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Synergetic Photocatalysis on Y_2O_3 doped $\text{NiZnFe}_2\text{O}_4$ @rGO nanocomposite for efficient Sunset Yellow dye degradation

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Abstract

The paper describes a synthesis of Y_2O_3 doped $\text{NiZnFe}_2\text{O}_4$ /reduced graphene oxide (rGO) nanocomposite to be used in photo catalytics. The preparation of nanohybrid was performed by a cheap and widely used sol-gel auto combustion technique. The crystalline structure, functional groups, chemical bonds, morphology, and specific surface area of the obtained $\text{NiZnFe}_2\text{O}_4/\text{Y}_2\text{O}_3/\text{rGO}$ were characterized using XRD, FT-IR, UV-Visible, FESEM with EDX, HRTEM, BET and XPS. Other XRD and SEM analyses revealed the purity of the phases and the crystallinity and morphology of the nano-photocatalyst. The nanocomposite from the spectral analysis performed with the UV-visible devices exhibited a band gap of 3.1 eV. The studies also revealed that Y_2O_3 and $\text{NiZnFe}_2\text{O}_4$ particles were evenly attached onto reduced graphene oxide surface. Thus, the prepared photocatalyst could degrade Sunset Yellow dye in the visible light radiation. Operational parameters (pH, catalyst amount, initial pollutant concentration and electrolyte concentration) were varied in the experiments to understand the effect on photocatalytic performance. The findings showed that $\text{NiZnFe}_2\text{O}_4/\text{Y}_2\text{O}_3/\text{rGO}$ had a higher photocatalytic efficiency compared to pristine rGO with a degradation efficiency of 96% of Sunset Yellow dye. The conclusion from a scavenger study was that during the presence of ultraviolet light a reaction mechanism occurred and the electron-hole pairs created by the photogeneration process were effectively separated. All these results support the idea that $\text{NiZnFe}_2\text{O}_4/\text{Y}_2\text{O}_3/\text{rGO}$ nanohybrid has an outstanding photocatalytic ability, which can be used in advanced environmental remediation.

Keywords: Nanohybrid, sol-gel auto combustion technique, Photocatalytic material, Sunset Yellow.

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1.Introduction

There are a number of industries that use synthetic dyes such as food production, pharmaceuticals, textiles, and cosmetics [1]. These industries are frequently associated with wastewater that may contain a large amount of persistent dyes, which can be toxic and are not easily degraded in the environment, thus endangering aquatic ecosystems and public health [2]. The dyes used in most of these industries are synthetic dyes and due to their toxic nature and resistance to degradation can create serious and long-term environmental problems if discharged without treatment [3,4]. One of the most widely used azo dyes, sunset yellow, is found in a variety of food products such as those in drinks, sweets and medicines [5]. Sunset yellow is useful, but also linked to health problems, including allergic reactions, hyperactivity in children and even cancer. So, governments have established high limits for its consumption in food and drinks [6,7]. For this reason, an effective treatment method should be used for treatment before the waste water can be released into the environment [8]. There are several approaches that have been developed to remove the dyes from the wastewater such as physical (precipitation, membrane filtration), chemical (coagulation, advanced oxidation processes, chlorine, hydrogen peroxide and photocatalysis) and biological (aerobic, anaerobic and combination systems) [9,10].

Photocatalysis is a chemical reaction that involves the use of light energy to assist a reaction, usually through the help of a catalyst referred to as a photocatalyst [11,12]. The photocatalyst takes in light that produces electron-hole pairs that are involved in the chemical reaction to produce CO_2 and H_2O which are very reactive and effective to degrade the target pollutants [13]. Due to its natural occurrence, low cost and its superior catalytic activity, multiple oxidation states, Nickel Zinc Ferrites ($\text{NiZnFe}_2\text{O}_4$) have been much in the spotlight as photocatalytic catalysts [14]. $\text{NiZnFe}_2\text{O}_4$ is a mixed nickel–zinc ferrite, which belongs to the family of spinels, $\text{Ni}_{1-x}\text{Zn}_x\text{Fe}_2\text{O}_4$ [15].

Pristine $\text{NiZnFe}_2\text{O}_4$, however, has a few drawbacks, such as high electron-hole recombination rates, low active surface area and low utilization of visible light [16]. To improve its

photocatalytic activity, strategies need to be developed to increase light harvesting, charge separation, surface reaction kinetics, and stability [17]. The heterojunction formation, carbon coupling, loading of noble metals, defect engineering, particle-size control and optimization of Ni/Zn ratio are the most important ways to improve the photocatalytic property of NiZnFe₂O₄ [18]. Some of the types of resources that are broadly used in the present world to enhance the degradation efficiency are Nickel Zinc Ferrites with metal oxides and carbon [19]. The NiZnFe₂O₄/Metal Oxide/rGO is one of these methods that show potential due to the synergistic combination of superior charge separation, high adsorption capacity, and magnetic recyclability [20]. The photocatalytic activity of the metal oxide semiconductors, such as ZnO, CuO, CdO, TiO₂, NiO and Ag₂O, has been well investigated [21,22]. Among the rare earth metal oxides, it is found that Y₂O₃ is an excellent photocatalytic material upon optimal compositing with rare earth metal oxides [23,24]. However, in conventional hybrid systems, recombination centres form as a result of poor reassembling of the electron hole pair at the contact interface, thus diminishing the photocatalytic properties [25]. In order to overcome this, researchers are pursuing active development of integration of hybrid nanoparticles through interfaces engineering [26].

