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Synthesis of Silver, Chromium, Manganese, Tin and Iron Nano Particles by Different Techniques

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Abstract

Nano technology has a large number of applications in different fields of science and technology. Nano particle are the tiny particles which are being used in upcoming technology due to their small size and high capability. The properties of nanoparticles depend largely on their synthesis procedures. There are different methods for the synthesis of nano particles using different conditions. In this review synthesis of five nano particles namely chromium, manganese, silver, iron and tin has been discussed. Possible methods of their preparation, conditions and properties are the main concern of this review. The results from various investigations performed by different scientists using some methods have been summarized. The applications, conveniences and difficulties of each synthesis method are also discussed in-detail. Applications of nano particles and metal oxide are also elaborated.

Keywords: *Nano Particles, Synthesis Procedures, Chromium, Manganese, Silver, Iron and Tin.*

1. INRODUCTION

Nanoparticles have sizes between 1-100nm [1, 2]. Nanoparticles show novel properties which are contrast from those of mass materials. These properties change with the molecule size [3, 4]. On a basic level, the span of the nanoparticles can be controlled keeping in mind the end goal to furnish materials with particular properties. The two fundamental reasons why materials at the nano scale can have diverse properties due to expanded relative surface region and new quantum impacts. Nano materials have a much more prominent surface area to volume proportion than their traditional structures, which can prompt more noteworthy synthetic reactivity and influence their quality. Likewise at the nano scale, quantum impacts can turn out to be significantly more vital in deciding the materials properties and qualities, prompting novel optical, electrical and attractive practices. It is known that the sizes, phases and morphologies of nano materials have great influence on the properties and applications; therefore, many research efforts have focused on rational control of shape, phase, dimensionality and size of nano materials [5]. Nano biotechnology and their inferred items are exceptional in their treatment strategy as well as because of their uniqueness in molecule size, physical, substance, biochemical properties and wide scope of utilization too [6, 7, 8]. This present rising field of nano biotechnology is at the essential phase of advancement because of absence of execution of inventive strategies in substantial mechanical scale but then must be enhanced with the cutting edge innovations. Consequently, there is a need

to plan a monetary, economically plausible also earth feasible course of union of nano particles so as to take care of its developing demand in assorted segments. Transition metal oxide nanoparticles speak to a wide class of materials that have been looked into broadly because of their fascinating electronic and attractive properties. Transition metal oxide nanoparticles has numerous applications as catalyst [9], sensors [10, 11, 12, 13], superconductors, magnetic resonance imaging (MRI) [14], sun based cells [15] and adsorbents [16]. Metal-oxides constitute an imperative class of materials that are included in ecological science, electrochemistry, science, synthetic sensors, attraction and different fields.

2. SYNTHESIS METHOD OF NANO PARTICLES

Nanoparticles can be conveyed using an extensive variety of techniques, ordinarily named bottom up or chemical and biological methods and top down or physical strategies [17]. In the base up methodology, the structure of nanoparticles is produced by particles, atoms or group. In top-down methodologies, a mass bit of an obliged material is decreased to nano sized measurements utilizing cutting, grinding and scratching strategies, i.e. nanomaterials are readied from bigger elements without nuclear level control [18]. Physical methods for nanoparticles synthesis are laser pulse ablation [19], mechanical processing [20]. Vacuum vapor discharge [21], pulsed wire discharge (PWD) [22]. Chemical reduction [23], micro emulsion (colloidal) procedures [24], sonochemical reduction [25], electrochemical [26], microwave-assisted [27] and aqueous mixes [28] are the major frame works for the synthesis of nano particles through the manufactured philosophy considering large scale manufacturing and handling expenses, the flash disintegration technique, which is a so-called arc discharge method, can be considered a good example of the top-down approach [29, 30, 31]. The method involves applying pulsed voltage between electrodes that are immersed in a dielectric medium (or solvent) and become sources of nanoparticles through induced metal transfer. The best known case of the base up methodology is a diminishment blend technique utilizing a chemical wet procedure, including a polyol method [32, 33, 34]. This process comprises decomposition of the precursor and reduction of metal ions in the solvent. In the process, a surfactant (or capping agent) is usually added during the reduction synthesis to promote nucleation by metal ions, suppress agglomeration between nanoparticles, and prevent excessive oxidation with formed nanoparticles. A number of synthesis methods have been used for the preparation of metallic nanoparticles [35] such as prominent cases carry, electrochemical synthesis [36, 37], salt reduction [38], microwave dielectric heating reduction [39], ultrasonic irradiation [40], radiolysis [41], solvothermal synthesis [42], reverse micelles process [43] etc.

2.1. Precipitation Method

Precipitation technique is additionally called solvent displacement technique. It involves the precipitation of a preformed polymer from an organic solution and the diffusion of the organic solvent in the aqueous medium in the vicinity or vacancy of a surfactant [44, 45, 46, 47]. First of all dissolve precursor in di ionized water and stirrer then add reducing agent drop wise in this solution. After some time precipitates are formed and lay down in the bottom of beaker. Then drain that solution and separate precipitant and slag through filtration for further characterization of nano particles. This strategy includes the points of interest as it is a straight forward, low priced and one stage process easy control of particle size and composition can be made in this method and also there are various possibilities to modify the

particle surface state and overall homogeneity.

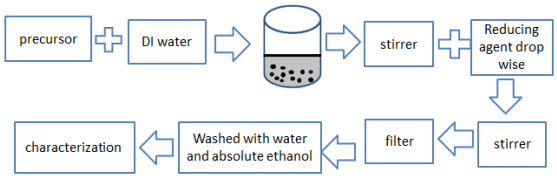

Figure 1. *Schematic of synthesis of nanoparticles by precipitation method.*

2.2. Solvothermal method

In solvothermal process the chemical reaction performed in a fixed vessel for example, bomb or autoclave, where solvents are conveyed to temperatures well above their boiling points [48]. In solvothermal method dissolves precursor and reducing agent in a beaker then place in an autoclave after the formation of nanoparticles characterize it. Solvothermal is a versatile, convenient and straight forward method using only a polar, oxygen-containing solvent and by applying heat but synthesis usually in closed vessels, so temperature-pressure-volume considerations are critical.

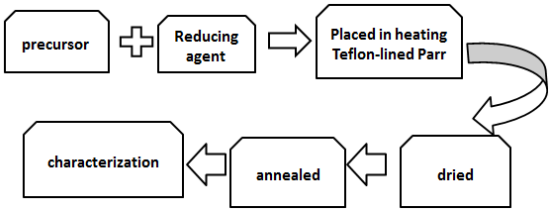

Figure 2. *Schematic of synthesis of nanoparticles by solvothermal method.*

2.3. Hydrothermal Method

Hydrothermal synthesis is mainly used for the synthesis of a single crystal that is in aqueous form. This process is also takes place in autoclave, in which a mineral is supplied along with water. Between the opposite ends of chamber a temperature grade is maintained. Hotter end dissolves the supplements and the cooler end deposits it on crystal to obtain the desired crystal [49]. Hydrothermal synthesis method is beneficial as it has the ability to synthesize large crystals of high quality and those crystals which are unstable near the melting point. But this process requires costly equipment and not suitable to monitor crystals [50].

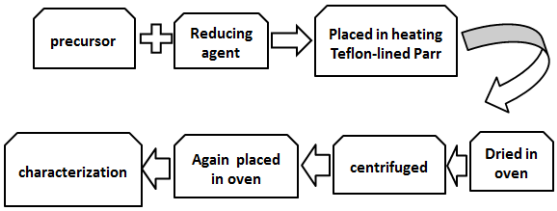

Figure 3. *Schematic of synthesis of nanoparticles by hydrothermal method.*

2.4. Sol Gel Method

Sol-gel method may be used to prepare materials with a variety of shapes, such as dense powders and thin films. It is easy and cost effective. In this method solution was added to the precipitate and, over a period of time, this mixture becomes clear sol and later turns to gel [51]. Sol gel have benefits i.e. it synthesize the nanoparticles method at room temperature and found to be an efficient and mild route for the large-scale industrial production of fine nano particles without any expensive chemicals or template but it is hard to control stoichiometry of doped oxide nano particles which is the drawback of this method [52].

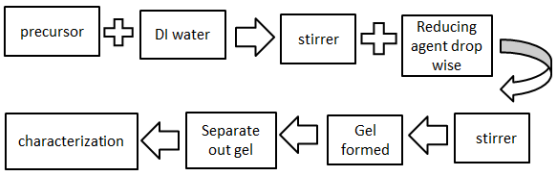

Figure 4. *Schematic of synthesis of nanoparticles by sol gel* method.

2.5. Green Synthesis

Green synthesis is a plant mediated synthesis of metal nanoparticles it is simple, easily available, fast and eco-friendly among all the synthesis methods [53]. The bio molecules which are present in the plant materials can increase the rate of nano synthesis or the stability of the product. In this method first of all collect plant extract from boiling leaves of regarding plant in di ionized water. Further use this extract with precursor and form

nanoparticles. Plant materials have an important role in surface morphology and size of the metals [54]. Extracts from plants may act both as capping and reducing agent in nano particle synthesis. But the particles formed by this method may have large sizes.

