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Synthesis of Metal Nanoparticles and Their Application in Degradation of Textile Dyes by Advanced Oxidation Process

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Abstract

The discharge of textile dye-containing wastewater into aquatic ecosystems is a major environmental concern because many synthetic dyes are chemically stable, non-biodegradable, and potentially toxic to aquatic organisms and human health. Conventional wastewater treatment methods are often inadequate for the complete removal and mineralization of these persistent contaminants. The present study focuses on the synthesis of metal nanoparticles and their application as efficient nanocatalysts for the degradation of textile dyes through advanced oxidation processes (AOPs). Metal nanoparticles, owing to their high surface area, unique physicochemical properties, and enhanced catalytic activity, offer considerable potential for the treatment of dye-contaminated wastewater. The synthesized nanoparticles are characterized using appropriate analytical techniques to determine their structural, morphological, optical, and physicochemical properties. Their catalytic performance is evaluated using selected textile dyes under controlled experimental conditions. Advanced oxidation is investigated through the generation of highly reactive oxygen species, particularly hydroxyl radicals, capable of breaking down complex dye molecules into simpler and less harmful products. The effects of important operational parameters, including nanoparticle concentration, initial dye concentration, pH, oxidant dosage, temperature, and reaction time, are examined to determine optimum degradation conditions. The degradation efficiency is assessed using spectrophotometric analysis, while reaction kinetics are investigated using suitable kinetic models. The study is expected to demonstrate that metal nanoparticles can significantly enhance the efficiency of advanced oxidation processes and promote rapid degradation of persistent textile dyes. The findings may contribute to the development of efficient, economical, and environmentally sustainable nanotechnology-based approaches for industrial wastewater treatment and the mitigation of aquatic pollution.

Keywords: *Metal nanoparticles; Nanoparticle synthesis; Textile dyes; Dye degradation*

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1. Instrumental Techniques

This section includes the basic principles and theories of the main analytical approaches and characterization techniques. This section of this chapter also concerns with the details of different instrumental techniques such as electron microscopy, XRD, Zetasizer, LC-MS, FT-IR spectroscopy, UV-Visible spectroscopy etc. adopted for characterization of nanoparticles and studies the degradation of dyes in the presence of nanocatalysts.

1.1 Ultraviolet-Visible Spectrophotometer

Ultraviolet–visible spectroscopy (UV-Vis or UV/Vis) is an absorption spectroscopy or reflectance spectroscopy. It uses light in the ultraviolet and visible range. The absorption or reflectance in the visible range directly affects the colour of the chemicals involved. In this region of the electromagnetic spectrum, atoms and molecules undergo electronic transitions. Fluorescence spectroscopy is opposite to absorption spectroscopy, in the fluorescence electrons transitions from the excited state to the ground state, while in absorption electrons transitions from the ground state to the excited state.

Molecules having π-electrons or non-bonding electrons (n-electrons) which are excited in higher anti-bonding molecular orbitals may absorb the energy in the form of ultraviolet or visible light. There are four possible types of transitions (π-π*, n-π*, σ-σ*, and n-σ*), and they can be ordered as follows: σ-σ* > n-σ* > π-π* > n-π* [1].

In analytical chemistry UV-Visible spectroscopy is used for the quantitative determination of different analytes, such as transition metal ions, highly conjugated organic compounds, and biological macromolecules and this technique readily allows determining the concentrations of substances, to study the rates of reactions, to determine rate equations for reactions, with the help of these a mechanism can be suggested. Generally, spectroscopic analysis is carried out in liquid solutions but solids and gasses may also be studied [1].

UV-Vis spectroscopy is useful for analysis metal nanoparticles dispersed in a solvent or embedded in the insulator matrix. In this analysis, absorption of incident radiation takes place due to surface plasmon resonance (SPR) of the metal nanoparticles. Surface plasmon resonance is essentially the light waves that are trapped on the surface because of their interaction with the free electrons of the metal [2].

For the kinetic study of reaction, occurring in solution must present colour or brightness shifts from reactants to products. The rate constant of a degradation of dye can be determined by measuring the UV-Vis absorbance spectrum at specific time intervals. If the reaction is first order, it would have the integral first order rate law: $\ln [A](\text{time } t) = -kt + \ln[A](\text{initial})$. Then, plot the graph of natural log (ln) of the concentration [A] versus time obtained a line with slope –k, which is negative the rate constant. According to the mechanism of the reaction different rate orders have different integrated rate laws. The degradation of dye was observed by changes in absorbance is useful in the kinetics study.