Graphene can increase the photocatalytic activity and decrease the electron recombination which consequently boost the photocatalytic efficiency in the presence of UV-visible light as an electron collector [27,28]. Reduced graphene oxide (rGO) has less oxygen groups in the structure than that of graphene and is less hydrophilic as compared to graphene [29,30]. The mechanical, optical, thermal and electrical properties of rGO can be beneficial for its potential application in photocatalyst [31,32]. The photocatalytic efficiency of rGO is, however, compromised by the factors such as low quantum efficiency and light harvest [33]. In order to alleviate these restrictions, hetero junctions with rGO have been produced by introducing rGO based nanocomposites comprising of different materials like metal oxides CuO, ZnO, CeO, TiO₂ etc.,[34]

In the present work, the authors have done a synthesis of NiZnFe₂O₄/Y₂O₃/rGO nanocomposite using a chemical precipitation method in an eco-friendly way. Photodegradation and adsorption of Sunset Yellow which is a representative member of the azo dye family was done on these nanocomposites. Therefore the adsorption properties of the said nanocomposites were studied under various operating conditions such as effect of PH, catalyst dosages, initial dye concentration and electrolytes. The degradation of the sunset yellow organic pollutant with these NiZnFe₂O₄/Y₂O₃/rGO was tested under the UV irradiation to determine their possible

use in the actual systems. This research aims at creating a low-cost and simple procedure of preparing $\text{NiZnFe}_2\text{O}_4/\text{Y}_2\text{O}_3/\text{rGO}$ by using no-toxic feedstock capping agent to apply to photocatalytic processes.

2. Materials and Methods

2.1 Chemicals

Materials such as Nickel nitrate hexahydrate $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, Zinc nitrate hexahydrate $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, Ferric nitrate nonahydrate $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, Yttrium nitrate hexahydrate $\text{Y}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$, Graphene oxide, Citric acid ($\text{C}_6\text{H}_8\text{O}_7$), Ethylene glycol ($\text{HOCH}_2\text{CH}_2\text{OH}$), Sodium hydroxide, Ethanol, Deionized water were purchased from Sigma Aldrich to fabricate $\text{NiZnFe}_2\text{O}_4/\text{Y}_2\text{O}_3/\text{rGO}$ composite.

2.2 Preparation of $\text{NiZnFe}_2\text{O}_4$ nanoparticles

The quantities of about 1.65 gm of $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, 1.49 gm of $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and 4.04 gm of $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ were weighed and each salt was dissolved in an appropriate amount of deionized water. The three solutions were then mixed together with constant magnetic stirring to get a uniform solution of metal ions. The citric acid was dissolved in deionized water individually and slowly added to the mixed metal-nitrate solution with continuous stirring for about 1-2 hrs. The pH of the precursor solution was adjusted to around 7-9 using controlled addition of dilute ammonium hydroxide solution. The homogeneous solution was brought to a gentle boil (around 70-80°C) with constant stirring. The heating and stirring were continued until a thick gel was obtained. The resulting gel was then heated to about 200-250°C at which point the nitrate species are oxidizing agents and the citrate species are fuel. After the combustion, a fluffy and porous precursor powder was obtained, and gently ground with an agate mortar and pestle to form a uniform fine powder. The precursor powder thus obtained was placed in an alumina crucible and calcined in a muffle furnace at 600°C for 3 hrs. A very gentle grinding was done again to get $\text{NiZnFe}_2\text{O}_4$ nanoparticles.

2.3 Preparation of Y_2O_3 nanoparticles

About 3.83 gm of $\text{Y}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ can be dissolved in about 50 mL of deionized water with continuous magnetic stirring. The solution should be stirred to ensure a clear and homogeneous solution is made. Citric acid monohydrate is first dissolved in a small amount of deionized water, and then slowly dropped into the yttrium nitrate solution with stirring. This

complexation allows for the yttrium to be uniformly distributed in the precursor. Use dilute NH_4OH solution for adjusting the pH to $\sim 6-8$. The solution is then heated up to $\sim 70-80^\circ\text{C}$ with constant magnetic stirring. The resulting gel is placed in an appropriate ceramic container, and then dried at around. The dried precursor powder is loaded into an alumina crucible and then burnt in a muffle furnace. The furnace is then cooled down to room temperature by natural cooling method after calcination. The obtained white or off-white powder is very softly ground to provide small particles of Y_2O_3 .

2.4. Synthesize of $\text{NiZnFe}_2\text{O}_4/\text{Y}_2\text{O}_3/\text{rGO}$ nanocomposite

The modified Hummer's method is used for the production of reduced graphene oxide. The salts were dissolved in DDI water while stirring to make a 0.1 M solution of both $\text{NiZnFe}_2\text{O}_4$ and Y_2O_3 nanoparticles. The uniformly dispersion were made by dissolving the rGO in 0.1 M deionized water for 30 minutes by ultrasonic dispersion. The rGO which was dispersed, was then mixed with the solution containing nanoparticles, and then step-wise addition of NaOH solution was made to decrease the pH to around 9 which resulted in the formation of heterogeneous precipitate. The mixture was placed inside a Teflon lined stainless-steel autoclave and hydrothermally treated at 180°C for 12 hr. The precipitate was brought to the normal temperature, collected, washed with the deionised water and ethanol and dried at 80°C . The dried substance was finally pyrolysed at 450°C for 3 hours to get the metal oxide nanocomposite for analysis. The whole synthetic method is shown in Fig. 1.

2.5. Photocatalytic experimental procedure

The experiment was performed in the presence of UV light using a 500 watts mercury lamp, in order to study the photocatalytic property of these materials in their as-prepared state, the Sunset Yellow dye solution was used. To put it simply, the dye solution used (Sunset Yellow – 100mL with 10mg per litre) was spiked with photocatalyst (1mg). The suspension was shaken for 30 minutes in the dark before it was exposed to the irradiation in order to achieve a balance between adsorption and desorption. Samples were taken at 15, 30, 45, 60, 75, 90 and 105 minutes after the irradiation process. The absorbance of the Sunset Yellow dye solution was then determined after this by using a UV-Vis spectra to determine it at the wavelength of 482nm.