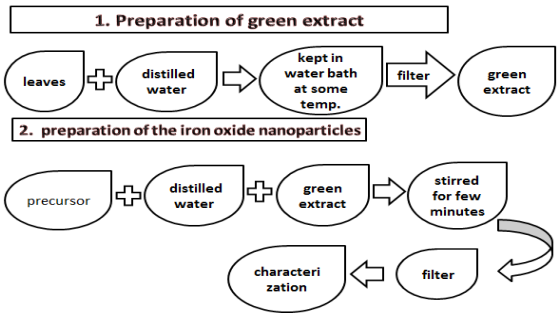

Figure 5. *Schematic of synthesis of nanoparticles by green method.*

2.6. Micro Emulsion Method

Water-in-oil (w/o) micro emulsions has the drops of water on the surface of oil and these drops of water become the source in the formation of nanoparticles [55, 56]. In this method form micro emulsion first which is either water in oil or oil in water. Then use this emulsion with precursor and characterize it. This method is useful because it formed particles of controlled size and there is no need of expensive instruments [57, 58]. W/o micro emulsions have been used to synthesize metal nanoparticles i.e. organic nanoparticles, metal oxides and silver halides.

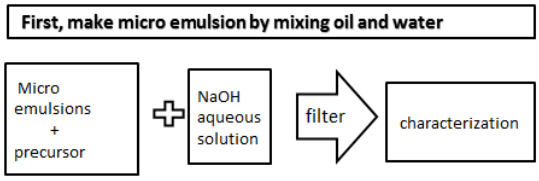

Figure 6. *Schematic of synthesis of nanoparticles by micro emulsion method.*

3. SYNTHESIS OF SILVER NANO PARTICLES

In ancient times silver and silver nitrate were used in medical science due to its antifungal properties. Among all the metals silver exhibits the highest thermal and electric conductivities [59]. Nanoparticles of silver have applications in thick film pastes, conductive inks [60] catalysis [61] as well as have gain attraction in photography and photonics [62]. Silver particles play an important role in the electronic industry. Recently, high demands of thin films and small width of integrated circuits employed a way to synthesis silver colloidal metal particles [63]. Silver nano particles exhibits antimicrobial activity which is used in medicines for the prevention of infection and bacterial effects [64], dental materials [65], human skin [66] catheters [67], stainless steel materials [68], vascular grafts [69]. Silver containing materials can be used for water treatment [70] and employ to erase microorganisms on textile fabrics [71, 72]. Silver nano particles are produced by using different methods electrochemical method [73, 74, 75], thermal decomposition [76], sonochemical synthesis [77], microwave irradiation and laser ablation [78]. In this way, in creating courses of amalgamation, an accentuation was made to control the extent of silver nano particles.

Table 1. *Summary of methods of obtaining silver oxide nanoparticles.*

Method	Material	Conditions	Properties	Applications	Reference
Chemical reduction method	$AgNO_3$, hydrazine hydrate, sodium dodecyl sulphate and citrate of sodium.	Reaction at room temperature	Face-centered-cubic silver nanoparticles with mean diameters of 9, 11, 24 and 30	Antibacterial activities	[79]

			nm		
	Silver nitrate, hydrazine hydrate, sodium formaldehyde sulfoxylate, Poly(vinyl Alcohol)	Reaction at room temperature. Heated: at 100 ◦c	Cubic silver nanoparticles with size of less than 10 nm	Opto-electronics and electronics	[80]
	Silver nitrate, glucose, sodium hydro oxide Polyvinyl pyrrolidone pvp solution	Heated: at 60 ◦c	spherical shape nanoparticles with size of 20–80 nm	To form the conductive films and printing the circuits on the basement	[81]
Green synthesis	silver nitrate, dextrose and polyvinyl pyrrolidone (pvp)	Reaction at room temperature	6-12 nm	Use as a new fungicidal material	[82]
	M.Balbisiana (banana), A. India (neem) and o. Tenuiflorum (black tulsi), Silver nitrate	Leaves Boiled for 20 min. Refrigerated: at 4°c Solution: at power of 300 w for 4 min	Banana: 50 nm Tulsi: 50 nm Neem: 20nm	Pollution remediation	[83]
	Leaf extract of ocimum sanctum and Silver nitrate	Leaves dried: for 10 days Heated: 600°c for 24-48 hours. Centrifuged: at 18.000 rpm for 25 min again heated at 50- 95°c.	Spherical shape with 50 nm size.	antibacterial and antifungal activities	[84]
	Carob leaf extract, silver nitrate	Boiled for 10 min	5-40nm with diff concentration.		[85]
Sol gel method	silver salt, Hydrolyzed silica Acids, hydrochloric acid, sulfuric acid and nitric acid	Room temp.	5-30 nm	In controlling the spontaneous silver precipitation	[86]
	Tetraethyl orthosilicate, Silver nitrate, ethanol and Nitric acid.	Stirred: 4 hours at room temp. Annealed: from 300 °c to 1000 °c for 1 hour	Spherical Shape Size: 20-40nm	Antibacterial activity	[87]
Reverse micelle method	Silver nitrate, glucose, hexane and dioctyl sodium sulphosuccinate.	Heating up to 60-70ºC.	Spherical shape and monodisperse particles Size: 10nm	In optics and medicine with smallest size	[88]
	silver nitrate, sodium dioctyl sulfosuccinate, sodium borohydride, isooctane, cetyltrimethyl ammonium bromide, quercetin, sodium bis(2-ethylhexyl) sulfosuccinate, chloroform,	Reaction at room temperature for 2-3 hrs.	5nm.	make possible the delivery of ionic silver during disinfection process	[89]

Micro emulsion method	Sodium dodecyl sulfate, Silver nitrate sodium, Borohydride 1-petanol and cyclohexane	Reaction at room temperature	16-29nm	Used as alternative to Conventiona l antibiotics.	[90]
	Sodium borohydride, Di-octyl sodium sulfosuccinate, Silver Nitrate, cyclohexane, isooctane	Reaction at room temperature. Stirred for 3min	Highly mono dispersive having average diameter of 5.5 nm.	Volatile organic compounds removal	[91]

3.1. Chemical Reduction Method

M. G. Guzman *et al* [79] used silver and hydrazine hydrate as a precursor and reducing agent respectively for the preparation of silver oxide nanoparticles. The antimicrobial susceptibility of silver nanoparticles was evaluated using the Kirby-Bauer or disc diffusion method zones of inhibition were measured after 24 hours of incubation at 35^{o}C. The results demonstrate that the colloidal silver nanoparticles shows anti-bacterial activities inhibited the growth and multiplication of the tested bacteria silver nanoparticles with mean diameters of 9, 11, 24 and 30 nm.

P. K. Khanna *et al* [80] prepared Silver/poly (vinyl alcohol) nano composites by reduction of silver salt by using two different reducing agents i.e. sodium formaldehyde sulfoxylate and hydrazine hydrate. They have done first time the reduction of silver salt in aqueous PVA with SFS. TGA showed that the composite has higher degradation temperature than the PVA alone. TEM show the particle size was found to be less than 10 nm.

H. Wang *et al* [81] took the solution containing polyvinyl pyrrolidone (PVP) and silver nitrate that reduced by the glucose. The TEM indicated that the particles dispersed better with the increase in PVP and the particles dispersed individually in a colloid form silver particles with 20–80 nm size and spherical shape were prepared if the weight ratio of PVP to $AgNO_3$ is no less than 1.5.

Chemical reduction is the most frequent method to obtain silver nano particles. They are colloidal dispersion and stable in water or organic solvents. Generally colloidal silver nano particles are generated by this method. This is the easiest route than all other methods. But the mixing speed is the main factor which may agglomerates silver nano particles if precautions are not followed. Moreover, silver nano particles quickly change their properties

3.2.Green Synthesis

Ghorbani *et al* [82] synthesized silver nano particles by a fast method and took silver nitrate as a precursor, poly vinyl pyrrolidone (pvp) as a stabilizing agent, dextrose as a reducing agent and water as the solvent. UV–Vis spectroscopy, dynamic light scattering (DLS) and transitional electron microscopy (TEM) were used to identify and analyze nanoparticles. All characterization revealed that the particles have the size of 6–12 nm.

Banerjee *et al* [83] prepared silver nanoparticles by the reaction of 1 mM silver nitrate and 5% leaf extract of each type of plant (Azadirachta indica (neem), Musa balbisiana (banana) and Ocimum tenuiflorum (black tulsi)) separately. The silver nano particles were characterized and tested for their antibacterial activity and toxicity. The silver nanoparticles prepared are safe to be discharged in the environment and possibly utilized in processes of pollution remediation. Silver nanoparticles prepared by this way may also be efficiently utilized in agricultural research to obtain better health of crop plants.

Y. Rout *et al* [84] synthesized silver nano particles from the leaf extract of Ocimum sanctum, as reducing agent. SEM analyses showed that the particles were spherical shape with 50 nm size. The main objective

of their study is to emphasize the use of plant medicinal to synthesize silver nanoparticle with anti-bacterial and antifungal effects. They also work on the urinary tract infections.

This is the environmental benignity and low cost synthesis thechnique for silver oxide nano particles. The plant extract is advantangeous and dis advantageous as well. If fresh plant extract is not available the particles wil not formed or the properties will be altered.

3.3. Sol Gel Method

Wu *et al* [86] prepared silver nanoparticles through sol gel method by using silver nitrate with different hydrolyzed precursors in different molar ratios. They also used HCl as catalyst (with different concentrations 0.2 M, 0.04 M and 0.004 M) for the formation of AgCl and subsequent formation of silver upon light exposure. TEM analysis shows that the size of nanoparticles is 5-30nm. Results showed that the solvent phase (methanol and intermediates) are the main cause of reducing capability encapsulated in the pores and not from the silica network.

D. P. Pham *et al* [87] prepared Ag/SiO_2 nano-composite powders with 5 mol% silver by sol-gel method. They do not use any protecting agents. TEM analysis showed that spherical shape sliver nanoparticles were prepared with 30 nm size. They showed that the decrease in crystallizing temperature of SiO_2 was due to the presence of metal nano crystals and the growth of silver particles with annealing temperatures has no effect on the densification of silica.

