

CHAPTER 3

MATERIALS AND METHODS

3.1 Experimental:

3.1.1 Materials:

All the starting reagents were of analytical grade and high purity (>99.9 %). Ammonium per-chlorate (NH_4ClO_4) of the average particle size of 300 μm was purchased from Pandian Chemicals Cuddalore. AP was brought down to average particle size of 45 μm using the air classifying mill. Copper nitrate tri-hydrate ($\text{CuNO}_3 \cdot 3\text{H}_2\text{O}$) was purchased from Sigma-Aldrich, Germany. Chromium Nitrate nonahydrate [$\text{Cr}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$] of laboratory grade was purchased from Sigma-Aldrich, Germany. Citric acid anhydrous $\text{C}_6\text{H}_8\text{O}_7$ was purchased from M/s Merck, India. However, ethanol ($\text{C}_2\text{H}_5\text{OH}$) was purchased from Merck, Germany and TiO_2 (P-25) of nano-metric size was purchased from Evonik Industries, Germany. Readymade industrial catalyst i.e. activated copper chromite (ACR): CuCr_2O_4 is purchased from BLI Kerala, India. The brief description of the chemicals is given in table 3.1.

Table 3.1: List of chemical along-with their respective manufacture/suppliers

S. No.	Chemicals	Quality	Manufacturer/ Suppliers
1.	Ammonium Perchlorate	Industrial Grade	Pandian Chemicals Cuddalore
2.	Industrial Copper Chromite	Industrial Grade	M/s BLI, Kerala
3.	Copper nitrate tri-hydrate	99% pure, AR	Sigma-Aldrich, Germany
4.	Chromium Nitrate nonahydrate	$\geq 99\%$ pure, AR	Sigma-Aldrich, Germany
5.	Citric acid anhydrous	$\text{MW}=1.3 \times 10^6 \text{ gm/mol}$	M/s Merck, India
6.	Titanium dioxide	99% pure, AR	Evonik Industries, Germany

7.	Graphite Powder	99% pure, AR	Graphite India Ltd.
8.	Sulphuric Acid	Analytical Grade	Sigma-Aldrich, Germany
9.	Potassium permanganate	99% pure, AR	M/s Merck, India
10.	Hydrogen peroxide	99% pure, AR	M/s Merck, India
11.	Orthophosphoric acid	99% pure, AR	Sigma-Aldrich, Germany
12.	Hydrochloric acid	99% pure, AR	M/s Merck, India

3.1.2 Methods:

3.1.2.1 Synthesis of Cu-Cr-O-nTiO₂:

In order to prepare Cu-Cr-Ti mixed oxides via sol-gel technique, firstly the mother solution of Cu-Cr-citric acid was prepared. The preparation of mother solution required 0.021 mol of copper nitrate tri-hydrate and 0.03 moles of chromium nitrate nona-hydrate added into 20 ml of ethanol. The mixture was stirred until the formation of homogenous solution. 0.042 moles of citric acid was added into the obtained homogeneous mixture followed by further stirring. The mother solution thus obtained was divided into three equal parts to prepare three different solutions by adding 0.86, 1.172, and 1.516 g of titanium oxide (TiO₂) nanoparticles respectively. Further, 5 ml of additional ethanol was added into each solution and stirred for 4 h to get them into transparent suspension-cum-solutions respectively. The solutions thus obtained were kept for aging at room temperature (30°C) for 48 h. After this initial aging, each suspension was again mixed thoroughly to ensure the homogeneity of suspended particles and heated with continuous stirring at 80 °C till the paste like material was obtained for each the suspension. The pastes thus formed were dried for 3 h in an oven maintained at temperature 130°C and obtained materials

were individually crushed to fine particles. Each of the three powders thus obtained was divided into two equal parts for their calcinations at two different temperatures. First part of each the sample was calcined individually at 300°C (below the crystallization temperature of Cu-Cr-O) in N₂ atmosphere for 3 h. However, the second part calcined individually at 1050°C (above the crystallization temperature of Cu-Cr-O) in the same atmosphere for 3 h. The crystallization temperature of Cu-Cr-O is 700 °C. We have now total six samples; Three Cu-Cr-O-nTiO₂ (n= 0.5, 0.7 & 0.9) samples calcined at two different temperatures 300 °C and 1050 °C. The basic schematic flow diagram of the synthesis process of Cu-Cr-O-nTiO₂ (n= 0.5, 0.7 & 0.9) is shown in Fig. 3.1 and Fig. 3.2.

