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Synthesis and Characterization of Nickel and Zinc Substituted Alumina

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Abstract: **These Nan composites are synthesized by Sol-Gel process named as Nickel-Alumina and Zinc-Alumina. They are calcined and sintered with high temperatures at 1000°C and are characterized by XRD and FTIR techniques to calculate crystalline size and lattice strain %. The following conclusions are drawn: Sol-Gel process via high temperature sintering methods is favourable for crystalline phase for both nickel and Zinc-Alumina and the sintering temperature supports the formation of MAl_2O_4 spinal Aluminates bulk Phases(M= Ni, Zn).**

Keywords: **Characterization of Nickel, Zinc Substituted Alumina, Nan composites.**

I. INTRODUCTION

Nan crystalline ceramics are attracting an increasing interest nowadays with much attention because they have a variety of interesting and novel physical properties which lead to prepare different kind of Nan composites. The properties of Nan composite materials will be depend on the properties, particle-size, surface area; Morphology, and Interfacial characteristics of individual parents to enhance its reactivity strength and electrical properties etc.

Sol-Gel method enables homogeneous samples to be obtained at low temperatures and the starting cationic composites to be maintained by using metal salts as raw material and mixing them in a liquid solution. The most obvious advantage of this Sol-Gel method is that reagents are mostly mixed in atomic level, which may increase the reaction rate and decrease the synthesis temperature [5].

The materials like $ZnAl_2O_4$ and $NiAl_2O_4$ [6,7] found enhanced properties such as thermal stability at elevated temperatures. The ability to scale up the synthesis to bulk scale will gain increasing importance as more applications are established.

The present investigation is envisaged to generate basic data on the nano composites of Nickel and Zinc-Alumina by adopting standard method to study

Synthesis by Sol-Gel process of Alumina with Nickel and Zinc. Preparation of Nan particles of the above composites at different speeds to study characters. Characterization of the composites of different combinations by the following methods: Crystalline by XRD, Lattice strain %, IR-bonding, the techniques employed was XRD analysis, and FTIR techniques.

II. EXPERIMENTAL PROCEDURE

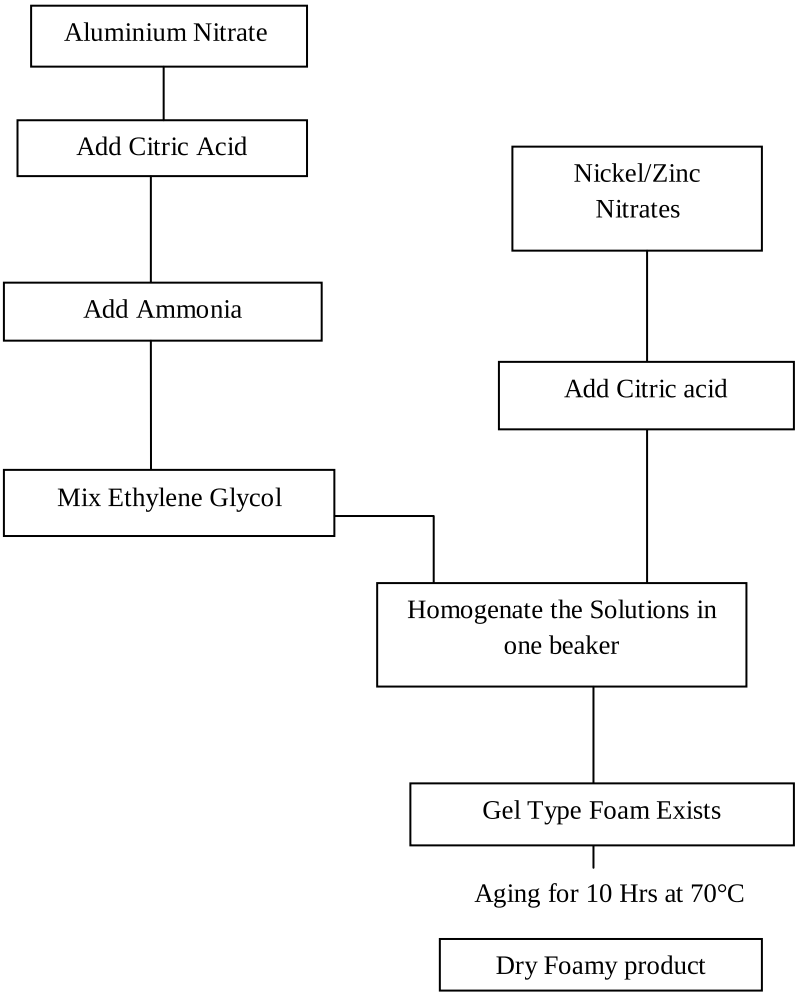
Nickel and Zinc Nitrates are mixed with Aluminum Nitrates were prepared by mixing calculated portions of Zinc Nitrate and Nickel Nitrate with definite amounts of citric acid . To prepare Nickel and Zinc alumina through Sol-Gel process on every combination 100 ml Ammonia and 20 ml Ethylene glycol is mixed. And the table with amounts of materials and Flow sheets were reckoned below.