The kinetic study of the degradation of dye and optical characterization of the synthesized metallic nanoparticles was observed in this research work with the help of a double beam 3000^{+} LABINDIA, UV-Vis spectrophotometer and a cell of 1.0 cm path length in the spectral range 200-800 nm. Effect of temperature on kinetic study of reaction was studied by a Peltier accessory (temperature-Controller) model PTC-2 is linked with the UV-Visible spectrophotometer. Two lamp

combinations are used in double beam spectrophotometer one is deuterium lamp used for UV part and second is tungsten lamp used for the visible part. Sample cell is made by Quartz and light beam travels a distance of 1 cm through
the sample. For obtained UV spectra of our sample first done baseline by reference so that other reagent peaks were nullified, after that obtain the spectra of reaction mixture. Spectra were plotted between wavelength v/s absorbance [3, 4].

1.2 Centrifuge

Centrifugation is a technique that advantages to separate mixtures by applying centrifugal force. A centrifuge is a device, generally driven by an electric motor that puts an object, like a rotor, in a rotational movement around a fixed axis. Sedimentation is the principle of centrifuge: Under the effect of gravitational force (g-force), materials separate according to their density.

In present research work Centrifuge (Remi C-854/6 Laboratory) with 8x15 swings out heads were used. For the preparation of metal nanoparticles purified pellets, nanoparticles were put in tubes and these tubes centrifuged at 3500 rpm for 15 minutes then pellets were re-dispersed in deionized water. This process (centrifugation and re-dispersion) was repeated three times to confirm better separation of free entities from the metal nanoparticles. Purified pellets were separate out by Whatman filter paper and then dried for further use in FT-IR and XRD.

1.3 Vacuum Oven

Centrifuged samples dried in vacuum oven at 70 °C for 2 hours (LABPRO INTERNATIONAL). The oven consist double wall with chambers size is 250 x 250 mm and 12 L capacity, inner round chamber made of stainless steel, exterior made of mild steel duly finished in white stoving enamel/powder coated paint with mat finished colour combinations. The oven work on temperature range 50 °C to 150 °C ± 3 °C. The body capable of withstanding the vacuum of 760 mm Hg, workable on 220 VAC 50 Hz single phase and dried samples were used for further characterization.

1.4 X-Ray Diffractometer

X-ray diffraction (XRD) is an analytical technique mainly used for phase determination of a crystalline material and give knowledge about unit cell dimensions. The analysed material is finely grind, homogenized, and average bulk composition is determined.

X-ray diffraction is depending on constructive interference of monochromatic X-rays and a crystalline sample. These X-rays are produced by a cathode ray tube and then filtered to produce monochromatic radiation, collimated to concentrate, and focussed toward the sample. The collaboration of the incident rays with the sample generates constructive interference when situations satisfy Bragg's Law ($n\lambda = 2d \sin\theta$). This law shows the relation among the wavelength of electromagnetic radiation, the diffraction angle and the lattice spacing present in a crystalline sample. These diffracted X-rays are detected, processed and counted. 2θ angles range is used for scanning the sample, all possible diffraction angles of the lattice should be achieved because of the random orientation of the powdered material. Change of the diffraction peaks to d-spacings permits identification of the mineral as each mineral has a set of unique d-spacings. Usually, this is gained by comparison of d-spacings with standard reference patterns [5].

X-ray diffractometers contain of three basic elements like an X-ray tube, a sample holder, and an X-ray detector. For X-rays are

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generated in a cathode ray tube by heating a filament to produce electrons, accelerating the electrons directed towards a target by applying a voltage, and bombarding the target material with electrons. When electrons have sufficient energy to remove inner shell electrons of the target material, characteristic X-ray spectra are produced. These spectra consist of several components, the most common being Kα and Kβ.

X-ray powder diffraction is most broadly used to determine of unknown crystalline materials. Identification of unknown solids is serious to studies in geology, environmental science, material science, engineering and biology [6].

