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SYNTHESIS, CHARACTERIZATION AND CATALYTIC APPLICATIONS OF NICKEL AND COBALT ALUMINATE NANO-CATALYST

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ABSTRACT

The spinel $NiAl_2O_4$ and $CoAl_2O_4$ nano-aluminates have been efficiently synthesized by using sol-gel combustion heating method by using glycine fuel. The nano structured nickel aluminate and cobalt aluminate spinels were confirmed by the calcination reaction temperature. The influence of Ni^{++} and Co^{++} ions over Al_2O_3 forces system, leads to the great position in the substitution and development on thermal, spectroscopic, structural, morphological then catalytic behaviors of nickel and cobalt doped aluminates have been studied. It was observed that the synthesized aluminate material acts as effective nano-catalyst for the conversion of organic compounds. Therefore, these practices were confirmed to be useful for investigation of the deliberated structures which produced the number of outcomes.

KEYWORDS

Sol-Gel Combustion Method, Nickel Aluminate, Cobalt Aluminate and Catalytic Applications.

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INTRODUCTON

Oxide spinels are a large category of compounds of mixed-metal oxides crystallizing into AB_2O_4 kind structure. These systems are of huge technological importance because of their numerous applications in sensors, transducers, microelectronics, magneto electronic devices, telecommunication systems and industrially important catalytic and photo-catalytic reactions. The nano-crystalline aluminates with the general formula MAl_2O_4 i.e. M = nickel, zinc, manganese, cobalt and magnesium so on captivates the analysis interest associated to their useful practical applications. Metal aluminates are a category of ternary metal oxides having a general formula MAl_2O_4, wherever M may be a bivalent metal particle. These materials possess interesting properties like high chemical, thermal and mechanical stabilities and even have fascinating electrical, magnetic and optical properties that make them appropriate for applications like photo-catalysis, sensing, optoelectronic devices, displays and imaging etc.

Their superior and impressive physico-chemical properties, namely high thermal and chemical stability, high mechanical resistance, optical uniqueness, hydrophobicity and low surface acidity have been the body of comprehensive theoretical and practical studies[1-13]. The Nickel and Cobalt aluminates is the sort of inverse spinel with the Ni^{++}or Co^{++}ion preferentially distributing over the octahedral site. Nickel and Cobalt aluminate spinel are often ready by several strategies such as solid-state reaction, sol-gel combustion technique, particle changed zeolites technique and microwave-induced technique[14-20]. In material chemistry, nickel aluminate is being considered in catalytic applications and proposed as an aspirant material in high-temperature fuel cells[21]. $NiAl_2O_4$is also a potential runner in the metal-ceramic composite because of its excellent strength and wettability with metal[22]. Cobalt blue is brighter and smaller number concentrated than the iron-cyanide centered pigment like Prussian blue. It is very stable and usually been used as a coloring agent in porcelains, nail paints, ceramics, jewelry, transparent glassespaint, cements, phosphor, papers, fibers, different colours and also unique optical properties tends to its wide-ranging use as colour filter for automotive lamps or pigment layer on luminous constituents in optical devices[23-28]. The colouring performance of the cobalt aluminate pigments is strongly depends on its thermal stability, chemical reactivity and coordination of Co^{++} ions within the spinel structure[29]. In this way, the rational fields of materials science, this research article has focused on the synthesis of aluminate spinel through sol-gel combustion method, describe them by different characterization and test out their synergist catalytic properties.

SYNTHESIS OF NICKEL AND COBALT ALUMINATE

MATERIAL AND METHODS

For the synthesis of cobalt doped nickel aluminate spinel system, commercially purchased chemicals of analytical grade obtained from Merck, India received and were used without further purification. Aluminium nitrate, Nickel nitrate, Cobalt nitrate and Glycine were used as the fuel. The samples were prepared with the addition of Ni^{++} and Co^{++} the molar ratios.

Experimental

The sol-gel combustion process was adopted for the synthesis of the nickel aluminate and cobalt aluminates spinels by using glycine fuel. The spinel systems nano-catalysts were successfully prepared by a simple sol-gel combustion method. All the aluminate spinel systems were formed at the higher temperature 900°C.

