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# VISIBLE LIGHT ASSISTED PHOTOCATALYTIC DEGRADATION OF CARCINOGENIC CONGO RED DYE USING GREEN SYNTHESIZED YTTRIUM AND ZINC DOPED NICKEL OXIDE NANOPARTICLES FROM CORIANDER LEAF EXTRACT

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**ABSTRACT**

The growth of effective green chemistry approaches for the production of metal nanoparticles has gained importance to discover an environmentally friendly method for the preparation of well-characterized nanoparticles. The NiO nanoparticles were synthesized by doping Yttrium and Zinc using Nickel (II) Chloride hexahydrate as a precursor using the *Coriandrum sativum* leaf extract as a reducing agent at room temperature under ordinary conditions. The synthesized nanoparticles were characterized using XRD, FT-IR, HRTEM, HRSEM, and EDAX studies. The degradation rate of Congo red dye was also studied with and without catalysts under visible light irradiation. From the kinetics study, the rate was found to be higher in the presence of 0.1 mol% yttrium doped NiO nanoparticles when compared to other photocatalysts proving its catalytic activity and found to follow first order.

**Keywords:** Green synthesis, Nickel oxide, Doping, Photocatalysis, Congo Red

RASĀYAN *J. Chem.*, Vol. 15, No.3, 2022

## INTRODUCTION

Nanoparticles have gained prodigious attentiveness because of their exceptionally smaller size and bigger surface-to-volume ratio, which leads to both physical and chemical differences in their different properties like mechanical properties, catalytic activity, electrical and thermal conductivity etc. Nanoparticles reveal shape and size-dependent properties which extended curiosity in various fields ranging from optics to catalysts and biosensing fields, computer transistors, chemical sensors, electrometers, antimicrobial activity, wireless electronic memory, and logic schemes.[1] Among the transition metal oxides, Nickel oxide (NiO) has grown significant focus in the past few years and it possesses different properties like mechanical, magnetic, optical, and electronic properties and has been commonly used as catalysts,[2,3] sensors,[4,5] detectors,[6] solar cells[7,8], and biological fields.[9,10] It has been extensively synthesized in different chemical methods and also through a green approach using different parts of plants.[11-14] Nickel oxide is a familiar p-type semiconductor having a wide band gap of around 3.5 eV which is widely used in the applications of photocatalytic applications. Even though other existing semiconductor materials comprise diverse photocatalytic abilities and possess a similar energy bandwidth range of NiO, it has been revealed that in terms of application NiO is superior to the other kinds of photocatalysts.[15,16] Green synthesis of nanoparticles aims at minimizing the waste which is generated and implementing sustainable processes. The green synthesis methods are an eco-friendly alternative to the chemical and physical methods of synthesis, which maintain low pH, low costs, and ambient temperature. In that list, *Coriandrum sativum* frequently recognized as coriander belongs to the Apiaceae family (Umbelliferae), has been widely cultivated over centuries in various temperate climates such as the Latin American, Asia, Middle East, and Africa are being widely used as a herbal medicine in the treatment of liver disease, cancer, diabetes, and hyperlipidemia. It has also been extensively used in the synthesis of several varieties of nanoparticles from the elements like gold, silver, copper etc.[17-19] Environmental pollution is one of the greatest harms faced by humans and other life forms because of industrialization, urbanization, and increasing population. Dyes are key pollutant that has been released from textile industrial effluent.[20-24] Dye molecules exist in the environment as many of them are not responsive towards acids, bases, oxygen, and light the color of the material turns out to be long-lasting. So the photocatalytic dye degradation is done by means of a UV lamp


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to produce energy for the formation of oxidizing radicals.[25-27] The free radicals will react with the dye molecules where the structure of the dye changes, color changes, and the dye starts degrading.[28-30] Congo red dye is a benzidine-based anionic diazo dye that is found to be deadly to numerous creatures and so-called as a mutagen and carcinogen. Synthetic dyes like Congo red are challenging to degrade because of the difficult aromatic structures that afford them thermal, optical, and physicochemical stability. Therefore, it is necessary to find new steps to eliminate such kinds of harmful environmental pollutants.[31-35] In this work, yttrium and zinc doped NiO nanoparticles were synthesized via the green synthesis method by using coriander leaf extract and then applied the same in photodegradation of carcinogenic Congo red dye.