The growth of nano particles through sol-gel process is highly depending on annealing temperatures. But in synthesis of silver nano particles the annealing temperature has no effect on the densification of silica. This will leads to decrease the specific surface area. Absorption peaks of silver nano particles can be changed in this method.

3.4. Reverse Micelle

K. Sigal *et al* [88] synthesized nano particles by reverse micelle method which is based on silver mirror reaction. They also used silver nitrate as a precursor with dioctyl sodium sulphosuccinate (Aerosol-OT) and hexane. They showed that the reverse micelle process is most efficient in order to obtained narrow sized nano particles not more than 10 nm sizes.

T. T. N. Dung *et al* [89] synthesized silver nano particles by reverse micelle technique by taking $AgNO_3$, $NaBH_4$, quercetin and SDOSS or CTAB as a silver ions source, reducing agents and surfactants respectively. They study on bactericidal activity of the nano silver products and show that the particle size of the nano silver products depends on their stoichiometric ratio and the concentration of the reaction components. They also showed that the good particle-size distribution and smallest nanoparticles ~5 nm is obtained by reaction system using AOT surfactant.

Reverse micelle method is advantageous because synthesis of silver nano particles through this method helped to obtaining particles of narrow sized that is not more than 10 nm. But dis advantage of this is that the binding reaction in this synthesis may alter the molecular structure of the macro molecules.

3.5. Micro Emulsion

S. Hossain *et al* [90] synthesized nanoparticles by reduction of aqueous silver nitrate and copper chloride solutions with sodium borohydride in anionic water-in-oil micro emulsions and sodium dodecyl sulphate (SDS)/ 1- pentanol/ cyclohexane/ water. Zone inhibition method using nanoparticles in ethanol suspension is useful to check antibiotic sensitivity of silver and copper nanoparticles against Escherichia coli (E. coli). Characterization reveal that the nano particles size is in the range of 16-29nm. Their results show that antibacterial activity decreases with the decrease of suspension in nano particles,

while the concentration dependence on antibacterial activity has been less pronounced.

J. N. Solanki *et al* [91] synthesized highly mono disperse silver nanoparticles by chemical reduction of one micro emulsion silver nitrate dissolved in water and another micro emulsion of sodium borohydride used as reducing agent dissolved in water. In their experiment they show that when mole ratio is greater than unity particle size increases. TEM show that the synthesized nanoparticles have average diameter of 5.5 nm.

This is a novel route for synthesis of silver nano particles with tunable dimension. This method provides the fascinating domain. But there is some drawback of this method i.e. formation of some clusters observed in TEM image which increase the wide size distributions and particle size.

4. SYNTHESIS OF CHROMIUM OXIDE NANO PARTICLES

Recently in the field of both science and technology chromium oxides has significance results. Chromium can form the different types of oxides because of its different oxidation states. Among all oxides of chromium, chromium oxide (Cr_2O_3) is important in specific applied applications such as in high temperature resistant materials [92], corrosive resistant materials [93], liquid crystal displays [94, 95], green pigment [96], catalysts[97-98] H_2 absorption material [99, 100], gas sensing and as a glaze in ceramics [101]. For the synthesis of chromium nano particles various techniques have been developed to synthesize Cr_2O_3 NPs [102, 103, 104] such as thermal decomposition [105], precipitation [106], precipitation gelation [107, 108], sol gel [109, 110, 111], mechano chemical reaction, oxidation of chromium in oxygen [112] and sono chemical method [113]. Here in below table we discuss some methods to prepare chromium oxide nano particles and compare their conditions and properties.

Table 2. *Summary of methods of obtaining chromium oxide nanoparticles.*

Method	Materials	Conditions	Properties	Applications	Reference
Precipitation method	Chromium sulphate Ammonium hydroxide Deionized.	Reaction :at room temp Dried: at 70°C for 24 hrs. Calcined: at 600°C for 5 hrs.	Hexagonal structure with 45 nm size.	Used in the process of stropping knives	[114]
	Chromium sulphate, Ammonium hydroxide, De ionized water	Dried: in oven at 70°C for 24hrs. Calcined at 600°C for 5 hrs. pH:10	Hexagonal structural nanoparticles with 20-70nm size.	Used in the process of stropping knives, glasses, inks, paints and precursor to the magnetic pigment	[115]
	$CrCl_3 \cdot 6H_2O$, Ammonia solution,	Reaction temperature at 5, 27 and 65°C. Dried at 120°C. Calcined at 500 ◦C for 3 hrs	Synthesized powder has small crystallite size	To investigate the sensing behavior of synthesized powder toward LPG.	[116]

Novel Solvent Free Method	Chromium chloride, Urea	Mix: 4000 rpm, 20-30 min Heating:35 to 40 min Frequency: 2.45 GHz and power 800 W. Calcined : 200, 400, 600 and 800^{o}C for 1h	N type transparent conducting oxide Nano particles with size of 20 – 58 nm	Coating, wear and corrosion wear resistance	[117]
Solvothermal method	Cr Acetylacetonate Acetone, Chloroform	Heating 200^{o}C For 72 h to one week. Dried: 12 h Annealing: 4 h at 500^{o}C in air.	XRD: Spherical, polydisperse, and amorphous shape with size of 18.5nm.	Magnetic resonance imaging (MRI)	[118]
	chromium acetylacetonate, 1-butanol, 1,4- butanediol, 1,5-pentanediol and 1,6-hexanediol	heated to 300 ^{o}C at a rate of 2.5 ^{o}C min–1 and held at this temperature for 2 h. Calcined at 350 ^{o}C for 1 h.	Single nanocrystalline of chromium oxide with size of 16–26 nm.	Used for thermal stability	[119]
Hydro thermal synthesis	CrO_3, anhydrous alcohol (C_2H_5OH) deionized water	Heating: at 190 ^{o}C for 1 h. Cooled at room temp. Dried: 10 h. Calcined: at 500 ^{o}C and 700 ^{o}C, respectively for 1 h.	Loosely agglomerated Cr_2O_3 nanoparticles with 29-60nm size.	Serving as catalysts and wear resistance materials.	[120]
	$Cr(NO_3)_3.9H_2O$, ammonia sol.	Reaction at room temperature. Centrifuged at 13,000 rpm for 10 min. Heat at 200 ^{o}C in oven for 12 h – 4 days.	Round-shape particles with narrow size-distribution of 45–69 nm.	Used in high-temperature resistant materials	[121]
Thermal decomposition method	$Cr(NO_3)_3.9H_2O$, ammonium polyacrylate	Dried: 1 h at 110 1^{o}C. Calcined : 480 ^{o}C for 120 min at a heating rate of 3 ^{o}C/min	Rhombohedral particles with 16 nm size.	Using for the doping in varistor compounds	[122]
	$Cr(NO_3)_3 \cdot 9H_2O$,	Heated at 550°C Dry at low temperature	Heated at 550°C Dry at low temperature.	Used in pigments	[123]

4.1. Precipitation Method

D. Ritu *et al* [114] by reacting $Cr_2(SO_4)_3$ and aqueous ammonia until the pH of the solution reached to 10. UV-Visible absorption and IR spectroscopy confirm the formation of Nano sized Cr_2O_3. TGA verifies that the Cr_2O_3 NPs are thermally

stable up to 1000 °C XRD studies show that chromium oxide was formed as Cr_2O_3 instead of the commonly formed CrO_2. And the resultant particles are highly stable and reproducible.

V. S. Jaswal *et al* [115] use the same experiment for chromium oxide nano particles by reacting $Cr_2 (SO_4)_3$ and aqueous ammonia

N. Kohli *et al* [116] prepared chromium oxide nano particles and then investigate sensing behavior of these Cr_2O_3 samples towards LPG. They initiated their experiment with the 0.2 M solution of $CrCl_3 \cdot 6H_2O$ in distilled water to which ammonia solution was added drop wise with continuous stirring and tailor nanoparticles of chromium oxide at various reaction temperatures i.e. 5, 27 and 65^{o}C. For obtaining precipitate, they used same procedure to prepare three different reaction mixtures and maintained their reaction temperature at 5, 27 and 65°C. The resulting precipitate in each case was separated from solutions, washed and dried at 120°C. Collected samples were calcined at 500^{o}C for 3 hours. They used XRD for characterization which show that the Cr_2O_3 nanoparticles synthesized at 27°C were smaller and have high sensing behavior as compared to those synthesized at 5 and 65°C.

Precipitation method is a rapid method to synthesize chromium oxide nano particles. It is low cost, eco-friendly and simple method. Nano particles prepared by this method are highly stable, reproducible and have greatest feasibility for further applications. But temperature and constant stirring is the main factor for the synthesis of nano particles through this method. If temperature changes then particles will be formed with large in size.

4.2. Novel Solvent Free Method

R. Meenambika *et al* [117] prepared nano particles of Cr_2O_3 via novel solvent free microwave irradiation technique by the reaction system of $CrCl_3.6H_2O$ and NH_2-CO-NH_2 with different molar ratios 1:1, 1:2 and 1:3 respectively. They first time synthesize the chromium nanoparticles by calcinations at 200, 400, 600 and 800°C for 1h. They also found that the molar ratio 1:3 of chromium chloride and urea is considered to be the best proportion to synthesis Cr_2O_3 nanoparticles.

There are many methods to prepare nano particles some of them are very complex or expensive to use that's why a new method is introduced to fulfill the demands of industrialization. Novel solvent free method is simple and cost effective among all new methods for silver nano particles. As it is a new method so more precautions are required and microwave irradiations influence on the particles size.