XRD analysis was done by X'PERT PRO Panalytical Instrument (SAIF/CIL, Panjab University, Chandigarh). In this instrument copper used as an anode, 2θ range starts from 10.0 to 89.9. For analysis, centrifuged and dried samples (2 g) were grind in mortar and pestle then powdered sample keeps into samples holder. The powder was pressed by glass slide for the powder to "stick" in well. Enter sample into XRD machine to identify unit cell dimensions [7].

1.5 Fourier Transform Infrared Spectrophotometer

Fourier Transform Infrared Spectroscopy (FTIR) is useful technique for identifying chemical bond in molecules. It can be utilized to know some components of an unknown mixture and for the analysis of solids, liquids, and gasses. In the Fourier Transform Infrared Spectroscopy, data is collected and converted from an interference pattern to a spectrum. It is a powerful tool for identifying types of chemical bonds in a molecule by producing an infrared absorption spectrum that is like a molecular "fingerprint". The wavelength of light absorbed is characteristic of the chemical bond as can be seen in IR spectrum [8].

Bonds are present in molecules are vibrate at several frequencies which is depend on the elements are present in molecule and the type of bonds. Any bond vibrates on several specific frequencies. Quantum mechanics illustrate that these frequencies related to the ground state (lowest frequency) and many excited states (higher frequencies). One way to cause the frequency of a molecular vibration to increase is to excite the bond due to absorb light energy. Transition between given two states, the energy equal to the difference in the energy between the ground state and the excited state [9].

For getting spectra between wavelength and transmittance ALPHA-T model, Bruker, Germany spectrometer was used range is 400-4000 cm^{-1} and resolution is 4 cm^{-1}. For preparation of pellet of sample first pellet holder, pestle, mortar, dies etc. were washing with alcohol and then 1:4 ratio centrifuged dried sample and KBr grind with a pestle and mortar. Place just enough grinded sample to cover the bottom in pellet dies and press at 0-10 Tones. Then carefully remove
the pressed sample from die and place in the FTIR sample holder for FTIR analysis. The pressed disc should be nearly clear if properly made [10, 11].

1.6 Ultrasonic Processor

The ultrasonic processor works on principle that ultrasound waves transfer energy into the sample, causing turbulence and friction in the liquid so that clear sample were prepared.

The ultrasonic processor was used to prepare the nanoparticles samples for characterization by the electron microscopy (Transmission Electron Microscopy (TEM), Scanning Electron Microscopy (SEM)) and Zetasizer analysis. The dispersed nanoparticles were sonicated in Ultrasonic processor (model - Ultramet 2005 (Buehler), USA) (115 VAC, 60

Hz) with a 9.5 L capacity quickly cleans samples to prepare for the next preparation step and 0- 60 minute is timer range.

1.7 Transmission Electron Microscopy

Transmission Electron Microscopy is a major analytical technique in physics, chemistry, and biological sciences. In nanotechnology the results of TEM analysis plays a most important role in imaging morphology and distribution of nanomaterial with high resolution and provide information about the structure, crystallography nature is studied by the diffraction mode.

The basic principle of TEM is based on Light Microscope, uses electrons instead of light and electromagnetic lenses to focus the electrons into a very thin beam rather than glass lenses focus the light. TEM use electrons as "Light source" and their lower wavelength, high energy makes it possible to get thousand times better resolution than Light Microscope [12]. Electron source present at the top of the microscope emits electrons that travel through a vacuum in the column of the microscope then travels through the specimen to form an image. The specimen is most often an ultrathin section less than 100 nm thick or a suspension on a grid at the bottom of the microscope the unscattered electrons hit a fluorescent screen. This screen gives rise to a "Shadow image" of the specimen with its different parts displayed in varied darkness according to their density [12].

In TEM analysis at first electrons emits by electron gum (these electrons accelerate and have a potential difference in the range 40-200 kV) and operates under vacuum because electrons are easily scattered at atmospheric pressure [13]. Electron gun below electron on two or more condenser lenses. These lenses are electromagnetic lenses. An aperture is present between the condenser lenses to control electron beam diameter as it hits the specimen. Now electron beam interacts with the specimen and unscattered beam passes through the objective lens. The work of the objective lens is either image or diffraction pattern made of the specimen. To identify the crystallographic structure of the material electron diffraction patterns is used. After beam passes through objective lenses, produced 50-100 times magnified image [13]. This is further magnified by a series of intermediate lenses and finally projected on to the fluorescent screen.