## EXPERIMENTAL

### Material and Methods

$Ni(NO_3)_2.6H_2O$ (Nickel (II) nitrate hexahydrate), NaOH (Sodium hydroxide), $Zn(NO_3)_2.6H_2O$ (Zinc(II) nitrate hexahydrate), $HNO_3$ (Nitric acid), $Y_2O_3$ ( Yttrium oxide), ethanol were purchased from Sisco research laboratories. Congo red Dye was bought from SD fine chemicals. Deionized water was used completely throughout the synthesis.

### Preparation of Plant Extract

Fresh coriander leaves were chopped into pieces which were washed and dried. 50 g of chopped coriander leaves were added to the beaker with 500 mL water. The leaves were boiled at 80 °C and stirred magnetically. The reaction was kept in heating for half an hour, further, the extract was filtered and collected. The filtered extract was stored under cool conditions and was used for the synthesis of NiO nanoparticles.

### Synthesis of Yttrium (0.1 mol%) and Zinc (0.1 mol%) doped NiO Nanoparticles

$Ni(NO_3)_2.6H_2O$ and $Zn(NO_3)_2.6H_2O$ was added with 40 mL water and stirred to get a homogeneous solution. 50 mL of plant extract was added slowly with vigorous stirring and heated at 30 °C for half an hour. NaOH solution was added to the reaction mixture dropwise till the pH reaches 8. The stirring was continued for another 1 hour at room temperature and the solution was kept undisturbed for one day. The reaction mixture was then centrifuged and washed with water and ethanol. The attained black precipitate was calcinated at 400 °C for 3 hours. The same procedure was followed to 0.1% yttrium doped NiO nanoparticles, in which yttrium oxide was initially dissolved in hot nitric acid and added with nickel nitrate solution followed by the further above-mentioned steps. Pure NiO nanoparticles were also synthesized for comparison without the addition of dopants.

### Characterization of Yttrium and Zinc doped NiO Nanoparticles

The synthesized Yttrium and Zinc doped NiO nanoparticles were characterized by different analytical techniques. X-ray diffraction (XRD) was studied using BRUKER USA D8 Advance, Davinci diffractometer using Cu-Kα radiation source from the 2θ range 10-80°. The elemental analysis and surface morphology was investigated using Scanning Electron Microscope SU1510, acceleration voltage of 30 kV attached with X-ray energy dispersive spectroscopy (EDAX). The Fourier Transform-Infrared Spectroscopy (FT-IR) studies were recorded in BRUKER Optical GmbH Spectrometer model TENSOR 27 from the spectral range 500 – 4000 $cm^{-1}$. The structure and inner morphology were examined using High- resolution transmission electron microscopy (HRTEM) JEOL Japan, JEM-2100 Plus instrument.

### Procedure for Photodegradation of Congo Red Dye

The visible-light-induced photocatalytic performances of Pure NiO nanoparticles, 0.1% Y doped and 0.1% Zn doped NiO nanoparticles were tested in an XPA-II photochemical reactor using the 300 W Xe lamp as visible light irradiation in Congo red dye degradation. To achieve adsorption equilibrium, 50 mg of pure NiO catalyst was dispersed in a quartz reactor containing 50 mL Congo Red Dye (20 ppm) and is allowed to stir in dark for 30 minutes to gain equilibrium adsorption. The reactor was further irradiated with visible light to begin the photocatalytic degradation process. At 30-minute intervals, 5 mL aliquots were collected from the reactor using filter membranes. The obtained samples were analyzed using a UV-VIS spectrophotometer (SHIMADZU, UV 2100) from the wavelength range between 200-600 nm. The same procedure was carried to all the other photo catalysts and for comparison, a control reaction without a catalyst was performed.