4.3. Solvothermal Method

A. L. Willis *et al* [118] employed the reactions of oxygen-containing solvents i.e. acetone (or cyclohexanone or ethanol) and metal acetylacetonate precursors in a Teflon-lined Parr acid-digestion bomb at 200^{o}C for 72 hours to one week to prepare metal oxide nanoparticles. TEM, XRD and elemental analysis were used to characterize the materials. Scherer equation showed that the average crystal size was 18.5 nm.

P. Praserthdam *et al* [119] reacted chromium acetylacetonate in various organic solvents at 300 ^{o}C for 2 h to yield an amorphous product. Calcination at 300 ^{o}C for 1 h was appropriate to obtained single nano crystalline chromium oxide. The ratio between calcination temperature and the correlation between thermal stability of synthesized crystallite size provided a good explanation of the effect of crystal size and calcination temperature on the thermal stability of chromium oxide which is single crystal. The crystallite size of product is in the range of 16–26 nm. They suggested the solvent used in preparation had no effect on thermal stability of chromium oxide the main influencing parameters tended to be the as-

synthesized crystallite size and calcination temperature.

A versatile, nontoxic, straight forward and convenient method to prepare nano particles of chromium oxide is solvothermal method. A single crystallite size can be varied by solvothermal method in which the solvent do not effect on thermal stability of chromium oxide nano particles. But calcination temperature and different mole ratios can effect on crystal size.

4.4. Hydrothermal Method

Z. Pei *et al* [120] prepared chromium oxide nanoparticles by reacting chromium anhydride (CrO_3) and anhydrous alcohol (C_2H_5OH) using hydrothermal approach. The process needs no stirrer and surfactant and it was easily controllable. The found that a higher calcination temperature tends to result in a smaller specific surface area and a larger average Particle size and a higher ratio of C_2H_5OH to CrO_3 always lead to a higher specific surface area and therefore a smaller average particle size of the products. FE-SEM shows the obtained nanoparticles were loosely agglomerated with the average size of 29–60 nm.

J. Yang *et al* [121] used Cr $(NO_3)_3.9H_2O$ and ammonia solution as precursor. They dissolved 15 g Cr $(NO_3)_3.9H_2O$ in 75 mL ultrapure water and 28% ammonia was diluted into 10% solution. The diluted ammonia solution (10%) was added at a rate of 1 mL min-1 into the ion solution with vigorous stirring at room temperature to form oxide colloid. In determining the nature of the final product of chromium oxyhydroxide nano materials the pH values and reaction duration of the precipitation process played an important role.

Hydrothermal process in the preparation of chromium oxide nano particles mostly needs no stirrer and surfactant. This method is so easily controllable, has short time approximately 1hour and low cost that it has the potential to be produce at large scale and to be industrialized. The drawback of this method is the formation of grains with large sizes.

4.5. Thermal Decomposition Method

R. F. K. Gunnewiek *et al* [122] synthesized rhombohedral chromium oxide nano particles readily via thermal decomposition of a polyacrylate/chromium complex. The rapid technique yielded the synthesis of crystalline nano particles by thermal decomposition at 480 ^oC for 2 h and no ageing step was required. Cr $(NO_3)_3$ $.9H_2O$ (Vetec) mixed into an ammonium in a proportion of 1:2. The resulting dark green resin was washed thrice in distilled water and dried for 1 h at 110 ^oC. This method allowed for the synthesis of highly crystallized chromium nano particles with an average calculated particle size of 16 nm with SEM analysis.

P. Gibot *et al* [123] prepared chromium (Cr_2O_3) oxide by thermal decomposition of chromium (III) nitrate nano-hydrate Cr $(NO_3)_3 \cdot 9H_2O$. The commercial reagent was first dissolved in a colloidal silica solution and reaction was heated at 550°C and then dried at a low temperature to slowly evaporate the aqueous solvent. The SiO_2/Cr $(NO_3)_3 \cdot 9H_2O$ weight ratio was varied from 0 to 2. The various Cr_2O_3 powders were characterized by FTIR, TEM, XRD, SEM and nitrogen adsorption techniques. TEM and XRD correlation showed that these pristine chromium oxide nanoparticles, with a slightly sintered sphere-shaped morphology, exhibited a mono crystalline character with 10nm particle size.

Among the entire polymer based synthesis methods polyacrylate complication is a novel and high degree of water based cations exchange route to prepare chromium oxide nano particles.one of them is thermal decomposition method. It is the low temperature, simple, effective and fast polymer based method to synthesize chromium oxide nano particles. Stability of resin is most important factor which makes this method sensitive.

5. SYNTHESIS OF MANGANESE OXIDE NANO PARTICLES

Among magnetic nanoparticles, manganese oxide (Mn_3O_4) is an important material as a magnetic transition metal oxide [124]. The properties of MnO_2 are fundamentally influenced by their stages and morphologies; additionally, the working properties of lithium-ion batteries (LIBs) also depend on the phase of MnO_2. So for the preparation of MnO_2 a large number of works has been coordinated with different phases and various shapes [125]. Molecular absorbent, varistors, ion exchange, medium, electrochemical material and as a catalyst are the applications of MnO_2 [126, 127, 128, 129, 130]. MnO_2 and Mn_3O_4 are charming composites and have been utilized as a part of wastewater treatment, super capacitors, catalysis, rechargeable batteries and sensors [131, 132]. Especially, MnO and MnO_2 nano materials have attracted great interest as anode materials in lithium-ion batteries for their special properties, low cost, high theoretical capacity, and environmental benignity [133, 134]. The magnesium oxide is exceptionally suitable material for protection application because of their high melting and low heat points. MnO_2 metal nanoparticles also have potential applications in the field of coating, nano wires, and bio science. For synthesis of manganese oxide a variety of methods including high temperature sol–gel [135], sonochemical [136], co-precipitation [137], solvothermal [138], hydrothermal [139], chemical bath deposition [140], oxidation [141-142], microwave irradiation [143] and surfactant-assisted methods [144], precipitation [145], electrochemical [146], electron spinning, combustion, template and carbo thermic reduction [147]. Each method has its own advantages and disadvantages. Here we have a comparison table to show the appropriate method.

Table 3. *Summary of obtaining manganese oxide nano particles.*

Method	Material	Conditions	Properties	Applications	Reference
Green synthesis	Ionic liquid 1-n-butyl-3-methylimidazolium hydroxide, [BMIM]OH $Mn(NO_3)2\times 4H_2O$, NaOH solution, H_2O_2	Reaction at room temp.	polycrystalline structure with particle size 45 ± 11 nm	Act as a catalyst	[148]
	$Mn(NO_3)_2\cdot 4H_2O$, NaOH, LiOH and KOH.	Reaction for 1-24 hrs.	XRD showed Philips X'pert MPD, CuKα1 =0.15406 nm	Ion exchange	[149]
	acidic ferric salt solution, ferric nitrate solution	Aging at 98 °C for 7 days.	The Mn_3O_4 nanowires have been synthesized	Molecular adsorbent	[150]
Co precipitation method	manganese(II) sulphate, manganese oxalate, NaOH solution, Ethanol	Reaction: 60^0C Stirring: 1 h dried: for overnight at 100°C. Heat: 500°C for	Purely crystalline particles with 25-30 nm.	Using in pharmaceutical industries and sensors.	[151]

		4 hrs.			
	Manganese acetate, etyltrimethyl ammonium bromide, NaOH, acetone, ethanol	Stirrer for 10 hrs. Dried at 70°C for 18hr	XRD: crystallite size of 60nm SEM: spherical with 500nm	Super capacitors.	[152]
Sol gel method	Manganese chloride dehydrate, Deionized water. ammonium hydroxide	Heated at 85°C for 12 hours.	Hyper fine structure with 32nm size.	Used for high density magnetic storage devices	[153]
	Manganese acetate, Ethanol, tetra butyl (Ti(OBu)4), acetyl acetone	Heated for 24 h at 353–473K.	Pure pyrophanite $MnTiO_3$ powder with pie shape morphology	Used in nonlinear optics	[154]
	Copper (II) acetate monohydrate manganese (II) acetate tetra – hydrate absolute ethanol mono-ethanol amine	Stirred: 1.5 h at room temperature Aging: 24 h Dried: 150°C for 20min Heated: 300-450°C for 1h Annealed:300°C to 450°C	orthorhombic phase of MnO_2 and cubic structure of Cu_2O	Used in high performance electrochemical electrodes	[155]
	Manganese chloride potassium permanganate manganese oxide	Heated at different temperatures 80, 200, 400, and 600°C	Meso-porous structure	Used in sensors	[156]
Hydro thermal method	$KMnO_4$, CTAB, urea	Stirred: 30min Heat: oven for 12h at 150°C. Dried: for 12h at100°C.	Orthorhombic shape with 12nm size.	Electro chromic films	[157]
	sodium manganese oxide (NaBir), H_2O_2, NaOH, $Mn(NO_3)_2$, NaBir, MLiCl, KCl, $MgCl_2$, $MBaCl_2$	Sample heated at 150°C.	Pyrolusite and Ramsdellite-type manganese oxides were prepared. The size of the resulting tunnels corresponds to the size of the templates.	Useful for the chemical synthesis of inorganic material	[158]

5.1. Green Synthesis

Z. Durmus *et al* [148] used green chemical route by using an ionic liquid 1-n-butyl-3-methylimidazolium hydroxide ([BMIM] OH) at room temperature. ILs is organic salts which are environment-friendly and exhibiting high thermal stability. XRD show crystallite size of 45±11 and phase purity. TEM tell about crystalline nature. EPR measurements shows that Long-range interactions are

might be due to the wide range of particle sizes and morphologies.

