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RESEARCH ARTICLE

Photocatalytic dye degradation using nickel ferrite spinel and its nanocomposite

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Abstract

Coloured wastewater is a major issue of today for human health and ecology. Among all available processes such as physical, chemical, biological and electrochemical methods, photocatalysis can be a promising solution because of its ability to degrade colour-causing compounds completely by converting them into simpler molecules (H_2O, CO_2) depending on dye structure. In this work, $NiFe_2O_4$ was synthesized by the co-precipitation method. Furthermore, the composites of $NiFe_2O_4$ with TiO_2 were synthesized by varying amounts of TiO_2. The spinel and composites were characterized by XRD, ZETA analysis and UV–DRS. Their photocatalytic activities were investigated using the photocatalytic degradation of reactive turquoise blue 21 (RB 21) dye as model pollutants under sunlight. The increased absorption of the visible light and the enhanced separation of the electron–hole pairs due to the relative energy band positions in $NiFe_2O_4$ and TiO_2 are considered as the main advantages. Our results showed that $NiFe_2O_4$-based nanocomposites could be used as an effective and magnetic retrievable photocatalyst.

Keywords Nanocomposites · Photocatalytic dye degradation · Spinel · Sunlight

Introduction

Wastewater is one of the biggest challenges to the scientific community. Because of increasing industrialization has questioned the quality of water. A very small part of water available on earth is useful for human uses (nearly 0.03%). Chemical and typical dyes and dying industries consume maximum water and produce a large amount of wastewater, which adversely affects aquatic life because of its colour-causing compounds, which hindered the sunlight penetration through the surface of water bodies (Ahmed et al. 2012; Osman 2014; Rajaram and Das 2008; Robinson et al. 2000; Seow et al. 2016; Singh 2015). The main constituents of these types of effluents are dyes. Dyes have mainly four parts namely skeleton, chromophore, auxochrome and soluble part. Dyes could be classified into two basic types: based on structure and based on application (Benkhaya et al. 2020; Forgacs et al. 2004; Jonstrup et al. 2011; Khehra et al. 2006; Kiernan 2001; Klaus 2008; Popli and Patel 2015; Sandhya et al. 2005).

Dyes from wastewater could be removed via different methods such as physical (Hethnawi et al. 2017; Katheresan et al. 2018; Yagub et al. 2014), biological (Chacko and Subramaniam 2011; Manavi et al. 2017; Mojsov et al. 2016; Srinivasan and Viraraghavan 2010), chemical (Forgacs et al. 2004; Gusain et al. 2019; Joshi et al. 2004; Nidheesh et al. 2018). Physical methods do not destroy the dyes but carry out the mass transfer from the liquid phase into the solid phase commonly, hence there are the chances of colour regain after some time, while in biological processes only limited dyes can be degraded and have a limited range of pH and temperature. Even though biological processes are used to a large extent for effluent treatment in ETP plants, too long operation time makes it unfavourable for dye degradation. And chemical processes are faster than biological processes, hence these processes can be a promising option for dyes removal. Typically, with AOPs complete

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mineralization and partial oxidation to inert and less concerning products such as H_2O, CO_2 and other simpler products (Anwer et al. 2019; Babuponnusami and Muthukumar 2014; Hodges et al. 2018). Among all AOPs, photocatalysis serves as a promising method as it makes use of sunlight and relatively cheaper process than other methods as separation of catalyst is easy (Anwer et al. 2019; Babuponnusami and Muthukumar 2014; Joseph et al. 2009; Paździor et al. 2018; Krishnan et al. 2017; Verma and Samanta 2018).

Semiconductors are widely used photocatalysts, such as TiO_2, ZnO, ZnS, Fe_3O_4, CdS, C_3N_4 and many others (Chen et al. 2020; Cui et al. 2018; Davar et al. 2015; Hu et al. 2020; Mahmoodi 2014; Saravanan et al. 2016; S. Wang et al. 2014; Ye et al. 2012). But among widely explored are TiO_2 and ZnO. But problems associated with semiconductors are wide bandgap, nonmagnetic nature. Because of these properties, semiconductors can be only used with UV radiation and are difficult to remove after completion of degradation, while this problem can be resolved by using spinel catalyst which has a narrow bandgap, and if ferrite spinels are used it shows better magnetic properties than semiconductors (Eiichi et al. 2003; Kirankumar and Sumathi 2020). While even spinels are not as effective as semiconductors even under UV radiation, combination can be a promising solution to the hurdles of both types of catalysts (Mamba and Mishra 2016). Composites of semiconductors and spinel have advantages of easy separation, utilize almost whole UV and visible spectrum of sunlight which is nearly 50% (combine) of sunlight, higher surface area and a large number of active sites (Gawande et al. 2015; Govan and Gun'ko 2014; Johnson 2017; Parsons et al. 2009; Sun et al. 2019; Wang and Astruc 2017; Zhang et al. 2010).