The formula used to determine the decomposition effectivity was:

$$\frac{C_0 - C_t}{C_0} \times 100\%,$$

Here, C_0 is the absorbance of the dye after dark adsorption, and C_t is absorbance at the time t that the light is being applied.

3. Results and discussion

3.1. XRD analysis

The phase integrity and crystalline configuration of the prepared $\text{NiZnFe}_2\text{O}_4/\text{Y}_2\text{O}_3/\text{rGO}$ nanocomposite are confirmed by XRD technique. Cubic phases of yttrium oxide (Y_2O_3), $\text{NiZnFe}_2\text{O}_4$ and reduced graphene oxide (rGO) are present in the diffraction pattern, whereas no unwanted impurity phases are found. The presence of reduced graphene oxide (rGO) usually indicates the presence of a wide and low diffraction peak at about $24-26^\circ$, which reflects the (002) plane of graphitic carbon (Fig. 2a) [35]. The characteristic reflections of the spinel $\text{NiZnFe}_2\text{O}_4$ phase are observed at approximately 30.03° , 35.46° , 42.84° , 53.57° , 56.94° and 62.89° , which can be indexed to the (220), (311), (400), (422), (511) and (440) planes, respectively. Such reflections are typical of the cubic spinel ferrite structure (Fig. 2b) [36]. The diffraction peaks of Y_2O_3 are indexed as belonging to the cubic crystal structure of bixbyite (space group Ia-3, JCPDS No.41-1105) [37]. The prominent diffraction peaks generally appear at 2θ values of approximately 20.59° , 29.13° , 31.04° , 39.96° , 43.57° , 47.92° , 57.60° , and 64.25° , corresponding to the (211), (222), (400), (332), (431), (440), (622), and (444) crystallographic planes, respectively (Fig. 2c). The aforementioned peaks at 2θ of 20.75° , 29.30° , 33.87° , 43.52° , 48.62° , 53.19° , 57.59° , 64.92° , 71.26° and 78.71° corresponds to (211), (111), (400), (431), (440), (311), (222), (444), (400) and (331) were identified in the XRD pattern of $\text{NiZnFe}_2\text{O}_4/\text{Y}_2\text{O}_3/\text{rGO}$ nanocomposite (Fig.2d). The hetero structured nanocomposite of highly conductive rGO and crystalline Y_2O_3 and $\text{NiZnFe}_2\text{O}_4$ has effective charge transfer pathways and the good interface contact between the two.

3.2 FT IR Analysis

FTIR spectroscopy methodology was selected for this study to analyse the chemical bonds and functional groups which acted as chelating and reducing substances in the chemical reaction. The FTIR spectrum is shown in Fig. 3. The absorbance band noted at 651, 827, 1044, 1329, 1624, and 3296 cm^{-1} for Y_2O_3 (Fig. 3c) [38]; 1413, 1554, 1641, 2152 and 3347 cm^{-1} for $\text{NiZnFe}_2\text{O}_4$ (Fig. 3b) [39]; 1210 and 1567 cm^{-1} for rGO (Fig. 3a) [40]; 464, 565, 837, 1062, 1396, 1520 and 3376 cm^{-1} for $\text{NiZnFe}_2\text{O}_4/\text{Y}_2\text{O}_3/\text{rGO}$ (Fig. 3d). The frequency of the stretching vibration mode of the O-H monomer was found to be around 3350 cm^{-1} for all the compounds. This property is related to the ability to adsorb hydroxyl molecules present in the atmosphere. The bands in the spectral range 1000-1400 cm^{-1} can be linked to the main functional groups containing oxygen atoms in rGO. The characteristic vibrations of Y_2O_3 are dominated by the lower number of wavenumbers. Vibrational modes of Y-O can be identified in the approximate 800–900 cm^{-1} region. The characteristic metal-oxygen vibrations in the low-frequency region

confirm the formation of the $\text{NiZnFe}_2\text{O}_4$ spinel ferrite phase. The dominant absorption band around $550\text{--}600\text{ cm}^{-1}$ is due primarily to the Fe-O stretching vibration in the tetrahedral metal-oxygen sites in the spinel lattice of the ferrite. Overall, the FTIR spectrum shows that both the O-H and C=C, C-O-C stretching modes of rGO and the Y-O and Fe-O/metal-oxygen stretching modes of $\text{NiZnFe}_2\text{O}_4$ are present.

3.3. UV-Vis analysis

The UV-vis spectrum was used as illustrated in Fig. 4 in order to determine the optical properties of the $\text{NiZnFe}_2\text{O}_4/\text{Y}_2\text{O}_3/\text{rGO}$ nanocomposites that were synthesized. The spectrum was well revealing high absorption in the region of the UV light spectrum for all the samples. When Y_2O_3 is added to $\text{NiZnFe}_2\text{O}_4$ (Fig. 4a) an obvious shift in the absorption edge towards longer wavelength range ($\sim 465\text{ nm}$) is observed [41]. The absorption edge in the composite sample has shifted towards the red, indicating a reduction in the band gap energy of the samples. Moreover, the calculated values of the band gaps are presented by Tauc plots as shown in Fig. 4b. The band gap of $\text{NiZnFe}_2\text{O}_4/\text{Y}_2\text{O}_3/\text{rGO}$ nanocomposite is narrowed to $\sim 3.1\text{ eV}$, which is less than that of Y_2O_3 ($\sim 3.3\text{ eV}$), $\text{NiZnFe}_2\text{O}_4$ ($\sim 3.6\text{ eV}$) and rGO ($\sim 4.0\text{ eV}$) (41). The band gap of the ternary nanocomposite is lower, this is beneficial to its ability to absorb visible light, and it has a better photocatalytic effect. The ternary nanocomposite of Y_2O_3 , $\text{NiZnFe}_2\text{O}_4$, and rGO was fabricated, and the significant redshift in the absorption edge was observed, which suggested a decrease in the optical band gap. This band gap lowering effect may be attributed to the synergistic effect between Y_2O_3 and $\text{NiZnFe}_2\text{O}_4$ and the excellent characteristic of electron conducting of rGO that can be used to facilitate the charge transfer and the reduction of electron-hole interaction. These two properties are responsible for the ability of the electron conducting of rGO.