L. Balan *et al* [149] synthesized manganese oxide nanoparticles with different crystalline phases and shapes. They tuned many experimental parameters, i.e. hydroxide type, the nature of the precursor salt, irradiation time, hydroxide concentration and the atmosphere also structure of the nanoparticles was studied and the influence of these parameters on the morphology was studied. They used TEM, Raman spectroscopy and XRD to characterize the materials. Their approach is based on the visible light irradiation i.e. 445 nm of an aqueous solution of a manganese (II) salt in the presence of an alkaline hydroxide (LiOH, KOH or NaOH) at room temperature. This synthetic approach can be readily extended to other transition metal oxides. They successfully control the shape and the size of the nanoparticles. They obtained a variety of nanoparticles morphologies ranging from one dimensional to three-dimensional nano structures i.e., nano rods, spherical, nano cubes, and nano flowers.

H. Veeramani *et al* [150] also report a green room-temperature synthesis of polycrystalline Mn_3O_4 nanowires. They started as aqueous Mn (II) was oxidized under circumneutral conditions by atmospheric oxygen in the presence of an inexpensive catalyst namely nano crystalline iron oxide (α-Fe_2O_3). The iron oxide nano crystal catalyst was synthesized by forced hydrolysis of an acidic ferric salt solution ($Fe(NO_3)_3 \cdot 9H_2O$) Fisher Scientific, 99.4% pure, ACS, method involves aging of a ferric nitrate solution at 98 °C for 7 days followed by dialysis to remove residual salts. The iron oxide catalyst was characterized by TEM and XRD. This synthesis method is novel due to its minimal energy, potential scalability, simplicity and waste output.

Over all green synthesis of manganese nano particles is a minimal energy input and small waste output method. It is fast, eco-friendly and economically alternative to traditional routes. But toxic organic solvents are used in the synthesis of manganese oxide nano particles. Large size of particles is formed in this method.

5.2. Precipitation Technique

H. Kumar *et al* [151] used precipitation method with green chemistry thrust to develop eco-friendly procedure for MnO nano particles by taking two salts of equal concentration which are manganese (II) sulphate and manganese oxalate. Characterization techniques are FTIR, UV and XRD showed the fine and most applicable nanoparticles. The UV-visible absorption showed sharp absorption at 339.60 nm due to metal nanoparticles. XRD spectra predicted the average size of 25-30 nm. These MnO_2 metal nano particles have applications in electrode materials in different rechargeable batteries.

S. Vijayalakshmi *et al* [152] used co-precipitation method from the decomposition of metal acetate and synthesize CTAB-Mn_3O_4 nanoparticles with the spinel structure. Nano particles were characterized by XRD, FT-IR and SEM. Their studies reveal that the size, morphology and shape of precursors and oxides vary significantly with the method of synthesis. FTIR and XRD analyses confirmed the composition and structure of CTAB-Mn_3O_4 i.e. Tetragonal Hausmannite and spinal shape.

Manganese oxide nano particles prepared by co precipitation method have many advantages like rapid, simple and easily control of particle size. And there are also possibilities to change the surface state. But there is dis advantages of this method is the variation in absorption peaks which is because of functional group vibration. Furthermore manganese sulphate which used as a starting agent is a bit toxic.

5.3. Sol Gel Synthesis

A. Baykal *et al* [153] used sol gel method for the preparation of Mn_3O_4 nano particles. They used manganese chloride

dehydrates as a precursor with ammonium hydroxide for the synthesis of nano particles. They studied different morphological and optical studies which showed that Mn has hyper fine structure with particles size of 32nm.

Z. Q. Song *et al* [154] synthesized manganese titanate ($MnTiO_3$) powders by sol gel hydrothermal method. They used low temperature below than 473K. They used manganese acetate with tetrabutyl ($Ti(OBu)_4$). They used auto clave and centrifuge for batter results. Their results show the good pie shape morphology of particles. And the absorption of particles lies in the visible region. NaOH play an important role in the crystallization of nano particles.

S. S. Falahatgar *et al* [155] check the effect of annealing temperature on the microstructure, optical properties and morphology of Cu-doped nanostructured MnO_2 thin films. They used sol-gel spin-coating technique on glass substrates on different annealing temperatures. They explain that the XRD patterns revealed that the annealed at 300°C exhibited its best performance. MnO_2 showed the orthorhombic phase Cu_2O showed the cubic structure.

T.F. Kuznetsova *et al* [156] Study morphology and adsorption properties of manganese oxide nano particles through sol gel method. They obtained nano particles by the reduction of potassium permanganate with manganese (II) chloride. They used different temperatures (at 80, 200, 400, and 600°C). surface area of particles decreases at 600°Cand increases at 200-400°C and. These temperatures help in the formation and selective control of nano particles.

Sol-gel process provides manganese oxide nano particles good optical and morphological studies. Absorption will be lying in the visible region. But the size of nano particles is not appropriate.

5.4. Hydrothermal Method

A. Geetha *et al* [157] synthesized manganese oxide, nickel oxide nano particles and nickel-manganese oxide nano composites by hydrothermal method using potassium permanganate and nickel chloride hexa-hydrate as a starting materials. From X-ray diffraction study using Debye-Scherer formula it was found to be orthorhombic shape with 12nm for MnO, 14nm for NiO and 5.5nm for NiO/MnO Nano composite. They found that the band gap of NiO/MnO increased with decreasing particle size.

Q. Feng *et al* [158] used soft hydrothermal synthesis of manganese oxides with tunnel structures. They prepare a precursor with layered structure and insertion of templates (structure-directing agents) into its interlayer space by a soft chemical reaction and then transform the template-inserted precursor into a tunnel structure by hydrothermal treatment. The application of that process was demonstrated by synthesizing six kinds of tunnel manganese oxides from a birnessite-type manganese oxide with layered structure. The lithium, sodium, potassium, magnesium, barium, and manganese contents in the manganese oxides were determined by atomic absorption spectrometry after dissolving the samples in a mixed solution of HCl and H_2O_2. The tunnel size can be controlled by the size of template ions. The hydrothermal soft chemical process is useful for the chemical synthesis of inorganic materials. The other new types of tunnel manganese oxides may also be obtained by this process and this process may not be limited to the manganese oxides.

A simple method to prepared manganese oxide nano particles by using hydrothermal treatment is most effective because under hydrothermal conditions (at low temperature) transformation reaction can be possible to prevent the destruction of metastable tunnel structures. New type of tunnel manganese oxides may also prepare by this method. The problem in that

method is a lengthy process and weak peaks are observed in the synthesis of manganese oxide nano particles.

6. SYNTHESIS OF TIN NANO PARTICLES

Among the nano metal oxides, tin (IV) oxide is a versatile oxide having more extensive applications in the field of gas detecting and catalysis and as a straightforward directing oxide. For its potential application in straightforward conductive cathode for sunlight based cells a gas detecting material for gas sensors gadgets, straightforward directing terminals, photochemical and photoconductive gadgets in fluid gem show, gas release show and lithium-ion batteries [159]. Tin oxide also used for catalytic and electrochemical purposes, like active catalyst for partial oxidation and amino oxidation of olefins and solid state gas sensors for environmental monitoring because of its unique electrical and catalytic properties [160]. In the field of electronics tin is used in fabricating solar cells, electrode materials, gas sensors, and doped semiconductors, optoelectronic devices and catalysts for redox reactions [161]. Numerous procedures have been produced to the synthesis of SnO_2 nanostructures like sol gel, hydrothermal method, spray pyrolysis, thermal evaporation of oxide powders and chemical vapor deposition. Different precursors may be used as starting materials for preparation of tin oxides but here we see some methods and its results in table using tin chloride as a precursor.

Table 4. *Summary of methods of obtaining tin oxide nanoparticles.*

Method	Materials	Conditions	Properties	Applications	Reference
Chemical Precipitation	Stannous chloride, dehydrate Ammonium solution.	Dried: at 80ºC for 24h	XRD: 8-10nm SPM: 73nm and tetrahedral structure	Gas discharge display	[162]
	$SnCl_2.2H_2O$, distilled water, ammonia	Stirrer: at 25ºC for 30 min, washing and heating at 100ºC for 1h. Calcination at different temperature.	Tetragonal rutile structure with size of 9 - 43 nm	Transparent conducting electrode.	[163]
	Tin (II) chloride Dehydrate, oxalic acid dehydrate, distilled water	Dry in oven at 120ºC for 24 hours, heating at 120ºC/h and soaking at 800ºC for 1 h.	Spherical shape with 75nm size.	Suitable for making optoelectronic and sensor devices.	[164]
Sol gel	Titanium tetra chloride, Ammonium hydroxide, Deionized water	Stirred: 70 ºC for 24h Dried: 40 ºC Calcined: 400 ºC for 3h.	Crystalline phases with average size of 7.7nm	Use in chemical to faster the rate of destruction	[166]
	Tin(IV) chloride Tetra hydrate, ammonium hydroxide, NaOH,	Reaction at room temperature for 24 h, drying at 80ºC for 24 h	FTIR, BET, XRD and TEM: 4-5.6nm	Electro chemical and catalytic applications	[167]
	$SnCl_2.2H_2O$, polyethylene	Stirrer: 30 min Refluxed: 80 ºC for 5h.	16–32 nm	solid state gas sensors	[168]