## RESULTS AND DISCUSSION

### Characterization of Synthesized Nanoparticles

The XRD spectrum of the obtained NiO nanoparticles is shown in Fig.-1(a, b, and c). The XRD peaks were matched with the JCPDS number and its crystal structure was predicted. The crystallite size of the synthesized nanoparticles was predicted using the Debye- Scherrer equation.

$$d = \frac{0.9\lambda}{\beta \cos\theta}$$

Where, *d* is the crystallite size, λ corresponds to the wavelength of Cu Kα radiation (0.1541 nm), β attributes to the full width at half maximum (FWHM) of the diffraction peak, and θ corresponds to diffraction angle. From the spectra, the diffracted peaks at 2θ were obtained at 37.28°, 43.30°, 62.89°, 75.41°, 79.34°, 95.06° indexed to (101), (012), (110), (113), (202), (024) planes of pure NiO (Fig.-1a), 37.19°, 43.18°, 62.79°, 75.17°, 79.24°, 95.04° indexed (101), (012), (110), (113), (202), (024) planes of 0.1% yttrium doped NiO nanoparticles (Fig.-1b) and 31.73°, 43.39°, 56.32°, 75.17° indexed to (101), (012), (110), (113) planes of 0.1% zinc doped NiO nanoparticles (Fig.-1c) correspond to the characteristic Rhombohedral structure (JCPDS No. 00-044-1159). The calculated crystallite size was about 40 nm, 22 nm, and 36 nm for pure NiO, 0.1% Yttrium, and Zinc doped NiO nanoparticles respectively.

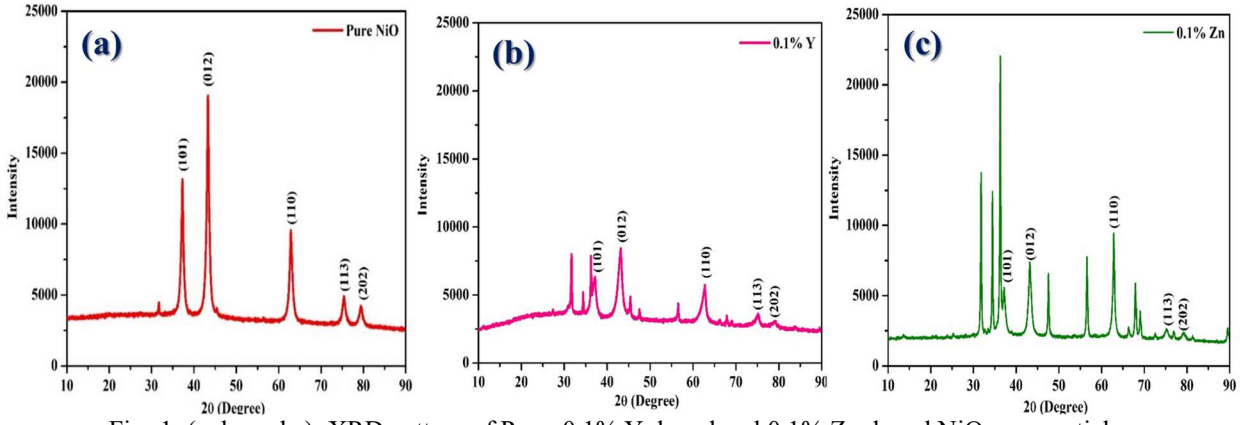



Fig.-1: (a, b, and c): XRD pattern of Pure, 0.1% Y doped and 0.1% Zn doped NiO nanoparticles

The FT-IR pattern is represented in the Fig.-2(a, b, and c). The peak at 3394 $cm^{-1}$ may be due to the -OH stretching as the calcinated powder can tend to absorb water. Similarly, the absorption peaks at 1637 $cm^{-1}$, 2360 $cm^{-1}$, and 1100 $cm^{-1}$ correspond to the bending vibration of -OH, $CO_2$, and linkages of phenol, acids, and flavonoids respectively. The peak around 670 $cm^{-1}$ attributes to the Ni-O bond vibration.