CHARACTERIZATION OF NICKEL AND COBALT ALUMINATE

TGA analysis of the samples

The degradation of the compounds was administrated by TGA study for the weight loss, thermal behavior and structural destruction of the synthesized aluminate spinel systems $NiAl_2O_4$ and $CoAl_2O_4$ with in the calcination treatment. The final weight loss of the samples occurs at temperature 500-800ºC regions which confirmed that the oxidation treatment above 800ºC acts as calcination temperature because of the dehydration by oxidation as a complete decomposition of the samples.

FTIR study of the samples

FTIR results indicated that the decrease in intensity of the absorption band thereby suggesting the occupation of Al^{3+} ions at the octahedral B site. FTIR spectrums showed the characteristics peaks spinel section just about 900-500 cm^{-1} ranges that ensure the cuboidal spinel structures. The metal-oxygen (M−O) stretching frequencies are reportable within the range 900 cm^{-1} to 500 cm^{-1} accompanying with the vibrations of Al−O and M−O−Al (M=Ni, Co) bonds.

Structural morphology of the samples

The surface morphology of the samples was achieved at desired magnification with a Joel JSM 6360 scanning electron microscope (SEM) equipped with energy dispersive X-ray for elemental composition analysis. The structural and morphological of aluminate samples nanoparticles were confirmed by the SEM images of both the aluminate samples 20kV at 500 magnifications for 50μm range respectively as shown in the Figure No.2. Consequently it was found that, the most important particle size distributions were the characteristic ranges which were measured and comparatively good appearances for the mixed metal aluminate spinel systems.

CATALYTIC STUDY OF THE SYNTHESIZED ALUMINATES

Catalytic Study of the Samples by Homogeneous Phase

The study of nickel aluminate ($NiAl_2O_4$) and cobalt aluminate ($CoAl_2O_4$) catalyst utilized on the hydrogen peroxide (H_2O_2) decomposition was succeeded under pseudo-first order state. Reaction was studied by titration with the un-decomposed H_2O_2 with standardized $KMnO_4$ solution at different time intervals. The impact of cobalt aluminate nanoparticle on H_2O_2 decomposition was successively studied and the rate constants were calculated by using equation,

$$k = \frac{2.303}{t}\log_{10}\frac{a}{a-x}$$

Where,

a = Initial concentration of H_2O_2,

(a-x) = concentration of H_2O_2 at time *t*

The temperature influence was conjointly calculated for $NiAl_2O_4$ and $CoAl_2O_4$ at four totally different temperatures and also the rate constants are calculated. From these rate constants and temperatures the energy of activation value was studied by using Arrhenius equation

$$2.303\log_{10}\frac{k}{k1} = \frac{Ea}{R\frac{T2-T1}{T1\times T2}}$$

The rate constant (k) values for influence of $CoAl_2O_4$ content on H_2O_2 decomposition is 0.8×10^3 sec^{-1} and 0.8×10^3 sec^{-1} with the effect of temperature on $NiAl_2O_4$ and $CoAl_2O_4$ catalyzed H_2O_2 decomposition at 27°C and 29°C with Ea = 50.64 ± 1 kJ mol^{-1}.

Catalytic Study of Samples Heterogeneous Phase (With Bet)

In the present study, the liquid phase catalytic reaction of phenyl methanol into benzaldehyde was applied using the $NiAl_2O_4$ and $CoAl_2O_4$ nanoparticles as a catalyst. The catalytic studies revealed that the character and concentration of the dopant ions had a considerable effect on each the conversion and products selectivity. Mostly, the catalyst with a high specific covers integrates a hopeful result on the catalytic activity surface area and also the absorbance capacity of spinel $NiAl_2O_4$ and $CoAl_2O_4$ nano-catalysts, BET analysis was carried out as shown in the Figure No.3. BET area studies seeming the specific relationship between the concentration of the sorbate and its sorption gradation point into the adsorbent surface of the samples.

