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A Research on the Synthesis of Cuo-Ceo2 Bimetallic Oxide Catalyst: Some Methodologies

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Abstract – We have likewise effectively fixed up the creation procedure for the impetus for example Co3O4 for groups of 1000gm to 5000gm; along these lines making the procedure functional and impetus accessible. The structures of the impetuses delivered have been found out by the utilization of FTIR, XRD and TEM. The particles of CuO delivered by the either procedure are monoclinic and that of Co3O4 are spinel: though the blended morphology has been watched for the composites of CuO/Co3O4 created by both of the procedure i.e substance ignition, sol-gel auto burning, aqueous and warm disintegration.

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INTRODUCTION

The successful commercialization of nanomaterials is possible only when the material production and application development proceed together. Material production is a thought-provoking process as the surface functionalities of nanoparticles have to be tailored keeping in mind the applications. For example, through surface modification, a number of properties of nanoparticles like dispersability in a suspension, compactability during subsequent consolidation,
colour in case of metal and semiconductor quantum dots and compatibility with the matrix material in the case of nano composites can be altered. In general practice, multiple synthesis techniques have to be assessed for each application. Other challenges in synthesis include consistency in product quality, cost of raw material and equipment, yield of the product, safety of the process, waste disposal and environmental issues.

PREPARING CUO-CEO2 BIMETALLIC OXIDE CATALYST

CuO-CeO2 bimetallic oxide catalysts were prepared by co-encouraging copper (II) nitrate (GR grade, Merck, Darmstadt, Germany) with cerium (III) nitrate (GR grade, Merck, Darmstadt, Germany) using K2CO3 (0.2 M) at four molar ratios: 6:4, 7:3, 8:2 and 9:1. The pH of the coprecipitating aqueous solution was 11.5 ± 0.2. After filtration, the co-accelerating aqueous solution was washed five times with deionized water, and dried at 393 K. These compounds were then calcined at 773 K in an air stream for 4 hours. The powder thus produced was formed into tablets using acidic corrosive as a folio. The tablets were later reheated at 573 K to consume the fastener out of the CuO-CeO2 bimetallic oxide catalyst. They were then crushed and sieved into particles of various sizes from 0.15 and 0.25 mm, for later use.

Characterizing the Solid Phase -

X-ray diffractograms were obtained using a Diano-8536 diffractometer with CuKα radiation as the source. During analysis, the sample was scanned from 20 to 80° at a pace of 0.4°/min. Diffuse reflectance FTIR spectra of species adsorbed on the catalyst were measured at room temperature using a Bruker Vector 22 FTIR spectrometer, equipped with a diffuse reflectance connection with a resolution of 4 cm-1 (Bruker, Germany). The changes in the sizes of the catalytic particles were measured using a laser light-scattering molecule size analyzer (PSA, Coulter LS100, USA). Scanning electron microscopy, using a vitality dispersive X-beam spectrometer (SEM/EDX, JEOL, JSM-6400, Kevex, DeltaII), yielded the morphology of the catalysts and provided data on the distribution of copper and cerium on the surfaces of the catalysts.

Response System -

Experiments were conducted on a cylindrical fixed-bed stream quartz reactor (TFBR). Two streaming gases, NH3 and O2, were used to set up the feed blend in the weakening gas, helium, which flowed into the bay of the reactor. A mass stream controller was used to control freely the flows of smelling salts and oxygen. Amazingly unadulterated helium was used as a transporter gas at a stream rate from 8 to 13 L/min, controlled using a mass stream meter (830 Series Side-TrakTM, Sierra, Monterey, CA, USA). The mass of every catalyst was 1 g and the vacant bed volume was around 1.2 cm3 . An inactive material formed from (hydrophilic and latent) γ-Al2O3 spheres was used to increase the interfacial region

between the solid and the gas phase to increase the mass transfer of alkali from gaseous streams. This approach resembled that of Huang (2000), who conducted experiments on the catalytic oxidation of smelling salts. A response tube with a length of 300 mm and an internal width of 28 mm was placed inside a split cylinder heater. The cylinder that contained the catalyst was placed in the same heater. The temperature was measured using two sort K thermocouples (KT-110, Kirter, Kaohsiung, Taiwan), each with a distance across of 0.5 mm, these were located before and behind the catalytic bed. The thermocouples were also connected to a PID controller (FP21, Shimaaen, Tokyo, Japan) to keep up the temperature in the cylinder inside ±0.5%. The concentration of the feed gas (GHSV, 92,000 ml/h-g) was maintained at 1,000 ppm NH3 and the O2 concentration was 4%. The catalyst was not deactivated during testing. Fig. 1 schematically depicts the rounded fixed-bed response system (TFBR).

Analyses -

When the response, samples were naturally injected through a sampling valve into a gas chromatograph (Shimadzu GC-14A), equipped with a warm conductivity identifier. A stainless-steel column (Porapak Q 80/100mesh) was used to separate and decide the concentrations of N2O isothermally at 100 ℃. The areas associated with the signals were electronically measured using an information integrator (CR-6A, Shimadzu, Kyoto, Japan). Weaken sulfuric corrosive was used to scrub the residual smelling salts in the vapor gas and the sum present was measured using a Merck pack (Merck, Spectroquant Vega 400, Darmstadt, Germany). The concentrations of NO, NO2,, and O2 in the gas samples were monitored continuously during combustion at a specific area, using a versatile vent gas analyzer (IMR-3000, Neckarsulm, Germany). Information were collected when the SCO response was in a steady state, ordinarily after 20 min at every temperature. Every temperature was maintained for 90 min to enable the system to enter a steady state. Most experiments were repeated once to ensure reproducibility, and similar results were always obtained.

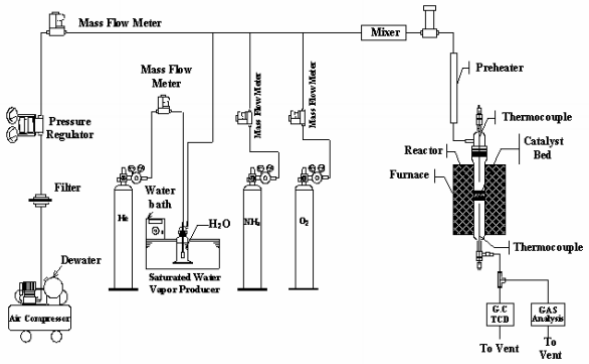

Fig. 1. Schematic diagram of the tubular fixed-bed reaction (TFBR) system.

MATERIALS

Copper ferrite and cobalt ferrite were prepared by a precipitation method. Iron nitrate Fe(NO3)3 9H2O (Merck, 98%), copper nitrate Cu(NO3) 3H2O (Sigma Aldrich, 99%), and cobalt nitrate Co(NO3)2 6H2O (Sigma Aldrich, 99%) were used as precursor salts. Sulfuric corrosive (H2SO4) (Mallinckrodt, 98%) was used so as to wash the composites. Nitric corrosive (HNO3) and hydrofluoric corrosive (HF) (Merck, 68%), were employed in corrosive disintegration tests. Silver nitrate (AgNO3) responsive evaluation (Scharlau) was employed with the expectation of complimentary cyanide measurement by titration. Sodium cyanide (NaCN) and sodium hydroxide (NaOH) of a specialized evaluation were employed for oxidation essays.

SYNTHESIS OF COPPER FERRITE

For the synthesis of copper ferrite (CuFe), a solution of Cu(NO3) 3H2O and Fe(NO3)3 9H2O receptive evaluation was used with a Cu:Fe molar proportion of 1:2 [24]. At that point, a 1.0 M NaOH solution was added until the blend reached a pH estimation of 6 with constant attractive stirring for 2 h. The encourage was then filtered and washed with distilled water. Afterwards, the hasten was dried at 110 C for 16 h and after that calcined for 4 h at 750 C. At long last, a corrosive wash was carried out with 2% v/v sulfuric corrosive to dispose of the soluble copper oxides.

