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Eco- Friendly Synthesized Of Copper Nanoparticles For Methyl Orange Degradation Via Neem Extract

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Abstract :-

A bio synthesis or Green synthesis of copper nanoparticles (CuNPs) is an interesting aspect in the field of these technology and is also inexpensive method for nanoparticles biosynthesis. But inrecently utilization of secondary metabolites from plant leafage excerption has emerged as a new technology for the synthesis of various nanoparticles. we are focused on green synthesis,the synthesis of copper nanoparticles and comparison the chemical process in CuNPs/peroxodisulfate (PDS) and CuNPs/peroxomonosulfate (PMS) process for degradation of Methyl Orange (MO). copper nanoparticles were synthesized by the Liquid leaf excerpt of *Azadirachta indica* (Neem). In Green synthesis Dye, initial pH and high temperature rapidly promoted the degradation of molecule. The degradation of MO in CuNPs/ Peroxosulfates system is sculptured as pseudo-first order mechanics and parameters were also determined. Sulfate radicals (SRs) (SO4%−) were also identified with copper as oxidative species using circumstantial alcohols. Moreover, nanoparticles of copper oxide are characterized through UV-Vis spectra, FTIR, XRD In the present study the formation and stability of the reduced CuNPs was monitored by absorbance spectra of UV-visible photometer at different stages during the synthesis process. Metal nanoparticles in the solution were monitored by UV–Vis spectro photometer analysis. Their peaks of CuNPs were determined with size and crystal shape and characterization. Apply green synthesis of CuNPs to enhance oxidation capacity of peroxosulfates for degradation of MO is a novel, efficient, promising and eco-friendly process and it does not require costly chemical agents.

Keywords:- neem extract, green synthesis , copper radical, A. indica leaves, Cu NPs

Introduction :- when we synthesized NPs by physical method which is include evaporation, vaporization, laser ablation. And when we used second method named as chemical method in which the metal ion in solution is reduced under favoring conditions the formation of small metal oxides. Here chemical method mainly grouped into two, one is classical (by radiation chemical). other third method is biological synthesis. This is also a form of chemical method that are eco friendly and naturally occuring reducing agent such as plant extract, fungi, bacteria and many other used. It was noticed that the metal NPs formation is as the result of anti oxidants and reducing property [1].

Fig a - Azadirachta indica (Neem)

Here in this method we should also know that about this magical antibacterial medicial plant name as azadirachta indica, these imaginary properties of this three and their extract induced we to discuss about this tree: -

Fig a: A fresh Azadirachta indica's (Neem) leaves

Azadirachta indica (Neem); is a tree in mahogany family. we used in

controlling blood sugar level,antibacterial property also clean blood and in soap formation. Its amazing medicinal property from Neem are believed to be cleaning bound of body, antifungal, antidiabetic, antibacterial, antiviral, Neem Plant Leaves extract is used to produce Iron contraceptive and medication Various plant part can be used in the synthetic thinking of metal nanoparticles i.e. the leaf, stem, root, flower and seeds, bark power and fruits.

Family Name: Meliaceae

Binomial name: Azadirachta indica

Common name: Neem

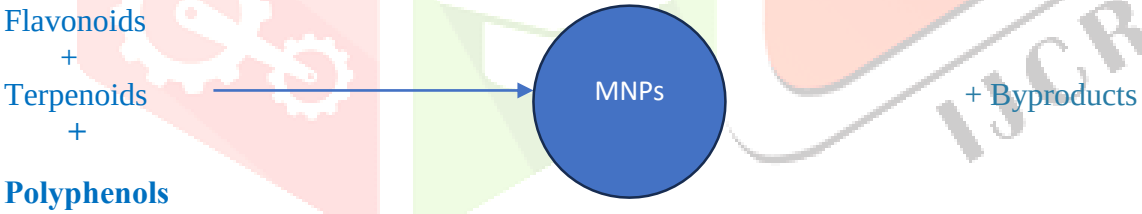
Extract taken part : Leaves

lower toxicity. The formed reactive species sulfate radicals depend on the catalytic activity an The reduction as well as oxidation technology is the most effective chemical oxidation method and currently applicable in water treatment process [2]. Determined by the demand and seeking for a procedure, that present powerful oxidants than hydroxide radicles (OH%), ozone, Fenton settled activity [3], so this procedure works research the period of very strong oxidizing form of species (sulfate radicals) through with the transition metal mediated activity of peroxosulfates [4]. then also this procedure is the modification of Fenton reagent since an oxidant is coupled with a transition metal in a correspondent behavior. since many more study reports [5] that sulfate radicals are not existence more efficient than hydroxyl in harmful organic compounds. Yet, the fact that the sulfate contains compounds were show the most effective oxidants to proves that sulfate radicals, generated by heat (6), ultrasound (7) and transition metal (8) are all-powerful oxidizing species. transition metal coupled oxidative process show greater removal efficiency for the degradation of carcinogenic contaminants into lower molecular weight andd oxidation state of transition metals. The copper catalyzed