The TEM analysis results presented in this thesis, transmission electron microscope Model- Tecnai G^2 20 (FEI) S-Twin with 200 kV energy has been used. The Tecnai G^2 supports a wide range of techniques including high resolution scanning STEM diffraction and chemical analysis with on BF/DF axis detectors provide the Z-contrast imaging. The high angle, annular dark field detector generates atomic resolution dark field STEM images. The equipment provides a point resolution of 0.24 nm, line resolution of 0.14 nm and STEM resolution of 1.0 nm. EDAX gives the elemental composition of the material and the facility available in Malaviya National Institute of Technology Jaipur, Jaipur (Rajasthan).

For TEM characterization the sample were prepared as follows: the drops of the sonicated sample were mounted on the copper grid using a micropipette, thin sections of material are sufficiently robust to spread out over the grid without additional support. This copper grid was allowed to stand for 2 minutes, the extra solution was removed using a blotting paper and the grid allowed to dry prior to TEM analysis [14].

1.8 Scanning Electron Microscopy

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The scanning electron microscope (SEM) analysis is same as the transmission electron microscope (TEM) analysis because both have a beam of electrons focussed at the specimen. The certain features, such as the electron gun, condenser lenses and vacuum system, are same in both instruments, though the ways in which the images are produced and magnified are totally different. The TEM analysis gives information about the internal structure of thin specimens whereas the SEM analysis is used to study the surface, or near the surface, the structure of specimens [13].

In the SEM instrument includes an electron source; a condenser lens; a deflector; an aberration correction device; a convergence aperture; and a detector [15]. In this technique, an electron beam is thermionically emitted from an electron gun. The electron beam, which has an energy ranging from 0.2 keV to 40 keV, is directed by one or two condenser lenses to a spot about 0.4 nm to
0.5 nm in breadth. In the final lance this beam passes over pairs of scanning coils or pairs of deflector plates in the electron column, typically in the final lens, which are responsible for deflection of the beam in the *x* and *y*-axes so that it scans in a raster fashion over the sample surface [16].

Accelerated electrons in an SEM carry significant amounts of kinetic energy, and this energy is dissipated as a variety of signals produced by electron-sample interactions when the incident electrons are decelerated in the sample. These signals include secondary electrons, backscattered electrons, diffracted backscattered electrons, X-Ray, visible light, and heat. Secondary electrons and backscattered electrons are commonly used for imaging samples: secondary electrons are most valuable for showing morphology and topography on samples and backscattered electrons are most valuable for illustrating contrasts in the composition in multiphase samples. X-Ray is used for elemental and crystalline structure analysis [17].

Nova Nano FE-SEM 450 (FEI) instrument used for SEM analysis in the laboratory of Malaviya National Institute of Technology Jaipur, Rajasthan. Ultra-high resolution provied by the instrument for characterization and analysis giving precise, true nanometer scale information. Advanced optics and detection, including beam deceleration, in lens ETD (SE), TLD (custom), a lens mounted DBS & LVD offer the best selection of information and image optimization. Beam landing energy can go down from 30 keV to 50 eV. It gives a resolution of
1.4 nm at 1 kV (TLD-SE) & 1 nm at 15 kV (TLD-SE). The FE-SEM is coupled to EDAX detector for measuring the elemental chemical composition of materials. For SEM analysis the samples were prepared as follows: 30µl aliquots of sonicated sample extracted and deposited on the glass slide or carbon tap by micropipette. The stub was dried and keeps it on the sample holder for SEM analysis [18].

1.9 Zetasizer

The Zetasizer Nano range of instruments provides the ability to measure two characteristics of particles or molecules in a liquid medium that is particles size and Zeta potential. This technique is work over a wide range of concentrations of solutions.

Size of particle determined by measuring the random changes in the intensity of light scattered due to their random motion from a suspension or solution. This technique is called as dynamic light scattering (DLS). Random motion of particles related to their size so smaller particles move faster than larger particles. The speed of Brownian motion is also

determined by the temperature, therefore precision temperature control is essential for accurate size measurement. To measure the diffusion speed, the speckle pattern produced by illuminating the particles with a laser is observed. The intensity changes are analyzed with a digital
autocorrelator which generates a correlation function and curve gives the information about size and the size distribution [19].