SYNTHESIS OF COBALT FERRITE

A 0.5 M solution of cobalt nitrate and iron nitrate was prepared with a molar proportion Co:Fe = 0.5, and a 0.4 M solution of sodium hydroxide. The nitrate solution was placed in a vessel and subjected to constant attractive stirring, while the hastening operator, sodium hydroxide, was added until the blend and reached a pH worth equivalent to 7. The stirring was carried out at room temperature for 2 h. At that point, the accelerate was filtered and washed with distilled water. Afterwards, the encourage was dried at 110 C for 16 h lastly calcined at 750 C for 4 h. So as to take out the possible cobalt and iron oxides present in the obtained item, a corrosive wash was carried out with a 2% v/v sulfuric corrosive solution until a transparent solution was obtained as a result of the filtration.

ACTIVATED CARBON-SPINLES COMPOSITES PREPARATION

The activated carbon-spinels composites were prepared by including activated carbon of 280 m2/g (AC280) and 1000 m2/g (AC1000) of specific surface in the copper-iron and cobalt-iron solutions. The mass proportion among ferrite and activated carbon was 1:1 and a 5.0 M sodium hydroxide solution was added so as to achieve a pH = 12

under continuous stirring for 1 h. At that point the obtained composites were dried medium-term at 110 C and employed for oxidation essays. An extra calcination stage was implemented as well, which consisted of warming for 2 h at 700 C in a pot with a cover, so as to avert the combustion of carbon. At long last, the corrosive wash was carried out with a sulfuric corrosive solution 2% v/v so as to dispose of all copper and cobalt oxides.

CHARACTERIZATION

Ferrites and activated carbon composites were characterized using a X-beam diffractometer (XRD) Bruker AXS D8 Advance model (Bruker, Karlsruhe, Germany) and a scanning electron microscopy (SEM)-electric dispersive scanning (EDS) Vega (TESCAN, Brno, Czech Republic), as well as a BET surface region with a Quantachrome Instruments Nova4200e (Quantachrome Instruments, Boynton Beach, FL, USA). So as to decide the impregnation of copper and cobalt ferrites on activated carbon, all composites were disintegrated with 6 mL of HNO3 and 2 mL of HF. Copper and cobalt in the solutions were analyzed by nuclear absorption spectroscopy with a Perkin Elmer AAnalyst 300 spectrometer (Perkin Elmer, Shelton, CT, USA). The impregnation level of copper and cobalt ferrites on activated carbon was determined from the accompanying condition:

$$\%\text{Impregnation} = \frac{\text{Copper or cobalt ferrite mass}}{\text{Active carbon mass}} \quad 100\%$$

CATALYTIC OXIDATION PROCEDURE

Catalytic movement of the ferrites and activated carbon composites was measured during all oxidation essays. The oxidation was carried out in a 500 mL volume reactor with continuous mechanical stirring of 480 rpm and air bay of 180 NL/h at room temperature (20 C). A 500 mg/L solution of NaCN was prepared, the pH was adjusted all through the oxidation procedure to an incentive above 10.5, with the expansion of NaOH. The proportion between the material and the cyanide concentration was maintained at 15 g composite/g CN/L. Samples of 5 mL were taken every hour to investigate the residual cyanide by titration with a 4.33 g/L AgNO3 solution. Likewise, a set of 10 mL samples were taken every hour for 8 h so as to measure the dissolution of copper and cobalt in the cyanide solution by nuclear absorption spectroscopy. The cyanide particle oxidation was determined with the accompanying condition:

$$\%\text{Oxidation} = \frac{[CN]_{t=0} - [CN]_t}{[CN]_{t=0}} \quad 100\%$$

The catalysts samples were named CuFe and CoFe for mass copper and cobalt ferrites, respectively. CuFe-AC280, CuFe-AC1000, CoFe-AC280, and CoFe-AC1000 for supported catalysts with activated carbon of 280 m2/g and 1000 m2/g of specific surface, respectively.

METHOD

The successful commercialization of nanomaterials is possible just when the material generation and application improvement proceed together. Material creation is an interesting process as the surface functionalities of nanoparticles must be tailored remembering the applications. For instance, through surface adjustment, various properties of nanoparticles like dispersability in a suspension, compactability during subsequent consolidation, color in case of metal and semiconductor quantum dots and compatibility with the grid material on account of nano composites can be altered. By and large practice, various synthesis techniques must be assessed for every application. Different challenges in synthesis incorporate consistency in item quality, cost of crude material and gear, yield of the item, safety of the process, waste disposal and ecological issues.

In short for a researcher, selection of material is significant task as each step of processing has to be deliberately monitored and controlled to get the best item and it is among the main considerations which choose the suitability/feasibility of chosen field of research work. It is must that materials to be used for synthesis of desired compound should be of high virtue, ease, easily accessible and storable, and should be non-lethal or if nothing else less poisonous or more each of the a trustworthy supply source.

Basically the entire research work was carried out in the parent University, Eternal University in the Laboratories of Akal School of Chemistry, Nanotechnology and for Analysis/Characterization of the material viz., XRD, FTIR, TEM, facilities from SAIF Punjab University Chandigarh (PU) and Himachal Pradesh University (HPU) were taken and every one of them gave overpowering response. All the crude materials, chemicals, glass and plastic product and instruments were purchased from reputed firms like SD Fine, Merck, Rankem, Tarson, Him Media, Perfit India and Avantor and so forth.

The objective of any synthetic method for nanomaterial is to create a material that exhibits properties that are a result of their characteristic length scale being in the nanometer extend (~1 - 100 nm). Accordingly, the synthetic method should show control of size in this range so that some property can be attained. The two basic approaches to making nanomaterials and the creation of Nano structures include either a top-down approach whereby an existing solid is bit by bit reduced in size using some outside radiation or potentially substance, and a Bottom up approach refers to the development of a material from the base: particle by iota, atom by atom or cluster by cluster. The two

approaches assume significant job in present day industry and in nano innovation. There are advantages and disadvantages in the two approaches.

Base up methods by and large fall into two categories: confused and controlled. Tumultuous processes include lifting the constituent atoms or molecules to a confused state and after that suddenly changing the conditions so as to make that state unstable. The collapse from the confused state can be troublesome or impossible to control thus collective statistics frequently oversee the resulting size distribution and normal size. Examples of Chaotic processes are: Laser removal, Exploding wire, Arc, Flame pyrolysis, Combustion, Precipitation synthesis techniques.

Controlled processes include the controlled conveyance of the constituent atoms or molecules to the site(s) of nanoparticle arrangement such that the nanoparticle can develop to a prescribed size in a controlled way. For the most part the states of the constituent atoms or molecules are never a long way from that needed for nanoparticle arrangement. Examples of controlled processes are, self-constraining development solution, self-restricting compound vapor precipitation and shaped pulse femtosecond laser techniques, profound and extraordinary UV lithography.

Steady loss or Milling is a regular top down method in making Nanoparticles. The biggest issue with top down approach is the flaw of surface structure and significant crystallographic harm to the processed patterns. These imperfections lead to additional challenges in the gadget design and manufacture. Be that as it may, this approach leads to the mass creation of nano material. Regardless of the defects produced by top down approach, it will continue to assume a significant job in the synthesis of Nanostructures. In spite of the fact that the base up approach regularly referred in nanotechnology, it's anything but a more up to date concept. All the living beings in nature observe development by this approach just and furthermore it has been in industrial use for over a century. Examples incorporate the generation of salt and nitrate in substance industry. At the point when structures fall into a nanometer scale, there is somewhat chance for top down approach. Base up approach also promises a superior opportunity to get nano structures with less defects, progressively homogeneous substance composition. On the contrary, top down approach most likely introduces inward stress, notwithstanding surface defects and contaminations. Herewith we have chosen to pursue the base up strategy to synthesize the desired nano structures.