decomposition follows a sulfate radical based mechanism, the same was suggested when nickel, cobalt, iron, ruthenium and silver transition metals were used [8–11].

Fig b : A boi synthesis of nanoparticles

metal nanoparticles are attracting the great attention of present day or future in field of nanometer scale leads to specific intrinsical properties for the material that execute them very promising for utilization in catalytic activity. The importance of transition metal NPs as a accelerator for hydrogenation [12], hydrosilation [13] as well as redox [14] and other electron transfer process [15] were reported. Among the metal nanoparticles, copper nanoparticles are very attractive due to their excellent
physical and chemical properties and low cost of synthesis, have been of great interest. Copper nanoparticles have wide applications in heat transfer system [16], anti-microbial materials [17], super strong materials [18] and catalysis [19].



Metal nanoparticles

(chemical constituents of plant extract act as

Reducing as well as capping agents)

Fig : 1 constituents of plant extract responsible for the bioreduction of metal ions.

A green synthesis of nanoparticles vary importance than physical and chemical methods of metals degradation. [20–23] reason is that the chemical method are costly and hazardous comparison to eco-friendly method. So for nanoparticle bio-synthesis, researchers used plant extract and microorganism for synthetic thinking [24–27]. other literary study reports present synthesis of CuNPs through with a green path which also includes the T. arjuna bark solution or raw extract [28],

the potential of copper nanoparticles was investigated, and the application of plant-based copper nanoparticles in the development of antibacterial nanoparticles was carried out by agar disc diffusion method against microorganisms[29].The production of the copper nanoparticles through bio-reduction of copper ions

by azadirachta indica leaf extract Capricious zeylanica leaf broth [30], Ocimum sanctum leaf broth [30,31], Syzygium aromaticum (cloves) aqueous extract [32] and vitis vinifera leaf broth [33] as well as various other plant extract used as reducing and capping agent. Fig. 1 shows the possible constituents of plant extract responsible for the bio reduction of metal ions and their development and stabilisation [34,35].

properties of nanoparticles affected by the source of the plant extract like bark, fruits etc [36] where as each plant extract consist unique property and combining of organic reducing factors [37]. purity of CuNPs in an aqueous phase still challenge for the researcher. Then also, it is of interest to obtain monodispersed CuNPs by a simple and green route, very less known about the size dependent performance of CuNPs as a suitable catalyst. Here, we have developed a rapid, eco-friendly and convenient green route for the synthesis of CuNPs from copper chloride using leaf broth of Indian medicinal plant namely A. indica (Neem). It belongs to Meliaceae family and found
abundantly in India and in nearby subcontinents. The study also highlights the synthesis of CuNPs with various experimental parameters and corresponding morphology changes of CuNPs. A textile azo dye, here we target the Methyl Orange (MO) which is chosen as main compound when we studies about this content to employ MO as a model primarily because it is a widely used dye and resistant to degradation by conventional methods [38,39]. As far as we are concerned, nano copper based catalyst/PDS and catalyst/PMS method for degradation of moleculer orbital was still exposed in the environmental field of area. Consequently the main objectives of this prose are -

(i) for the synthesis the stable CuNPs by green synthesis and less costly method will be of great attraction for countering waterborne disease and national wellness.
ii) by the degradation of moleculer orbital, we can determine the effect of the various concentration of CuNPs, PDS, PMS, optimum tempreture and PH.
iv) The operation deviation of CuNPs/PDS and CuNPs/PMS system was compare systamatically .