3.4. SEM with EDX analysis

The FESEM was used to investigate the surface morphology of the synthesized $\text{NiZnFe}_2\text{O}_4/\text{Y}_2\text{O}_3/\text{rGO}$ nanocomposite. The FESEM micrographs showed a rough and heterogeneous structure with the $\text{NiZnFe}_2\text{O}_4$ and Y_2O_3 nanoparticles connected and embedded in the rGO matrix [42]. The oxide nanoparticles were dispersed on the wrinkled and folded rGO sheets, which meant that there was good interaction between the inorganic nanoparticles and the carbonaceous support (Fig. 5a-b). The $\text{NiZnFe}_2\text{O}_4$ nanoparticles showed quasi-spherical and irregular shape and had an aggregation tendency, while the Y_2O_3 nanoparticles were distributed among the ferrite nanoparticles and rGO sheets. With the introduction of rGO, the dispersion of the nanoparticles was significantly enhanced, and the tendency of excess

aggregation between particles was avoided because of the anchoring points provided by rGO. In addition, the interlocked 3D architecture of rGO with relatively high accessible surface area was further obtained due to the wrinkled structure. Due to the intimate contact between NiZnFe₂O₄, Y₂O₃, and rGO, it is believed that charge transfer may occur at the interface and that it will offer a large amount of active sites for photocatalytic reactions.

The elemental composition of the prepared nanocomposite NiZnFe₂O₄/Y₂O₃/rGO was studied using the EDX which was attached to the FESEM system. The characteristic peaks of Ni, Zn, Fe, yttrium (Y), oxygen (O) and carbon (C) are clearly visible (Fig. 5c). The carbon peak is attributed to the reduced graphene oxide support, oxygen peak is attributed to the presence of oxide lattices of Y₂O₃, NiZnFe₂O₄ and in the case of the presence of functional groups of oxygen in rGO. There were no impurity peaks, indicating that the synthesized material is very pure. The absence of any additional components detected from any residues from substrates, or contaminating substances that could be introduced during the synthesis/purification process, is shown by the effective performance of the fabrication and purification processes.

3.5. HRTEM analysis

The synthesized NiZnFe₂O₄/Y₂O₃/rGO nanocomposite is expected to show the nanosized NiZnFe₂O₄ and Y₂O₃ particles anchored and distributed over the thin, wrinkled sheets of rGO in the HRTEM images [43]. The presence of the dark contrast nanoparticles on the relatively light and transparent carbon sheets can be explained by the higher electron density of metal oxide phases (Fig 6a-c). The rGO sheets serve as the two-dimensional surface structure for dispersing the oxide nanoparticles and then spreading them over the surface. The micrographs obtained in HRTEM are expected to exhibit relatively good lattice fringe definition, thus validating the crystallinity of both NiZnFe₂O₄ and Y₂O₃ phases. Furthermore, the SAED patterns show additional proof of the crystallinity of the nanocomposite. The SAED image shows bright concentric diffraction rings, and a large number of diffraction spots, meaning the nanocomposite material has a polycrystalline nature (Fig. 6d).

3.6. BET analysis

In this experiment, we also carried out N₂ adsorption/desorption to investigate the surface area and porous property of carbonaceous adsorbents. The N₂ adsorption-desorption isotherm for NiZnFe₂O₄/Y₂O₃/rGO nanocomposite is displayed in Fig.7a. The execution of the samples is mesoporously indicated by a type IV isotherm with an H3 hysteresis loop, as the curve makes evident. The BET surface area for NiZnFe₂O₄/Y₂O₃/rGO nanocomposite sample is 84.6 m²/g and BJH pore size distribution is 28.9 nm [44]. This is compared with bare NiZnFe₂O₄, there

is a large surface area ($57.1 \text{ m}^2/\text{g}$) and large pores (44.9 nm) (Fig. 7b). The specific area and porous properties of the composite sample are enhanced by the interaction between nanoparticles of Y_2O_3 and rGO.

3.7. XPS analysis

The wide-scan XPS spectrum is expected to show characteristic peaks for Fe, Ni, Zn, Y, C and O confirming the successful preparation of the ternary $\text{NiZnFe}_2\text{O}_4/\text{Y}_2\text{O}_3/\text{rGO}$ nanocomposite (Fig. 8a). The lack of strong additional elemental signals suggests high purity of the synthesized material. Fe 2p peaks give evidence for the iron ions in the spinel $\text{NiZnFe}_2\text{O}_4$ phase. Generally the Fe $2p_{3/2}$ and Fe $2p_{1/2}$ peaks are seen in the region of 710.32 eV and 723.96 eV respectively (Fig. 8b) [45]. The Ni 2p spectrum is likely to show peaks at around 852.21 eV , 872.33 eV for Ni $2p_{3/2}$ and Ni $2p_{1/2}$, respectively, along with satellite peaks at higher binding energies (Fig. 8c) [46]. There are two characteristic peaks for Zn $2p_{3/2}$ and Zn $2p_{1/2}$ at about 1021.19 eV and 1044.13 eV , respectively (Fig. 8d) [47]. The presence of the yttrium oxide in the composite is confirmed by the spectrum Y 3d. The Y 3d region is expected to exhibit spin-orbit-split components of Y $3d_{5/2}$ and Y $3d_{3/2}$ which are typically located at 157.71 eV and 159.56 eV , respectively (Fig. 8e) [48]. The C 1s spectrum can be deconvoluted into multiple components. The main peak at $\sim 284.30 \text{ eV}$ is assigned to graphitic C–C/C=C bonds of the rGO framework structure (Fig. 8f). It is generally accepted that the O 1s spectrum mainly contains contributions from lattice oxygen and surface oxygen species (Fig. 8g). The lower-binding-energy component around 531.16 eV can be attributed to metal–oxygen (M–O) bonds, such as Fe–O, Ni–O, Zn–O and Y–O.