It is observed that the physical and synthetic properties of the nanomaterials rely on various parameters such as synthesis techniques, compound composition, microstructures, and so forth. Out of all the above parameters, synthesis methods assume a vital job in modifying the size, morphology and various properties of nanomaterials. Various methods for the synthesis of nanomaterials are Mechanosynthesis method [Sepelak et al. 2007], Conventional fired method [Shaheen and Ali 2001; Goya et al. 1998, Kenfack and Langbein 2004], RF sputtering method [Desai et al. 2002], Egg white precursor method [Maensiri et al. 2007], Combustion response method [Ahlawat et al. 2011], Thermal decomposition method, Sol-gel method [Malik et al. 2010], Reverse micelle method [Kale et al. 2004], Aerosol method [Singhal et al. 2005], Co-precipitation method [Maaz et al. 2010], Mechanical processing method [Kodama et al. 1996], Citric corrosive combustion method [Zhu et al. 2006], Organic gel warm decomposition method [Guo et al. 2010], Hydrothermal method [Li et al. 2010], Self-propogation [Cross et al. 1999], Solvothermal method [Wang et al. 2009], and so on.

Each method has its very own benefits and limitations. Contingent on the individual and distinctive features, four unique methods were choosen from the previously mentioned methods to synthesize nanomaterials that can go about as catalyst. The four methods used were Thermal decomposition method, Hydrothermal method, Sol-gel method, and Chemical combustion method. Compound combustion method involves simple calculations to get ready nanomaterials. Homogeneous and unagglomerated powder can be synthesized from inexpensive crude materials no sweat and comfort. Aqueous method is a low temperature synthesis course used to synthesize nanomaterials with various structures like nanorods, nanowires, nanospheres, nanoparticles, and so forth. Warm decomposition method produces nanomaterials by the compound decomposition caused by warmth. It is commonly done at the temperature at which substance artificially decomposes. The response is usually exothermic as warmth is required to break concoction bonds. Sol-gel method produces nanomaterials of high physical consistency with respect to the distribution of components and porosity which is possible by having complete control over molecule interactions. This method is ready to create fine powder materials with various morphologies and properties.

Chemical Combustion method: -

Concoction combustion synthesis is also known as 'Self-propogating high temperature synthesis' (SHS) and fire or furnaceless synthesis. The process involves the redox concoction response occurring among metals and nonmetals and the idea of the response is exothermic. The method has discovered its use in the arrangement of huge number of oxides (attractive dielectric, semi conducting, headstrong oxides, insulators, catalysts, sensors, and so forth.) and non-oxide (carbides, borides, silicides, and so forth.) materials. This method is simple, fast, and vivaciously

economic and yields high virtue products as compared to the conventional routes.

Substance combustion synthesis is one of the concoction method that showed promising results in the development of oxide compounds at lower temperature [Aruna and Mukasyan 2008]. By playing out the trial under right conditions, it is possible to get unadulterated metal oxide legitimately with no toughening [Ianoş et al. 2008]. This method does not permit the arrangement of item by the outer warmth sources instead; the item is formed by an exothermic self-proliferating combustion response that occurs between aqueous solutions of the desired metal nitrate and fuel mixtures at low temperatures (~300oC). The compatibility between the metal nitrates and fuel is tentatively determined [Ianoş and Lazău 2009]. The results of the response are by and large CO2, H2O and NOx and are considered eco-accommodating.

Combustion method involves a self-sustained response in homogeneous solution of metal nitrates (oxidizers) and fuels (eg. urea, glycine, hydrazides). Solution combustion synthesis may happen as either volume or layer-by-layer engendering combustion modes relying on the sort of the precursors as well as the conditions used for the process association. Inspite of yielding nanosize oxide materials, this method also allows homogeneous doping of follow measure of uncommon earth polluting influence ions in a single step [Aruna and Mukasyan 2008].Chemical combustion synthesis has several advantages such as the start temperature is lower than the phase transition temperature and the arrangement of as synthesized powders with small mean molecule size and huge surface areas [Ianoş and Lazău 2009]. In addition, this method is simple and inexpensive to synthesize fine materials and between metallic compounds using just one step process [Manoharan et al. 1992; Manoharan and Patil 1993; Jose et al. 1999], it is a solution process and thus it has control over the homogeneity and stoichiometry of the products, it is possible to incorporate desired debasement ions in the oxide hosts and get ready industrially useful materials such as pigments, phosphors as well as high Tc cuprates and SOFC (Solid oxide fuel cell) materials and being simple and fast process, it doesnot need any special gear as in the self-propogating high-temperature synthesis [Patil et al. 1997].

Single step synthesized nano-ZnO/carbon composite is observed to have more capacitance as compared to micron sized ZnO powder [Yan et al. 2008]. Nano titania synthesized by combustion method was applied as a dainty film in color sensitized solar cells and had shown a high light to power conversion yield. Also nano TiO2 synthesized through combustion method has shown higher catalytic action for cancer-causing hexavalent chromium Cr (VI) as compared to Digussa P-25 TiO2 [Aarthi and Madras 2008]. Nano crystalline MgO synthesized by combustion synthesis had shown 97% evacuation of fluoride present in water as compared to 76% by regenerated MgO and 17% by commercial evaluation MgO [Nagappa and Chandrappa 2007]. The incorporation of solution combustion synthesized nanoparticles (such as zirconia, alumina, ceria, alumina-zirconia, and so forth.) into metal network during electrodeposition had shown better results such as enhanced grid properties including microhardness, wear resistance and corrosion resistance [Aruna et al. 2006]

It has been entrenched that fuel is a significant component for the arrangement of oxides by combustion synthesis. Urea and glycine are considered as the most prominent and appealing fuels for yielding exceedingly uniform, complex oxide powders with precisely controlled stoichiometry [Aruna and Mukasyan 2008]. Because of the hazardous idea of nitrogen oxides and CO formed (as by products) while using glycine as fuel, several new organic compound are explored as fuels such as alanine, asparagine, serine, methyl cellulose, ammonium acetic acid derivation, ammonium citrate and ammonium tartarate [Ianoş et al. 2008; Edrissi and Norouzbeigi 2007]. In this method, response occurs in homogeneous solution containing various oxidizers (metal nitrates) and fuel (urea, glycine, and so on.) in stoichiometric amounts. For the most part nitrates of the corresponding metals are preferred because they serve as low temperature oxidant source for synthesis [Selvan et al. 2003]. Fuel lowers the decomposition temperature of metal nitrates. Thusly the selection of fuel is basic in the process. Response completes with the development of huge volume of gases which leads to the arrangement of metal oxide nanopowders. Setkol et al. what's more, Yu et al. proposed that the advancement of gases limits the event of agglomeration [Sertkol et al. 2010; Yu et al. 2006]

Preparation of Co3O4 nanomaterial **-** Crude materials used to plan Co3O4 are cobalt nitrate [Co(NO3)2.6H2O], urea [NH2CONH2], ethylene glycol/polyethylene glycol [(C2H4O)/(C2H4O)n+1H2O], methanol [CH3OH] and twofold distilled water. Nitrates go about as oxidizers, urea as fuel whereas EG/PEG go about as reducing agents.

Cobalt nitrate (0.1mol; 29.103 g) and EG/PEG in stoichiometric proportions were taken in a 500 ml measuring utencil and were stirred without warming for homogeneous blending for 10 minutes. The solution so obtained was then poured into a 1000 ml measuring utencil which was continued stirring for 10 minutes at 30oC. Further stirring was done at 40oC for an additional 10 minutes. After that urea (0.1mol, 6.0g) was added in stoichiometric proportion and temperature was raised upto 70oC. As the response was completed, enormous volume of gases accompanied by dark frothy mass/powder was obtained The powder was crushed, grounded and afterward washed with methanol and water three times each to expel any unwanted impurities. The item obtained was then dried and further annealed at

400oC, 500oC and 600oC in artistic crucibles in a programmed heater. It is observed that the savage conduct of the response between metal nitrates and urea is especially controlled by the expansion of EG/PEG which might be because of the development of complex between CO gathering of EG/PEG with metal ions.

Preparation of CuO nanomaterial **-** To synthesize CuO nanomaterial, crude materials used were Cupric nitrate [Cu(NO3)2.3H2O], urea [NH2CONH2], ethylene glycol/polyethylene glycol [(C2H4O)/(C2H4O)n+1H2O], methanol [CH3OH] and twofold distilled water. The item synthesized by this method was annealed at 400oC, 500oC and 600oC. Methodology to set up the samples was similar as explained in the above section.