The Zetasizer Nano series calculates the zeta potential by determining the Electrophoretic Mobility with applying the Henry equation. The development of a net charge at the particle surface affects the distribution of ions in the surrounding interfacial region, resulting in an increased concentration of counter ions to the surface. Thus an electrical double layer exists around each particle and the potential exists at this boundary is known as the Zeta potential. The magnitude of the zeta potential gives an indication of the potential stability of the colloidal system. If the particles present in suspension have a large negative or positive zeta potential then they will tend to repel each other and there is no tendency to flocculate. However, if the particles have low zeta potential values then there is no force to prevent the particles coming together and flocculating [19].

In this research work Zetasizer ver. 7.11 (Malvern), UK was used to analyze the average size and zeta potential of the colloidal solution of metal nanoparticles. This analysis was done in the laboratory of Malaviya National Institute of Technology Jaipur, Jaipur (Rajasthan). For this analysis, sonicated sample poured in the disposable folded capillary cell then the cell was placed in the zetasizer instrument and obtained the electronic output of average size. To determine zeta potential value, the sonicated sample pour in dip cell of the instrument [20].

1.10 Liquid Chromatography – Mass Spectroscopy

Liquid chromatography-mass spectrometry (LC-MS) is an analytical chemistry technique which is based on the combinations of the physical separation abilities of liquid chromatography with the mass analysis capabilities of mass spectrometry (MS). The typical LC-MS system is a combination of HPLC with MS using interface (ionization source). The sample is separated by LC, and the separated sample species are sprayed into the atmospheric pressure ion source, to ions converted in the gas phase. The mass analyser is used to categorise ions
according to their mass to charge ratio. The separated ion identifies and quantifies directed to a photo or electron multiplier tube detector amplifies the signal generated from each ion. Result of mass spectrum (a plot of the ion signal as a function of the mass-to-charge ratio) is generated, which is used to determine the elemental nature of a sample, the masses of particles of molecules, and to elucidate the chemical structures of molecules [21, 22].

The advantages of this technology like sensitivity, specificity, and precision as analysis is done at the molecular level. This technique can be capable to analyse inorganic compounds, organic compounds and biochemical compounds commonly found in complex samples of environmental and biological origin [22].

An interface is present in LC-MS that efficiently transfers the separated components from the LC column into the MS ion source. While the mobile phase in an LC system is a pressurized liquid, the MS analysers commonly operate under vacuum. Therefore, the eluate directly pumped from the LC column into the MS source is not possible. LC-MS

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interfaces are grounded on atmospheric pressure ionization (API) strategies such as electrospray ionization (ESI), atmospheric pressure chemical ionization (APCI), and atmospheric pressure photo-ionization (APPI) [23].

Intermediates and end products obtained during dye degradation process were detected by LC-MS technique. Mass/charge ratio is used to determine the molecular ions of unknown products. When fragmentation voltage of LC-MS increases more fragments are observed to facilitate product determination. This study, for LC-ESI-MS analysis Waters, Micromass Q-TOF micro was used with parting Module: Waters Alliance 2795; LC Column: Unisol YVR C18; Ionization: Electro spray Positive (ES+); Acquisition: MRM, unit resolution; Injection Volume: 20 micro liters; Flow rate: 0.5 ml per min and for Mass spectrometer, Desolvation Gas: 550 Lts/Hr; Cone Gas: 30 Lts/Hr; Desolvation Temperature: 300 °C; Source Temperature: 110 °C; Capillary Voltage: 3000 V; Cone Voltage: 30 V; Collision energy 4 ev; Gases used N_2 and Argon; Mobile Phase used:

20/80:H_2O: MeOH; 6-7 bar (90-100 psi) pressure is used for N_2 supply and fro Argon is 5-6 bar etc. parameters were used. LC-MS characterization was done from SAIF/CIL, Panjab University, Chandigarh [24].

1.11 Chemical Oxygen Demand (COD) and Biological Oxygen Demand (BOD)

To determine the COD, BOD the Winkler method with azide modification was used.

Chemical Oxygen Demand (COD) is the total measurement of all chemicals (organics and inorganics) in the water samples.

Twenty ml sample was diluted to 40 ml with distilled water then add pumice stone, 10 ml $K_2Cr_2O_7$, 30 ml H_2SO_4 containing Ag_2SO_4. Now the flask of the mixture connected to condensor and reflux for minimum 2 h. After that cool the mixture and titrate excess $K_2Cr_2O_7$ with ferrous ammonium sulphate using ferroin indicator and sharp colour change from blue green to wine red indicates end-point. The same process was applied for the blank sample using distilled water instead of sample.