Tables with materials:

Aluminium Nitrate $Al(NO_3)_3$	Nickel Nitrate $Ni(NO_3)_2$	Zinc Nitrate $Zn(NO_3)_2$	Citric acid
12.50	9.69		57.6
12.50		9.91	57.6

General Flow sheets of Ni and Zn/ Al:

SOL-Gel Process for Nickel and Zinc Substituted Alumina



Calcination Process:

The obtained materials are calcined at 600^0C for 3 hours, to remove the nitrates and unwanted solvents.

Sintering process:

The precalcined Nickel and Zinc – Alumina composites are chosen for sintering process. The Nanocomposites were subjected to IR-spectra and XRD analysis. The sintering process was operated at 1000^0C for 6 hours.

III. RESULTS & DISCUSSION

Preparation of precursor Sol-Gel and calcinations to avoid porosity and agglomeration. The work done for this project has been divided into two sections. First section consists of synthesis of nanocomposites by adopting Sol-Gel method and calcinations of the two composites. Second section deals with the characterization of Nan composites through the properties such as crystallinity, lattice strain and IR absorbency by adopting the techniques XRD and FTIR.

Section I:

General Observation:

The dissolution with Ethylene glycol: citric acid (EG:CA) and Inorganic metal salts with low temperatures makes the precursor powders predominantly a mixture of homogeneously distributed metal oxides in an intermediate single phase compound with stoichiometric of the metal ions. The viscosity of the solution drastically increases during the polymer chain augmentation. The nature of the cations affect the rate of viscosity increases, which indicates that metal ions play an important role in cross-linking the polymer chains by means of complex formation. During the first stage of the polymer growth in the solution it provides necessary environment to prevent cation segregation, and later relatively rigid polymer network traps cation preserves the initial homogeneity of the solution.

Citric acid is well soluble in Ethylene Glycol, which provides a wide range of CA: EG ratios with the sol-gel process and makes it possible to tune the conditions of synthesis for each particular system. Chemical Interaction between Citric acid and Ethylene Glycol with metal nitrates are occurs in the room temperature where the used ammonia is used as a fastest evaporation of vaporizable phases. The solvent removal and the nitrates are decomposed during the low temperature range of decomposition. The gelation, with a slow grain growth and a particular phase of alumina and copper, zinc, Nickel oxide substituted alumina's are formed during the Sol-Gel synthesis. he obtained precursors are calcined to remove the Nitrates, particular dissolved solvents and wet content of the composites, so they are calcined at 600^{o}C for 3 hrs.

Section II:

High Temperature Sintering process and characterization by XRD and IR spectra:

After the preparation of precursor of the four composites by Sol-Gel process, the precursor is subjected to the calcined process at 600°C for 3 hours and allowed the same to the sintering at 1000°C for 6 hours. We obtained the XRD patterns and FTIR measurement for all the (Ni-alumina, Zn-alumina) composites to characterize them.

Precalcined Nickel-Alumina:

The precalcined precursors $NiOAl_2O_3$ are calcined at 1000°C and analysed with X-ray diffraction analysis and IR spectra to characterize.