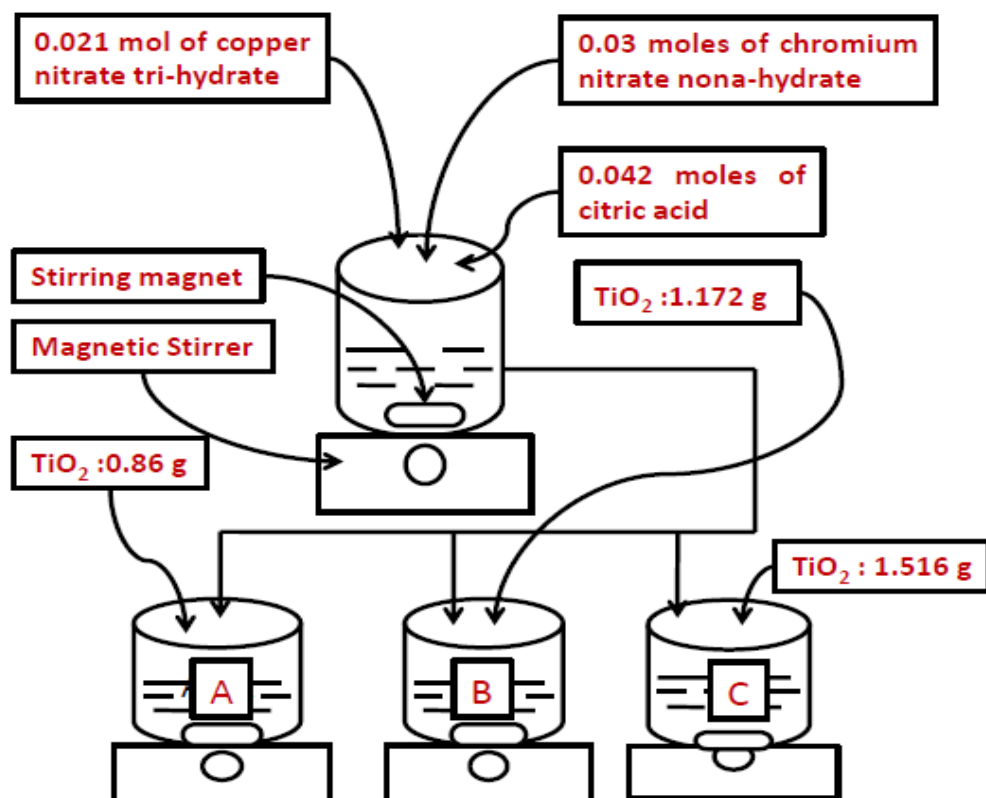


Fig. 3.1: Schematic Diagram for Synthesis of Cu-Cr-O-nTiO₂

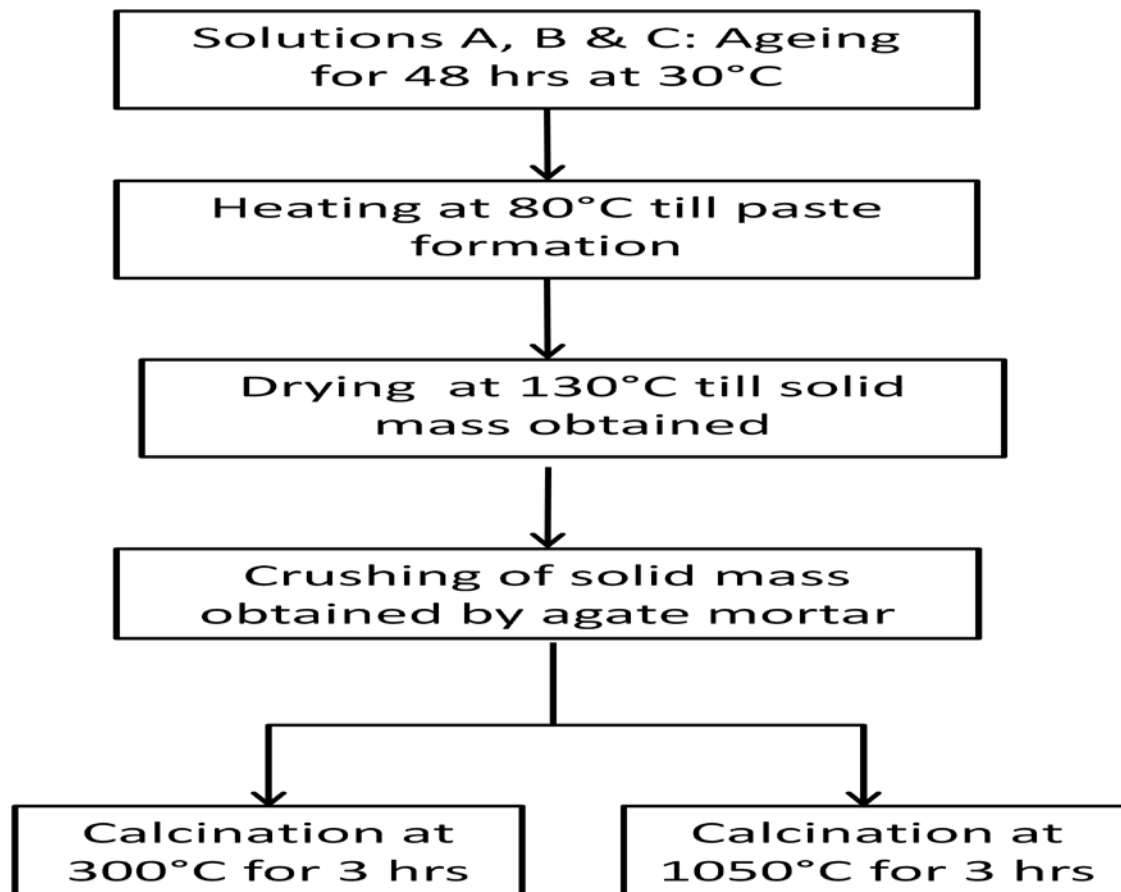


Fig. 3.2: Flow Diagram for Synthesis Of Cu-Cr-O-nTiO₂

3.1.2.2 Synthesis of Reduced Graphene Oxide (rGO):

In order to get 1g of rGO product, 120 ml of H₂SO₄ and 20 ml of H₃PO₄ were added into a beaker with continuous stirring using magnetic stirrer. After 10 minute of stirring, 1g of graphite powder was dispersed into the beaker having mixture of two different acids. The dispersion of the added graphite powder of average particle size of 40 μm was allowed for further 30 minutes of continuous stirring. The color of the mixture in beaker was dark black due to graphite. After the homogeneous mixing of graphite particles, 3 g of KMnO₄ was added slowly into the beaker which leads to the color change of graphite added acids from black to the greenish blue. In due course of time, the color change from greenish blue to pink was observed