Calculation:

$$COD\ mg/l = \frac{(a-b)\ x\ N\ x\ 8000}{ml\ of\ Sample\ taken}$$

Where:

a = ml. of Ferrous ammonium sulphate used for titration of blank

b = ml. of Ferrous ammonium sulphate used for titration of sample N = normality of $(NH_4)_2Fe(SO_4)_2$ (Ferrous ammonium sulphate)

Biochemical Oxygen Demand (BOD) is a measure of, the amount of oxygen that require for the microorganism to degrade the organic components present in water samples.

Preparation of dilution water: First aerate the required volume of distilled water in a container by bubbling compressed air for 1-2 days and maintain the temperature near 20 °C. Then add 1 ml each of phosphate buffer, magnesium

sulphate, calcium chloride and ferric chloride solution for each liter of dilution water.

Dilution of sample: First neutralize the sample, and then remove chlorine content by using Na_2SO_4. Take four BOD bottles and add 10 ml sample in two bottles, then fill the remaining volume with dilution water. Remaining two BOD bottles fill with dilution water for blank experiment, immediately close the bottles to

avoid the air bubble in the bottles and shifted to the incubator (Labline Incubator Sun Instruments Pvt. Ltd. Ahmedabad (India)) at 20 °C for 5 days. Analyse dissolve oxygen immediately and after 5 days incubation.

Test of dissolved oxygen: In BOD bottles add 2 ml manganous sulfate ($MnSO_4.H_2O$) solution followed by 2 ml of the alkali-iodide-azide reagent and allow reacting the solution with the present oxygen in the sample. When precipitates are settled down at the bottom add 2 ml of concentrate sulfuric acid, mix well to dissolve the precipitates. Take sample from BOD bottle into an Erlenmeyer flask, titrate immediately with sodium thiosulfate solution using starch indicator until blue color disappear.

Calculation:

Blank correction = B.R. for blank at D_0 – B.R. for blank at D_5

BOD mg/l = [(B.R. for sample at D_0 –D_5)– blank correction] x dilution factor

$$\textit{Dilution factor} = \frac{\textit{Bottle volume (300 ml)}}{\textit{Sample volume}}$$

Where:

B.R. = burette reading D_0 = Initial
D_5 = Day five after incubation

BOD and COD measurments done from Rajasthan state pollution control board, Kota, Rajasthan.

1.12 Electronic Balance

Citizen electronic balance (model- CX 220) was used for weighing reagents. The weighing range of balance is 0.0001 mg and 220 g respectively.

1.13 pH – Meter

To determine the pH of the synthesized nanoparticles and the reaction mixtures, Systronic digital pH meter. Model MAC (MSW-552) was used with maximum uncertainty in pH of ±0.01 unit. To determine the pH of solution first calibrate the pH meter using buffer solutions of different pH such as 7.0, 4.0, 9.2. Thoroughly wash the pH electrode between measurements with distilled water to avoid carryover impurity of the tested solutions. Softly blot the electrode on a tissue paper to remove the excess rinse water and do not rub the bulb to avoid build-up static charge.

1.14 Magnetic Stirrer with Hot Plate

Magnetic stirrer with the hot plate was used for the synthesis of nanoparticles (MSW-313, MAC) at 600 rpm (range 0-1200 rpm), stirrer work on 220/230 volts AC supply and temperature 0-100 °C. The synthesis process of nanoparticles and size of nanoparticles are also affected by speed of stirring and reaction temperature.

Section II

1.15 *Materials*

The section deals with the details of all reagents employed in this study and the details regarding methods of preparation of various solutions. Chromic acid and doubly distilled water was used for cleaning glassware, which were used for handling chemicals in practical work and double distilled water also used for preparing all solutions.

1.16 Silver Nitrate

Silver nitrate is an inorganic compound and colourless crystalline solid. Lunar caustic is another name of silver nitrate. A solution of silver nitrate (E.

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Merck) was prepared by dissolving an required amount of $AgNO_3$ with the addition of double distilled water. Prepare solution were kept in dark bottle to avoid the photodecomposition and also kept the solution 4 °C temperature so that it do not show any other decomposition for appreciable longer period.