Jing et al.(Jing et al. 2016) have investigated photocatalytic performance of Ag/Ag_3VO_4 and 5% $CoFe_2O_4/Ag/Ag_3VO_4$ composite. The result has shown that 49.75% and 61.48% tetracycline was photo-degraded, respectively. This shows that incorporation of $CoFe_2O_4$ improves the activity, and even composite shows magnetic separability. They have confirmed the better performance for several runs. Photocatalytic activity of core–shell structured $Fe_3O_4/SiO_2/TiO_2$ nanocomposites synthesized using the sol–gel process was compared with SiO_2 TiO_2 by Ye et al. (Ye et al. 2010). This composite shows additional advantages of high chemical stability, fast magnetic separation and maintenance of the photocatalytic activity for at least eighteen cycles. Similarly, Wang et al. (Wang et al. 2012) synthesized $Fe3O_4/SiO_2/TiO_2$ NCs using the sol–gel method and investigated its photocatalytic activity on MB that was present in an aqueous solution at room temperature and pH = 10. Under UV irradiation, the NCs showed higher photodegradation of MB (78%) within 5 min. Here, in Table 1 some of the other composites are briefed. Other works on $CoCr_2O_4$ / TiO_2 for methyl orange degradation under UV light by Shojaei et al. (Shojaei et al. 2013), while $Ni_{0.65}Zn_{0.35}Fe_2O_4$ / r-GO has been investigated as photocatalyst by Javed et al. (Javed et al. 2019).

Table 1 COD degradation percentage for different catalysts

Sr. No	Catalyst	%COD Degradation
1	TiO_2	23.07
2	$NiFe_2O_4$	40.23
3	$NiFe_2O_4/TiO_2$ (90%-10%)	38.46
4	$NiFe_2O_4/TiO_2$ (50%-50%)	33.69
5	$NiFe_2O_4/TiO_2$ (10%-90%)	26.07

In this work, dye degradation performance of photocatalyst nickel ferrite and nickel ferrite/ titanium dioxide synthesized via co-precipitation method has been investigated. Materials from two different generations are combined to obtain photocatalyst with desired properties to sequester ultraviolet and visible ranges from whole solar spectrum reaching to the earth, better dye decolouration and mineralization along with effective separation at the end of the process. Some of the characterizations have been done to investigate the properties of prepared catalysts.

Materials and methods

Material

All chemicals used were analytical or laboratory-grade reagents which were supplied by Ranbaxy, Spectrochem, HPLC, NICK, CDH, MERCK. The detailed purity and phase of precursors and other reagents are as follows: citric acid (Ranbaxy, 99.9%), ethylene glycol (Spectrochem, 99.9%), sulfuric acid (HPLC, 98%), sodium hydroxide (NICK, 99.9%), potassium dichromate (HPLC), Ag_2SO_4 (NICK, 99.9%), ferroin indicator (HPLC), ferrous ammonium sulphate (HPLC, 99.9%), $HgSO_4$ (HPLC, 99.9%), Fe_2O_3 (MERCK, 99.5%), distilled water (MERCK), iron nitrate—$Fe(NO_3)_3 \bullet 9H_2O$ (HPLC, 99.0%), nickel nitrate—$Ni(NO_3)_2.6H_2O$ (HPLC, 99.0%), TiO_2 (HPLC, 80% anatase and 20% rutile), Na_2CO_3 (NICK, 99.9%).

Reactive turquoise blue 21 (RB21), a copper phthalocyanine reactive group with (molecular weight = 1282.97 g/mol), was generously supplied by Avni Dye Chem Industries, Ahmedabad (Fig. 1).

Preparation method

Preparation of $NiFe_2O_4$

The co-precipitation method was adopted for the synthesis of $NiFe_2O_4$ and $NiFe_2O_4$/ TiO_2. A stoichiometric amount of

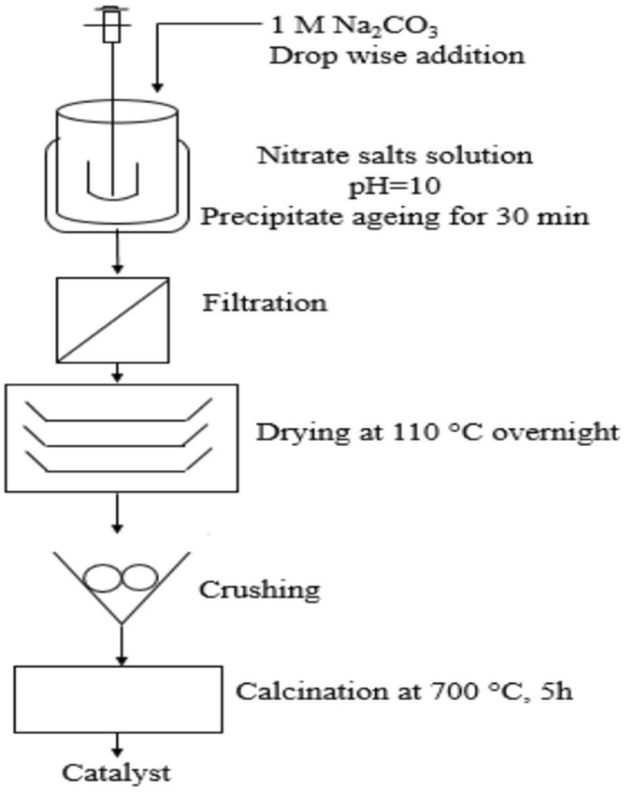

Fig. 1 Schematic diagram for spinel catalyst preparation by co-precipitation method