Preparation of Co3O4/CuO nanomaterial **-** To synthesize CoCuO nanomaterial, crude materials used were cobalt nitrate [Co(NO3)2.6H2O], Cupric nitrate [Cu(NO3)2.3H2O], urea [NH2CONH2], ethylene glycol/polyethylene glycol [(C6H6O2)/(C2H4O)n+1H2O], methanol [CH3OH] and twofold distilled water. Both cobalt nitrate and cupric nitrate were taken in 1:1 proportion and rest of the procedure is similar to the procedure in the above section. The nanomaterials obtained were annealed at 400oC, 500oC and 600oC. The entire procedure of concoction combustion is shown schematically in the stream graph given underneath:

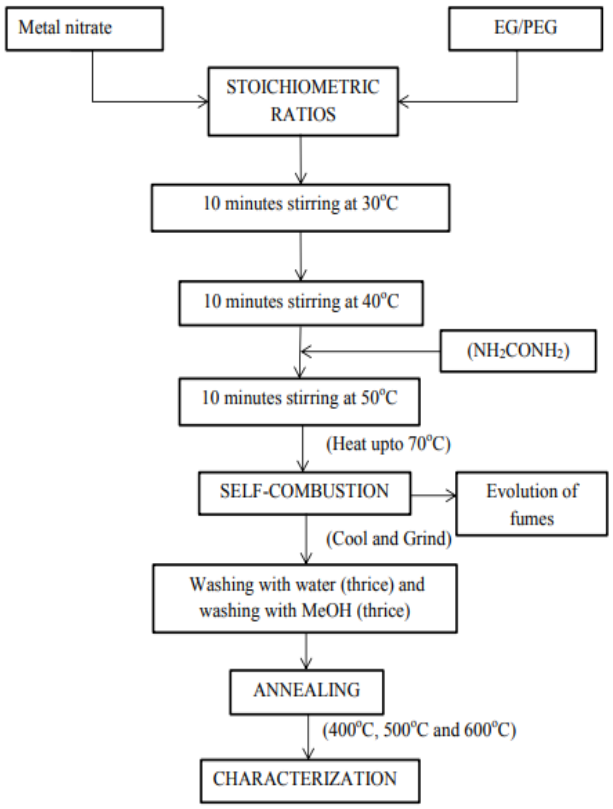

FIGURE 2: Preparation of nano catalysts via chemical combustion method

Sol-gel autocombustion: -

Sol-gel auto-combustion method (also called low-temperature self-combustion, auto-start or self propogation, as well as gel-warm decomposition) involves the combination of concoction sol-gel and combustion process. It might also be defined as the method that involves the arrangement of precursor gels from aqueous solutions of metal nitrates and an organic complexant such as citrus extract. The nitrate-citrate gels, when heated in a hot heater, consume in a self-propogating process, quickly converting the precursor mixtures legitimately into products [Cannas et al. 2006]. This method is found to have incredible potential in the synthesis of spinel type nanomaterials. This method is commonly known as solution combustion method [Aruna and Mukasyan 2008]. During the last decade, this method has been used for the synthesis of in excess of 20 diverse spinel ferrite compounds [MFe2O4], where M could be Ni, Zn, Co, Cu, Li, Mg, Cd, Mn ions or its combination [Sutka and Mezinskis 2012]. As of late this method has also been used to synthesize 1D and 2D nano structures [Guo et al. 2010]. The synthesized products from this method have discovered numerous applications in the field of electronics [Azadmanjiri 2008], science [Biasi et al. 2007], attractive [Shukla et al. 2008], radar absorbing materials [Aphesteguy et al. 2009], magnetoelectric composites [Atif et al. 2011], gas sensing [Sutka et al. 2012], photocatalytic movement [Guo et al. 2004] and catalysis [Costa et al. 2006]. This method involves the exothermic and self-sustaining thermally-induced anionic redox response of xerogel, which is obtained from aqueous solution containing desired metal salts (oxidizer) and organic complexant (reductant). Nitrate salts are considered progressively good because of water-soluble low temperature NO3-oxidant source for synthesis [Selvan et al. 2003]. In some cases, metal nitrates and complexant are legitimately mixed together using stirring followed by warming without including water. Since metal nitrates are hygroscopic in nature, they easily absorb moisture and become slurry. This sort of sol-gel auto-combustion is known as Flash-combustion method [Mangalaraja et al. 2003]. The xerogel combustion leads to the quick development of enormous volume of gases accompanied by extraordinary mass loss during the arrangement of ferrite nano powders. Advancement of gases limits the event of agglomeration [Yu et al. 2006].

This method is observed to have numerous advantages such as great substance homogeneity (blending of cations of desired composition at atomic level); high item immaculateness and crystallinity; fine molecule size and tight molecule size distribution; it is easy to control stoichiometry; dopants can be easily introduced into the last item; simple hardware and readiness process; low processing time; low outside vitality consumption (process initiates at low temperature) and different

steps are not involved [Costa et al. 2007]. It has been discovered that the synthesis of spinel nanocompounds involves various kinds of complexant agents (such as citrus extract, urea, glycine, hydrazine, ethylene glycol, carbohydrazide, alanine, acidic corrosive, acrylic corrosive), out of which urea [CO(NH2)2], glycine [NH2CH2COOH] and citrus extract [C6H8O7] are considered most prevalent. Complexant agents with carboxylate or amine groups assume a significant job in water-soluble complex precursor synthesis course. Thus citrus extract contains carboxylate gathering, glycine contains carboxylate and aliphatic amine groups while urea contains aliphatic amine groups [Wu et al. 2006]. During the synthesis, the complete valencies of metal salts should be balanced by all out valencies in the complexant [Costa et al. 2008]. By choosing suitable complexing specialist, oxygen equalization worth or concentration and sort of compound additives, as well as warming source and atmosphere, it has been possible to control system stability, xerogel smaller scale structure, response temperature, rate and volume of gases generated.

Citrus extract is one of the most as often as possible used complexant in huge assortment of sol-gel auto-combustion synthesis. It is inexpensive and is preferable complexing specialist over hydrazine and glycine in delivering fine powder with smaller molecule size. As a rule, citrus extract is frequently accompanied by the expansion of specific amounts of smelling salts which might be attributed to their use in overcoming drying stresses and contributes to the porosity and strength of the sol-gel organize [Sutka and Mezinskis 2012].

Hua et al. synthesized nonporous iron metal foams by sol-gel auto-combustion method. Extremely low density and very enormous saturation polarization has been observed in synthesized samples [Hua et al. 2012]. Yue synthesized NiCuZn ferrite powders using sol-gel autocombustion method and found that the innovation has a favorable position of minimal effort, simple arrangement process and results in nano-sized dynamic powders. The item obtained has fine-grained microstructure highlight and possesses great recurrence stability and amazing element yet low porousness factor compared to those of the samples prepared by conventional course [Yue et al. 2000].

Preparation of Co3O4 nanomaterial: - Every one of the chemicals used were of investigative evaluation. Cobalt chloride and citrus extract were used as starting materials. Ethanol and twofold distilled water were mixed (1:5) using attractive stirrer for 30 minutes and the homogeneous solution prepared was used as dissolvent. Stoichiometric measure of cobalt chloride (0.1M) and citrus extract were dissolved in (1:5) ethanol: water solution. The solution was continuously stirred using an attractive stirrer. At that point the pH of the solution was adjusted to 7 using smelling salts alongside stirring. At that point the mixed solution was poured into a dish and heated and stirred continuously to transform it into xerogel. At a legitimate temperature, start started and the dried gel consumed it in a self-propogating combustion way until all the gel was worn out completely to frame a loose powder. The desired powder was calcined at 450oC for three hours.

