International Research Journal of Natural and Applied Sciences
ISSN: (2349-4077)
Impact Factor 7.032 Volume 10, Issue 02, February 2023
Website- www.aarf.asia, Email : editor@aarf.asia , editoraarf@gmail.com

Synthesis and Characterization of Silicon Cobalt mixed Nano Ferrites

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Abstract-

Silicon Cobalt mixed oxide sample, with different compositions from 0.0 to 2.0 at 20^0C, 40^0C and 60^0C cobalt wt% were synthesized using a modified Wet-Chemical and Sol-Gel method.Proposed composite materials which are based on transition metal nano particles expressed in surface area to volume matrix gives different properties as optical, adsorptive, magnetic and catalytic.This sample also dispersed in proposed gas sensor on catalyses for different reactions. The synthesis method in dispersion of metal oxide articles and generally on chemical properties of such systems. The wet chemical and sol-gel procedure for the advantage of synthesis condition which allow the surface chemical properties. In this work, we obtained the sample prepared by a modified sol-gel and wet chemical properties and their structure.

Keywords: *Cobalt, Nickel nanoand mixed ferrites, magnetic properties, solvent*

Introduction-

Silicon Cobalt mixed oxide sample prepared with different compositions of 0.0 to 2.0 at 20^0C, 40^0C and 60^0C cobalt wt% were synthesized using a modified Wet-Chemical and Sol-Gel method. The materials that characterised in order to correlate with structural and chemical properties to synthesize. TG/DTA measurements and X-ray diffraction XRD were used to follow the thermal evolution and the crystallization behaviour of the samples. The textural and red-ox properties were evaluated by N2 adsorption, desorption isotherms and TPR measurements. Lewis's acidity and redox properties of cationic sites were studied by FTIR spectra of the exposed sample to NO. It was found that the nature of Cobalt oxide species and their interaction with silica matrix depends on the Cobalt content. No Cobalt

oxides were observed in 20^0C sample while Co_3O_4 was present as a segregated phase in the samples at higher Cobalt loading. TPR measurements strength of observations takes place unless 20^0C sample 40^0C and 60^0C shows to TRP peaks in the range from Room Temperature to 300^0C.The attribution of two-step reduction in Co_3O_4 phase followed by a single at about 250^0C that is the probably due to the reduction of Co species are more strongly bonded to silica. N_2 adsorption and desorption measurements indicate that the Co content affects the micropore and mesopore volumes. FTIR spectra recorded after NO adsorption at different temperature which show two-week acid Lewis sites due to Co_2 surface. Yet, the presence of NO weekly adsorb over Co_3 cannot be excluded.

Experimental Setup- Cobalt oxide prepared by sol-gel and wet chemical method using cobalt nitrate hexa hydrate $Co(NO_3)2.6H_2O$ and tetraethoxysilane TEOS as starting materials. Tetraethoxysilane (TEOS) sample hydrolysed for 2 hours at 100^0C using nitric acid without alcoholic solvent. The ratio of all solvents such TEOS:H_2O:HNO_3 are in 2:8:0.02 and this solution cooled at room temperature with a definite quantity of $Co(NO3)H_2O$ which added slowly on magnetic stirrer. From this method three samples with 20^0C, 40^0C and 60^0C cobalt wt% obtained. After 24 hours a transparent and pink gel were obtained in each composition. This obtained gel sample kept in room temperature for 72 hours. The obtained gels dried at 120^0C in electric oven for 24 hours then obtained sample slowly heated upto 300^0C. Thermogravimetric/differential TG/DTA carried out by using simultaneously thermos analyser STA at 210^0C in Al_2O_3 using reference materials. The obtained sample as dried gel found in the nature of Amorphous. The crystallizing phase obtained by XRD determination by diffractometer using monochromaticradiations at 0.75 and 2θ at 200^0C N2 adsorptions desorption obtained.The isotherms elaborated by α-plot and BET method using N2 as silica reference. Cobalt oxides catalysts characterized using temperature programmed reduction (TPR) using 4% H_2/Ar as reducing gas. During this process the consumption of H_2O was measured by TCD at room temperature to 300^0C. The prepared catalyst sample of weight 200mg and temperature range 20^0C /min from FTIR spectra.