4. Photocatalytic analysis

The Sunset yellow (SY) dye was decomposed under the Visible light using $\text{NiZnFe}_2\text{O}_4$ and $\text{Y}_2\text{O}_3/\text{rGO}$ nanocomposite. The experiment was done using a Philips ultraviolet light lamp with 200 W . The reaction mixture was prepared by adding 100 mL of catalyst, 100 mL of dye solution (10 ppm) and 1 mg of catalyst. The distance of the container from the illumination source was 25 cm . Studies were done at ambient temperature. The results of the UV-Vis spectra, which was used in the wavelength of $200 - 800 \text{ nm}$, were to determine the minimum pass filter emission of the material over the $500 - 650 \text{ nm}$ wavelength with the highest intensity of its emission being a 482 nm wavelength in the UV spectrum [49]. The reduction in the intensity was observed by recording this at a certain time at 482 nm with certain intervals at 15 mins . In

the experiment, a decrease in the concentration of the dye with time was noticed. Assessment of the photocatalytic degradation efficiency was basis on the following equation,

$$\text{Degradation (\%)} = \frac{C_0 - C}{C_0} \times 100$$

In this study, the effect of the decomposition of Sunset Yellow dye on the sun and synthesized NiZnFe₂O₄/Y₂O₃/rGO nanocomposites as photocatalysts are investigated. The SY dye had the best absorption rates at 482 nm of UV light. Nevertheless, the absorption maxima of the dye solutions started decreasing as soon as the catalyst was added. In a UV experiment, 3 mL reaction samples were also taken for every 15 mins and the UV-Vis spectra were recorded. This gives reason to believe that the catalyst had a great impact regarding the absorption rate of the colorants. And lastly, the Fig. 9a cleared up the fact that SY dye maximum was at 482nm reduced almost to zero in 105 mins, which means that the degradation rate of the SY dye was discovered to be approximately at 96% accordingly. It is the spectrum of absorption in Fig. 9c also helped in explaining the process of degradation of the doped catalyst. This is further elaborated using graphs of degradation efficiency (%) of absorption and investigate the kinetics of photosynthesis and the effectiveness curves of degrading. Since the analysts wanted to compare the catalytic activity of NiZnFe₂O₄/Y₂O₃/rGO photocatalyst the researchers examined the decomposition rate of SY pollutant.

To study the behaviour associated with the process of decomposition, the Langmuir rate formula was used to find the rate of the reaction. By this rate, it was found that the first-order rate was following the Langmuir model as given in the Equation.

$$k = -\ln(C_t/C_0)$$

In this specific case, the outcomes of pseudo-first-order kinetics, the rate constant and the variable chosen to determine the investigations were discussed. The results of these research can be deemed to be a positive outcome in the degradation process of SY, which is present in the nanocomposite investigated.

It is called as C₀, it is the concentration of the dye solution at the time period (t). At the same time, the symbol k represents the kinetic constant, and the symbol C_t represents the concentration of dye solution at a given time (t). The rate of degradation (k) of the SY dye in the photocatalytic degradation process using NiZnFe₂O₄/Y₂O₃/rGO photocatalyst was obtained as 0.0306min⁻¹ (Fig. 9b). These rates of change show that the photocatalytic degradation of SY dye with the irradiation of the UV light was highly photocatalyzed by the NiZnFe₂O₄/Y₂O₃/rGO photocatalyst.

Optimization analysis

Other parameters that affect the process of photodegradation are also studied: pH level, catalyst dosage, concentration and effect of electrolytes. The reusability and the mechanism of the nanocomposite are also described. The parameters are varied continuously, enabling researchers to gain enough knowledge about the functioning of the composite under various conditions, to determine the effect of the parameters on the efficiency of the photo degradation process.

4.1. Effect of pH

The degradation of Sunset Yellow dye at 10 ppm concentration was studied at 20 mg of nanocomposite at the following 3, 5, 7, 9, and 11 pH. The results showed that the percentages of SY dye degradation of NiZnFe₂O₄/Y₂O₃/rGO at pH 3, 5, 7, 9, and 11 were 35.5, 55.5, 94.5, 75.5 and 38.5, respectively (Fig. 10a). These results have shown that the performances of photo decomposition was optimal at pH 7 as the pH increases from 7 to 11, the degradation efficiency decrease. The increased charge density of nanocomposite increases their capacity to react with dye molecules especially at lower pH. With the increase of pH the interaction between dye molecules and nanocomposite decreases, because of negative surface of the nano composite which repels the interaction. Also, the minimum of absorbance is quite similar at low and high pH. Regarding the curved shape of the absorption curve is observed at close wavelengths, there is no significant change in the molecular conformation despite the fact that the accurate wavelength can be slightly different.