The reaction was accomplished by the equal quantity of 10m mole phenyl-methanol, 10m mole hydrogen peroxide and 10m mole of acetonitrile heated in the three naked round bottom flask refluxed on the temperature 80ºC for few hours maintained by using the 0.5g of cobalt aluminate nano-catalyst samples. The BET surface area, conversion and selectivity percentage for the oxidation of the phenyl-methanol to benzaldehyde were confirmed by the 38.96 m^2/g, 87.69% with 100% selectivity in the reaction conditions by the application of cobalt aluminate nano-catalyst. GCMS was carried out to identify the transformation and percentage of the product formation. Thus, it is proposed that the samples nano-particles with very smaller particle size distribution would be the most likely economical catalyst for the chemical reactions. The catalytic studies showed that the character and concentration of the dopant ions consumed the considerable influence on each the conversion and products selectivity with the BET surface area in the heterogeneous reaction phase were recorded in the Table No.1.

The catalyst with a high specific incorporates a favorable result on the catalytic activity surface area and also the absorbance ability of nano-catalysts carried out by BET analysis. BET area exposes the evident and accurate relation between the concentration of the sorbate and its sorption point against the adsorbent surface of the samples. The graphical representation of the BET surface area, conversion and selectivity percentages of the samples illustrated below;

RESULTS AND DISCUSSION

In this study, the aluminate spinels systems were synthesized by sol-gel combustion synthesis technique. TGA was carried out for the sol-gel precursor. The weight loss was offensively negligible at on highest temperature of 900 ºC that indicates that the synthesized aluminate spinels was nearly stable and TGA curves showed the single part material FTIR study indicated that the decrease in intensity of the absorption band thereby suggesting the occupation of Al^{3+} ions at the octahedral B site.

The metal-oxygen (M−O) stretching frequencies are reportable within the range 900 cm^{-1} to 500 cm^{-1} accompanying with the vibrations of Al−O and M−O−Al (M=Ni, Co) bonds. The structural and morphological study of spinel aluminate samples nanoparticles consequently found that, the most important particle size distributions were the characteristic ranges were measured and relatively good appearances for the mixed metal aluminate spinels. In the homogeneous reaction, the Nickel aluminate ($NiAl_2O_4$) and cobalt aluminate ($CoAl_2O_4$) catalyst utilized on the hydrogen peroxide (H_2O_2) decomposition was succeeded under pseudo-first order state by using potassium permanganate. The catalytic activities of aluminate spinels by heterogeneous liquid phase catalytic reaction of phenyl-methanol into benzaldehyde were applied using the $NiAl_2O_4$ and $CoAl_2O_4$ nanoparticles as a catalyst.

Table No.1: BET surface area, conversion and selectivity aluminates

S.No	Nano Catalyst	BET Surface area (m^2/g)	Conversion (%)	Selectivity (%)
1	$NiAl_2O_4$	57.34	87.12	100
2	$CoAl_2O_4$	40.61	86.73	100