2. Experimental method :-

2.1. Chemicals and materials

We have to use in this type of green experimental method because its not harmfull for environment. Copper chloride dihydrate ($CuCl_2 \cdot 2H_2O$) (E. Merck), Potassium Peroxodisulfate ($K_2S_2O_8$) (Sigma-Aldrich), Peroxomonosulfate ($2KHSO_5 \cdot KHSO_4 \cdot K_2SO_4$ 95%) , Methyl Orange (MO) and other reagents were of analytical grade. Neem (Azadirachta Indica) leaves were collected kaimur (bhabhua) bihar (India), kaimur (district) situated in 25.054 degree north and 83.677 degree east in state of bihar. We collected fresh 20 g leaves of neem with 100 mL H_2O stirred on a magnetic stirrer at 80 °C for 20 min. after preparation of extract filtered twice through Whatman paper and stored at 4 °C temperature further experiments. We have to use disttiled water or Deionized water for experiment.

2.2 Instrumentation :-

Identifying optical properties of the CuNPs were noted periodically to support completion of bioreduction of the CuCl2 solution, which is follow by UV–vis spectra on a double beam spectro photometer (3000+ LABINDIA) having resolution 1 nm. And The morphology of the CuNPs was determined using Scanning Electron Microscopy (SEM, Nova Nano FE-SEM 450, FEI), US and Transmission Electron Microscopy (TEM, Tecnai G2 20 (FEI) S-Twin, US) operative at 200 kV. The activity of the sample for SEM analysis dispersed NPs were centrifugate and ultrasonically for 40 min then 30 μL aliquots were extracted and deposited on the stub and for TEM analysis ultra-sonically spread mixture mounted on standardized carbon-black-coated Cu control grid and then air-dry under IR lamp. The crystalline structure of nanoparticles was characterized by X-ray Diffractometer (XRD, X'PERT PRO P analytical, Netherlands) with Cu Kα radiation (λ = 0.1540 nm) in the 2θ scanning range from 10° to 89° and a scanning rate of 2°/min. Fourier Transformation Infra-Red (FTIR, ALPHA-T Bruker, Germany) was used to detect of functional groups of biomolecules in neem leaf broth was identified by spectrometer using KBr pellet technique and

transmittance mode operating at a resolution of ± 4 cm−1 . Analysis of oxidative degradative products of MO was detected by Liquid Chromatography-Mass Spectrometry (LC–MS, XEVO G2-XS QTOF, Waters India Ltd.) and the mass spectrometer was operated with a quaternary pump, Quadropole detector.

2.3 Synthesis of Cu NPS :-

In order to prepration of aqueous solution of 7.5×10^{-3} mol dm^{-3} copper chloride ($CuCl2 \cdot 2H2O$) was taken in round bottom flask after that heated at 85 °C in the oil bath with magnetized stirring and by droper drop by drop added 20% leaf broth in this solution. Slowly slowly after some time the color of the reaction mixture was step by step changed from green, yellow, orange, radish brown, finally brown to dark brown with the number of mediate stages. After that we wait for resultant dispersal was centrifugate for 15 min and receive supernated was placed at 4 °C temperature. And we can also discribe this reduction process of copper salt into CuNPs by Azadirachta indica leaf broth at 85 °C is given by (Eq. (1)).

When Stirring at 85 degree centigrade then we get

$Cu^{+2}_{(aqu)}$ + Neem leaf broth ----------→ [Cu/Neem leaf broth]$^{+2}$

then Stirring for 28h at 85 degree centigrade then

----------→ [Cu/neem leaf broth] (1)

2.4. Kinetic measurements

desirable concentration of Moleculer solution and other reactants placed in stoppered type Erlenmeyer flask arround off 30 °C temperature and degraded of MO was initiated with adding a known dilution of PDS or PMS mixture. The proportion of decolonisation was get in position of alteration in intensity level at the dimension peak 465.1 nm wavelength in period of time interval studied by UV–vis Photometer attached with Peltier accessory (Temperature- controlled). All the kinetics runs were followed up to 90% completion of the reaction. A plot of log(C/C0) versus time was found liner which indicates pseudo first order kinetics. And plot of pseudo first order rate constant (k_{obs}) were deliberate from the slope of these plots. The rate constants values k_{obs} were consistent within ± 5 %.