Result and Discussion-

The prepared sample characterized by XRD and FTIR methods. The prepared sample gel hydrolysis slowly aqueous acid environment according to modified Sol-Gel and Wet Chemical method. According to the process of prepared wet gel sample was formed by Siloxane network with cobalt spices trapped. This process takes place until pinkish colour of all wet gels by all studied compositions exhibits for 24 hours. Thus, the prepared sample selected on the basis of the result of thermal analysis.

XRDStudies-In this result the TG curves shows 36wt% for 20^0C, 39wt% for 40^0C and 48wt% for 60^0C. The wt% loss in each case obtained at room temperature to 200^0C. In this range the evaporation of solvents, subsequent and residual organic molecules are overlapped the decomposition of $Co(NO_3)2.H_2O$ upto 230.The XRD pattern for this given range of 15, 25 and 35^0C samples which heated upto 300^0C shows in figure. As 20^0C was keeps as amorphous nature. The crystallization of mixed sample of cobalt oxides Co_3O_4 annealing at 300^0C. For the formation of Co_2SiO_4 crystallites in all three samples. These results shows that the prepared gel composition and calcination plays an important role to find out the nature of cobalt and interacts with silica matrix.

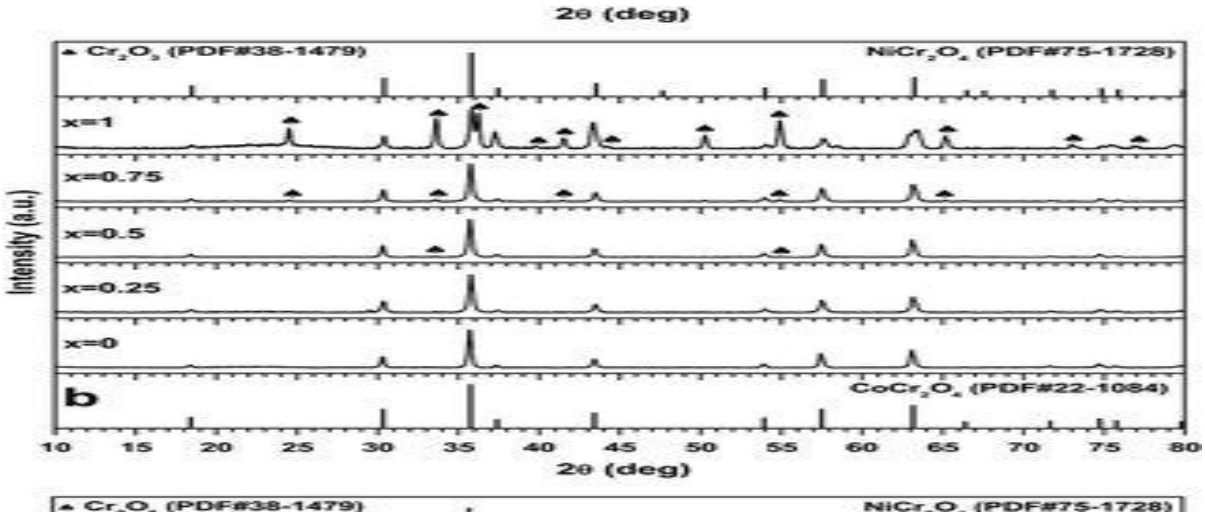

Figure-*XRD pattern of prepared gel sample from room temperature to 300^0C as Co_2SiO_4*

FTIRStudies-The catalysts of prepared sample characterized from room temperatue to 300^0C as the maximum temperature at which it is found that the catalysts reduced at 300^0C as the maximum temperature. The interaction of NO with Co_3O_4 is very complex and gives different species at different temperature. Below 300^0C the transformation characteristic of prepared sample molecularly adsorbed NO bands found in the range of 1700 to 1400cm^{-1}.