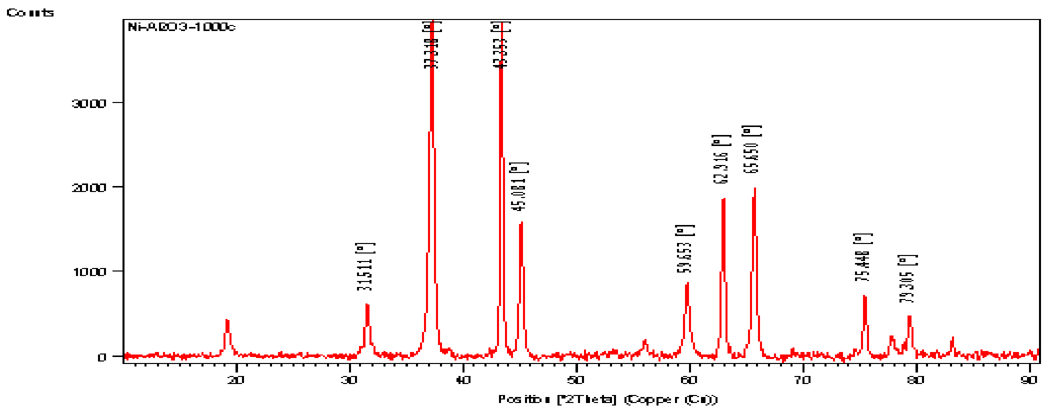


Fig 1.a XRD image of precalcined $NioAl_2O_3$ at 1000°C

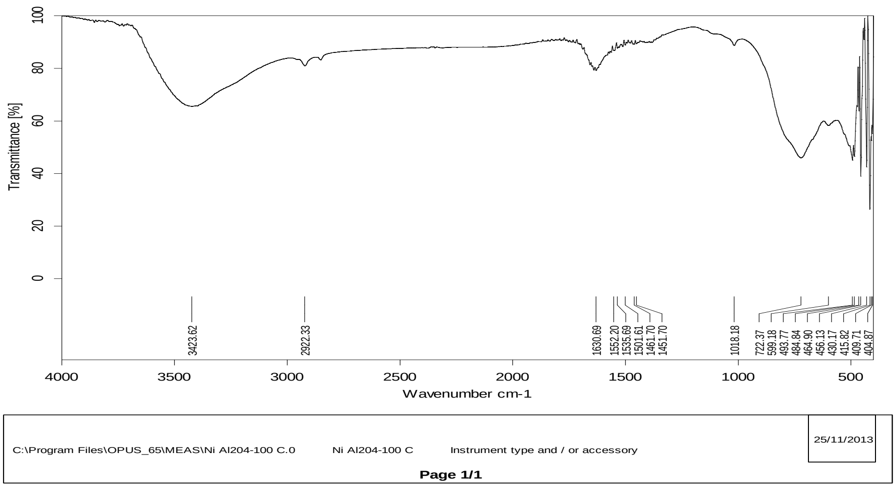


Fig 1.b IR-Spectra 500-4000 cm^{-1} of precalcined $NioAl_2O_3$ at 1000°C

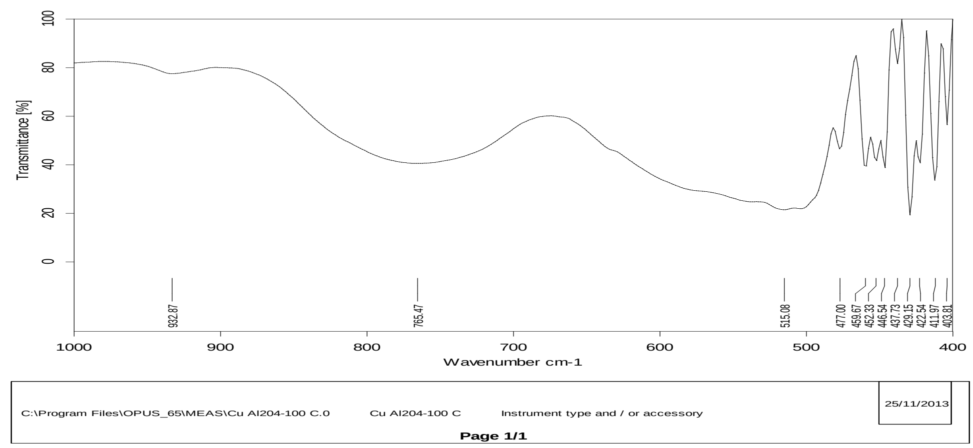


Fig 1.c IR-Spectra 400-1000 cm^{-1} of precalcined NioAl$_2$O$_3$ at 1000°C

Figure 1.a show the XRD patterns obtained for NiOAl$_2$O$_3$ powder prepared by Sintering process at 1000°C for 6 hours. All the peaks in the XRD pattern are very sharp showing the well crystalline behaviour of the heat treated powder. Obtained XRD pattern is compared with reported data. The identified phases present in the patterns of NiOAl$_2$O$_3$ are of cubic spinel-type (ICDD: 71-0963) [4]. The crystallite size calculated for NiOAl$_2$O$_3$ are small reported earlier (nearly 14 nm) whereas the crystallite size obtained from our XRD pattern is 33.3nm with lattice strain 0.342%, which is coinciding the particulate size of crystallite with urea as fuel. FTIR spectra are also support the same pattern.

Precalcined Zinc alumina (ZnOAl$_2$O$_3$):

The precursor was calcinated at1000°C. Then Crystallite size and Lattice Strain% are determined by X-ray diffraction (XRD) analysis recorded on an X-ray diffractometer system using CuKα radiation and absorbency with FTIR