4.2. Effect of catalyst dosage

The amount of the catalysts was adjusted from 3 to 20mg and the percentage of decomposition was measured to find the lowest amount of catalyst needed for the successful photocatalyst decomposition of the Sunset Yellow dye molecule. 10ml of 10ppm solution of neutral pH 7.5 was used in the studies. After being exposed to the ultraviolet light, the results are as shown in Fig. 10b. The decomposition percentage of SY dye on 5 mg, 10 mg, 15mg, 20 mg of the SY dye are 87, 94, 95 and 97% respectively with an initial augmentation in catalyst dosage, thereafter, reducing to 90% at 30 mg. When the dose of the photocatalyst transferred to 20 mg (97%), a significant decrease in proportion breakdown was not observed. If the concentration is high, the photocatalyst particles scatter the incident light rays, thus inhibiting the starting of the photoexcitation process by the incident light by the photocatalyst particles. Therefore, the best dosages of 10 mL of the solutions being analysed were determined as 10 mg of catalyst of Sunset Yellow dye molecule.

4.3. Effect of concentration

It was also examined how different initial concentration of Sunset Yellow dye molecules affect the catalytic activity of the $\text{NiZnFe}_2\text{O}_4/\text{Y}_2\text{O}_3/\text{rGO}$ nano photocatalyst with the help of ultraviolet light when exposed to nano photocatalyst. The concentration of the potential organic compounds were observed to be changing between minimum of 5 ppm to a maximum of 30 ppm, as would be observed in Fig. 10c. The above determined neutral pH 6.5 was used in conjunction with 10 mg of SY dye catalyst. The concentration was increased at the first level (5 ppm to 30 ppm). The decomposition rate of the SY dye decreased by 99 to 61 in this period. Such conclusion is reasonable, as the light that can reach the surface catalytic regions decreases in solutions with a higher concentration. With the age of the light exposure, organic compounds in solutions of high concentration may well be completely broken down.

4.4. Effect of Scavengers

In order to investigate the species that are active of $\text{NiZnFe}_2\text{O}_4/\text{Y}_2\text{O}_3/\text{rGO}$ nanocomposite in the decomposition of Sunset Yellow solution, BQ, TBA, EDTA-2Na and were used as h^+ , O^{2-} and OH scavengers, accordingly. The chemically catalytic effectivity of $\text{NiZnFe}_2\text{O}_4/\text{Y}_2\text{O}_3/\text{rGO}$ nanocomposite has been shown in the presence of various scavengers as shown in Fig.10d. When EDTA-2Na and TBA added to the $\text{NiZnFe}_2\text{O}_4/\text{Y}_2\text{O}_3/\text{rGO}$ nanocomposite sample, the Sunset Yellow decomposition was reduced which proved that the catalytic decomposition involved h^+ and OH. Nevertheless, the photocatalytic effectivity of the chemical catalyst was found to be significantly reduced with the introduction of BQ and this suggests that the major active species in the degradation reaction was the $\cdot\text{O}^{2-}$.

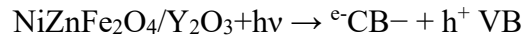
4.5. Reusability analysis

This is the potential to recycle photocatalysts, which is an important factor of the sustainability of photocatalysts. The efficacy of $\text{NiZnFe}_2\text{O}_4/\text{Y}_2\text{O}_3/\text{rGO}$ photocatalyst as catalysts during five cycles was measured, which is depicted in Fig. 11. The first cycle percentage removal of Sunset Yellow (SY) dye was 96% in the initial cycle of nano photocatalyst and dropped marginally to 94% in the second cycle and also achieved 98% in the last cycle. The results showed that the catalytic activity was very stable during the cycles throughout the course of the catalytic process with SY dye. Such experiments have shown that the catalytic activity was not significantly diminished after each use of the catalyst and catalyst good stability under the photodegradation reaction.

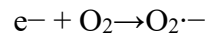
4.6 Mechanism

Based on the photocatalytic activity, the resultant $\text{NiZnFe}_2\text{O}_4/\text{Y}_2\text{O}_3/\text{rGO}$ solid solution is attributed to the synergistic effect of the magnetic spinel ferrite, the wide-band-gap

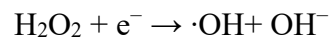
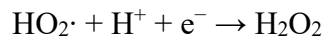
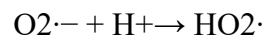
semiconductor Y_2O_3 and the conductive reduced graphene oxide (rGO). When the UV/visible light photons have energy equal or greater than the band gap of the semiconductor component ($NiZnFe_2O_4/Y_2O_3$), they promote electrons in the valence band (VB) to the conduction band (CB) of the semiconductor component [50]. These generated electrons and holes are the major charge carriers involved in the subsequent redox reactions.



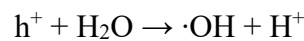
$NiZnFe_2O_4$ is a relatively narrow band gap spinel type semiconductor compound which has magnetic properties. It can be incorporated to expand light-response region and offer more active sites. The photogenerated electrons may be able to reduce oxygen.



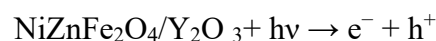
The superoxide radical can then produce other reactive oxygen species (ROS) like hydroxyl radicals.

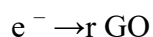


These are the reactive species which break down molecules of organic dyes. Thus, Y_2O_3 can be considered as an oxide semiconductor part and is responsible for the formation of an interfacial heterojunction with $NiZnFe_2O_4$. The difference in electronic structures between $NiZnFe_2O_4$ and Y_2O_3 allows for the direction migration of charge carriers generated by photons. The Y_2O_3 is also responsible for the presence of surface hydroxyl groups, which may be involved in the generation of hydroxyl radicals [51]:

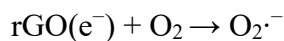


The highly reactive $\cdot OH$ radicals can oxidize the organic pollutants that have been adsorbed. In particular, the GO improves the charge separation. It has a long π -conjugated structure and high electrical conductivity which are efficient pathways for photogenerated electrons. The process of charge transfer can be simplified to:

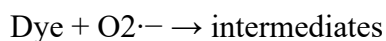
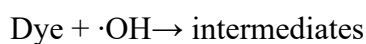




The rGO sheets thus serve as an electron acceptor and a conductive electron transfer network, minimizing the chances of electron-hole recombination. The transferred electrons on rGO can react with dissolved oxygen:



This results in a longer lifetime of photogenerated holes and electrons and thus in an increased photocatalytic efficiency. The overall degradation of a model organic dye is described in Fig. 12.