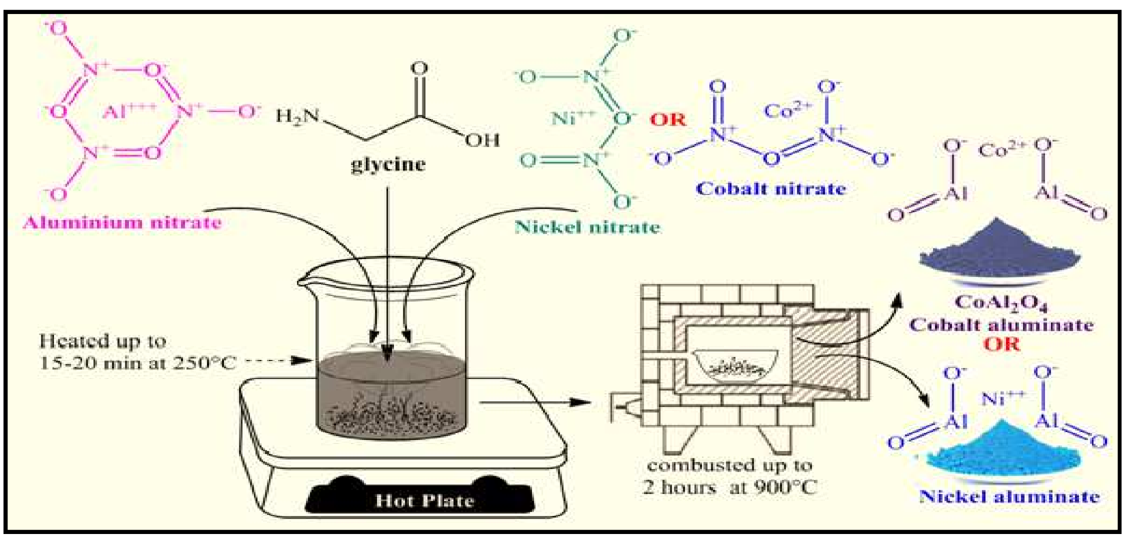


Figure No.1: Synthesis of Nickel and Cobalt Aluminates

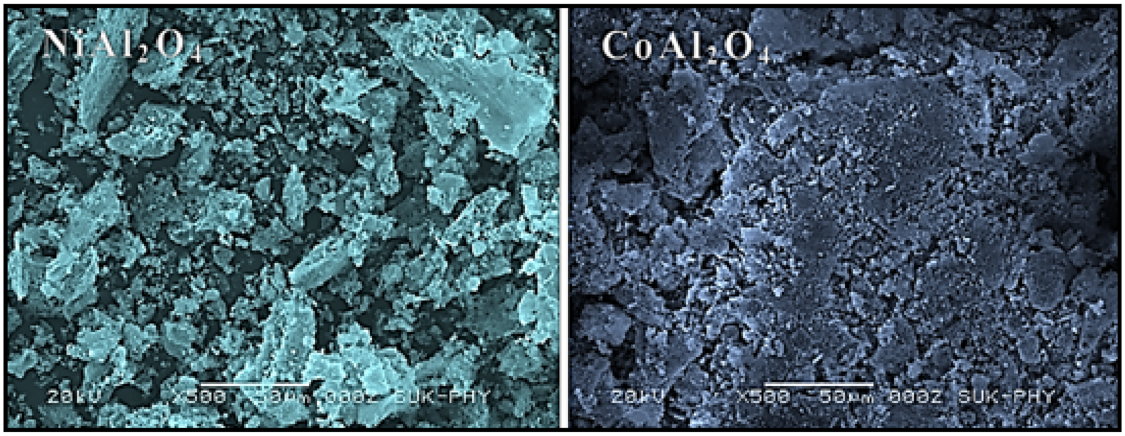


Figure No.2: SEM Images of the Prepared Aluminates

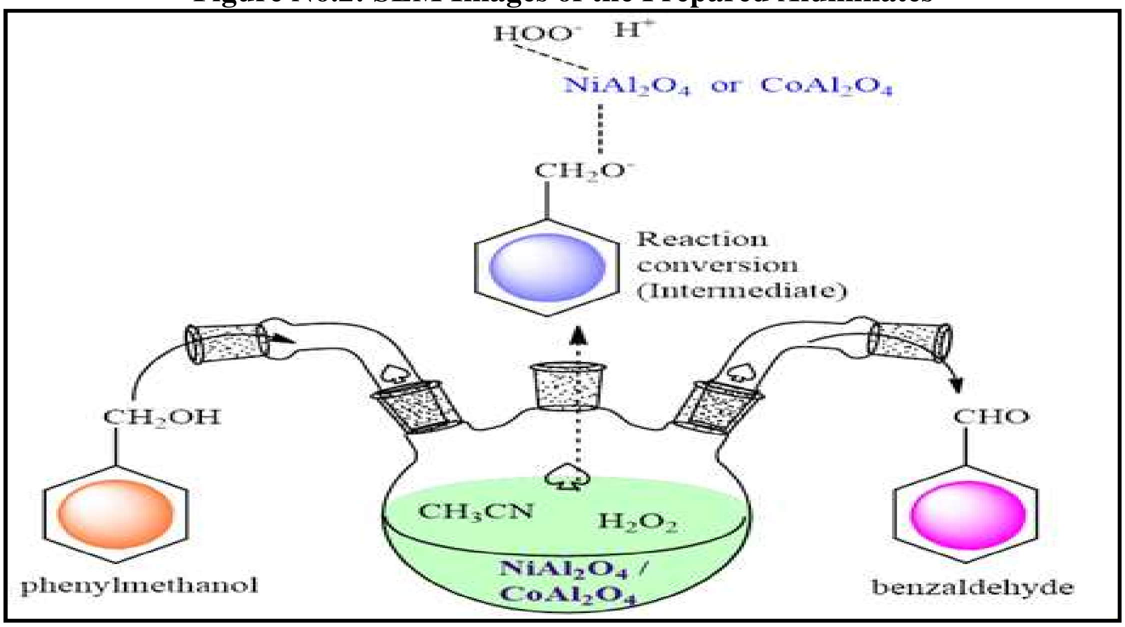
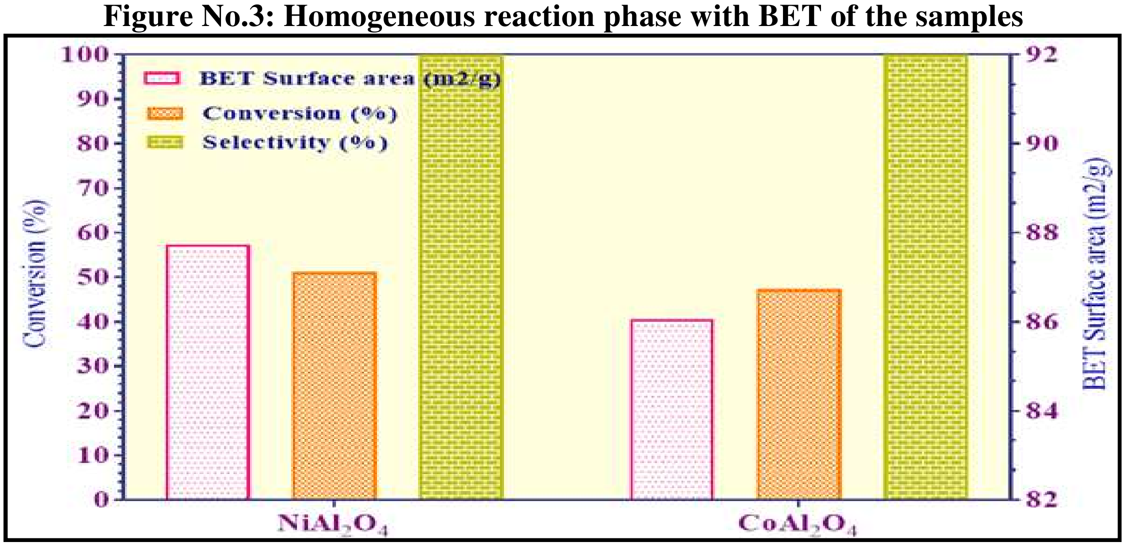


Figure No.3: Homogeneous reaction phase with BET of the samples



Graph No.1: BET surface area, conversion and selectivity percentages of aluminates

CONCLUSION

The Nickel aluminate ($NiAl_2O_4$) and cobalt aluminate ($CoAl_2O_4$) spinels were successfully synthesized by sol-gel combustion synthesis method and confirmed by TGA, FTIR and SEM studies. It has been demonstrated that the characterization properties of every single property such as, structural, spectral, morphological and catalytic activities of the prepared nanoparticles were studied. It was also revealed that the synthesized aluminate material also acts as efficient nano-catalyst for the conversion of organic compounds by homogeneous and heterogeneous phases.

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CONFLICT OF INTEREST

We declare that we have no conflict of interest.

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