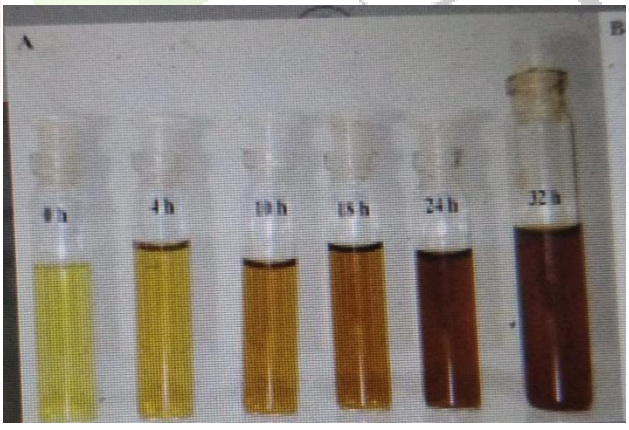


Fig.2 (a) the time evolution of the dispersion photographs

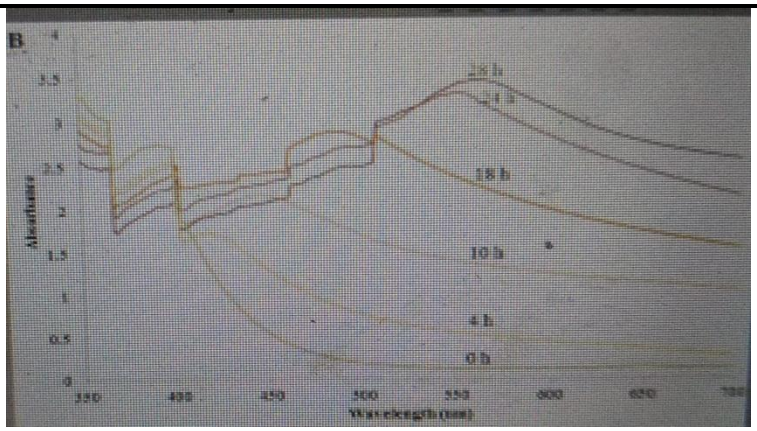


Fig 2 (b) UV-vis spectra during the synthesis process.

3. Outcome and discussion

3.1 characterization of copper nanoparticles

Using this one step synthesis process for the formation of CuNPs was confirmed by the color dispersion turned from light green to brownish and plot show in UV–vis spectroscopy in(Fig. 2). spectra of uv- visible were note down after different time intervals from the initiation of reaction and the intensity of SPR peak increased as the transit of time. the continued reduction of copper ions into CuNPs and then SPR band identifying of CuNPs was detected about 560 nm (Fig. 2b). we also read the morphology of CuNPs was confirmed by TEM and its indicate cubic shape with the size arround 48 nm (Fig. 3). when we discuss on XRD spectra which showed a sharp peak at $2\theta = 43.5°$, 49.5° and 74.01° orderly related to (111), (200) and (220), which indicate the
the face-centered cubic structure of copper (Fig. 4). after recored the result data we seen that particle size decrease from 73.51 nm to 48 nm with increase in the leaf of extract solution from 6% to 20% further increases in percentage then the concentration was agglomerate. surface of particles, at the high percentage of leaf broth excess number of nuclei generated and interacts with each other so secondary reduction process occurs on the surface of the performed nuclei then particles were agglomerated. effect of temperature on the reduction of copper ion is another important parameter for the synthesis of nanoparticles. Its also observe the below 65 °C of reaction temperature the reduction of copper salt was not completed. The reduction rate considerably increases by increasing reaction temperature from 65 to 85 °C but at the higher temperature (90 °C) the synthesis rate is too high to control particle size so NPs formed with higher average size were collective. The effect of initial concentration of $CuCl_2$ illustrates with Fig. S1. Thus the optimal conditions are 7.5×10^{-3} mol dm^{-3} concentration of copper chloride ($CuCl_2 \cdot 2H_2O$), 20% of leaf broth and 85 °C temperature for synthesis of NPs. More over CuNPs synthesis with antimicrobial activity also known by use of Azadirachta indica [41]. and the FTIR spectra of bio reduced synthesized CuNPs exhibits peaks at 2922.46 cm−1 (O-H stretching of phenolic group), 2372.07 cm−1 (C-N stretching of aromatic amine), 1631.99 cm−1 (C-O stretching), 1457.26 cm−1 (C-C stretching), 1384.50 cm−1 (aldehydic C-H stretching) suggests the presence of flavonoids and terpenoids, that may be responsible for reduction as well as stabilization process [29,42] (Fig. S2). The stability of synthesized CuNPs was also determined by zeta potential value −16 mV suggests that synthesized CuNPs are highly stable.