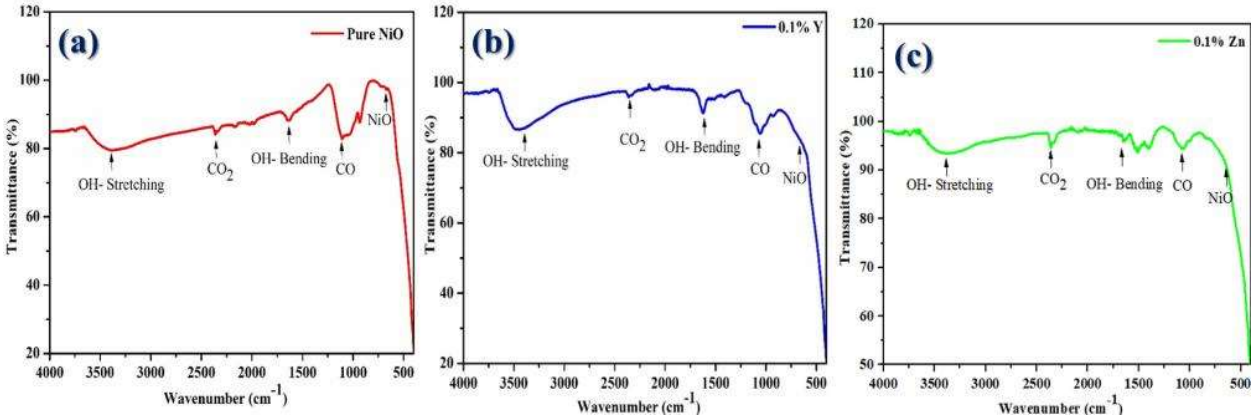



Fig.-2: (a, b, and c): FT-IR Spectra of Pure, 0.1% Y doped and 0.1% Zn doped NiO nanoparticles

The HRSEM image of synthesized NiO nanoparticles is shown in Fig.-3(a, b and c). The existence of bigger nanoparticles is due to the fact that particles have the affinity to form clusters and agglomerate because of

their high surface tension and surface energy of the resulted nanoparticles. The results indicate that the synthesized nanoparticles in each case were Rhombohedral in shape which agrees with the XRD results. Also, it can be clearly seen that there is a difference in morphology of pure NiO structure (Fig.-3a) compared to the doped NiO nanoparticles as we could witness the flakes of dopant on the rhombohedral structure of Yttrium and Zinc doped NiO nanoparticles (Fig.-3b and c).

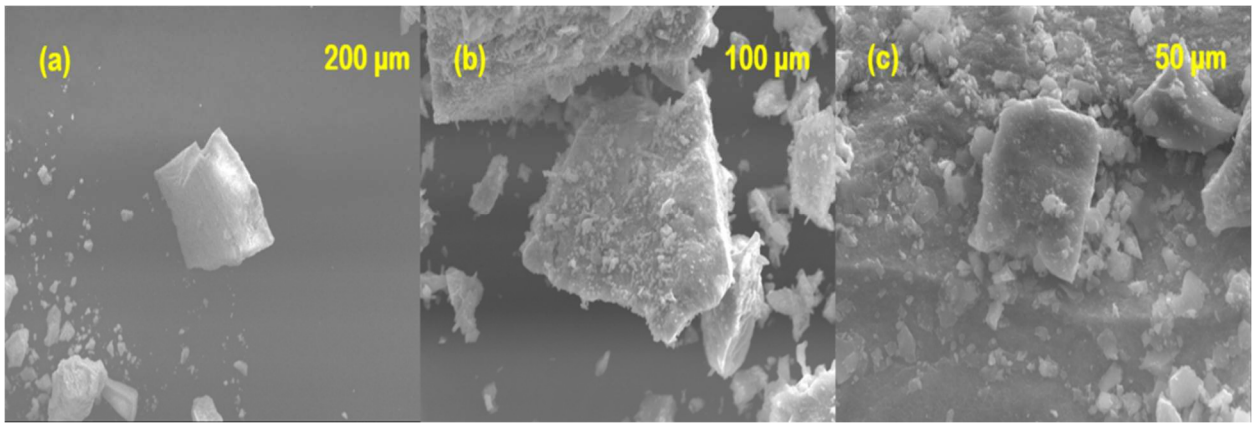
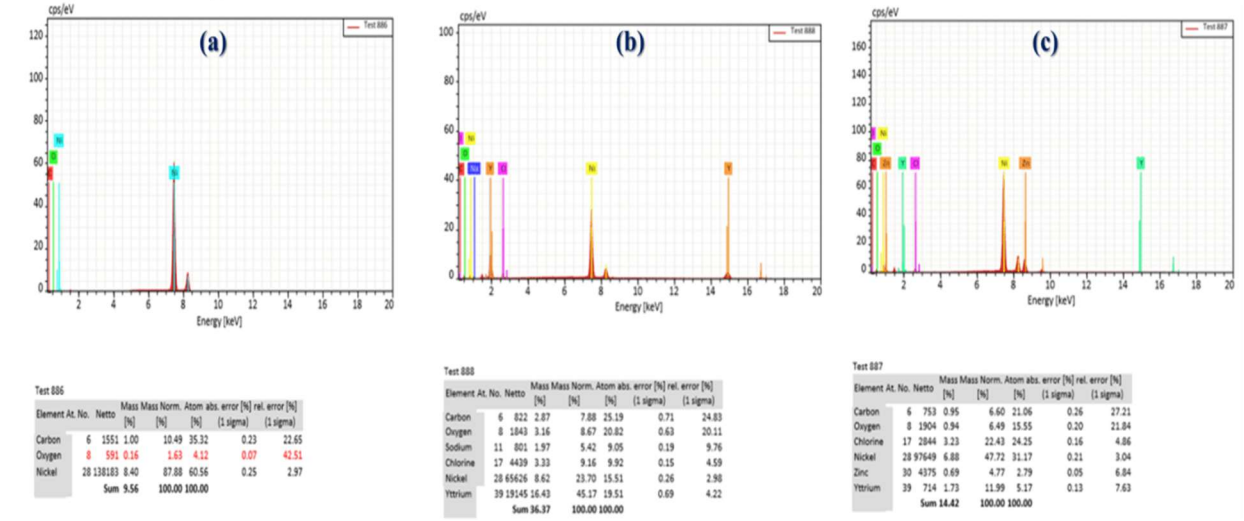



