

# Synthesis, characterization and application of nanocomposites as burn rate modifiers in solid composite propellant



Thesis submitted in partial fulfillment  
for the Award of Degree

**Doctor of Philosophy**

By

*Harish Kumar*

DEPARTMENT OF CHEMICAL ENGINEERING & TECHNOLOGY  
INDIAN INSTITUTE OF TECHNOLOGY  
(BANARAS HINDU UNIVERSITY)  
VARANASI - 221 005

Roll No. 13041004

August, 2017

**CHAPTER 5****5 CONCLUSION AND RECOMMENDATIONS****5.1 Conclusion:**

The catalytic activity of the burn rate modifier greatly depends upon its composition, formation phase, particle size and shape, and method of preparation. The burn rate of ammonium perchlorate (AP) based solid composite propellant (SCPs) is a surface phenomenon. It is controlled by the thermal decomposition of AP, which is greatly influenced by the particle size, the catalyst composition and dispersion. With decrease in the particle size of the catalyst, its specific surface area increases and provides the adsorption sites for the oxidizer to decompose.

The spinel structure of Cu-Cr-O composite plays an important role in determining its catalytic activity. The spinel structure depends upon the Cu/Cr molar ratio. It is achieved by keeping the Cu/Cr molar ratio  $>0.5$  and exist as  $\text{CuCr}_2\text{O}_4$  and  $\text{CuO}$ , whereas when  $\text{Cu/Cr} < 0.5$ , the phase composition of oxide phases changes to  $\text{CuCr}_2\text{O}_4$  and  $\text{Cr}_2\text{O}_3$  [Li *et al.* 2007]. Similarly, the anatase phase of  $\text{TiO}_2$  has excellent catalytic activity because of the presence of lots of points and linear defects on its surface.

In the present study, effect of  $\text{Cu-Cr-O-TiO}_2$ , and  $\text{Cu-Cr-O.TiO}_2$  modified with reduced graphene oxide (rGO) on burn rate of AP based solid composite propellant were investigated. Sol-gel method and modified Hummer method incorporating sol-gel technique were used to synthesize the nano-composite of  $\text{Cu-Cr-O.nTiO}_2$  and rGO based  $\text{Cu-Cr-O.0.7TiO}_2$ . The synthesized catalysts were characterized by TGA, XRD, FT-IR, SEM, EDAX, TEM, XPS and Raman spectroscopy to determine the calcination temperature, phase identification and crystallite size, attachment of functional groups, particle size and shape, elemental composition, shape of

the particles, oxidation state and binding energy and formation of G, D and 2D bands of reduced graphene oxide, respectively. The catalytic effect of the synthesized catalyst on thermal decomposition of AP was determined using the TG-DTA technique. Further, the impact of the best optimized catalyst i.e. rGO based Cu-Cr-O.0.7TiO<sub>2</sub> on the burn rate of SCPs was investigated using the Strand burner.

Based on the present results following the significant conclusions have been drawn.

- The amorphous phase of the synthesized catalyst was observed when calcined at 300°C where as crystallinity increased with increase in the calcinations temperature to 1050°C.
- The particle size exhibited the formation of nanoparticles of the size 20-100 nm of spherical and needle like shape in the catalyst calcined at 300°C, where as the particle size changed to 1 μm, the shape transformed into cuboids-like particles at calcinations temperature of 1050°C.
- Among three compositions of Cu-Cr-O.nTiO<sub>2</sub> (n= 0.5, 0.7 and 0.9) calcined at 300°C, Cu-Cr-O.0.7TiO<sub>2</sub>, showed the best catalytic activity on AP of particle size 45μm, and exhibited the excellent single step decomposition of AP<sub>45μ</sub> at 306 °C, which is 79°C lower than that for the pure AP<sub>45μ</sub> indicating a significant change in HTD.
- The reduced graphene oxide has the ability to permit superior catalytic activity due to faster rate of electron transfer, and higher thermal and mechanical stability.
- The 5.0 wt% of Cu-Cr-O-0.7TiO<sub>2</sub>-rGO catalyst added into AP exhibited an excellent catalytic activity, and accelerated the decomposition of AP.
- This 5% wt synthesized catalyst delivered the best result by exhibiting the single step decomposition at a much lower temperature of 97°C.

- A very large hike in the burn rate of SCP, having AP modified with synthesized catalyst was observed as compared to the SCP having AP modified with industrial catalyst (ACR) as well as pure AP.
- Burn rate of the synthesized catalyst assisted SCP was observed to be 133 % and 175 % higher as compared to industrial catalyst and pure AP assisted SCP.
- The activation energy, pre-exponential factor and reaction rate constant of thermal decomposition of ammonium perchlorate in presence of 5.0 wt% of Cu-Cr-O-0.7TiO<sub>2</sub>-rGO catalyst was observed as 100.266 kJ/mol,  $1.18 \times 10^7 \text{ (s}^{-1}\text{)}$  and  $0.006827 \text{ (s}^{-1}\text{)}$ , respectively.

## 5.2 Recommendations for Further Studies:

The followings aspects should be considered for further study:

- Effect of catalyst surface morphology on the burn rate of SCPs should be investigated.
- Investigations of the effects of surfactant concentration during catalyst synthesis to avoid the agglomeration of particles will be useful for coming out with the optimum concentration.
- A comprehensive knowledge of the thermal decomposition kinetics of AP based SCPs in presence of rGO based transition metal oxide catalyst will be helpful in explaining the role of rGO.
- The effect of catalyst concentration on other prominent oxidizers i.e. ammonium dinitramide (ADN) and CL-20 etc should also be evaluated.