during the synthesis process of rGO. Vigorous stirring of the obtained solution was allowed for further 36 hrs at the room temperature. The color of the solution became muddy yellow. 100 ml of distilled water was then added into the obtained yellowish solution followed by addition of 30ml of 30 % v/v H₂O₂. Exothermic reaction was observed and finally golden yellow colored particles were seen in the solution. The impurities of thus obtained solution were removed through washing by the water followed by the 30% v/v of HCl and C₂H₅OH. The solid particles were removed from the solution by centrifuging it at 5000 rpm. Thus obtained solid particles were dried up in the oven at 60 °C for 6 hrs to get GO. GO was transformed to rGO by thermal treatment method as reported in literature [D. Zhan *et al.* 2011]. Finally, rGO was calcined at 1050 °C to improve its activity by reducing the attached functional groups. The rGO thus obtained was ready for further use. The basic schematic flow diagram of the synthesis process of graphene oxide using modified Hummer method [W.S. Hummers *et al.* 1958] is shown in Fig.3.3.

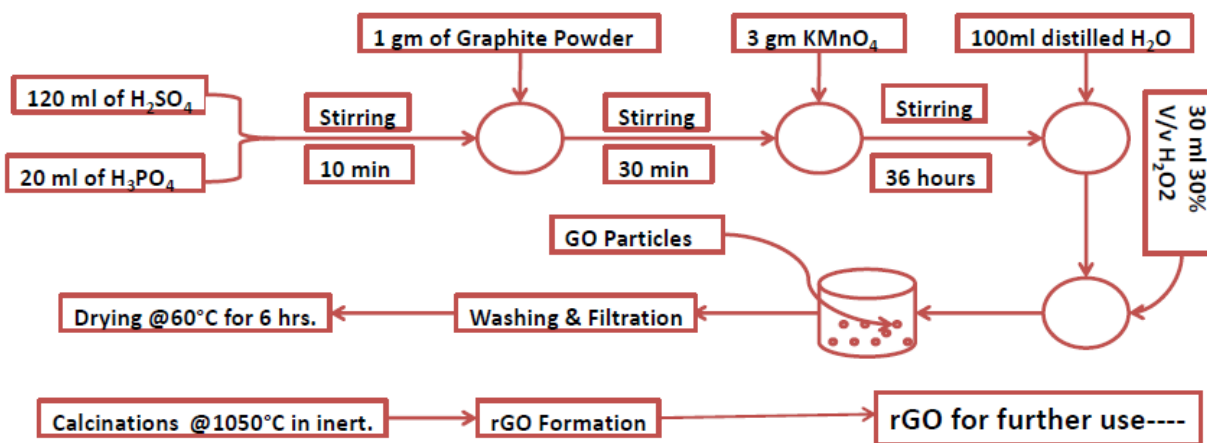


Fig. 3.3: Schemetic Diagram for Synthesis of Graphene Oxide

3.1.2.3 Synthesis of Cu-Cr-O-0.7TiO₂-rGO:

Among the total six compositions of tri-metallic oxide with three varying concentrations (0.5, 0.7 and 0.9 mol %) of TiO₂ calcined at two different temperatures 300 and 1050°C, the Cu-Cr-O-0.7TiO₂ composite calcined at 300°C was found to be best catalyst which had remarkably enhanced the burn rate of AP. The AP with particle size of 45 μm (AP_{45μm}) exhibited better thermal decomposition behavior rather than the AP having particle size of 300 μm (AP_{300μm}) as reported in literature [Kumar *et al.* 2017]. Therefore, we have used AP_{45μm} in our present study however, it is represented by AP. When 0.7 mol% of TiO₂ added Cu-Cr-O composite calcined at 300°C was used as catalyst in AP, a very sharp single step decomposition of AP_{45μm} at 306°C was obtained, which is 79°C lesser than the pure AP. Since, the catalyst sample Cu-Cr-O.0.7TiO₂ calcined at 300°C had achieved remarkable enhancement in thermal decomposition of AP when used as modifier in pure AP, therefore the further modification by rGO in this composition may offer more significant results. Figure 3.4 shows the basic schematic flow diagram of the synthesis process of the composites of Cu-Cr-O.0.7TiO₂ using sol-gel technique.