1.17 Copper(II) Chloride Dihydrate

Copper(II) chloride dihydrate (Cupric Chloride) is a blue-green crystalline solid. A fresh solution of copper chloride dihydrate (E. Merck) was prepared by dissolving an appropriate amount of $CuCl_2.2H_2O$ with the addition of double distilled water, make up to required volume in a volumetric flask.

1.18 Neem Leaf Broth

The plant *Azadirachta indica* (neem) was selected from Kota (Rajasthan) India, on the basis of cost-effective, easily available and medicinal property. Collected fresh and healthy leaves and rinsed thoroughly first with tap water followed by deionized water to remove all the dust and unwanted particles, leaves were dried in an oven for 15 minutes at 50 °C temperature and cut into small pieces. 10 g of finely incised leaves was transferred into 250 ml beaker containing 100 ml deionized water and stirred on magnetic stirrer at 80 °C for 20 minutes. The extract was then filtered twice through Whatman filter paper, then refrigerated (4 °C) in Erlenmeyer flasks for further experiments. In each and every step of the experiment, sterile conditions were maintained for the effectiveness and accuracy in results.

1.19 Methyl Orange

Methyl Orange is a mono-azo, water-soluble dye. Methyl orange is widely used in the textile, printing, paper manufacturing, pharmaceutical, food industries and also in research laboratories. This dye is mainly used as an acid-base indicator in the analytical chemistry laboratories [25]. The solutions of required concentration of this dye were prepared by dissolving the requisite amount of methyl orange (Sigma-Aldrich) in the known volume of double distilled water.

1.20 Orange G (Acid Orange 10)

Orange G is a synthetic anionic mono-azo dye used in dying of textile, food, paper industry, preparation of colour marker, and in many staining formulations [26]. Solutions of required concentration of orange G were prepared by dissolving the requisite amount of orange G (Sigma-Aldrich) in a known volume of double distilled water.

1.21 Peroxomonosulphate Solution

Peroxomonosulphate (PMS) was purchased from Sigma Aldrich by the name “Oxone”. The solution of peroxomonosulphate (Sigma-Aldrich) was prepared by dissolving the requisite amount of its potassium salt in doubly distilled water and was kept in the black volumetric flask to avoid the photo-decomposition. It is a triple salt with the composition of $2KHSO_5{\cdot}KHSO_4{\cdot}K_2SO_4$ and form with higher stability. PMS is found to be 96% pure when analyzed by both ceremetrically and iodometrically [27]. This reagent was used without further purification because permanganate tests showed the absence of free hydrogen peroxide. However, tests for free H_2O_2 were negative. Freshly prepared solution of the peracid always used in kinetics as well as analytical studies.

1.22 Peroxodisulphate Solution

The peroxodisulphate (PDS) is a white powder. A fresh solution of PDS (Sigma-Aldrich) was prepared by dissolving an appropriate amount of PDS with the addition of double distilled

water, make up to required volume in a volumetric flask.

1.23 Sodium Hydroxide

Sodium hydroxide solution was prepared by dissolving approximately weighed pellets of NaOH (BDH Analar) in double distilled water and solution was standardized by titration the known aliquot sample against the standard oxalic acid solution using phenolphthalein indicator [28, 29]. However, sodium hydroxide solution was always used after standardizing because these solutions decline on standing at ambient temperature.

1.24 Sulfuric Acid Solution

For preparation of sulphuric acid (Merck) solution first dissolves the requisite volume of sulfuric acid in double distilled water and second, the solution was standardized against pre standardized solution of sodium hydroxide employing phenolphthalein as an indicator. The standardized solution was kept stoppered as a stock solution.

1.25 Ethanol (EtOH)

Ethanol is miscible with water and is a good general purpose solvent. Ethanol (E. Merck) is a volatile, colourless liquid that has a slight odour. It was used for testing free radicals formed in reaction mixture.

All other reagents such as hydrochloric acid, potassium bromide etc. were either of (BDH AnalaR) grade or (E. Merck) guaranteed grade and used as supplied. Double distilled water, second distillation being from alkaline permanganate solution in a glass assembly, was employed in all the solution preparations and kinetic studies. All glass vessels were used for storing the chemicals solutions and for kinetic studies of degradation process were either of pyrex or borosil makes. Synthesis process of nanoparticles and kinetic procedure and analysis of results were discussed in concerned chapters.

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