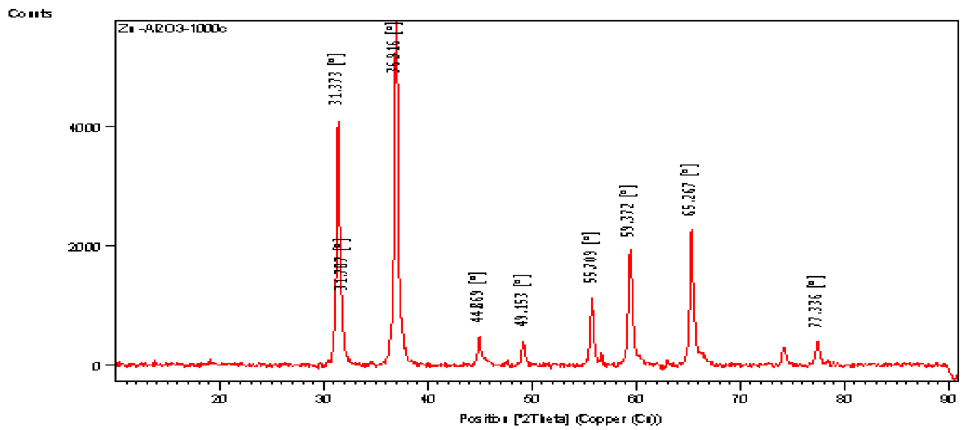


Fig 2.a XRD image of precalcined ZnoAl$_2$O$_3$ at 1000°C

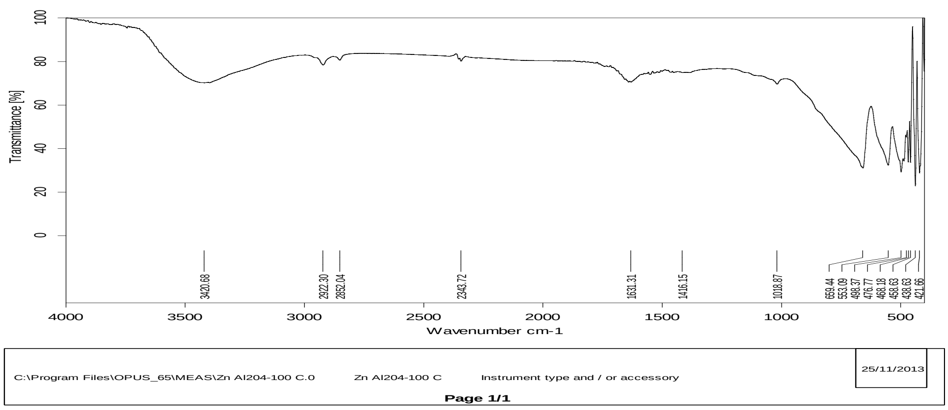


Fig 2.b IR-Spectra 500-4000 cm^{-1} of precalcined ZnoAl$_2$O$_3$ at 1000°C

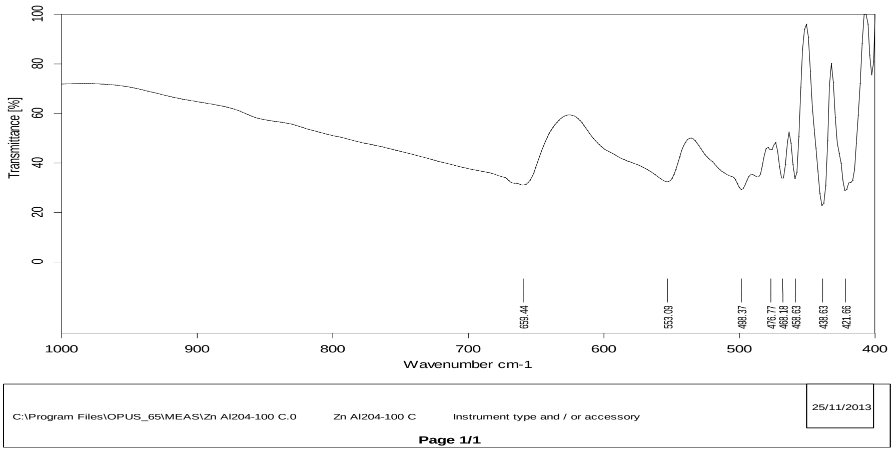


Fig 2.c IR-Spectra 400-1000 cm^{-1} of proclaimed $ZnoAl_2O_3$ at 1000°C

Figure 2.a represents the XRD patterns the compound $ZnOAl_2O_3$. All peaks of the diagram are well defined pointing to the high crystalline nature. All the diffraction peaks in good match with the reported data of Abdul Jamal et al 2011[3]. It is also supported the formation of spinel phase. The crystalline size is determined with Scherer method as 32 nm with lattice strain% 0.407. The FTIR spectrum is presented in the figure 2.b and 2.c a broad absorption band is observed at 3420 cm^{-1} and it indicates to OH group, which is an indication of slight water content in the crystallites. The bands at 1630 cm^{-1} 1410 cm^{-1} can be attributed to OH group in the metal alkoxides present in the gel [3]. Three sharp absorption peaks visible in the figure5.4.4c at 650, 550 and 498 cm^{-1} arise from the stretching vibrations of tetrahedral and octahedral bonds in the spinel [3] a small absorption peak noticed at 2343 cm^{-1} may be attributed to the presence of oxygen-oxygen bonds in the FCC crystal lattice of oxygen atoms [3].