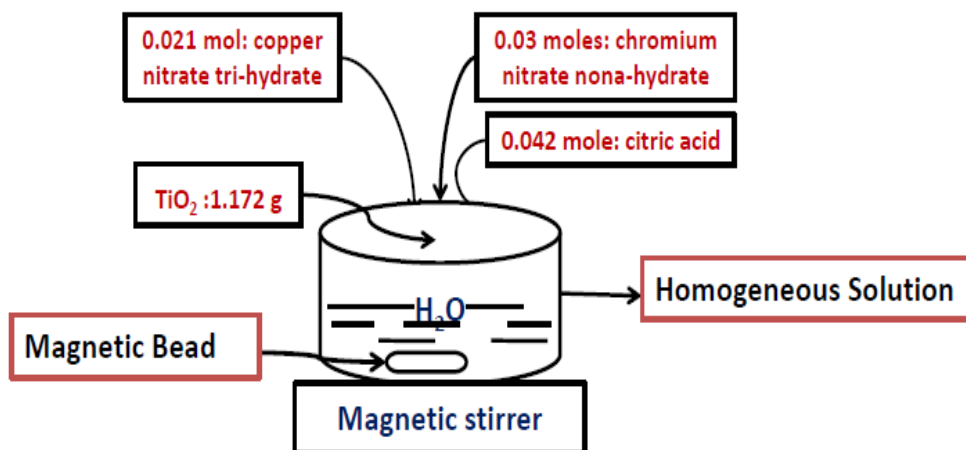


Fig. 3.4: Schematic Diagram for Synthesis of Cu-Cr-O.0.7TiO₂

In order to synthesis rGO incorporated Cu-Cr-O-0.7TiO₂ composite, 10 wt % of rGO was added homogeneously with 90 wt% of Cu-Cr-O-0.7TiO₂. 10 mg of already synthesized rGO was poured into 400 ml ethanol taken in beaker. The rGO suspension was kept on magnetic stirrer for continuous stirring and Cu-Cr-O.0.7TiO₂ powder was then allowed to add slowly into this suspension. The rGO modified Cu-Cr-O-0.7TiO₂ mixture was continuously stirred at 80 °C till the paste-like material was obtained. The paste obtained was dried at 130°C for 3 h in an oven. The material thus obtained was then crushed by mortar pestle to get homogeneous fine powder of Cu-Cr-O-0.7TiO₂-rGO. This powder was further calcined at 300°C for 4h in the N₂ atmosphere to form desired Cu-Cr-O-0.7TiO₂ composite materials having excellent catalytic properties on thermal decomposition of AP. Figure 3.5 shows the basic schematic flow diagram of the synthesis process of the composites of reduced graphene oxide based Cu-Cr-O.0.7TiO₂ using sol-gel assisted Hummer method.

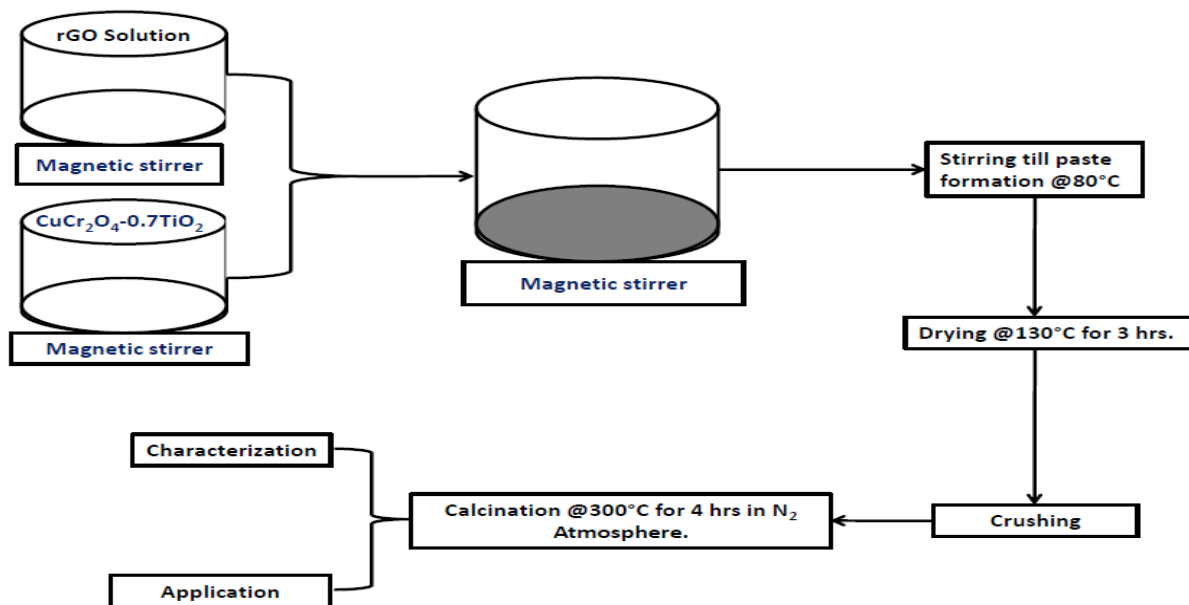


Fig. 3.5: Schematic Diagram for Synthesis of rGO/Cu-Cr-O.0.7TiO₂

3.1.2.4 Synthesis of Propellant:

(a) Mixing:

All the ingredients were weighed using an electronic weighing machine with a least count of 0.1 mg. while synthesis the solid composite propellant, all the mentioned ingredients are hand mixed in a mortar pastel. Firstly HTPB and DOA which are in liquid form were mixed together in the required compositions for 2 min. after achieving the homogeneous solution, burn rate modifier was added and mixed for 5 min. In the similar manner AP in the bimodal form and Al(P) were added to the mortar and mixed for 5 min at each addition. Finally TDI, curator was added to the obtained semi solid of propellant for the curing reaction to be occurred. The composition of propellant is given in the following Table 3.2.