Fig.-3: (a, b and c): HRSEM images of Pure, 0.1% Y doped and 0.1% Zn doped NiO nanoparticles

The Energy Dispersive X-Ray Analysis (EDAX) was analyzed which is given in Fig.-4(a, b & c). From the EDAX spectra, it could be confirmed that the nanoparticles were found to be pure without any impurities and indicated the presence of the elements in them.




Test 886

| Element | At. No. | Netto | Mass [%] | Mass Norm. [%] | Atom abs. error [%] (1 sigma) | rel. error [%] (1 sigma) |
|---|---|---|---|---|---|---|
| Carbon | 6 | 1551 | 1.00 | 10.49 | 35.32 | 0.23 | 22.65 |
| Oxygen | 8 | 591 | 0.16 | 1.63 | 4.12 | 0.07 | 42.51 |
| Nickel | 28 | 138183 | 8.40 | 87.88 | 60.56 | 0.25 | 2.97 |
| | | Sum | 9.56 | 100.00 | 100.00 | | |

Test 888

| Element | At. No. | Netto | Mass [%] | Mass Norm. [%] | Atom abs. error [%] (1 sigma) | rel. error [%] (1 sigma) |
|---|---|---|---|---|---|---|
| Carbon | 6 | 822 | 2.87 | 7.88 | 25.19 | 0.71 | 24.83 |
| Oxygen | 8 | 1843 | 3.16 | 8.67 | 20.82 | 0.63 | 20.11 |
| Sodium | 11 | 801 | 1.97 | 5.42 | 9.05 | 0.19 | 9.76 |
| Chlorine | 17 | 4439 | 3.33 | 9.16 | 9.92 | 0.15 | 4.59 |
| Nickel | 28 | 65626 | 8.62 | 23.70 | 15.51 | 0.26 | 2.98 |
| Yttrium | 39 | 19145 | 16.43 | 45.17 | 19.51 | 0.69 | 4.22 |
| | | Sum | 36.37 | 100.00 | 100.00 | | |