nickel nitrate (hexahydrate) and ferric nitrate (nonahydrate) are mixed. This mixture is stirred until it became a homogenous solution. Na_2CO_3 (sodium hydroxide, 1 M) was used as a precipitating agent and added dropwise until pH ≥ 10. Once the desired pH is obtained solution is kept for ageing for 1 h at 80 ℃. After ageing, filtration, several times washing of precipitates has been carried out and precipitates dried at 110 ℃ overnight and calcined at 700 ℃ for 5 h. Refer to Fig. 2.

Preparation of $NiFe_2O_4$ / TiO_2

A typical procedure for preparing nanocomposite $NiFe_2O_4$/TiO_2catalysts was prepared by adding nickel ferrite into suspension of TiO_2, TiO_2 was added in varying amounts for preparation of NF: TiO_2 = 90:10, 80:20, 50:50, 10:90. The suspension was stirred further for proper mixing. Heating was given to the solution then it was calcined in a muffle furnace under 700 °C (ramp = 10 °C/min) for 5 h to form $NiFe_2O_4$/TiO_2. Experimental set-up for the $NiFe_2O_4$/TiO_2 nanocomposite is reported in Fig. 3. Samples are named as follows: NFT90, NFT80, NFT50, NFT10, NF, T for NF:TiO_2 = 90:10, 80:20, 50:50, 10:90, and last two for bare nickel ferrite and TiO_2. Refer to Fig. 3.

Characterization

Some of the basic characterizations were carried out such as FTIR, zeta sizer analysis and UV–DRS analysis,

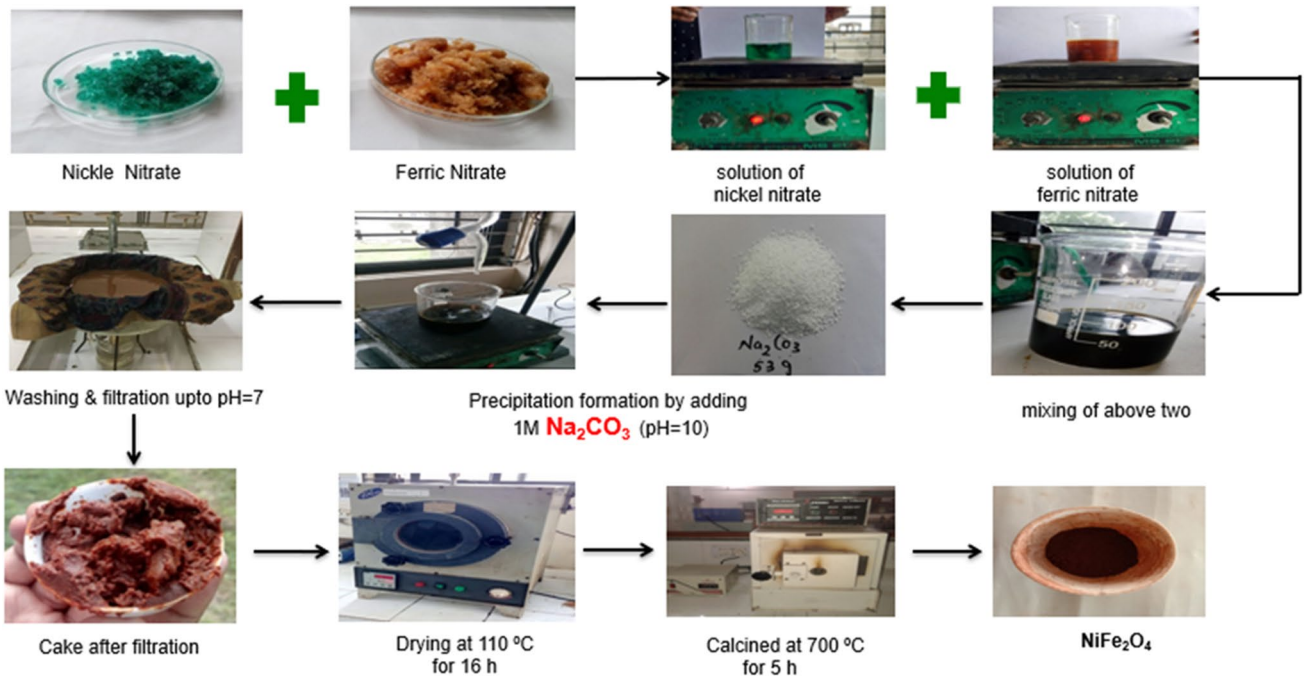

Fig. 2 Experimental set-up for $NiFe_2O_4$ spinel catalyst preparation by co-precipitation method