Table 3.2: Chemical compositions of Solid Composite Propellant

Qty, g	Base mix		Catalyst (0.1%)		Catalyst (0.2%)		Catalyst (0.3%)		Catalyst (0.4%)		Catalyst (0.5%)		Catalyst (0.5%)	
	%	Qty, g	%	Qty, g	%	Qty, g	%	Qty, g	%	Qty, g	%	Qty, g	%	Qty, g
Raw Materials														
HTPB	10	1.5	10	1.5	10	1.5	10	1.5	10	1.5	10	1.5	10	1.5
DOA	3	0.45	3	0.45	3	0.45	3	0.45	3	0.45	3	0.45	3	0.45
AIP	18	2.7	18	2.7	18	2.7	18	2.7	18	2.7	18	2.7	18	2.7
AP (Coarse)	45.3	6.8	45.2	6.7	45.2	6.7	45.1	6.77	45.0	6.76	45	6.75	44.9	6.7
	3		6	9	8	3		6					3	3

AP	22.6	3.4	22.6	3.3	22.6	3.3	22.5	3.38	22.5	3.37	22.5	3.37	22.4	3.3
(Fine)	7		3	9		9	7	5	3			5	7	7
TDI	1	0.1	1	0.1	1	0.1	1	0.15	1	0.15	1	0.15	1	0.1
		5		5		5								5
Burn rate modifier	0	0	0.1	0.0	0.2	0.0	0.3	0.04	0.4	0.06	0.5	0.07	0.6	
				15		3		5				5		
Total	100	15	100	15	100	15	100	15	100	15	100	15	100	15

(b) Propellant Sample Preparation:

The propellant samples for burn rate evaluation were obtained by pouring the propellant into the mould of aluminum sheet. The size of the mould was 110 (mm) L X 6 (mm) B X 6 (mm). The propellant strands are shown in Fig. 3.6.

(c) Curing Cycle:

The curing of as obtained propellant was carried out to obtain the solid composite propellant in the cubical oven. The curing process involve the heating and cooling cycle for the specified time. In this synthesis process, the heating temperature was kept 50°C for 120 hours and 24 hours for cooling purpose. The propellant samples are shown below.



Fig 3.6. Samples of AP Based Solid Composite Propellant

3.1.3 Characterization:

The phase transformation of the catalyst precursor was confirmed by thermo-gravimetric analysis (TG-DTA) using the Perkin Elmer Thermal Analyzer (Model no.: SPA-6000). Crystal structure of powders was investigated by X-Ray diffraction technique using X-ray Diffractometer (Rigaku Miniflex-II, Desktop X-Ray D) with Cu- K_{α} radiation ($\lambda=1.5418 \text{ \AA}$) and Ni filter operated at 30 KV and 15 mA. The scanning speed and scanning range were kept $3^{\circ}/\text{min}$ and $20-80^{\circ}$ respectively. Surface morphology and particle size analysis were monitored by scanning electron microscopy (SEM) (FEI Quanta 200F). Elemental analysis of synthesized powder was carried out with energy dispersive X-ray analysis method (EDAX) inbuilt in the SEM. Chemical bonding and molecular interactions analysis was understood by using Fourier Transform Infrared (FT-IR) spectra recorded in the wave number range from 4000 to 400 cm^{-1} using Perkin Elmer 577 FT-IR. TEM (TECNAI 20 G²) was employed for interior

morphology/structure of the sample. The oxidation state and elemental analysis of the samples were carried out using X-Ray photon spectroscopy (XPS- model PHI5000, Versa Probe II, physical Electronics) with Al α for monochromatic X-ray of energy $E= 1486.6$ eV, Ar ion Gun sputtering, Beam Dia- $10\ \mu\text{m}$, 6-25 W and 15 KV, surface clean sputtering-2 KV, $2\text{mm} \times 2\text{mm}$ for 1 min. The XPS patterns of various compositions were taken at 187.5eV pass energy for detection of all possible elements with atomic concentration limit above 0.5%. To confirm the oxidation states of constituent elements the core level analysis is done at 11.75 eV pass energy at a step size of 0.1eV with X-ray rating of 100 micron diameter, 25W and 15kV. The electron neutralizer and Ar ion gun is used during experiment for charge loss compensation which occurred during XPS process. The core level spectra are recorded under measured under ultra high vacuum (UHV) conditions of about 1-10 torr. The adventitious carbon is recorded for reference from the surface and then sputtered by Ar. ion gun at rating 2kV 2X2 at a sputter rate of 10nm/min of SiO₂ standard. Figure 3.7 shows the graphical view of characterization technique used in characterizing the synthesized catalysts.