Preparation of CuO nanomaterial: - Cupric chloride and citrus extract were used as starting materials. Ethanol and twofold distilled water were mixed (1:5) using attractive stirrer for 30 minutes and the homogeneous solution prepared was used as dissolvent. Stoichiometric measure of cuprous chloride (0.1M) and citrus extract were dissolved in (1:5) ethanol:water solution. The solution was continuously stirred using an attractive stirrer. At that point the pH of the solution was adjusted to 7 using alkali alongside stirring. At that point the mixed solution was poured into a dish and heated and stirred continuously to transform it into xerogel. At an appropriate temperature, start started and the dried gel consumed it in a self-propogating combustion way until all the gel was worn out completely to frame a loose powder. The desired powder was calcined at 450oC for three hours.

Preparation of Co3O4/CuO nanomaterial: - Cobalt chloride, Cupric chloride and citrus extract were used as starting materials. Ethanol and twofold distilled water were mixed (1:5) using attractive stirrer for thirty minutes and the homogeneous solution prepared was used as dissolvent. Stoichiometric measure of cobalt chloride (0.1M), cuprous chloride (0.1M) and citrus extract were dissolved in (1:5) ethanol: water solution. The solution was continuously stirred using an attractive stirrer. At that point the pH of the solution was adjusted to 7 using alkali alongside stirring. At that point the mixed solution was poured into a dish and heated and stirred continuously to transform it into xerogel. At an appropriate temperature, start started and the dried gel consumed it in a self-propogating combustion way until all the gel was worn out completely to shape a loose powder. The desired powder was calcined at 450oC for three hours. The entire procedure for sol-gel autocombustion is shown schematically in the stream outline given beneath:

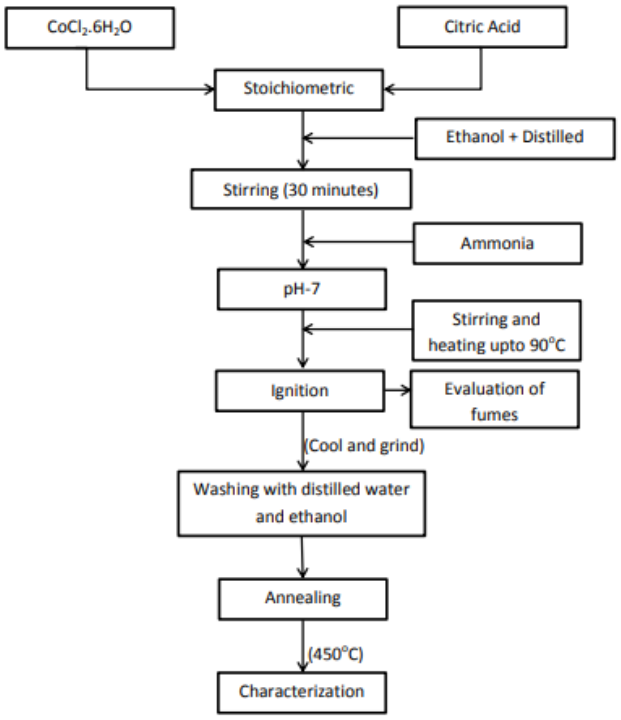

FIGURE 3: Preparation of nano catalysts via sol-gel autocombustion method

HYDROTHERMAL METHOD:

Hydrothermal synthesis might be defined as a process that utilizes single or heterogeneous phase reactions in aqueous media at elevated temperature (T > 25oC) and pressure (P > kPa) to cryatallize materials legitimately from solution. Maximum breaking point of hydrothermal synthesis may broaden upto 1000oC and 500 MPa pressure [Roy 1994] however mellow conditions having temperature less than 350oCand pressure less than 50 MPa are preferred. With the passage of time, intensive research has significantly led to better understanding of the process with significantly reduced response time, temperature and pressure for the crystallization of materials (T < 200oC, P < 1.5 MPa) [Yoshimura et al. 2000; Gersten et al. 2002].

Hydrothermal method is one of the methods used to get ready nanomaterials with homogeneity and consistency. In addition this method can be used to synthesize assortment of nanostructures with various morphologies like rods, spheres, wires, and so on and that too in a reproducible way. To synthesize the nanomaterials, a hydrothermal apparatus was designed and fabricated by the division with limit of delivering 10-15 g sample for every clump. The mechanism of the reactions in a hydrothermal method follows a liquid nucleation model. The guideline involved comprised theories of substance harmony, synthetic kinetics and thermodynamic properties of aqueous systems under hydrothermal conditions.

The apparatus required to do hydrothermal reactions are autoclaves or high pressure reactors. At these sub-supercritical conditions (180o-200oC) water acts as reactant and accelerates the kinetics of the reactions. Ionic species are soluble in water and the solubility increases with increase in temperature and the lower viscosity of water exhibits more prominent portability. The increase in the portability of ions promotes Ostwald's aging to continue at a fast pace in this manner increasing the consistency of the material. Time and temperature have pronounced impact in hydrothermal reactions for size and morphological control. Also phase immaculateness of the nanomaterials is controlled by response conditions of precursor material and pH.

The development of nanoparticles occurs by means of Ostwald's aging. Hastening agents plays a significant job in choosing the crystallinity of the nanostructures. It is discovered that size of the spectator particle of the encouraging operator has inverse connection with crystallinity for example bigger the size of the spectator particle less fortunate the crystallinity would be. The wonder is seemingly because of an adjustment in the electrostatic capability of the metal hydroxides. Lower the electrostatic capability of the metal solution, more noteworthy is the possibility that the metals sol coagulate. Because of the increase in side reactions during coagulation, stirring is frequently used to separate coagulating sols. Stirring results in consistency and phase virtue. This method provides an immediate, low temperature course for the synthesis of nano-oxide powders with thin size distribution maintaining a strategic distance from the calcination step. Also, the benefits of the method incorporate ease for instrumentation, vitality and starting materials [Inoue et al. 1998; Bae et al. 1998].

Hydrothermal synthesis has several advantages for various systems such as in clay powder creation there are less time and vitality consuming processing steps since high temperature calcination, blending and processing steps are either not necessary or minimized. Additionally the precipitation of the officially crystallized powders straightforwardly from solution regulates the rate and consistency of nucleation, development and maturing which leads to progress in control of size and morphology of crystallites with significant reduction in conglomeration levels [Pugh and Bergstrom 1993]. This method also has a favorable position of synthesizing materials with high virtue than the starting materials which might be attributed to the way that hydrothermal synthesis itself is a self-decontaminating process in which the developing crystals/crystallites will in general reject the impurities present in the development condition and thus the impurities are removed from the system together with the crystallizing solution [Suchanek and Riman 2006].

Hydrothermal synthesis can happen in a wide assortment of combinations of aqueous and solvent blend based systems. In comparison to solids, liquids permit the possibility for increasing speed of diffusion, adsorption response rate and crystallization under hydrothermal conditions [Yoshimura et al. 2000]. Substitution of water should be possible either by appropriately adjusting the synthesis conditions or by use of non-aqueous solvents (solvothermal processing). Because of its bit of leeway for continuous materials generation, it very well may be especially useful in continuous creation of artistic powders [Dawson 1988]. This method is considered as one of the earth benevolent process because of the reasons such as vitality conserving low processing temperatures, absence of processing, capacity to reuse waste, safe and convenient disposal of waste that can't be recycled [Adschiri et al. 2000]. Thermodynamic variables such as temperature, pH, concentration of reactants and additives, decide not just the processing space for a given material yet additionally impact both response and crystallization kinetics including shape and size.

Hydrothermal synthesis has the ability to control the structure of nanomaterials running from nanoparticles to nanorods or nanourchins to nanotubes by appropriately choosing the parameters such as temperature, time and solvent [Subramanian et al. 2005]. Bar cylinder like Mg(OH)2 nanocrystallites were synthesized by means of hydrothermal method. Results demonstrated that the crystallite size, shape and structure of the obtained nanoparticles could be controlled by this method even with the most commonly used magnesium slats or magnesium powder. The control over the morphology of nanocrystals with well-defined shape and uniform size remains a significant objective of present day synthetic chemistry, because shape and size controlled nanocrystals are promising candidates as dynamic components in a wide scope of innovative applications and are model systems for basic research. Thus synthesis of nanocrystals with novel shapes and guaranteed alluring physical and substance properties has become a matter of interest [He et al. 2004]. Very stable, 0D and 2D cubic spinel Co3O4 nanospheres and hexagonal platelets were synthesized using solvothermal method. The adjustment in morphology from 0D to 2D is observed to be response time subordinate. The results have shown that Co3O4 is a superior capacitive material as shown by the high capacitance achieved in the trial [Liang et al. 2009].