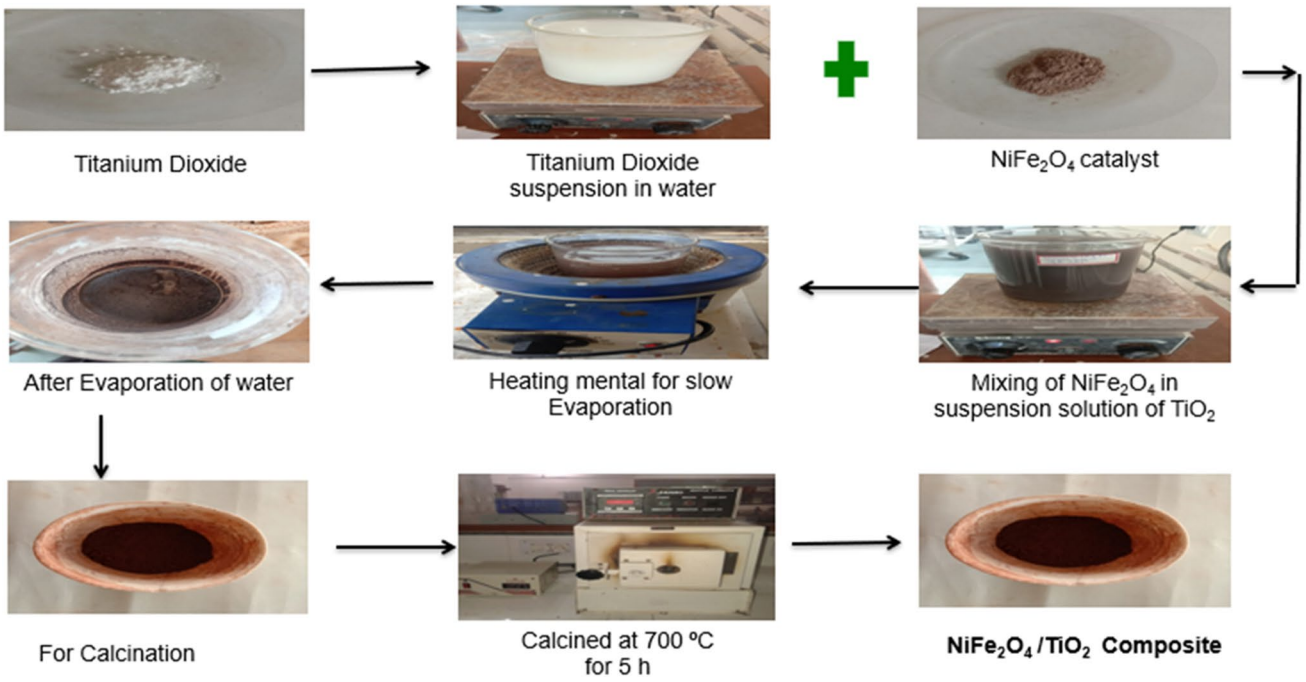

Fig. 3 Experimental set-up for $NiFe_2O_4$/ TiO_2 spinel-based nanocomposite

respectively, for phase check, practical size and for bandgap measurements Fourier transform infrared spectroscopy was carried out to check bond stretching and whether the prepared spinel is inverse type or normal. Zeta potential analysis was carried out to check the particle size of the prepared catalyst. It must be within the nanometric size range to give higher surface area and better catalytic activity. To find out the bandgap of catalyst, which is essential to be known for predicting whether the catalyst activation is possible under sunlight or not. As discussed above, it is observed that if