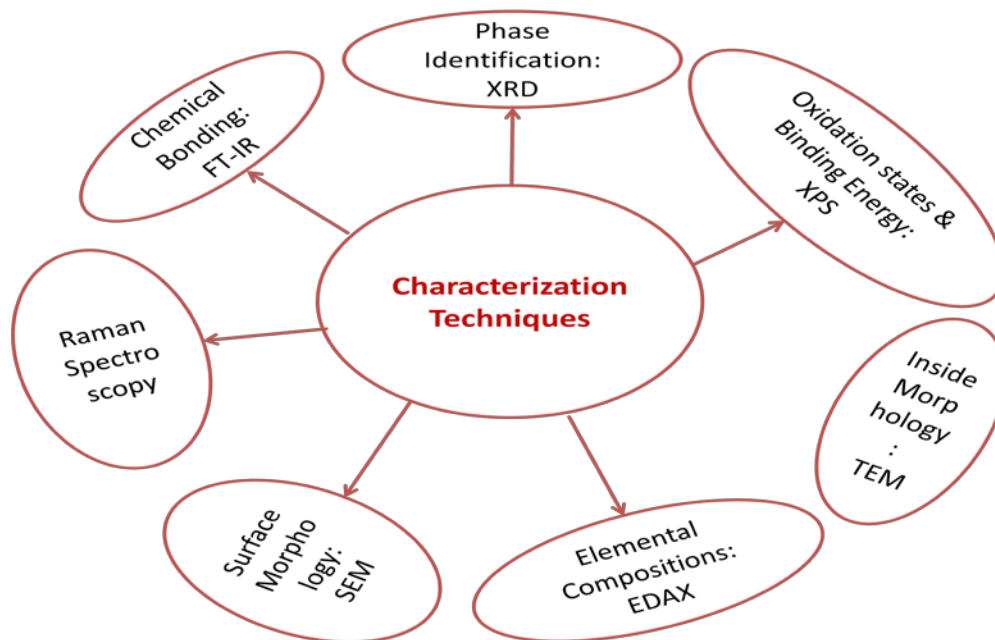


Fig.3.7: Characterization Techniques for Synthesized Catalyst

3.1.3.1 X-Ray Diffraction (XRD) Analysis:

Bruker D8 advanced wide-angle X-ray diffractometer with a graphite monochromator was used for performing X-ray diffraction using $\text{CuK}\alpha$ source with a wavelength of 0.154 nm. The generator was operated at 40 kV and 20mA. The powder or thin sheet of samples; nanofibres, foam were placed on silica sample holder at room temperature ($\sim 25^\circ\text{C}$) and were scanned at diffraction angle 2θ from 10° to 80° at the scanning rate of 2°min^{-1} . The peak width at half maxima in the XRD peaks was used to determine the crystallite size as per Scherrer formula:

$$D = \frac{K\lambda}{\beta(\cos\theta)} \quad (2.1)$$

Where, $K = 0.9$, (X-ray wavelength) $\lambda = 0.154$ nm, β is full width at half maximum and θ is the Bragg's diffraction angle.

3.1.3.2 Raman Spectroscopy:

Raman spectroscopy is one of the spectroscopy technique based on the polarizability (dipole movement) of the molecules present in an organic or inorganic compound under electric field (laser beam). The spectrograms were obtained at room temperature in backscattering geometry using an Ar⁺ excitation source having a wavelength 488 nm coupled with a Labram-HR800 having a resolution of 4 cm⁻¹.

3.1.3.3 Fourier Transform Infrared (FTIR) Spectroscopy:

IR Spectroscopy is one of the most common techniques, used to determine functional groups in organic compounds. Each functional group absorbs a characteristic frequency of IR radiation. The advantage of FTIR spectroscopy over the conventional dispersive IR Spectroscopy is that this offers a faster and simpler analysis. The FTIR measurements were performed in transmittance mode at room temperature from 400 to 4000 cm⁻¹ using Thermo Nicolet 5700, USA with a resolution of 4 cm⁻¹. Nanoparticles spectra were recorded by making pellets with 10 % particles in KBr powder and direct spectra was taken for powder samples.

3.1.3.4 Scanning Electron Microscopy Analysis:

It is another technique to know the sample morphology and particle size. FESEM has a higher resolution than conventional SEM. A field emission cathode in the electron gun of a scanning electron microscope provides narrower probing beams at low as well as higher electron energy, resulting in both improved spatial resolution and minimized sample charging and damage. Under vacuum, electrons generated by a field emission source are accelerated in a field gradient. The beam passes through electromagnetic lenses, focusing onto the specimen. As a result of this bombardment different type of electrons are emitted from the specimen. A detector catches the secondary electrons and an image of these secondary electrons so the scanning

primary electron beams. Finally the image is displayed on a monitor. Smaller area contamination spots can be examined at electron accelerating voltages compatible with energy dispersive X-Ray spectroscopy and high quality, Low voltage images are obtained with negligible electrical charging of samples. The high resolution reached by FESEM reached by FESEM (~ 2 nm) allows the study of very small microstructural details, morphology analysis (particle shape and size) fracture studies, interface behavior, quantitative and qualitative elemental analysis, also grain orientation textural and phase identification can be done.

In this thesis sample micrographs were recorded using, Carl Zeiss Supra 40 field emission electron microscope.

The morphology of the as synthesized catalyst powder were examined using a high resolution Scanning Electron Microscope (SPURA 40, Zeiss) operated at an accelerating voltage of 2-5 kV. Prior to imaging by using FESEM, samples were sputter coated for 50s with gold using a JEOL JFC-1200 fine coater to make specimens conducting. Based on the FESEM micrographs, powder diameters were analyzed using an image visualization software Image-J developed by the Upper Austria University of Applied Sciences. For Transmission Electron Microscopy nanoparticles samples were prepared by small amount of powdered sample was redispersed in water, dispersion was sonicated and one drop was poured on Cu grid and dried in air. The samples were viewed at high magnification using Tecnai 20 G2 (FEI make) TEM at an accelerating voltage of 100 kV.

