the fact that nitrates are microwave susceptible and hence yielded maximum ferritization. However in the case of lithium ferrites, one of the starting materials used is not nitrate. Complete ferritization has not taken place in both microwave route and conventional heating route.

**Scope for future work:**

Microwave synthesis is a promising method to decrease power inputs and synthesis time. It is efficient and hence can be used to synthesis functional materials economically. Modern microwave furnaces give precise control over the heating and cooling schedules for the samples. Temperatures as high as 1800°C can be easily attained with microwave heating and hence microwave heating can be employed for sintering studies. Zeolites, which are very valuable, can be effectively and economically synthesized from fly-ash, a waste product of thermal power plants.