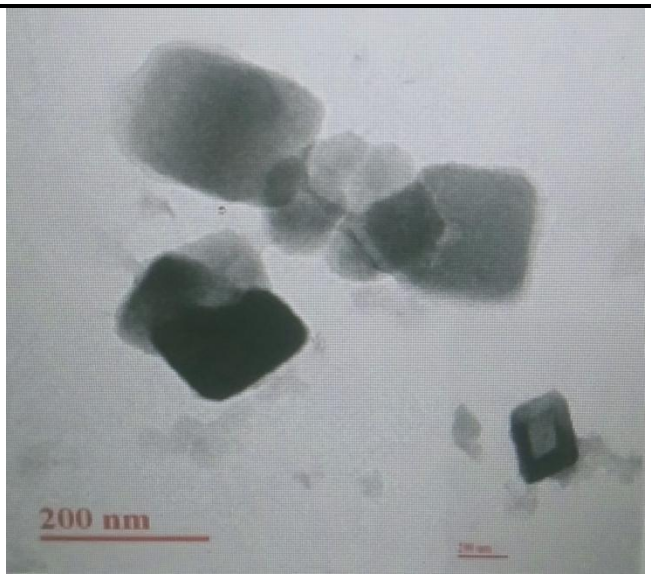


Fig3. TEM image of synthesized copper nanoparticles.

3.2. **Effect of experimental conditions**

3.2.1. Dye dependence

the initial concentration of MO was varying from 1.0×10^{-5} to 1.0×10^{-4} mol dm^{-3} at 30 °C temperature and other reactant concentrations were constant. Oxidation rate was found to increase with increasing concentration of MO in both CuNPs/PDS and CuNPs/PMS system. This is may be due to the increase in the concentration of dye, the reaction rate was increased as more molecules of dye was present for degradation. But after the certain concentration of dye 5×10^{-5} mol dm^{-3} , the oxidation rate was decreased. This can be described that at constant oxidant concentration the availability of SO_4^{-} radicals is less, so degradation of MO slowed down sig-nificantly.

3.2.2**. Peroxodisulfate and peroxomonosulfate dependence**

The oxidants such as persulfate and peroxomonosulfate are generally used in the sulfate radical based advanced oxidation process and having the standard redox potential of E° = 2.1 V, 1.82 V respectively. The degradation experiment was performed in the presence of CuNPs at different concentration (1.0×10^{-4} –1.0×10^{-3} mol dm^{-3}) of PDS and PMS respectively. Both oxidants are dominated by SO_4%− radicals (SRs) based mechanism and radicals were generated by the catalytic activation of PDS Eq. (2)) and PMS Eq. (3)

$$2S_2O_8^{2-} + Cu^0 \text{ -----} \rightarrow 2S04 - + 2SO_4^{2-} + Cu(II) \quad (2)$$

$$2HSO_5^{-} + Cu^0 \text{ ------} \rightarrow Cu\ (II) + 2SO_4^{-} + 2OH- \quad (3)$$

Once the SRs are formed it can produce a rapid attack on MO molecules and convert into end products. The rate of degradation initially increases with increase in the concentration of both oxidant.

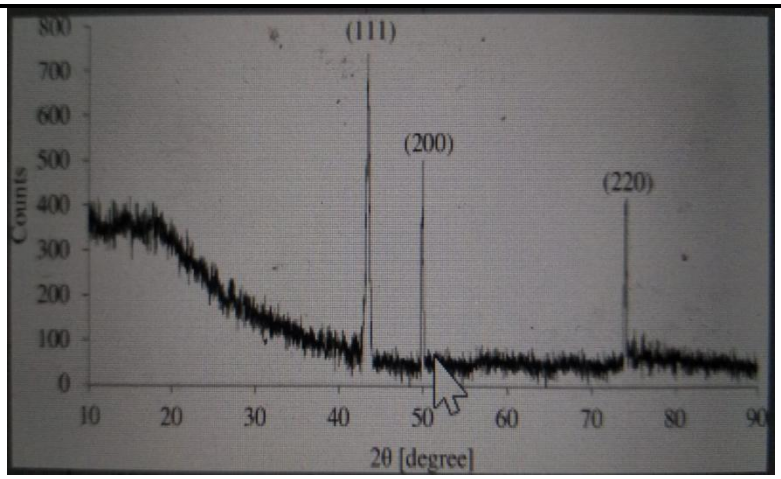
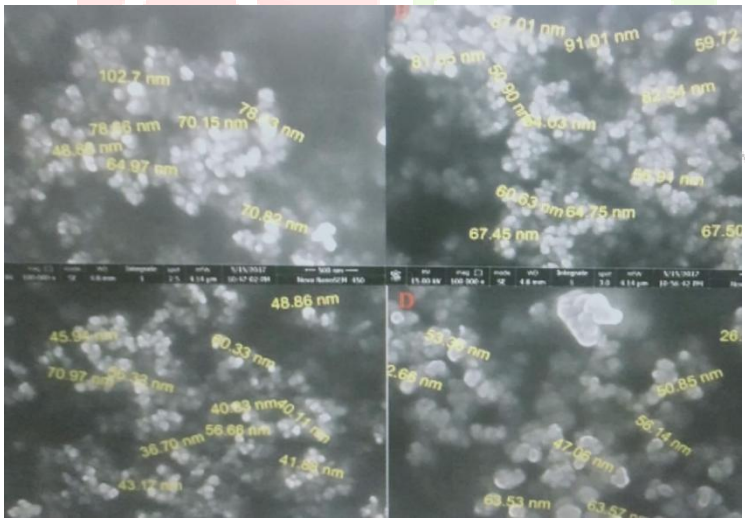