For the past couple of years, there has been an interest in structure structures with open crystallite lattices or expandable layer structures that can experience redox reactions and in this manner can be used in electrochemical devices such as batteries, displays and sensors. Such structures are less prone to be thermally stable under high temperature solid state conditions. So, there is need of low temperature approaches. One of this low temperature approach is hydrothermal and is usually performed between 100oC to 200oC. So as to understand the chemistry of the products, number of factors (such as pH of the response medium, its temperature and pressure and cations in the solution) are required to be studied [Whittingham 1996].

As of late researchers have developed a method known as supercritical hydrothermal synthesis which involves the supercritical water in the response process. Supercritical water provides amazing response environments for hydrothermal crystallization of metal oxide particles. At basic purpose of water there have been drastic changes in properties such as density, dielectric constant and ionic item. Thus, the phase conduct for the supercritical water-tight gas (O2, H2, and so forth.) system and response harmony rate can be varied to synthesize new materials or morphologies.

This method has a bit of leeway of fluctuating the response rate and response balance by shifting the dielectric constant and solvent density with temperature and pressure and thus giving benefits such as high response rates and small molecule sizes [Adschiri et al. 1992]. So as to make new materials or upgrade the response kinetics, researchers are using hydrothermal cross breed techniques. Part of efforts are being made to hybridize hydrothermal method with microwaves (microwave-hydrothermal processing), electrochemistry (hydrothermal-elecrochemical synthesis), ultrasound (hydrothermal-sonochemical synthesis), mechanochemistry(mechano substance hydrothermal synthesis),optical radiation (hydrothermal-photochemical synthesis) and hot processing (hydrothermal hot processing).

Adschiri et al. have synthesized Barium hexaferrite, YAG/Tb phosphor and LiCoO2 metal oxide particles by means of hydrothermal method at supercritical conditions and found that LiCoO2 is formed in a single phase at supercritical condition and Co3O4 was the primary item at supercritical conditions which implies that viable oxidation could be achieved in supercritical water because of the arrangement of a homogeneous phase for oxygen gas and water [Adschiri et al. 2000].

Hydrothermal crystallization affords incredible control of morphology (for example spherical, cubic, fibrous and plate like), size (from a couple of nanometers to tens of microns), and level of agglomeration. So as to control the above characteristics following thermodynamic parameters are used such as response temperature, types and concentration of the reactants, notwithstanding non-thermodynamic (active) variables such as stirring speed. The compound composition of the powders can be easily controlled from the perspective of stoichiometry and arrangement of solid solutions. In the twentieth century, hydrothermal synthesis was plainly identified as a significant innovation for material synthesis predominantly in the fields of metallurgy

and single crystal development [Byrappa and Yoshimura 2012].

Cobalt nano oxides at two unique pHs were synthesized by means of hydrothermal methods. The methods incorporate the response of the crude material in the hydrothermal apparatus having Teflon cup in stainless steel autoclave useful at 180o – 200oC lined autoclave at low temperature for a requisite period.

Co3O4 nanomaterial (Ethanol: Water) -

Every one of the chemicals used were of investigative evaluation and were subjected to analysis before usage. Crude material used to get ready Co3O4 were cobalt chloride [CoCl2.6H2O], ethanol [C2H5OH], potassium hydroxide [KOH] and twofold distilled water [H2O].

0.05 M of CoCl2.6H2O was taken in a recepticle alongside the expansion of 40 ml ethanol: water blend (ethanol: water:: 70:30, prepared by stirring for thirty minutes at room temperature). Stirring was accomplished for around 1 hour to set up a homogeneous solution. Alongside stirring, potassium hydroxide [KOH] was added as encouraging specialist and to increase the pH. As the pH increases, precipitation increases. KOH was added up to pH-13 alongside stirring. Further, the sol was stirred for thirty minutes to set up a homogenous sol. The entire material was then transferred to a teflon vessel of limit 100 ml and was kept in a stainless steel autoclave which thus was placed in a programmed heater at 180oC for 48 hours. The precipitates so obtained were washed three times with water and ethanol to expel the suspended impurities and were subjected to warming for 8 hours at 80oC in a programmed stove.

Co3O4 nanomaterial (Water) -

0.05 M of CoCl2.6H2O was taken in a measuring utencil alongside the expansion of 40 ml twofold distilled water. Stirring was accomplished for around 1 hour to set up a homogeneous solution. Alongside stirring, potassium hydroxide [KOH] was added as encouraging specialist and to increase the pH. As the pH increases, precipitation increases. KOH was added up to pH-13 alongside stirring. Further, the sol was stirred for 30 minutes to set up a homogenous sol. The entire material wass then transferred to a teflon vessel of limit 100 ml and was kept in a stainless steel autoclave which thus was placed in a programmed heater at 180oC for 48 hours. The precipitates so obtained were washed three times with water and ethanol to expel the suspended impurities and were subjected to warming for 8 hours at 80oC in a programmed broiler. The entire procedure for hydrothermal method is shown schematically in the stream diagram given beneath:

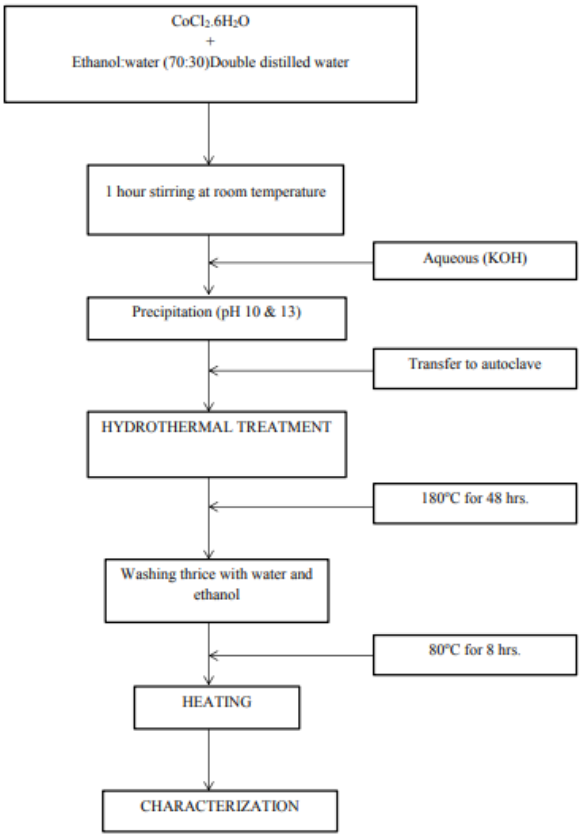

FIGURE 4 Preparation of nano catalysts via hydrothermal method

THERMAL DECOMPOSITION:

Warm decomposition method is known as one of the novel method for the synthesis of nanocrystallites and is a quickly creating research zone [Yin et al. 2005]. It is a lot faster, cleaner and economical. The procedure offers several novel advantages over different methods including easy stir up, low temperature processing, short response time and creation of inorganic nanomaterials with tight size distribution [Farhadi et al. 2013].