Test 887

| Element | At. No. | Netto | Mass [%] | Mass Norm. [%] | Atom abs. error [%] (1 sigma) | rel. error [%] (1 sigma) |
|---|---|---|---|---|---|---|
| Carbon | 6 | 753 | 0.95 | 6.60 | 21.06 | 0.26 | 27.21 |
| Oxygen | 8 | 1904 | 0.94 | 6.49 | 15.55 | 0.20 | 21.84 |
| Chlorine | 17 | 2844 | 3.23 | 22.43 | 24.25 | 0.16 | 4.86 |
| Nickel | 28 | 97649 | 6.88 | 47.72 | 31.17 | 0.21 | 3.04 |
| Zinc | 30 | 4375 | 0.69 | 4.77 | 2.79 | 0.05 | 6.84 |
| Yttrium | 39 | 714 | 1.73 | 11.99 | 5.17 | 0.13 | 7.63 |
| | | Sum | 14.42 | 100.00 | 100.00 | | |

Fig.-4: (a, b and c): EDAX spectra of Pure, 0.1% Y doped and 0.1% Zn doped NiO nanoparticles

The HRTEM images are represented in Fig.-5(a, b, and c) The Rhombohedral shape of the particle can be confirmed with smooth and uniform particle morphology which is in good agreement with the XRD and HRSEM data. Also, we could observe that there is a change in morphology of nanoparticles when compared to pure NiO (Fig.-5a) and that of 0.1% Y and 0.1% Zn doped NiO nanoparticles (Fig.-5b and c).

**Efficiency in Photodegradation of Congo Red Dye**

The photocatalytic behavior of Pure NiO nanoparticles, 0.1% Y doped NiO nanoparticles, and 0.1% Zn doped NiO nanoparticles were evaluated based on their ability to degrade Congo red dye using visible light irradiation as shown in Fig.-6(a, b, and c). In Fig.-6(a), the degradation of dye due to the addition of co-catalyst Pure NiO nanoparticles gives 73% degradation efficiency under visible light irradiation for 120 minutes. In Fig.-6 (b & c) the degradation efficiency for the photocatalysts 0.1% Y doped NiO nanoparticles and 0.1% Zn doped NiO nanoparticles was found to be 96% and 57% respectively, under the same conditions and time span of 120 minutes.

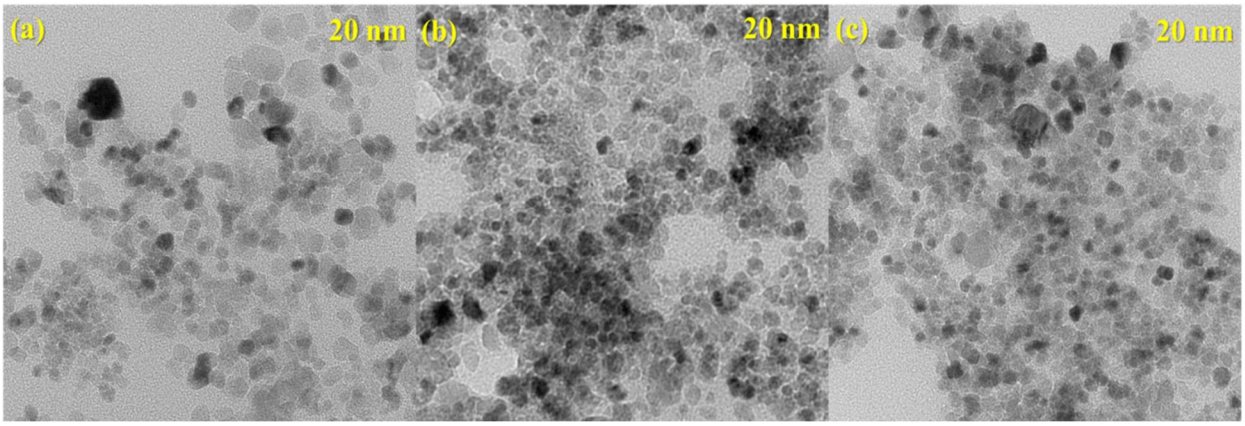



Fig.-5: (a, b, and c): HRTEM images of Pure, 0.1% Y doped and 0.1% Zn doped NiO nanoparticles