3.1.3.5 Transmission Electron Microscopy (TEM) Analysis:

It is the unique technique to know the sample morphology, particle size and also the crystal phase. In this technique a beam of electron is transmitted through an ultrafine thin sample, interacting with the sample as they pass through. An image is formed from the

interaction of the electrons transmitted through the sample, which is magnified and focused onto an imaging device, such as fluorescent screen, as is common in the most TEMs. On a layer photographic film or to be detected by a sensor such as a CCD camera. TEM is capable of imaging at a significantly higher resolution because of the small de Broglie wavelength of electron. This enables the instrument to be able to examine fine details even as small as a single column of atoms. At low absorption of electrons in the sample, due to the thickness and composition of the composition of the sample. At higher magnification complex wave interactions modulate the intensity of the image, requiring adequate and proper analysis of observed image. This technique is used to observe modulation in chemical identity, crystal orientation, electronic structure and sample induced electron phase shift as well as the regular absorption based imaging.

The most common mode of operation for TEM is the bright field imaging mode. In this mode the contrast formation is due to occlusion and absorption of electrons in the sample (classically). Thicker regions of the sample or region with higher atomic number will appear dark, whilst regions with no sample in the beam path will appear bright (hence the term bright field).

Selected area electron diffraction (SAED or SAD) is a crystallographic experimental technique that can be performed inside TEM. In this case, electrons are treated as wave like rather than particles. Since the wavelength of high energy electron is the order of nanometer and the spacing between atoms in a solid is only slightly larger, the atoms act as a diffraction grating to the electron, which are diffracted. As a result the image on the screen of the TEM will be a series of spots (selected area diffraction pattern, SADP) and each spot correspond to a satisfied diffraction condition of the sample's crystal structure. If the sample is tilted, the same

crystal will stay under illumination but different diffraction condition will be satisfied and different diffraction spots will appear or disappear. For thin crystalline samples, this produce an image that consists of a series of dots in the case of a single crystal, or a series of rings in the case of a polycrystalline sample. For the single crystal case, the diffraction pattern is dependent upon the orientation of the sample. This image provides information about the space group, symmetries in the crystal and the crystal's orientation to the beam oath.

To examine the morphology of the sample, TEM was done using Technai 20 G² (Philips) ultra high vacuum TEM. For this, suspension of powder was obtained and thereafter 2 drops of suspension were poured on copper grid (50 micron thick) and dried for half an hour. Then sample were mounted on sample holder and transferred to the TEM morphology and crystal structure were investigated by using real space imaging that is bright field TEM mode and reciprocal space imaging by selected area electron diffraction pattern (SAED).

3.1.3.6 X-ray Photoelectron Spectroscopy (XPS) Analysis:

X-ray photoelectron spectroscopy is a quantitative spectroscopic technique that measures elemental composition and gives empirical formula, chemical state and electronic state of the elements. The XPS spectra are obtained by irradiating a material with a beam of X-rays under high vacuum, while simultaneously measuring the kinetic energy and number of electrons that escape from 1 to 10 nm top layer of the material of atomic number three or above being analyzed. XPS measurement was made in an ultrahigh vacuum AMICUS photoelectron spectrometer equipped with MgK_α X-ray for primary excitation and KRATOS VISION 2 software, all samples were analysis in triplicate for error free area fitting. All binding energy (BE) values reported in the present work with reference to carbon C 1s core level at 284.6 eV.

The curve fitting of the high resolution spectra were performed with combined Gaussian-Lorentzian functions.

3.1.3.7 Thermogravimetric Analysis and Differential Thermo gravimetric Analysis:

Thermogravimetric analysis (TGA) is a technique for measuring the change in weight of a substance as a function of temperature or time. The sample usually a few milligrams in weight is heated at a constant rate, typically in the range 1 to 20°C min⁻¹ and has a constant weight until it being to decompose at a certain temperature. Under dynamic heating decomposition occurs over a range of temperature and after certain temperature no weight loss is observed leading to the completion of decomposition reaction. The weight losses are fundamental properties of the sample and can be used for quantitative calculation of compositional changes etc.

Differential thermal analysis (DTA) is a technique which predicts thermal behavior of sample under study. In DTA, the material under study and an inert reference are made to undergo identical thermal cycles, while recording any temperature difference between sample and reference. The differential temperature is then plotted against time or against temperature, known as thermogram, changes in the sample, either exothermic or endothermic can be detected relative to the inert reference. Thus a DTA curve provides information about the transformation that occurred, such as glass transition crystallization, melting. A DTA instrument consists of sample holder containing thermocouples, sample container, a furnace, a temperature programmer and a recording system. In this method the material under test, in the form of fine powder is placed in a small capsule, often alumina or other suitable refractory material. Adjacent to the test sample, a second capsule containing an inert powder such as α -Al₂O₃ powder and are connected so that their e.m.f are opposed; the net e.m.f therefore represents the temperature difference between the sample powder and the inert α -Al₂O₃. To know the temperature at which completion of reaction

occurred and no significant weight loss observed, differential thermal analysis (DTA) and thermo gravimetric analysis (TGA) were carried out on the collected catalyst mixed ammonium perchlorate powder sample (prior to the ignition) employing the instrument Diamond TGA/DTA Perkin Elmer, USA at a heating rate of $10^{\circ}\text{C min}^{-1}$ upto 500°C in air.