	glycol, pure ethanol, acetyl acetone	Aged for 72 h at 30 ^{o}C. Dried:100 ^{o}C for 30 min calcination at 450 and 600 ^{o}C for 1 h.			
	$SnCl_4$, ethylene glycol	Stirring: 80 ^{o}C heated: to 120 ^{o}C Dried: at 150 ^{o}C for 24 h. Final: Heat treatment at 600 ^{o}C for 2 h.	XRD: Crystallization occurs at low temperature as 250 ^{o}C with 12nm size.	Amino oxidation of olefins	[169]
Green synthesis	leaves of Cleistanthus Collinus plant, ethanol, tin oxide aqueous solution	Heated: at 60^{o}c for 2 hours, after that heated at 80^{o}C for one hour.	49.26nm	Used in antioxidant activities	[170]
	Matelic tin, plant extract of Ficus Benghalensis, Baringtoria acutagularis, Annona Squamosa Linn, Cyclea Peltata	Reaction at room temp Calcined: at a running temperature of 400^{o}C.	Tetragonal Phase with size 27nm.	Used for catalytic and sensor applications	[171]
	Plectranthus amboinicus plants, double distilled water, zinc nitrate solution H_2SO_4	Heated: to 200°C and maintained for 3 h in an oven, cool down to the room temperature. Calcined: at 300°C for 2 h	XRD: high purity of SnO2 nano particles with size of 63 nm.	Act as photo catalytic	[172]
Hydrothermal process	Tin chloride ($SnCl_4$.$5H_2O$), Cetyl trimethyl ammonium bromide, Urea $(NH_2)_2CO$, ethanol(C_2H_5OH)	Heated: in an oven at 100^{o}C for 24 hours, dried at 80^{o}C overnight and at last calcination at 600^{o}C for 2 hrs	XRD: 19 nm with Spherical shape and tetragonal structure	Fabricating solar cells	[173]
	cetyltrimethylammonium bromide hydrated tin chloride ethanol urea	Heated: 100 ^{o}C for 24 h. Dried: at 80 ^{o}C overnight. Grind: at 400 ^{o}C for 6 h Calcination: 24 h at 120 ^{o}C.	Reflection peak for 24, 48, and 72 h is in the range of 9.98, 6.98 and 5.37 nm, respectively.	Catalysts for redox reactions	[174]
	$SnCl_4.5H_2O$ NaOH, sodium dodecyl sulphate (SDS)	Heat: at 160^{o}C, 180^{o}C, and 200^{o}C for different times then cool at room temperature and	SnO_2 have diameters of 200 to 900 nm and shell thickness of 60	Rechargeable Li-batteries	[175]

	hexanol,	dried at 60°C for 12h.	to 70 nm Spherical shape		
Hydrolysis reaction	$SnCl_2.2H_2O$, ethanolic nitric acid, tetrabutylammonium hydroxide	Reaction at room temp.	4 nm with absorption spectroscopy in the ultra-violet range.	Optoelectronic nano devices	[176]
Chemical reduction method	tin(II) acetate, tin(II) chloride, tin(II) sulfate, tin(II) 2-ethylhexanoate , diethylene glycol polyvinylpyrrolidone , sodium borohydride	Stirring: 90 min 6000 rpm for 30 min heating: from 30°C-250°C at the rate of 10°C/min.	Sn nanoparticles were highly diverse with size ranges of 3 nm and ~6 nm,	To lower melting points.	[177]

6.1. Precipitation Method

A. N. Naje *et al* [162] prepared SnO_2 nano powders by precipitation method they used stannous chloride dehydrate ($SnCl_2.2H_2O$) and ammonia solution. The XRD pattern of the prepared sample is indexed to the tetragonal structure of SnO_2, and the calculated particle size in the range 8-10 nm. SPM estimated the average particle size 73 nm. The optical band gap of the SnO_2 was found to be 3.78 eV and 4.3 eV.

L. C. Nehru *et al* [163] studied photoluminescence nanocrystalline tin oxide by using $SnCl_2.2H_2O$, distilled water and ammonia as source. Crystallographic parameters such as crystallite size, lattice parameters and dislocation density in SnO_2 nanocrystalline powders were calculated by Rietveld analysis. Nano particles with crystallite size of 9 - 43 nm was found for SnO_2 powders by controlled heat treatment process. Results showed that the heating temperature can also affect the luminescence process and the emission resulting in the decrease in the oxygen vacancies.

A. Banisharif et al [164] had work on TiO_2/Fe_3O_4 nano composites for Enhancement of Photo-Decolorization of Congo Red Dye. For this purpose they used ultrasonic-assisted deposition-precipitation method. The 16/1 ration of TiO_2 /Fe_3O_4 showed the optimum magnetism and photo-decolorization activity. Results of their work showed that the band gap of the nano composite decreases with the increase of Fe_3O_4 content.

H. Taib *et al* [165] prepared the tin oxide nanoparticles by using Tin (II) chloride dehydrate, oxalic acid dehydrates and distilled water. Solution of oxalic acid with different concentration added in solution of tin chloride of fixed concentration under magnetic stirring. Formed precipitate washed and dried for removal of chloride ions. Tin oxide nanoparticles appear after the washing and drying process. By XRD analysis particle size is 75nm. In their paper, some samples are design which sample consisted of imperfect particles due to these imperfections, ionic collision frequency decrease and diffusion distance began to decrease.

Preparation of nano particles of SnO_2 in industrial scale at low cost is a challenge in material production. The main benefits of this method are a simple synthesize route, low cost materials. But repeated washing of the precipitate using distilled water could alter the stoichiometry of the intended metal oxides.

6.2. Sol Gel Method

Z. Ahmadi *et al* [166] improved the photo-catalytical activity of TiO_2 /SiO_2 by loaded the platinium particles on it. They also control the acidity of synthesis for more batter results. There results showed that the doping is not homo-genius and rate

of destruction is three times faster by using Pt doped catalysts than that of TiO2 /SiO2 catalysts

R. Adnan *et al* [167] used the Tin oxide nanoparticles as catalyst that prepared by Sol gel method with the help of Tin (IV) chloride tetra hydrate, ammonium and sodium hydroxide as source. During the preparation, the following reaction takes place by increasing the concentration of ammonia by gradually increasing from 1.07- 10.67M results that increase of the pH from1- 10.2, due to this, the particle size increases slightly from 4.2- 5.6 nm. It declared that the surface area of nanoparticles decreases with increasing ammonia concentration.

M. Aziz *et al* [168] synthesized tetragonal phase SnO_2 nanoparticles by sol gel technique using tin chloride ($SnCl_2.2H_2O$) and polyethylene glycol at different calcination temperatures. $SnCl_2.2H_2O$ dissolved in pure ethanol (C_2H_5OH). The solution was stirred for 30 min in a closed three necked flask and about 5 ml of acetyl acetone was added drop wise for the hydrolysis of SnO_2. After another 30 min, the solution was refluxed at 80 ^oC for 5h continuously to form the SnO_2 sol solution. 1 ml polyethylene glycol PEG was applied as a chemical modifier reagent was added in the SnO_2 solution and was aged at 30 ^oC for 72 h. Before calcination process at 450 and 600 ^oC for 1 h the sol was dried at 100 ^oC for 30 min. The size, purity and phase of the resultant products were characterized by XRD. Increase in molecular weight of PEG by decreasing in temperature caused the reduction of particle size. The results showed that molecular weight of PEG and the calcination temperatures play a significant role in determining the size of SnO_2 nanoparticles synthesized via this method. Calculated the particles size by Scherer equation was in the range of 16–32 nm.

G. Zhang *et al* [169] used a sol-gel process starting with tin tetrachloride and ethylene glycol as precursors. Crystallization of tin oxide starts at about 250 ^oC and growth continues at higher temperatures as the organics are removed. Fine powders with average particle size of 12 nm are obtained upon firing at 600 ^oC for 2 h. Results suggest that the -$OHCH_2CH_2OH$- prevent Cl^-ions from access to tin ions due to steric effect and hence increase the stability of the sol solution. Sol gel process for tin oxide nano particles offers many advantages of better homogeneity, simple to control, and the solution is extremely stable even in the presence of high concentration of hydrochloride. But there are two main disadvantages of this process. First, it is difficult to control the stoichiometry of a doped tin oxide. Second, residual Cl^- ions not only retard the kinetics of gelation but also affect the stability of the solution.

6.3. Green Synthesis

P. Kamaraj *et al* [170] prepared the SnO_2 nanoparticles by using cleistanthus collinus plant extract. Dried powder of leaves add with ethanol and after filtration tin oxide aqueous solution is added. The greenish yellow color of solution indicates the preparation of tin oxide nanoparticles. The preparations of tin oxide nanoparticles were definite by XRD, SEM and EDAX (Energy dispersive spectroscopy (EDS). SEM analysis show that the nanoparticle having the size which ranges from 20-40nm but according to this EDS the average crystallite size of SnO_2 nanoparticles was 49.26nm. Results show that when the concentration of SnO_2 nanoparticles start to increased, the antibacterial effect goes to increase.

S. Sudhaparimala *et al* [171] used five potent plant extracts Baringtoria acutagularis, Ficus Benghalensis, Annona Squamosa Linn, and Cyclea Peltata. The equivalent preparation is widely used as a drug formulation in the treatment of spectrum of infectious diseases. The overall research study provided an alternative route of preparing nontoxic nano tin (IV) oxide composite with good

biological activity apart from its already proven catalytic and sensor applications.

L. Fu *et al* [172] synthesized the photo catalytic activity toward rhodamine degradation by using the plectranthus amboinicus plants which prepared the tin oxide nanoparticle. By aggregation effect, the size of the tin oxide nanoparticle cannot be stately visually. XRD showed the average size of the SnO_2 NPs is 63 nm. The properties of light absorbance of tin oxide NPs were explored using UV spectroscopy. It's evidently detected that the observed tin oxide NPs shows a higher absorbance than commercial SnO_2. Tin oxide nanoparticles have the average size are 3 nm were successfully synthesized through green synthesis method using Saraca indica flower extract which use as a reducing agent. Biogenic SnO_2 nanoparticles confirmed activity of antibacterial in contradiction of antioxidant and E. coli properties.