Fumitaka Yanaglsawa and Hitoshi Sakal have used this procedure to synthesize Barium sulfate-Vanadium Pentaoxide-silica glass mixtures for the arrangement of sulfur dioxide in sulfur isotope proportion measurements [Yanagisawa and Sakai 1983]. Taeghwan Hyeon et al. synthesized exceedingly crystalline and monodisperse γ-Fe2O3 nanocrystallites fabricated by the controlled oxidation of uniform iron nanoparticles which were generated from the warm decomposition of iron complex with over 80% yield [Hyeon et al. 2001]. Masaud Salavati-Niasari and Fatemah Davar have successfully synthesized Cu2O nanoparticles by means of warm decomposition method with a molecule extend between 8-10 nm. The method is observed to be inexpensive and reproducible process for enormous scale synthesis of copper oxide nanoparticles [Salavati and Davar 2009]. Esumi et al. have synthesized and characterized

bimetallic Pd-Cu colloids by means of warm decomposition method by their acetic acid derivation compounds using organic solvents. The normal width and the dispersion stability of these colloids are observed to be solvent ward [Esumi et al. 1990]. Liang et al. successfully synthesized single crystalline NiO nanosheets by warm decomposition of single crystalline β-Ni(OH)2 nanosheets at 400oC [Liang et al. 2004].

This method is one of the most common methods to deliver nanomaterials with great size control, slender size distribution and astounding crystallinity of individual nanoparticles [Roca et al. 2007] with the capacity of self-assembly [Simeonidis et al. 2007]. Numerous bio-medical applications like attractive resonance imaging (MRI), attractive cell separation and magneto reflaxometry rely upon molecule size and thus this method is conceivably used for these applications.

Synthesis of Co3O4 nanparticles: -

Every one of the chemicals used were of diagnostic evaluation and were subjected to analysis before usage. Crude materials used to get ready Co3O4 were cobalt sulfate heptahydrate [CoSO4.7H2O], sodium hydroxide [NaOH], sodium carbonate [Na2CO3] and twofold distilled water [H2O].

Cobalt sulfate heptahydrate (28.1 g; 0.1 M) of investigative evaluation was dissolved in demineralized water (100 ml) followed by slowl expansion, with stirring, an aqueous solution of sodium hydroxide:sodium carbonate (1:1 w/w) in demineralized water. The pH was adjusted to 8.0 ± 0.2. The precipitated hydroxy carbonate of cobalt was repeatedly washed with demineralized water to free from sulfate ions and afterward dried at 80–85oC. The dried cobalt hydroxy carbonate was calcined at 300–350◦C in an artistic pot for 2 h. The entire procedure is shown schematically in the stream graph given underneath:

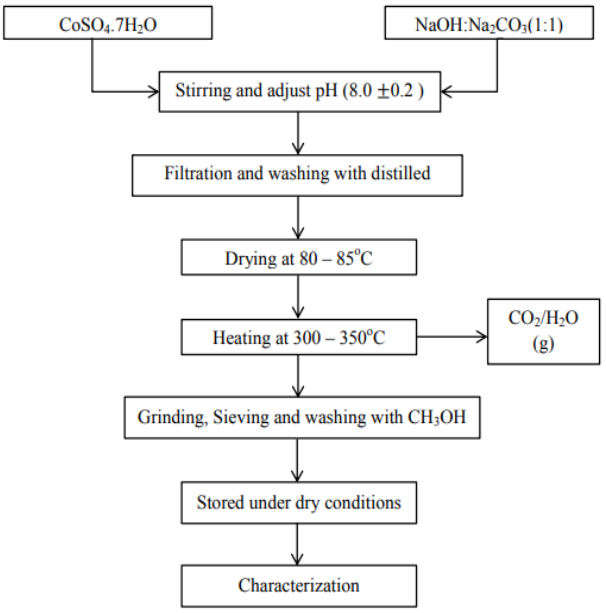

Figure 5: Preparation of nano catalyst via thermal decomposition method

GENERAL CHEMICAL REACTIONS

The general chemical reactions happening in the synthetic methods (chemical combustion, sol-gel autocombustion, hydrothermal and warm decomposition) are shown underneath in Figure 6:

Chemical Combustion

$$\underset{\text{Metal Nitrate}}{M(NO_3)_2.xH_2O} \xrightarrow[\text{Urea, }\triangle]{\text{EG/PEG}} MO/M_3O_4 + NO_x + xH_2O + xCO_2$$

Where MO ⟶ CuO

M_3O_4 ⟶ Co_3O_4

Hydrothermal

$$\underset{\text{Metal Chloride}}{MCl_2.xH_2O} + xKOH\ (aq) \longrightarrow M(OH)_2 + xKCl$$

$$\underset{\text{Metal hydroxide}}{M(OH)_2} \xrightarrow[O_2]{\text{Hydrothermal}} MO/M_3O_4 + xH_2O$$

Sol-gel auto Combustion

$$\underset{\text{Metal Chloride}}{MCl_2.xH_2O} + xNH_4OH \xrightarrow[C_2H_5OH:H_2O,\ \triangle]{\text{Citric acid}} MO/M_3O_4 + xNH_4Cl + xH_2O$$

Thermal decomposition

$$\underset{\text{Metal hydroxy carbonate}}{M(OH)_2.MCO_3} \xrightarrow{\triangle} MO/M_3O_4 + H_2O + CO_2$$

Figure *6 General Chemical reactions*

CATALYTIC OXIDATION EXPLORATORY STUDY:

The term catalysis was introduced by Berzelius in 1836. Ostwald defines catalyst as a species that accelerates a chemical response without influencing the position of the balance. A catalyst is bounded to a response during a catalytic process such that the reactants are bound to one structure the catalyst and the products are released from another, creating the underlying state for example the responding species are continuously adsorbed on to the empty sites and the products are released as soon as produced. Heterogeneous catalysis plays an importrant job in present day innovation. Since 80% of every single industrial chemical are manufactured by catalytic reactions [Hagen 2006].

Because of climbing prices of respectable metals, transition metal oxides are considered as catalyst of decision. Cobalt and copper oxides as catalyst have shown better results in numerous reactions however these reactions are performed mostly under high temperature and intense conditions. We have synthesized cobalt and copper oxides and studied the oxidation of formaldehyde, oxalic corrosive and benzaldehyde using these oxides as catalyst in aqueous medium, under mellow temperature and pressure conditions by using simple apparatus. The goal is to study the oxidation of simple atom by the high surface region catalyst (nanomaterials) in a greener way.

The properties of nanomaterials rely on the chemical composition, the grain size (<100 nm), the grain boundaries, the free surface and communication between the domains and an interchange of these factors make them appealing material as catalyst [Boakye et al. 1994]. As of late much consideration has been focused on their readiness. As the molecule size decreases the an ever increasing number of receptive sites are exposed and the catalytic proficiency increases. The stoichiometry of the reactants, the method of synthesis, which incorporate the pH, temperature and timespan of the response, alongwith the post treatment contribute to the effectiveness of the catalyst.

Oxidation of formaldehyde -

Every one of the chemicals used were of scientific evaluation. Crude materials used to study the oxidation of fromaldehyde are cobalt oxide [Co3O4], Copper oxide [CuO] and Cobalt oxide/copper oxide composite [Co3O4/CuO] (synthesized by means of chemical combustion method, sol-gel autocombustion method and warm decomposition method) formaldehyde [HCHO], potassium dichromate [K2Cr2O7], Mohr salt [(NH4)2Fe(SO4)2•6H2O], sulphuric corrosive [H2SO4], diphenyl amine [C12H11N] and twofold distilled water.

