

Chapter I

Introduction



In the search for improved properties of catalytic materials, great interest has focused on spinel-type structures. Among these materials, aluminate seems to be a good option because of their properties such as high thermal stability, high mechanical resistance, hydrophobicity, and low surface acidity. These properties make them interesting materials as catalysts and carriers for active metals to substitute the more traditional systems. Furthermore, it is known that some aluminate spinels, including zinc aluminate, tend to prevent sintering of noble metals due to a strong metal-support interaction [3,4,9]. The sintering resistance and chemical stability of catalytically phases are a very important problem for high-temperature processes.

Zinc aluminate spinel $ZnAl_2O_4$ may be used as well as a ceramic material. So far, it has been used as a catalyst for the double bond isomerization process of alkenes[3,4,9], for the dehydrogenation of saturated alcohols to olefins [3], preparation of polymethylbenzenes [3], methanol and higher alcohols synthesis [3], or the synthesis of styrenes from acetophenones[4], and foremost as a support for alkane dehydrogenation catalyst[4]. Zinc aluminate catalysts or supports and ceramics are commonly prepared by high temperature calcination (above 1000 K) of mixed aluminium and zinc oxides [9], coprecipitated products [9] or products of impregnating a porous alumina having a high surface area with a solution of zinc compound [9]. A disadvantage of such materials for catalytic applications is the low surface area, usually about $20\text{-}50\text{ m}^2\text{g}^{-1}$ [3], where conventional catalyst or supports are porous materials whose surface area require comprised between $100\text{ and }300\text{ m}^2\text{g}^{-1}$.

Zinc gallate (ZnGa_2O_4) is a double oxide with a spinel structures and is one of the new transparent and conductive materials. Many fundamental studies have been reported, particularly on the electrical and optical properties of ZnGa_2O_4 . In addition, ZnGa_2O_4 is also useful as a powder application for phosphors and has been investigated for its excellent properties for cathodoluminescent material. There is a growing interest in ZnGa_2O_4 as low-voltage cathodoluminescent phosphor for field emission displays (FEDs) and vacuum fluorescent displays (VFDs)[5,6,7,8]. Additionally, it is a possible alternative to the ZnS-based low-voltage cathodoluminescent phosphor presently used in VFDs.

In most of the synthetic processes, the alkoxides are hydrolyzed in an alcoholic solution yielding amorphous (or hydrated metal oxide with large-surface areas; however their surface areas are drastically decreased on calcinations at the temperatures where corresponding oxide begin to crystallize). Recently, Inoue et al. have examined the thermal reaction of metal alkoxide in glycols (glycothermal reaction) or other organic media and demonstrated that a number of novel or characteristic crystalline products can be obtained directly without bothersome procedures such as purification of the reactants or handling in inert atmosphere.

As mentioned above, the novel method for the synthesis of metal oxides in organic media may be a new route to prepare micro- and nanocrystalline metal oxides. Physical properties of the product can be controlled by the choice of the organic solvent, reaction conditions, and strengthen of the alkyl group of the metal alkoxides. In this study, the novel method was applied in the system zinc gallate and zinc aluminate and focus on:

1. The effect of starting Zn/Ga and Zn/Al molar ratio on the physical properties and thermal stability of zinc gallate and zinc aluminate samples.

2. The effect of the reaction of crystalline formation (type of organic solvent) on the physical properties and thermal stability of zinc gallate and zinc aluminate samples.

The present study is arranged as followed:

Chapter II presents a literature reviews of the novel synthesis of several metal oxides in organic media.

The theory related to this work, physical and chemical properties of zinc, zinc oxide, zinc gallate, zinc aluminate, preparation processes, zinc oxide uses, gallium, gallium oxide, gallium oxide uses, metal alkoxide, spinel structure, and others are described in chapter III.

Chapter IV presents the experimental systems and the catalyst preparation.

Chapter V shows the experimental results of the characterization of catalyst samples. The X-ray Diffraction (XRD) patterns, BET surface area, crystallite size, morphology and chemical composition of the products are explained.

In the last chapter, the overall conclusion emerged from this research is given.

Finally, the sample of calculation of the catalyst preparation, crystallite size, and chemical composition are included in APPENDICES at the end of this thesis.