In this way the pollutant is continuously degraded to smaller intermediates and to mineralization.

5. Conclusion

The work successfully prepared NiZnFe₂O₄/Y₂O₃/rGO nanocomposites by sol-gel auto combustion method and further analysed by various other laboratory techniques. On the basis of XRD result, the nanocomposites were successfully synthesized and the 2θ value matches with the standard XRD pattern which showed that there are perfect interface interactions between the rGO sheets, Y₂O₃ nanoparticles and NiZnFe₂O₄ nanoparticles. Based on morphological analysis, the particles had a heterogeneous, rough, and interconnected morphology, which are well-anchored on the rGO sheets. The better absorption of UV light is indicated by the presence of the absorption edge at 465 nm that helps to form reactive radicals, which are significant for the degradation of pollutants. When the photocatalytic experiment was performed with UV illumination for 105 min, degradation efficiency of 96% Sunset yellow dye was observed. Moreover, in the degradation of three cycles of the pollutant, the photocatalyst showed outstanding stability and recyclability with preserved effectiveness. The findings of this study clearly indicate that NiZnFe₂O₄/Y₂O₃/rGO composites can prove to be useful in several forms as highly stable and effective photocatalysts in removing hazardous materials in water containing pollutants upon illumination by UV light, as in the case of Sunset

Yellow dye. The materials are of great interest in terms of application potential in the degradation and technology of pollutants in water.

Conflicts of interest

The authors declare that there is no conflict of interest regarding the research work reported in this manuscript.

Author contributions

S. Rajasekaran, N. Balamurugapandian, M. Nagoor Meeran: study conceptualization and writing (original draft) the manuscript, R. Vennila, M. Jothi: data curation, formal analysis and writing (review & editing).

Data availability statement

The data that support the findings of this study are available from the corresponding author, upon reasonable request.

Funding Declaration

The authors have no relevant funding interests to disclose.

Ethical approval

The authors declare that there are no human cells and tissues are used in this manuscript.

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Fig. 1 Schematic representation of synthesis of $\text{NiZnFe}_2\text{O}_4/\text{Y}_2\text{O}_3/\text{rGO}$ nanocomposite

Fig. 2 XRD of a) rGO NPs b) $\text{NiZnFe}_2\text{O}_4$ NPs c) Y_2O_3 NPs d) $\text{NiZnFe}_2\text{O}_4/\text{Y}_2\text{O}_3/\text{rGO}$ nanocomposite

Fig. 3 FTIR of a) rGO NPs b) $\text{NiZnFe}_2\text{O}_4$ c) Y_2O_3 NPs d) $\text{NiZnFe}_2\text{O}_4/\text{Y}_2\text{O}_3/\text{rGO}$ nanocomposite

Fig. 4 UV-Vis Absorption spectra of a) $\text{NiZnFe}_2\text{O}_4/\text{Y}_2\text{O}_3/\text{rGO}$ nanocomposite b) Kubelka-Munk model of $\text{NiZnFe}_2\text{O}_4/\text{Y}_2\text{O}_3/\text{rGO}$ nanocomposite

Fig. 5 (a) - (b) FESEM images of $\text{NiZnFe}_2\text{O}_4/\text{Y}_2\text{O}_3/\text{rGO}$ nanocomposite

with different magnifications; **(c)** EDX image of $\text{NiZnFe}_2\text{O}_4/\text{Y}_2\text{O}_3/\text{rGO}$ nanocomposite

Fig. 6(a-c) TEM image of $\text{NiZnFe}_2\text{O}_4/\text{Y}_2\text{O}_3/\text{rGO}$ nanocomposite and **(d)** SAED of $\text{NiZnFe}_2\text{O}_4/\text{Y}_2\text{O}_3/\text{rGO}$ nanocomposite

Fig. 7 BET study of $\text{NiZnFe}_2\text{O}_4/\text{Y}_2\text{O}_3/\text{rGO}$ nanocomposite a) surface area b) pore size

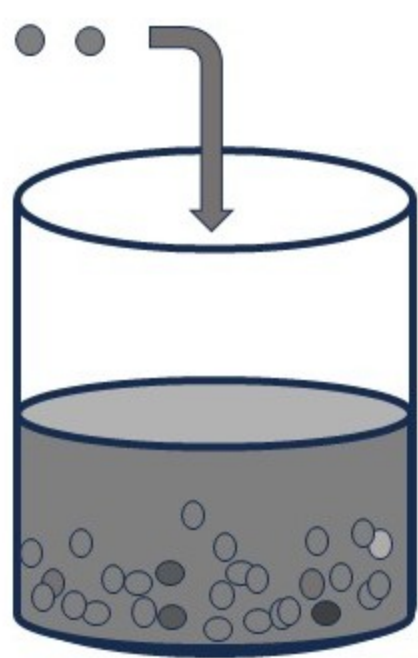
Fig. 8 XPS of $\text{NiZnFe}_2\text{O}_4/\text{Y}_2\text{O}_3/\text{rGO}$ nanocomposite a) survey spectra b) Fe 2p $3/2, 1/2$ c) Ni 2p $3/2, 1/2$ d) Zn 2p $3/2, 1/2$ e) Y 3d $3/2, 5/2$ f) C 1s g) O 1s