A convenient, non-toxic, mild and natural product yield method to produce metal oxide nano particles is green synthesis. In this method fungi, bacterial and plant extract are three main routes for biosynthesis of nano particles. Tin oxide nano particles form this method show crystals of big size and small intensity.

6.4. Hydrothermal Method

S. Blessi *et al* [173] prepared the tin oxide nanoparticles by hydrothermal approach using the tin chloride from sigma. First of all, hydrated tin chloride and CTAB add in distilled water. After this urea added drop wisely to checked pH at 8, results that mixture convert into Teflon-lined autoclave. After heating, drying and calcination process, tin oxide nanoparticles appear. The crystallite grain size that appears by using XRD analysis of SnO_2 is 19 nm. By Field Emission Spectroscopy (FESEM), samples that obtained are in spherical shape but having particles smaller in size whereas TEM showed 400-800nm in size of Tin oxide.

M. A. Farrukh *et al* [174] fabricated Tin oxide nano balls and nano plates by using a cationic surfactant of cetyltrim ethyl ammonium bromide (CTABr). They compare hydrothermal and conventional heating method they also check the effect of different reaction parameters (temperature, time and ratio of $Sn^{4}+$ to CTABr) on morphology, particle distribution and particles size. They observed that the morphology of nanoparticles synthesized via the conventional heating method was influenced by changes in the reaction parameters (temperature, surfactant ratio and time). While with the hydrothermal method no significant change was observed.

L. Tan *et al* [175] used hydrothermal method and described the optical property of Tin oxide nanoparticles with the help of precursor are $SnCl_4.5H_2O$, NaOH (5.0 M), sodium dodecyl sulphate (SDS) and 9.0mL hexanol and 30.6mL heptane. XRD's result of nanostructured SnO_2 by different time and hydrothermal temperature showed that all the products are the mono-phasic with good crystallinity. FT-IR analysis concludes that because of the FTIR samples were kept and ground in air the high surface area of the nano size materials outcomes in quick adsorption of water from the atmosphere. The diameter of SnO_2 hollow spheres changes from 200-900 nm with increasing reaction time under 160^{o}C means that with increasing reaction time, the characteristic ratios of SnO_2 nanorods increase that discovered by SEM analysis. Energy band gap are different in nano size and bulk material. TEM and high-resolution TEM investigated that tin oxide hollow spheres are in the scale of 400 to 800nm in diameter. By shell thickness, they are in the range of 60–70 nm.

Among all of methods to prepare hydrothermal method is widely used to prepare tin oxide nano particles. Because of its simplicity, low cost, high efficiency and low temperature method. It is one of

the best methods for producing fine oxide powders. On the other hand, changing experimental parameters can tune the nanostructure morphologies. The use of expensive autoclave is a big disadvantage of hydrothermal synthesis.

6.5. Hydrolysis Method

C. Ribeiro *et al* [176] used tin chloride and ethanol to prepare tin oxide nanoparticles by hydrolysis reaction. They vary the concentration of tin from 0.0025 to 0.1 M. They also check the effect on nanoparticles by changing the PH. They use TEM and OAS for characterization. Their results show that higher initial ion concentrations and agglomeration lead to larger nanoparticles i.e. with 0.025 M concentration tiny particles have 4nm size and it increases with increasing concentration.

As in hydrothermal synthesis, water is used as a catalyst. Higher agglomeration and initial ion concentrations lead to larger nanoparticles. They explained the concentration effect by enhancing growth due to a higher super-saturation of the liquid medium. And they observed that the agglomeration of the nanoparticles in suspension by the oriented attachment mechanism. But there is a difference in final particles size which is related to growth effect.

6.6. Chemical Reduction Method

S. S. Chee *et al* [177] used four tin precursor agents: tin (II) acetate, tin (II) chloride, tin (II) sulfate, and tin (II) 2-ethylhexanoate for the synthesis of tin nanoparticles. They set synthesis conditions according to precursor type. Particles synthesized with tin(II) sulfate or tin(II) 2-ethylhexanoate displayed characteristics of mono-dispersity at reduced size having an average diameter of 3nm and 6nm and exhibited melting points of 102.2°C and 131.1°C, . Results are Depending on the precursor type, the sizes and size distributions of the synthesized Sn nanoparticles were highly diverse.

There is a much role of chemistry in materials. For tin oxide nano particles chemical reduction method is very easy and fast but chemical methods lead to the presence of some toxic chemicals, which adsorbed on the surface of nano particles that may have adverse effects in environment and applications.

7. SYNTHESIS OF IRON NANO PARTICLES

Iron oxide nanoparticles have mechanical, compound, physical and warm properties that are better than customary mass iron oxide. The significant character of the particles depends on the basis of their shape, size and virtue of these nanoparticles utilized for recording media. These particles ought to be single area of unadulterated stage having high coercivity and medium polarization. These properties make $CoFe_2O_4$ nanoparticles suitable for attractive recording applications, for example high-thickness computerized recording circles, sound and tape and so on. Iron oxides nanoparticles assume a noteworthy part in numerous regions of science, material science and materials science. Iron oxide nanoparticles are better than bulk iron oxide due to its mechanical, chemical, physical and thermal properties. Fe_3O_4 (magnetite) is one of the magnetic nanoparticles which is used for recording media. Iron oxides nanoparticles play a major role in many areas of chemistry, physics and materials science. Iron has vast applications in pigment for paint industry, magnetic resonance imaging (MRI), material nano composites and drug delivery [178]. But there is need for developing fabrication processes that are relatively simple and yield controlled particle sizes. Conventional techniques for preparation of nanoparticles include evaporation condensation, hot spraying, laser-induced, sol-gel processing, vapor phase reactions, matrix isolation and aerosols. The utilization of these nanostructures in pharmacy includes real necessities on the grounds that oxide

nanoparticles must be super paramagnetic, with a discrete appropriation and restricted size dissemination in a reach littler than 20 nm, which is a condition for keeping up these specific physical and concoction properties. A wide mixture of routines have been accounted for in the writing for the combination of Fe_3O_4 nanoparticles such as hydrothermal process [179], sonochemical method, micro-emulsion technique , electrochemical route and co precipitation method . The literature survey reveals that some of the reports are in availability on green synthesis of Fe_3O_4 nanoparticles. Now a day's synthesis of iron oxide nanoparticles using phytochemicals has attracted much attention due to their simplicity, environmental benignity and low cost.

Table 5. *Summary of methods of obtaining iron oxide nano particle.*

Method	Material	Conditions	Properties	Applications	Reference
Micro emulsion	Dioctylsulfosuccinate sodium salt, Sodium hydroxide , iron (III) Chloride, AOT (Sigma-Aldrich Fine Chemical Isooctane	Stirrer: 25 °C and certain rpm Shake: at 300 rpm and 25 °C for 6 h	With the increase in surfactant concentration the colloidal nanoparticle size increased.	Work is in the production of in-situ nano catalysts which finds application in in-situ heavy oil upgrading.	[180]
	Span 80, Tween 60, potassium borohydride, ferrous sulfate heptahydrate, n-Butanol, Isooctane.	Reaction for 30 min Stirring: at 3000 rpm	Particles have spherical shape with average diameter about 80-90 nm.	High quality magnetic materials	[181]
Co precipitation method	DI water, NaOH, gluconic acid(GA) lacto bionic acid(LBA) or polyacrylic acid(PAA) sol, $FeCl_3$,$FeCl_2$	Stirred and heated to 60 °C. Reaction at 60 °C for 20 min	These nanoparticles Showed a much larger hydrodynamic size.	Biological and biomedical	[182]
	Ferric Chloride, ferrous chloride. sodium hydroxide	Stirring for 60 minutes at 30 °C Dried in oven at 60-70 °C	5-20nm	Removal of procion Dye	[183]
Wet chemical method	Ferric chloride, cobalt chloride, Sodium hydroxide, Oleic acid of HPCL, Double distilled, de-ionized water	Liquid reaction: Temperature of 80°c and stirred for one hour Centrifuged for fifteen minutes at 3000 rpm. Dried overnight at 100°c Heating at 600°c for ten hours	XRD and TEM: 15-48nm	Magnetic recording applications	[184]

	Ferric chloride, Urea, aqueous-alkaline medium	Reaction at 150.0 °C and pH 8.33	spherical shape with the typical diameters (45.0 ±5.0 nm)	Magnetic resonance imaging (MRI)	[185]
	sodium boro-hydride, ferric chloride hexa-hydrate	Dried: at 50 °C in oven for overnight.	Particles have spherical shape with average size of about 8 nm.	To produce efficient chemical sensor development, active photo catalyst	[186]
Green synthesis	Ferric chloride, Green tea extract, Sodium alginate, Calcium chloride, Acid dye, Basic dye, Distilled water	Reaction at 60°C for 20 minutes Stirred for 5 minutes in magnetic stirrer	The average grain boundary size is 250nm.	Removal of both organic and inorganic pollutants (dyes) from aqueous solutions.	[187]
	The yellow P. Tripartita fresh, Ecuador. Ferric Chloride	Magnetic stirring for 4 h at 80 °C. Dried at 90 °C for 16 h Centrifuged At 7000 rpm for 15 · 2 min	Spherical shape with size below than 25 nm in diameter.	Catalyst for degradation of dye	[188]
	Ocimum sanctum, Milliq water	Boiled at 80°C for 10-15min, stored at 4°C,	XRD: 47 nm TEM: 20nm	Used to maintain cell structures	[189]

7.1. Micro Emulsion Method

N. Nassar *et al* [180] prepared iron oxide nanoparticles by w/o micro emulsions using iron chloride, sodium bis (2-ethylhexyl) sulfosuccinate (AOT), isooctane and water. Their technique provided an in situ nanoparticle synthesis technique focused at minimizing particle aggregation associated with particle transportation to required sites. They study the effects of water and AOT concentration to AOT mole ratio on the nanoparticle size. To measure particles size distribution they used TEM and UV spectrophotometry. Results showed that with the increase in surfactant concentration the colloidal nanoparticle size increased.