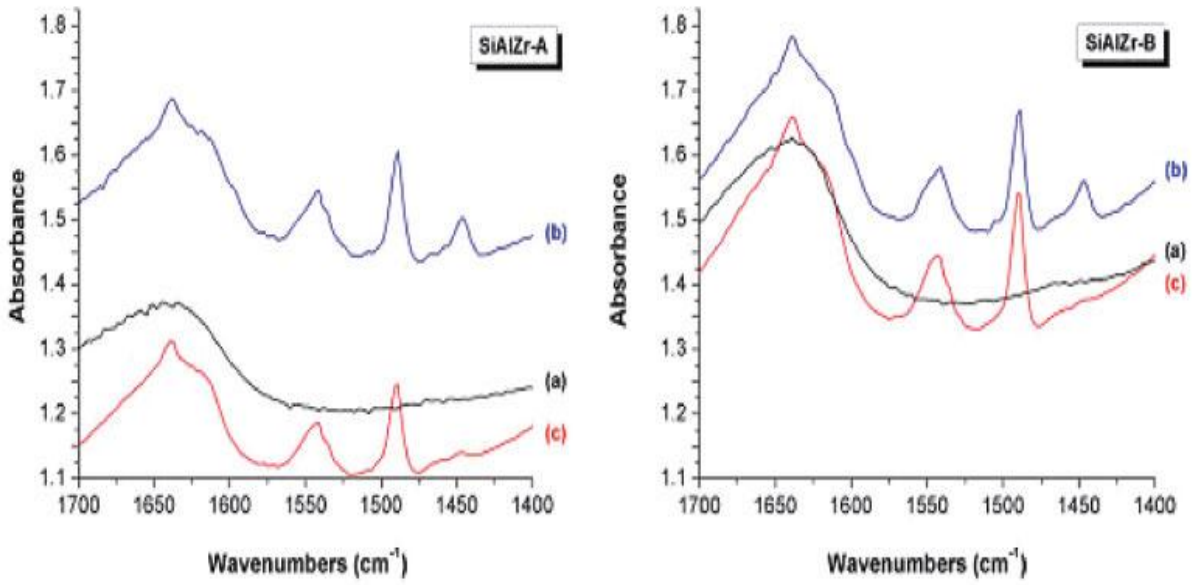

Figure 2-*FTIR spectra of adsorbed NO and NO_2 as 20^0C at Room Temperature to 300^0C as max temperature*

Diatomic molecule shows stronger in Infra-Red spectra at single band of 1620cm^{-1}. The weaker band is shifted of the NO stretching band of adsorbed nitrogen monoxide characteristics. It is found that a band probably coupled observed above 1700 $cm^{-1.}$ On increasing the temperature coupled band disappear. In the particular region below 1700cm^{-1} the complex absorption in the range 1700to 1400 cm^{-1} is due at different forms of nitrate also NO_2cannot be excluded. The prepared sample produced NO oxidation. The adsorption of NO at different temperature gives rise to very uniform and similar spectra. The overall difference causes the reduced transparency of the prepared sample and resolution of spectra. However, all the band at different ranges observed over 20^0C are detectable. So, the small change in similar intensities of same band can be detected.

Conclusion-

In conclusion, the x-ray diffraction patterns of all the samples confirm that single phase cubic spinal structure. By Sol-Gel method the cobalt ferrites displays much better result of degree of crystallinity than the Co-NO ferrites shows that these ferrites are slightly amorphous in nature. The prepared sample obtained by ball milling of Co-NO in HNO_3 ferrite samples experience smaller intensities and higher line broadening due to surface disorder introduced during Ball Milling and Sol-Gel method. The average particle size of prepared sample was discussed in XRD patterns and observed the range of the size of prepared sample from 20-84nm. This samples were resulting in lightly magnetisation and coercivity as the temperature varies from 100 to 300^0C. It was also observed that the Particle size and inversion parameters attributed at lower temperature. The FTIR spectra of prepared

sample found deduced such as resonance field and line width are in accordance with the particle size and magnetisation data of different systems. Further the measurements are underway to complement the particle size as well as superparamagnetic nature which could help us to understand the system easily.

Acknowledgements-I thankful to all my IIT and Universityfriends to carry out the characterization of thisresearch work.

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