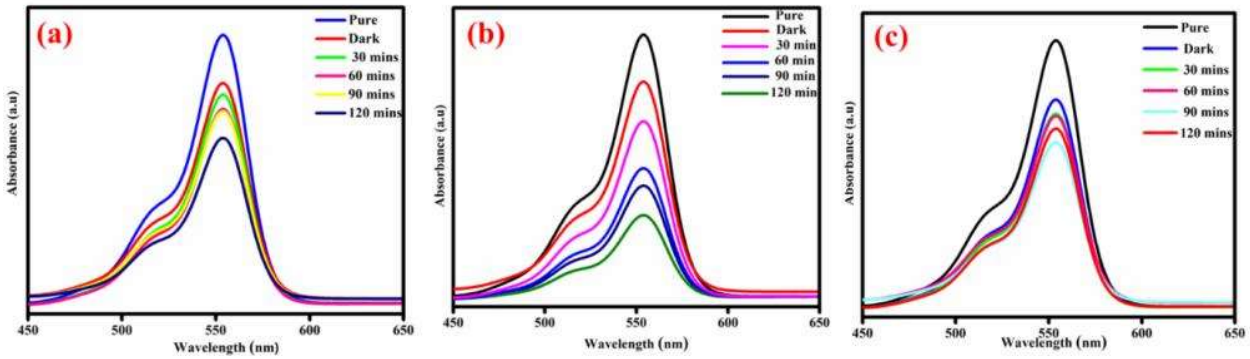



Fig.-6: (a, b, and c): Photocatalytic degradation of Congo red dye by Pure, 0.1% Y doped NiO, and 0.1% Zn doped NiO nanoparticles

**Kinetics of Dye Degradation**

The concentration of each sample for the specific absorbance was calculated as shown in Table-1. The rate constant was calculated using the formula,

$$k = 2.303/t \log (a/a - x)$$

Where k is the rate constant, t corresponds to time[min], a is the initial concentration of dye solution, and a-x corresponds to the actual concentration of dye solution at time t. Using the rate constant value calculated for each catalytic degradation, the rate of each catalytic degradation was calculated,

Rate = k * [concentration]

Table-1: Concentration of dye solutions at the respective time

| Time (Minutes) | Control 20 ppm (without catalyst) | Sample with Catalyst (Concentration ppm) | | |
|---|---|---|---|---|
| | | Pure NiO | 0.1% Y doped NiO | 0.1% Zn doped NiO |
| 0 | 20 | 20 | 20 | 20 |
| 30 | 19.812 | 17.185 | 15.981 | 17.251 |
| 60 | 19.516 | 14.951 | 14.122 | 16.512 |
| 90 | 19.411 | 12.128 | 12.301 | 15.771 |
| 120 | 19.028 | 11.122 | 7.312 | 15.613 |

The rate of degradation was found to be 1.106 $min^{-1}$, 1.195 $min^{-1}$, and 0.673 $min^{-1}$ for pure NiO, 0.1% Y doped NiO, and 0.1% Zn doped NiO respectively. These values thus show that 0.1% Y doped NiO nanoparticle was found to be the most efficient in Congo red dye degradation process compared to the other photocatalysts.

## CONCLUSION

0.1% Yttrium and 0.1% Zinc doped NiO nanoparticles were effectively synthesized via green synthesis from *Coriandrum sativum* leaf extract which is simple, cost-effective, and eco-friendly. From the various

analytical tools, the NiO nanoparticles formed were confirmed. The XRD spectra show that the nanoparticles are Rhombohedral in structure (JCPDS No. 00-044-1159). The size of the nanoparticles calculated was about 40 nm, 22 nm, and 36 nm for pure, 0.1% yttrium, and zinc doped NiO nanoparticles respectively. FT-IR spectra confirmed the characteristic Ni-O peak at 670 $cm^{-1}$. The HRSEM and HRTEM images clearly showed the rhombohedral shape of the synthesized nanoparticles which agrees well with the XRD results and also the change in morphology before and after doping was clearly understood. The EDAX spectra confirmed the presence of respective elements and show that the synthesized NiO nanoparticles were pure and free from other foreign materials. Photocatalytic degradation of Congo red dye was successfully applied and from the rate values calculated, it can be seen that 0.1% Yttrium doped NiO nanoparticles were found to be 96% efficient compared to other photocatalysts.

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[RJC-6745/2021]