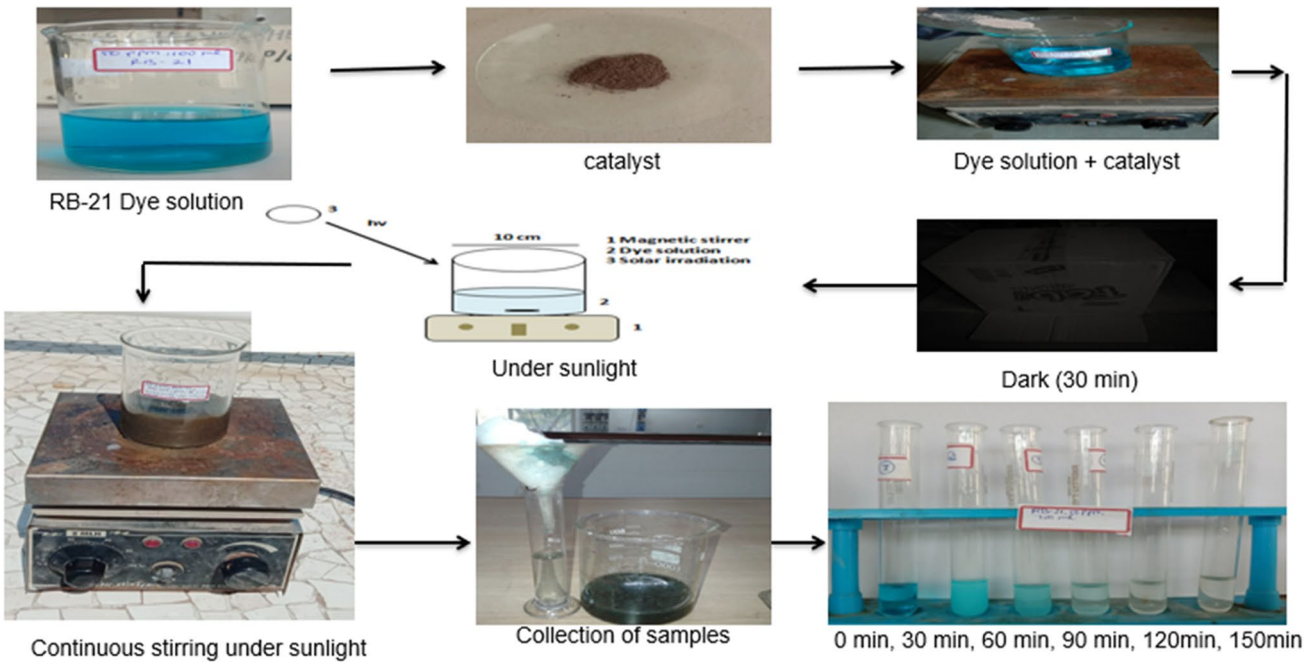

Fig. 4 Experimental set-up for degradation of dye under natural sunlight source

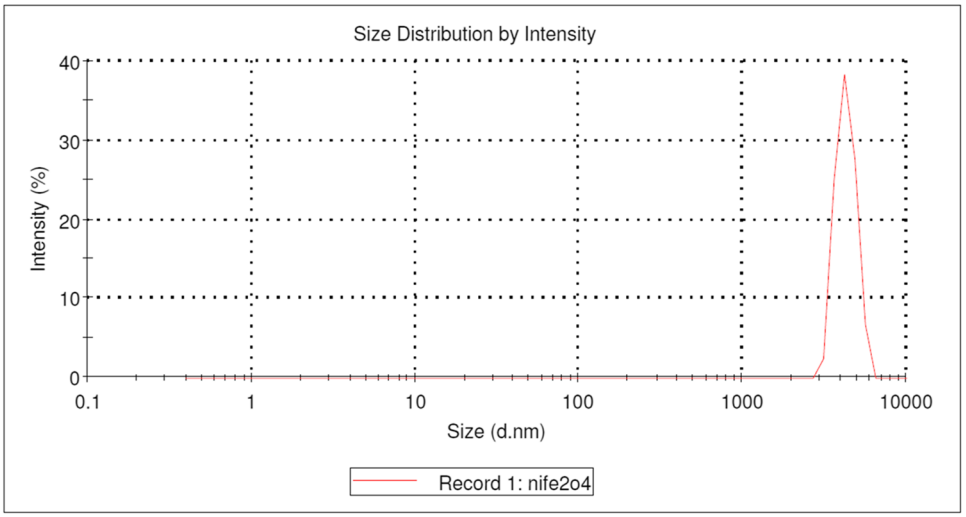

Fig. 5 Zeta sizer results for $NiFe_2O_4$

the catalyst bandgap is within 1.98 to 3.2 eV range it can be activated under sunlight.

Photocatalytic activity

Photocatalytic activity of spinel and nanocomposite were measured by degradation ofRB21dye under natural sunlight irradiation at neutral, acidic and basic pH values. A 150 ml of 40 mg/l RB21 dye aqueous solution and its corresponding dose of photocatalyst were added in a quartz glass container and stirred for 30 min in dark. After every 30 min up to 150 min irradiation time, 25 ml of the suspension sample was withdrawn and then solution and particles are separated by an extra magnate. The photocatalytic degradation process can be confirmed by UV–Vis spectrophotometer for colour removal analysis by checking absorbance at 645 nm and by COD degradation by APHA procedure—close reflux. Figure 4 shows the experimental set-up for photocatalytic dye degradation.