Tabular form of the peak position, crystallite Size and lattice Strain (%)

No	Element	B.Obs [2Th]	BStd [2Th]	Peak position [2θ]	B struct 2θ	Crystallite size [A°]	Lattice Strain [%]
1	$NiAl_2O_4$-1000°c	0.317	0.060	43.353	0.257	333	0.342
2	$ZnAl_2O_4$-1000°c	0.317	0.060	36.916	0.257	326	0.407

IV. CONCLUSION

Nanocomposites are prepared by Sol-Gel procedure named as Nickel Alumina and Zinc Alumina. The calcined composites are sintered at high temperatures i.e., 1000^{0}C. And they are characterized through XRD and FTIR spectra. Finally crystalline size and lattice strain % are calculated using Scherer's formula. Based on the above experimental data, XRD patterns and FTIR spectrum, the following conclusions are drawn from the study. XRD patterns of $NiAl_2O_4$ have shown that there is a formation of cubic spinel type with crystalline size of 33 nm with lattice strain % 0.342. XRD patterns of $ZnAl_2O_4$ supports that the formation of spinel Zinc aluminate particle with average crystalline size of 32nm with lattice strain % 0.407. FTIR spectra also support the structure spinel. The above conclusions are subjected to the range of concentrations and ratios of the chemicals are used in the present study.

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