

## Thesis Abstract

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This thesis work is based on heterogeneous catalyst synthesis and their application in ESR for hydrogen generation. The work was focused on non noble metal Ni and Co based catalyst. The deactivation of catalyst, reformulation of catalyst from the deactivated mixture and pre-treatment reaction effects were also studied. The best possible bimetallic catalyst with support were synthesized by novel route and optimized for ESR reaction. The entire work has been divided into nine chapters and their brief description was stated as follows:

**Chapter 1** It covers the brief description of hydrogen energy scenario and the complete literature survey on catalytic steam reforming of ethanol. On the basis of it the research objectives were fixed.

**Chapter 2** In this chapter the different methods opted for catalyst synthesis and the characterization techniques used for observation of physic-chemical properties were briefly described. The schematic diagram and photograph of the complete set up were marked with constituent part and described with their significant role in the ESR reactions.

**Chapter 3** In this part, active non noble metal Co, Ni and their bimetallic spinel ( $\text{NiCo}_2\text{O}_4$ ) were studied for ESR performance. The  $\text{NiCo}_2\text{O}_4$  catalyst has shown completion of ethanol conversion with higher hydrogen selectivity (62%) comparative to Ni and Co at 773K.

**Chapter 4** In this study, ESR was studied over barren Co metal ( $\text{Co}^0$ ) from oxalate precursor without any pre-reduction to find out its role in hydrogen and carbon nano-filament generation. The ethanol conversion was found to be 100% with 96.5% hydrogen

selectivity at 723K. The SEM with EDS analysis revealed that  $\text{Co}^0$  state was found in between the carbon nanofilament as well as at the tip of carbon nanofilament.

**Chapter 5** The  $\text{Co}_3\text{O}_4\text{-LaCoO}_3$  catalyst was synthesized from prepared Co-C nanofilament obtained by performing ethanol steam reforming over  $\text{Co}^0$ . The purpose of the work was to make it cost effective by waste utilization for catalyst synthesis, reutilization of catalyst and the role of composite ( $\text{Co}_3\text{O}_4\text{-LaCoO}_3$ ) during ESR performance. The ESR performance of  $\text{Ni/Co}_3\text{O}_4\text{-LaCoO}_3$  was found better as compared to  $\text{Co/Co}_3\text{O}_4\text{-LaCoO}_3$  and  $\text{Co}_3\text{O}_4\text{-LaCoO}_3$ . It was shown complete ethanol conversion at temperature 823K with 80% hydrogen selectivity.

**Chapter 6** To elucidate the role of Ni and Co with ZSM-5 support ethanol steam reforming (ESR) were performed. It had shown complete ethanol conversion at 773K but the selectivity in hydrogen generation was found higher for  $\text{Ni/ZSM-5}$  catalyst as compared to  $\text{Co/ZSM-5}$ . The  $\text{Ni/ZSM-5}$  catalyst has about 72 % hydrogen selectivity at temperature 873K. Nevertheless, the  $\text{Ni/ZSM-5}$  catalyst had shown its stability for high temperature (873K) ethanol steam reforming performance.

**Chapter 7** In this study, the pre-treatment effect of reduction and redox reaction on  $\text{Co/CeO}_2$  nano-cube are studied for ESR. The study reveals that the morphological properties of catalysts are not affected by the pre-treatment of  $\text{H}_2$ , air with  $\text{H}_2$ , and CO with air but their activities for ESR are considerably different. The  $\text{Co/CeO}_2$  treated with  $\text{H}_2$  has similar ethanol conversion with the catalyst treated with redox atmosphere (CO with air) but their  $\text{H}_2$  selectivity are found different.

**Chapter 8** In this chapter a significant route of precipitation for nano sized metal oxide synthesis was discovered from their nitrate precursor in the presence of ethanol solvent by solvothermal treatment. The bimetallic  $\text{Ni-Co/CeO}_2$  based catalysts were synthesized in

different concentration. The 20%Ni-Co/CeO<sub>2</sub> was found optimum for ESR performance. The optimization of different molar ratio of water and ethanol were also optimized on 20%Ni-Co/CeO<sub>2</sub>.

**Chapter 9** It includes the conclusion of whole work performed for different catalyst and operative conditions. The scientific contributions of the work in the area of ESR were also briefly marked. The recommendation for further study in this area was also described.