Results and discussion

Characterization

Zeta sizer results of catalyst show good agreement with JCPDS card no. 742081 for NF and JCPDS card #84–1286 as TiO_2 was used as prepared and NF was used as synthesized. As per analysis, particle sizes are in the range of 10^3 nm, refer to Fig. 4, while FTIR spectroscopy shows bond stretching of metal and oxygen for each site atoms (A and B both sites) for these results, refer to Figs. 5 and 6. The bandgap of the as-prepared composite is assumed to be in between bandgap of spinel and metal oxide and from the literature it is 2.69 eV for composites which may provide better efficiency under sunlight and it is lower than that of TiO_2 (3.2 eV) (Baig et al. 2020). As-prepared $NiFe_2O_4$ (NF) shows bandgap of 2.07 eV which is in agreement with what the other researchers have obtained, while composite shows bandgap of 2.927 eV, which is very

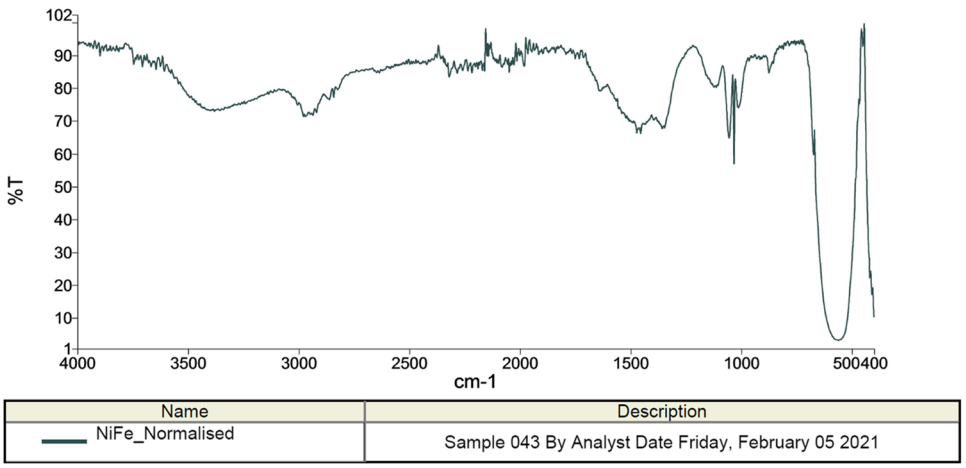

Fig. 6 FTIR of $NiFe_2O_4$

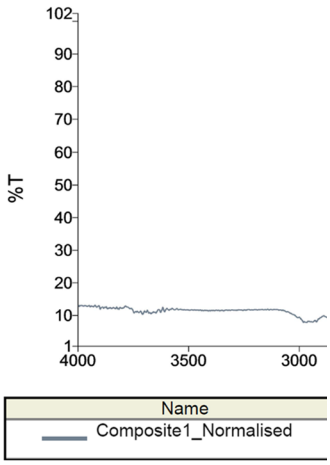

Fig. 7 FTIR of composite NFT90

close to bandgap of TiO_2 (Fig. 8). But not only bandgap is factor that affects the catalyst performance but other parameters such as separation ability and surface area are also important, here in the main objective to obtain better catalyst for dye gradation. Ease of separation of NF is also one of add-on benefit. And partially it was observed that even though bandgap of composite is quite higher than the NF but it shows better dye degradation compared to NF only that might be because of that TiO_2 can absorb in ultraviolet region, and spinel can absorb wavelengths from visible range of solar spectrum which to gather shares nearly half of the solar spectrum reaching to the surface. And even good magnetic properties of NF facilitate ease of separation of catalyst too. That is also true that some active sites are not available due to composite formation hence shows bit lesser dye degradation than theoretically assumed. FTIR analysis shows that pure phases are formed as no other vibrational spectra other than that related to Ni–O, Fe–O, O–O, Ni–Fe and Ti–O bonds could be observed. Major peaks were observed around 590 cm^{-1} and 400 cm^{-1} which shows stretching of metal oxygen bond those are present in spinel structure of NF sample, which is in agreement with other reported work as well (Li et al. 2014; Ojemaye et al. 2017) (Figs. 6 and 7).

Photocatalytic activity

Photocatalytic dye degradation was carried out under solar light. Refer to Table 1 for COD removal efficiency of composites and parent catalysts, i.e. NFTs and $NiFe_2O_4$ and TiO_2. As depicted in the table, it is clear that $NiFe_2O_4$ shows better COD removal but at the cost of colour removal efficiency. Referring to Fig. 9, it is clear that for colour removal TiO_2 performs far better than other catalysts but is accompanied by less COD removal. Hence, for optimum performance with the removal of both COD and colour composite NFT90 best fits the requirements.