3.1.4 Catalytic Activity Measurement:

The catalytic activity of catalyst was confirmed by differential thermo-gravimetric (DTA) and thermo-gravimetric analysis (TGA) in nitrogen atmosphere using LABSYSTM Setaram Instrumentation (serial no: 1-3347-1 DTA/TGA/DSC) from room temperature ($30\pm 2^{\circ}\text{C}$) to 600°C with heating rate of 10°C/min . The catalytic activity of as synthesized composite materials was determined over the thermal decomposition of AP. In order to analyze the catalytic Activity of the materials the measuring parameters were burn rate of solid composite propellant, temperature of crystallographic change, low temperature decomposition and high temperature decomposition. A known quantity of AP ($\text{AP}_{45\mu\text{m}}$) powder was mixed individually with the varying amounts (2.5, 5.0, 7.5 and 10 wt %) of synthesized catalyst (Cu-Cr-Ti-O-rGO). These three compositions with varying compositions having different concentrations of synthesized catalyst in AP were prepared homogenous (by ball mill method) to see the effect of synthesized catalyst on the burn rate of AP. The resultant compositions were analyzed by thermogravimetric and differential thermal (TG-DTA) characterizations at the heating rate of 10°C/min in N_2 environment. Nichrome wire was used to supply the 24 volt of power supply and the produced noise signal was captured through the acoustic measurement sensor. The burn rate of the SCP was achieved by using the strand burner and testing was operated at 33 bar in water.

3.1.5 Crawford Bomb Burn Rate Evaluation of Strand Burner:

Crawford and co-workers (1947) has developed this technique for burn rate measurement of solid composite propellant in 1947. While measuring the burn rate using this technique, the nichrome wires are embedded into the propellant strand. The schematic diagram displaying Crawford bomb for burn rate measurement setup is shown in Fig.3.8. The propellant strand is inhibited to exhibit only end burning. The inhibition is carried out by dipping the propellant strands into inhibiting solution composed of epoxy resin, epoxy hardener, diluents and antimony oxide and taken out. The nichrome wires are connected to an electronic timer and the propellant strand is mounted in a closed pressurized chamber, partially filled by the water. The nitrogen gas is used to develop the required pressure inside the chamber. The propellant strand is ignited at the top and the burning rate is calculated based upon the acoustic signal measurement. Klager *et al.* (1992) have reported the burn rate measurement error of the order of 2-3%) while using this technique. The photograph of Crawford bomb for burn rate measurement of solid composite propellant is shown in Fig. 3.9. For both the catalyst at one given catalyst loading, the burn rate determination experiments were conducted in triplicate and the variation in the observed values were found to be within 2-3% as reported by Klager *et al.* (1992).

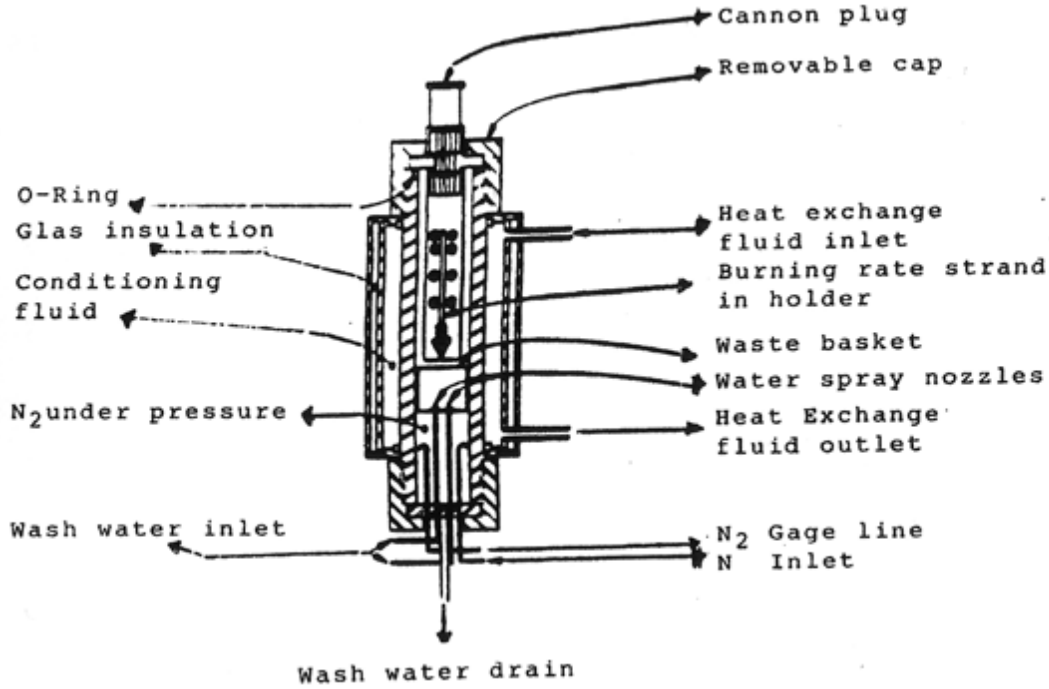


Fig 3.8. Diagram of Crawford Bomb



Fig.3.9: Photograph of Crawford Bomb for Burn Rate Measurement