Fig4 : -A sharp peak of XRD spectrum of bio synthesis Cu NPs

3.2.3. Effect of initial pH

In order to find the optimal pH for dye degradation, a series of experiments were conducted at different pH (2.5–10) in CuNPs/PDS and CuNPs/PMS system respectively. As pH increased from 2.5 to 6.5 the degradation rate dramatically increased further increase in pH shows the degradation rate dropped in both systems. This may be attributed to the zeta potential and surface charges of the catalyst. When CuNPs was dispersed in water than the surface become cationic nature, which would more coverage of hydroxyl groups from water [46] so uncharged surface hydroxyl groups of CuNPs were the main active sites for generate sulfate radicals (SO4%). Thus as pH increases, the degradation rate is also increased and reached the maximum at the pH6.5. After that, the catalyst surface become anionic and higher electronic force to repel the SO_4^- anion so less SO_4^- could reach the catalyst surface and rate of degradation decreased correspondingly at higher PH [fig 5].

Fig. 5- copper Nanoparticles.



3.2.4. Copper nanoparticles and temperature dependence

The catalytic activity of CuNPs was evaluated degradation of MO in both PDS and PMS system at various concentration 0.25 × 10−7 –2.0 × 10−7 mol $^{dm-3}$ at three temperature viz. 25 °C, 30 °C, and 35 °C respectively. In order to show the catalytic activity, a graph is plotted between the concentration of CuNPs and rate constant obtained at different three temperatures . The plot gives straight lines indicating the direct dependence of reaction rate on CuNPs concentration. This may be attributed to the fact that as the concentration of CuNPs is increased; the number of active radical species is also increased, which in turn increases the rate of oxidation. The synthesized CuNPs exhibited good catalytic activity in presence of small

concentration (1.0 × 10−7 mol dm−3). As the temperature increases the rate of generation of oxidizing species such as SO^4 − radicals and higher valent Copper species also increased, so the rate of degradation of MO was accelerated by the rise in temperature. The energy of activation was calculated from the plot of log (kobs) versus 1/T in both systems. The value of activation energy (13.19 kJ $^{mol-1}$) in CuNPs/ PMS system shows tha**n** CuNPs/PDS (21.64 KJ mol^{-1}).

4. Conclusion:-

From the above experimental results, we conclude that different size of CuNPs produced through bio-reduction of copper salt was strongly dependent on the process parameters like neem leaf broth concentration, the concentration of copper salt and synthesis temperature. It was confirmed by the different instrumental techniques that there was a full conversion of copperchloride to CuNPs and bioorganic components from neem leaf broth act as a stabilizer for the CuNPs. These synthesized CuNPs were used for activation of peroxosulfates for degradation of hazardous dye in a cost-effective manner. Cu° was the source of Cu+2, which was verified to be the efficient activating agent for peroxosulfates to produce sulfate radicals. Kinetic results reveal that CuNPs/Peroxosulfates system could induce a $0.81 \times 10^{-4} – 40.7 \times 10^{-4}$ s^{-1} folds increase in the degradation rate of MO at different concentration of CuNPs, peroxosulfate concentration, and initial pH. Moreover, the reactivity discrepancy of PDS and PMS followed the order of CuNPs/PMS > CuNPs/PDS in degradation of MO under similar conditions.

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