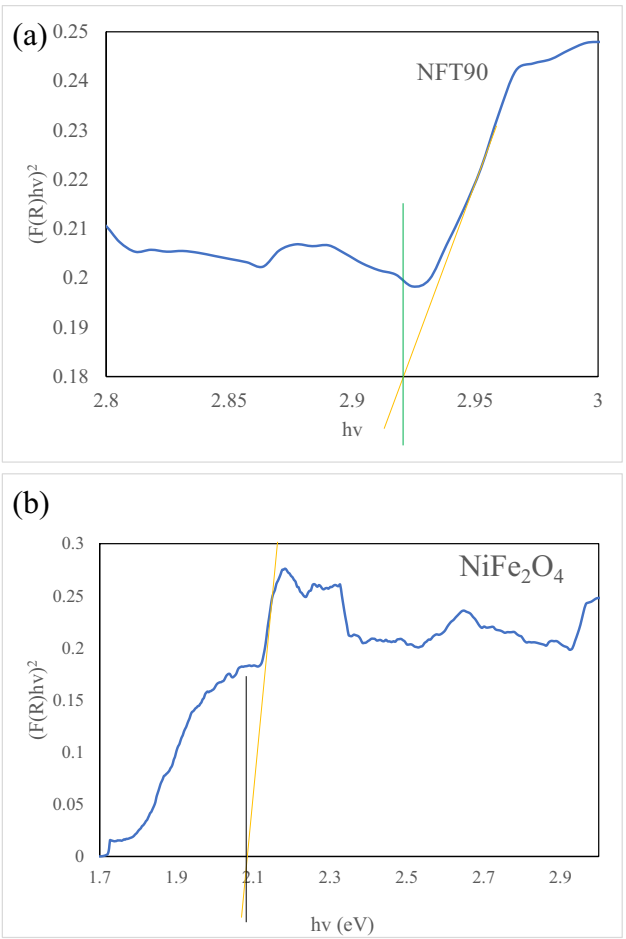

Fig. 8 UV–DRS spectra for nanocomposite—NFT90 (**a**) and NiFe2O4 (**b**) catalyst

Effect of various parameters is also investigated such

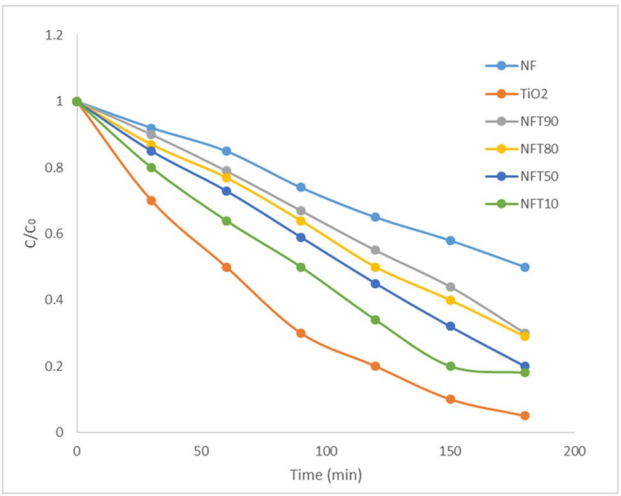

Fig. 9 Dye degradation efficiency of different catalysts (natural pH, room temperature, 40 ppm dye solution, 0.3 g catalyst dosage)

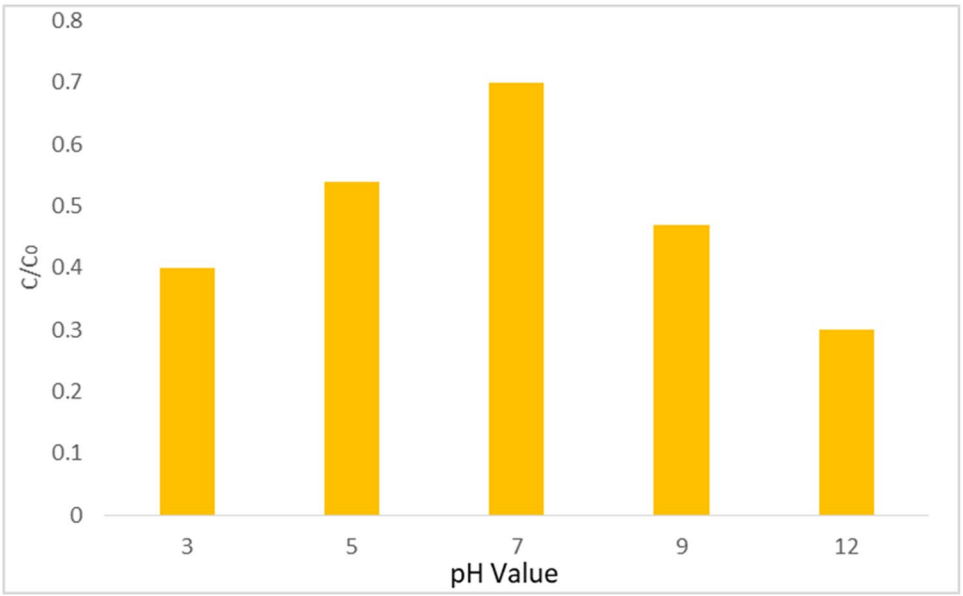

Fig. 10 Effect of pH on dye degradation efficiency of NFT90. (0.5 gm. catalyst dose, room temperature, 40 ppm initial dye concentration)