10 ml HCHO (1%), 20 ml K2Cr2O7 (0.1M) solution and 100 ml dil. H2SO4 were taken in a closed top 250 ml conical flask. To this flask, 100 mg of the synthesized metal oxide nano catalysts were added. The solution was ultrasonicated for 5 minutes at three distinct temperatures i.e.20oC, 30oC and 40oC. The solution was filtered using sintered glass crucibles and the filtrate was titrated against 0.1M mohr salt solution using diphenyl amine as marker. Appearance of green color gives the end point. The procedure was repeated for Co3O4, CuO and Co3O4/CuO nano catalysts synthesized by means of chemical combustion and sol-gel autocombustion method, warm decomposition method and hydrothermal method. The comparison was finished with the clear observation for example without the catalyst. The readings obtained for the titration were recorded and are shown Figure 7. The entire procedure is shown schematically in the stream graph given underneath:

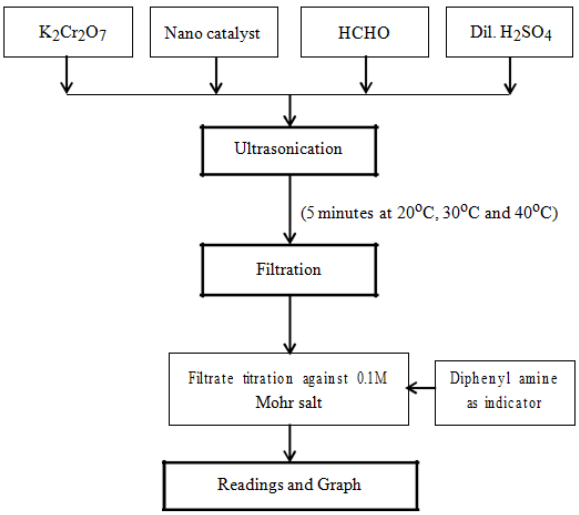

FIGURE 7: Oxidation of formaldehyde

The chemical reaction taking place in this experiment is shown in the scheme below:

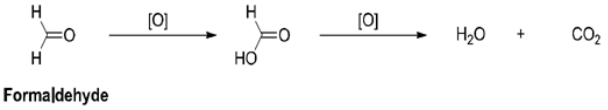

Figure *8: Oxidation of formaldehyde*

Oxidation of oxalic corrosive: -

Every one of the chemicals used were of systematic evaluation. Crude materials used to study the oxidation of oxalic corrosive are cobalt oxide [Co3O4], oxalic corrosive [H2C2O4], potassium permanganate [KMnO4], sulphuric corrosive [H2SO4] and twofold distilled water.

100 mg Co3O4 synthesized by means of warm decomposition method was taken in 100ml closed top measuring flask alongside 780 mg H2C2O4 and was filled sufficient using twofold distilled water.

Series of five unique sets were prepared and ultrasonicated with a hole of 15 minutes each at 25oC, 35oC, 45oC, 55oC and 65oC. Ultrasonication of each set was followed by a stay time of 2 hours. Following 2 hours, 5 ml of aliquot from each sample was taken and titrated against 0.01M KMnO4 solution using 25 ml distilled water and H2SO4 (20%). The readings observed for the titration were recorded and are shown in Figure 9.

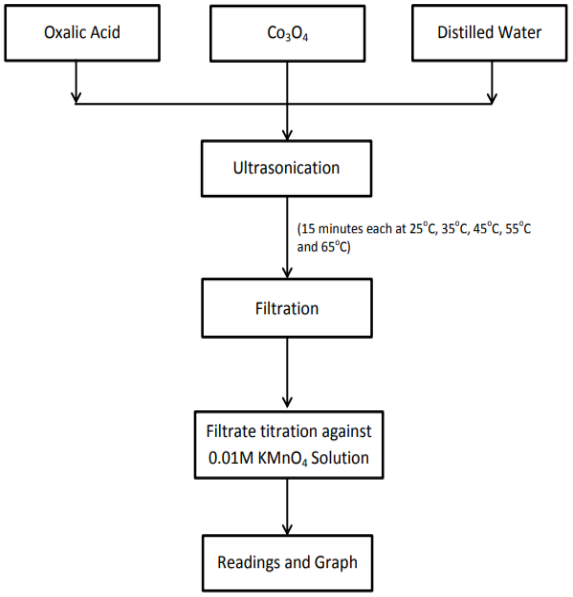

FIGURE 9: Oxidation of oxalic acid

The chemical reaction taking place in this experiment is shown in the scheme 3.3.2 below:

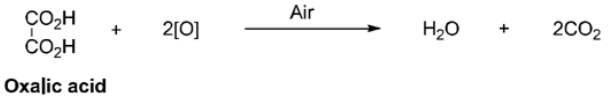

Figure *10: Oxidation of oxalic acid*

Oxidation of benzaldehyde: -

Every one of the chemicals used were of investigative evaluation. Crude materials used to study the oxidation of benzaldehyde are cobalt oxide [Co3O4], benzaldehyde [C6H5CHO] and twofold distilled water.

Co3O4 nanoparticles synthesized through warm decomposition method were used for the oxidation of benzaldehyde to benzoic corrosive. One pot liquid phase oxidation of benzaldehyde to benzoic corrosive was performed in a 100 ml flask. In each test, 0.2 g of the Co3O4 nanocatalyst was added to 2 ml benzaldehyde and 40 ml twofold distilled water. Reactions were carried out in aqueous conditions under refluxing using small condenser and carefully controlled stirrer at gentle temperature conditions (60oC) for 3, 4 and 6 hours. The crystals of the benzoic corrosive so prepared were separated, washed with distilled water and dried at 60oC – 70oC. The character of the item was confirmed by comparing their dissolving points and NMR information with those reported in writing. The so obtained yield. The entire procedure is shown schematically in the stream graph given underneath:

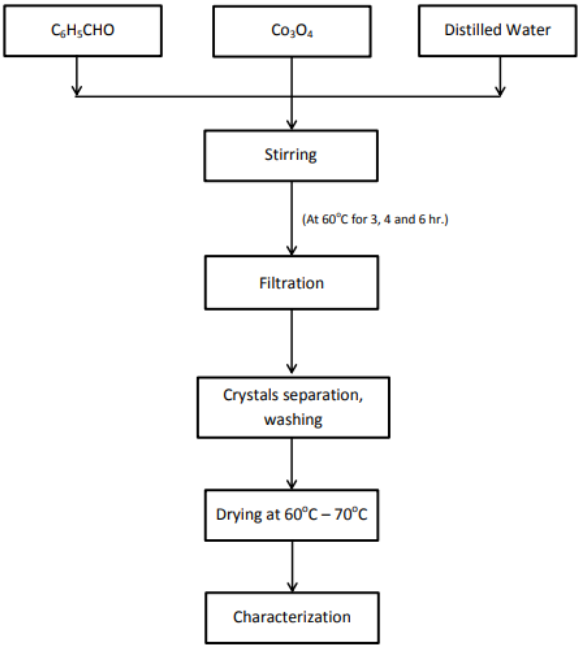

FIGURE 11: Oxidation benzaldehyde

The chemical reaction taking place in this reaction is shown in the scheme 3.3.3 below:

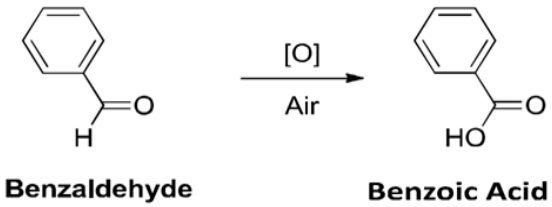

Figure *12: Oxidation benzaldehyde*

CONCLUSION

In co-cu alloy nanoparticles, the optimum composition for best performance was found to be cu0.15co2.84o4. Further it showed two linear ranges of detection with very high sensitivity of 4651.0 µ a mm^{-1} cm^{-2} up to 5 mm and 2581.7 0 µ a mm^{-1} cm^{-2} from 5 mm to 12 mm with a lower detection limit of 0.6 µm (s/n=3). Like the previous sensor this is also selective and its response towards blood serum was comparable with that of commercial glucose sensors. The work on cuo/pt/thns sensor is a pioneering one. The developed sensor exhibits very wide detection range up to 31 mm for glucose, in contrast to the previous two sensors (i and ii). Also the response time is as low as 3 s and the detection limit as low as 1 µ m and free of interference from other

bimolecular. Its performance in blood serum sample is satisfactory.

Experimental methodology and instrumentation have been discussed. Synthesis of nanosized metal/mixed metal oxides, their characterization and methods of chemical analyses has been studied. The metal/ mixed metal oxides have been synthesized by precipitation method using ammonia as precipitating agent and characterized using magnetic susceptibility measurements (VSM), TGA/DTA , X – ray diffraction patterns, Transmission electron microscopy(TEM) and Scanning electron microscopy(SEM). Experimental conditions and techniques used for the characterization of metal oxides/ mixed metal oxides have been given.

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