Z. Yunxia *et al* [181] used micro emulsion method for preparation of iron nano particles and modified the technique by applying Tween 60 and Span 80 as mixed surfactants. They used saturated Fe^{2+} solution to synthesize α-Feultra fine particles by redox reaction. They found the maximum content of water in the water-in-oil micro emulsion and appropriate forming conditions. 0.5 weight ratio of surfactants and co. surfactants and 2 weight ratio of surfactant and oil was the optimum condition for micro emulsion formation. TEM and XRD were used as characterization techniques and showed the average diameter of the particle is about 80-90 nm.

This technique serves as an in-situ nanoparticle synthesis technique used to minimize particle aggregation. Microemulsions systems for iron oxide are especially interesting as they provide

control over particle sizes and produce homogenous and mono dispersed nanoparticles. There is no need of expensive instrumentations. The production of nanoparticles in microemulsions is controlled mainly by the reactant exchange dynamics, for rapid reactions and reactant distribution in the water pools, for slow reactions. In this procedure the environmental friendly surfactants sorbitan monooleate (Span 80) used which is very important. Magnet properties of nano particles through this method are not more efficient.

7.2. Precipitation Method

D. Dozier *et al* [182] prepared iron oxide nanoparticles through co. Precipitation method coated with biological molecules e.g., gluconic acid, lacto bionic acid, or polyacrylic acid. They used ferrous chloride $FeCl_2$, ferric chloride $FeCl_3$ and NaOH for synthesis and then coated the surface with biological molecules. During synthesis, the concentration and amount of NaOH was varied to control the particle size. This work is useful for biological application. XRD show that the particles are highly water soluble because of hydrogen bonding and fully oxidized oxide form and narrow size distribution.

P. L. Hariani *et al* [183] Synthesized and talk about the properties of Fe_3O_4 nano particles by Co-precipitation method. They also used ferrous chloride [$FeCl_2$], ferric chloride [$FeCl_3$] and sodium hydroxide [NaOH]. Their main focus was on the suitability of iron oxide to remove dye in the water by a simple magnetic separation process. The optimum adsorption occurred at initial concentration of procion dye 100 mg L^{-1}, 6 Ph and 0.8 g L^{-1} dosage of Fe_3O_4 at room temperature for 30 minutes with color removal 24.40 % and adsorption capacity was 30.503 mg g^{-1} .SEM and TEM image of the Fe_3O_4 showed nanoparticles Fe_3O_4 have the mean diameter 5-20 nm.

Co-precipitation method is a method of synthesis of Fe3O4 which is easy to do with the success rate from 96 to 99.9%Chemical co-precipitation can produce fine, stoichiometry particles of single and multi-component metal oxides. The drawback of this method is that the adsorption is totally dependent on pH.

7.3. Wet Chemical Method

K. Maaz *et al* [184] synthesized magnetic cobalt ferrite nanoparticles by wet chemical method using stable ferric and cobalt salts with oleic acid as the surfactant. Formation of single phase cobalt ferrite nanoparticles confirmed by XRD and TEM in the range 15-48nm depending on the time and annealing Temperature. Results showed that with the increase in time and annealing temperature the size of the particles increased while the co-ercivity goes through a maximum peaking at around 28nm. The high field moment is observed to be small for smaller particles and approaches the bulk value for large particles.

M. M. Rahman *et al* [185] prepared nano particles in large-scale by wet-chemical method (150.0 ^oC and pH 8.33) using ferric chloride and urea as a starting materials in aqueous-alkaline medium. The physical, structural and optical properties are characterized using FT-IR, UV–vis spectroscopy and (XPS). The NPs size (average dia. 45 ± 5 nm) was measured by FE-SEM while the single phase of nano particles were exemplified using powder X-ray diffraction technique. The analytical performances of nano particles sensor were investigated that the sensitivity and stability of the sensor improved extensively using NPs thin-film on active silver surface. Their study has introduced a novel way for efficient chemical sensor development as well as active photo-catalyst using low-dimensional nano particles for the detection of environmental carcinogenic and hazardous compounds.

S. H. Chaki *et al* [186] also synthesized Fe_3O_4 nanoparticles by wet chemical

reduction method. They used Ferric chloride hexa hydrate ($FeCl_3.6H_2O$) and sodium boro hydrate ($NaBH_4$) for synthesis of Fe_3O_4 nanoparticles at ambient temperature. Structural properties and crystallite size were characterized by XRD using Scherer's equation was 7.20 nm and by Hall–Williamson's plot was 6.58 nm. Through SEM Surface morphology of synthesized Fe_3O_4 nanoparticles was studied which was composed of spheres having uniform flower-like features. UV spectroscopy revealed the optical absorption of the synthesized Fe_3O_4 nanoparticles and FTIS was used to study band gab in infrared region. Thermo gravimetric technique was used to calculate the thermal stability over all the average particles size is 8nm.

In summary wet chemical reduction method for synthesis of Fe3O4 nanoparticles at ambient condition can synthesized nanoparticles of do not contain any impurity elements and perfectly stoichiometric. The advantage of this method over the others is that the control of production of ferrite particles, its size distribution and is relatively easy and there is no need of extra mechanical or microwave heat treatments.

7.4. Green Synthesis

A. M. Paul *et al* [187] synthesized the iron nanoparticles (Fe NPs) using green tea leaves with Ferric chloride, Sodium alginate, Calcium chloride, Acid dye (textile dye), Basic dye (methylene green) and Distilled water. The green tea extract acts as both reducing and capping agent during the synthesis procedure. The iron nanoparticles formed was used for removal of both organic and inorganic pollutants (dyes) from aqueous solutions catalyst for degradation of dye. They check effects on dye by changing pH, concentration and dosage and ionic strength of the dye. Their result suggests that the iron nanoparticles are more efficient in decolorization of acidic than basic dye.

B. Kumar *et al* [188] synthesized iron oxide nanoparticles using an aqueous extract of Passiflora tripartita var. mollissima fruit with ferric chloride. They used mild conditions and form excellent results and also studied their catalytic effect for the synthesis of 2-arylbenzimidazole under room temperature. Dynamic Light Scattering (DLS) and Transmission electron microscopy (TEM) analysis show that the average particle size of spherical iron oxide nanoparticles is 22.3 ± 3 nm. Their TEM pictures showed Fe_3O_4 nanoparticles of below 25 nm in diameter with spherical shape.

M. G. Balamurughan *et al* [189] used an eco-friendly green synthesis of iron oxide nanoparticles using leaf extract of Ocimum sanctum was investigated. The XRD showed the average size estimated to be 47 nm. SEM micrographs at different magnification levels showed that the synthesized iron oxide nanoparticles were in the form of irregular shapes in aggregated form. TEM analysis confirms that the size of the iron oxide nanoparticles was found to be less than 20 nm. Thus, the green synthesis using ocimum sanctum leaf extracts can be economic and effective method for the synthesis of iron oxide nanoparticles.

Green synthesis using Ocimum sanctum and Passiflora tripartita leaf extracts is effective and economic method for the synthesis of iron oxide nanoparticles. This method is beneficial because the catalyst is easily separated by recyclable and magnet without significant loss of activity. The reaction in this synthesis is carried out under mild condition with excellent yields but this is slow kinetics reaction. Disadvantage of this method is the inefficiency of activity stability. If characterization takes long time the samples prepared by this method will easily alter their properties even at room temperature.

8. CONCLUSION

In summary, we discuss in detail the different synthesis methods available for preparation of chromium, manganese, silver, iron and tin nanoparticles All the materials and methodologies effect on the size and properties of nano particles are also explored while using bottom up and top down methods we cannot refers same method for the synthesis of all types of nano particles because all particles has their own properties and compatibility with synthesis method. So, for the selection of right method all its characteristics are counted its size, applications, toxicity and environmental effects.

For silver nano particles chemical reduction and green synthesis are easiest and one step method but stability factor of silver nano particles is weak. Sol gel method is important in respect of having no change with annealing temperature. Reverse micelle gives the small size not more than 10nm and on the other hand micro emulsion method gives the clusters which increase the size of nano particles.

For chromium oxide nano particles co precipitation method gives the most stable nano particles through a convenient way. Novel solvent free method is the new and inexpensive to use but if more radiations are used it changes the structure of nano particles. Hydrothermal method is convenient because it needs no stirring and temperature but the formation of large size of grains makes it inefficient for chromium oxide nano particles. Thermal decomposition method with polymer based synthesis is a good method to prepare chromium oxide nano particles.

For manganese oxide nano particles precipitation method is easy and rapid method but the absorption peaks are changes from other results. Hydrothermal method is very long method to prepare manganese nano particles whereas the green method is best for its synthesis.

Tin oxide nano particles prepared by sol gel method are sensitive toward stability and stoichiometry. Green synthesis is non-toxic and mild route but particles show big size and small intensity. In all hydrothermal methods an expensive auto clave used and the morphologies and growth mechanism are highly affected by the heating parameters. Over all precipitation method can fulfill all the properties if washed several time with distilled water.

Iron oxide nano particles are prepared by micro emulsion synthesis are feasible with respect to cost and time effect but magnetic properties are not more efficient. Green synthesis method is beneficial with respect to easy separation of catalyst but stability is not accurate in this method. On the other hand in precipitation method nano particles can synthesize of fine stoichiometry and 96% accurately. Wet chemical method need no extra mechanical or microwave treatments. This review papers provide an opportunity to select suitable method for the preparation of nanoparticles.

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