as pH, catalyst dosage, initial dye concentration and irradiation time. Refer to Figs. 10, 11 and 12 for the effect of pH, catalyst dosage and effect of initial dye concentration, respectively, for selected catalyst NFT90. Results show that at neutral pH (= 7) better performance was observed than other pH values (i.e. 3, 5, 9, 12). The main reason is the pK_a value of dye and catalyst, pK_a value for dye is 5.5 and the same for the catalyst is 8.5, which restrict the range of pH for better performance between 5.5 and 8.5. At lower pH better COD removal (nearly 30%) can be observed while at higher pH better colour removal (nearly complete removal of colour) is observed, because of repulsion of anionic dye molecules with negatively charged catalyst surface at higher pH and vice versa. Another parameter is catalyst dosage, generally, an increase in catalyst dosage improves the dye degradation performance. But observations suggest that as catalyst dosage increases above 0.5 g, solar light penetration is reduced because of a large amount of suspended solids particles. Higher dye degradation is observed for the lowest concentration of 10 ppm of dye. But all other parameters are checked for a dye concentration of 40 ppm. Further, as the time of irradiation increases, the dye degradation also increases but at a different rate for each catalyst.

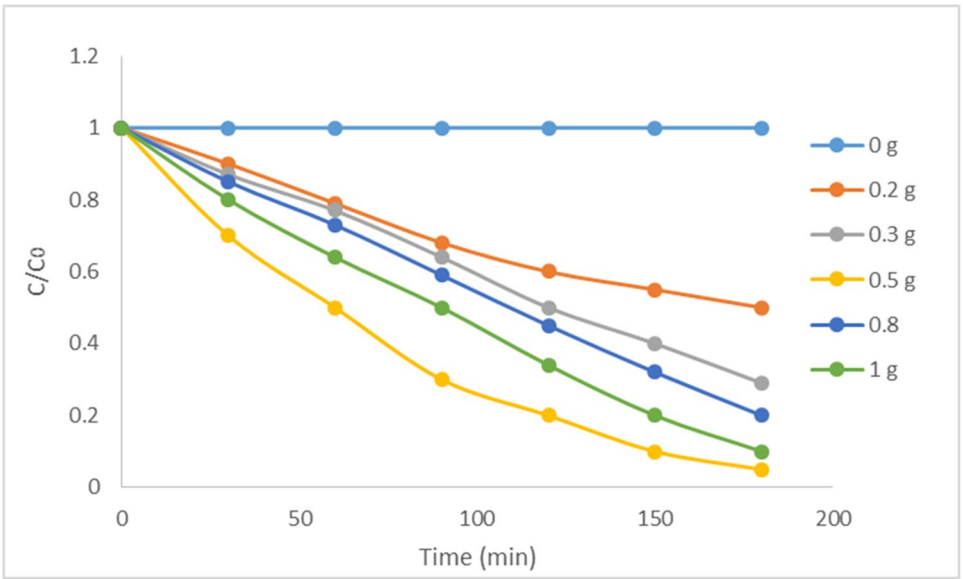

Fig. 11 Photocatalytic dye degradation with different catalyst dosages of NFT90. (natural pH, room temperature, 40 ppm initial dye concentration)

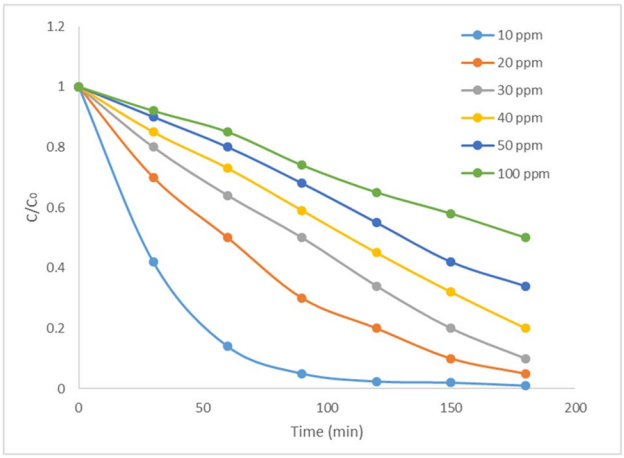

Fig. 12 Photocatalytic dye degradation of different initial dye concentrations. (natural pH, room temperature, 0.5 gm. catalyst dosage of NFT90)

Conclusion

Our observation shows that composites show better results for COD removal and dye degradation compared to spinel and TiO_2 alone, as this combination of photocatalysts from two different generations facilitates the use of a large part of the solar spectrum, better separation due to magnetic properties of $NiFe_2O_4$. From the observations, we can hypothesize that there can be the insertion of a new energy level in between the conduction band and valance band of TiO_2 and $NiFe_2O_4$ because of the difference in the bandgap of both the materials, which may also promote the separation of photoinduced electrons and photons. Over this lower dye concentration (~40 ppm) and neutral pH and 0.5 g catalyst dosage are favourable conditions for dye degradation under daytime sunlight, while other characterizations show agreement with other literature.

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Data availability All relevant data and material are presented in the main paper.

Declarations

Ethics approval and consent to participate Not applicable.

Consent for publication Not applicable.

Competing interests The authors declare that they have no competing interests.

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