Fig. 9. a) Photocatalytic measurement of Sunset Yellow dye; **b)** first order kinetic plot of SY dye; **c)** % of degradation efficiency

Fig. 10 a) Effect of pH **b)** Effect of catalyst dosage **c)** Effect of concentration **d)** Effect of scavenger

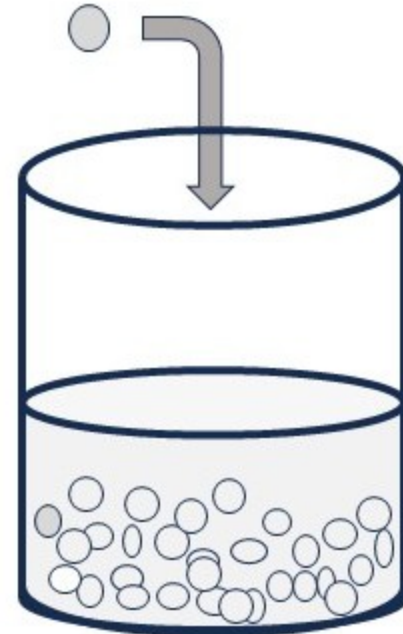
Fig. 11 Reusability analysis of $\text{NiZnFe}_2\text{O}_4/\text{Y}_2\text{O}_3/\text{rGO}$ nanocomposite at different cycles

Fig.12 Schematic illustration of mechanism of photocatalytic process NiZnFe₂O₄/Y₂O₃/rGO nanocomposite under UV irradiation



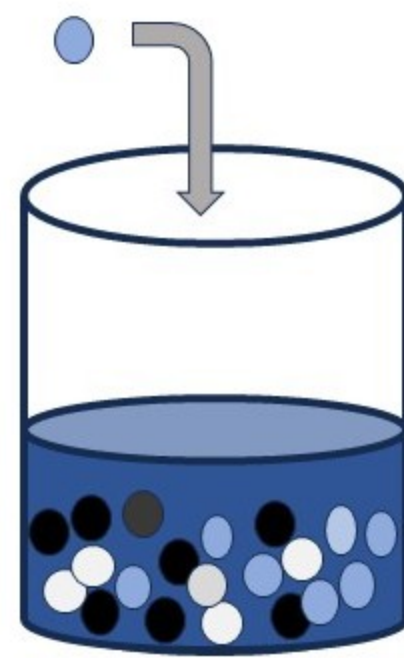
rGO + DI.water

Stirring
1 hr



Y₂O₃ + DDI water

Stirring
2 hrs



NiZnFe₂O₄ + DDI water

NaOH
pH - 9



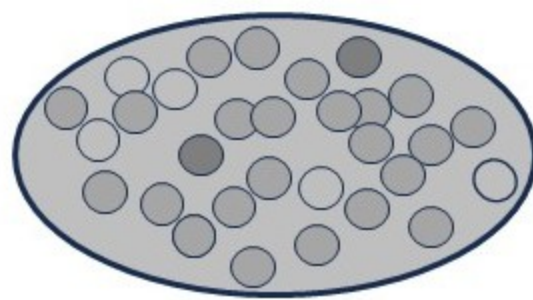
12 hrs **180°C**



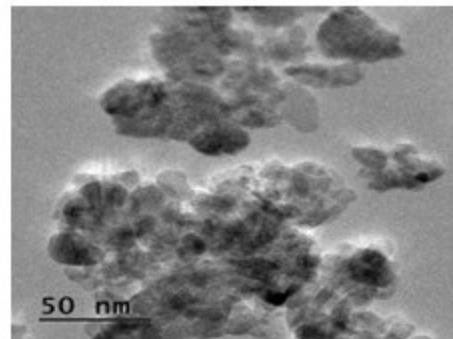
80°C
12 hrs

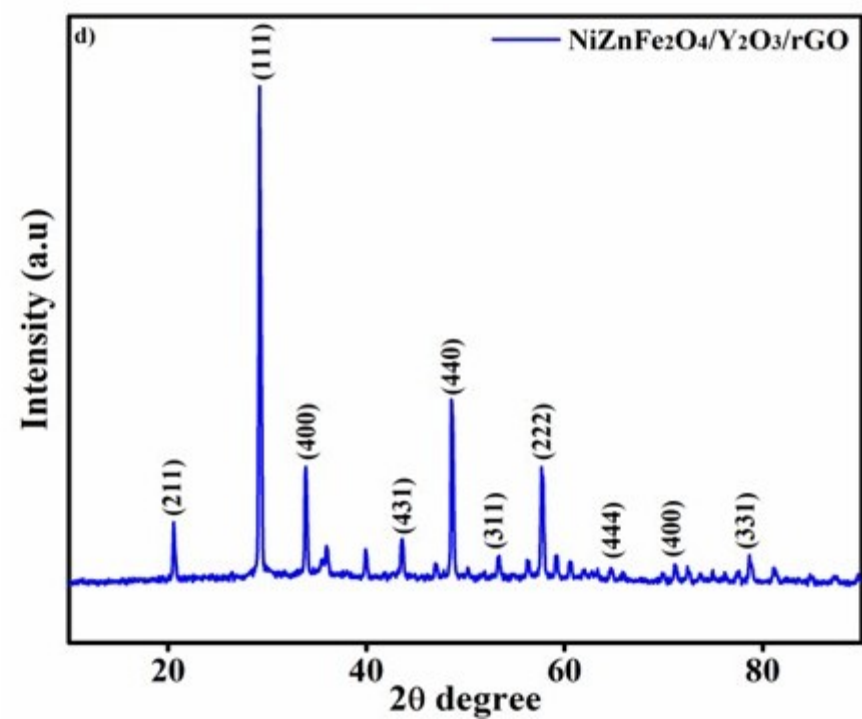
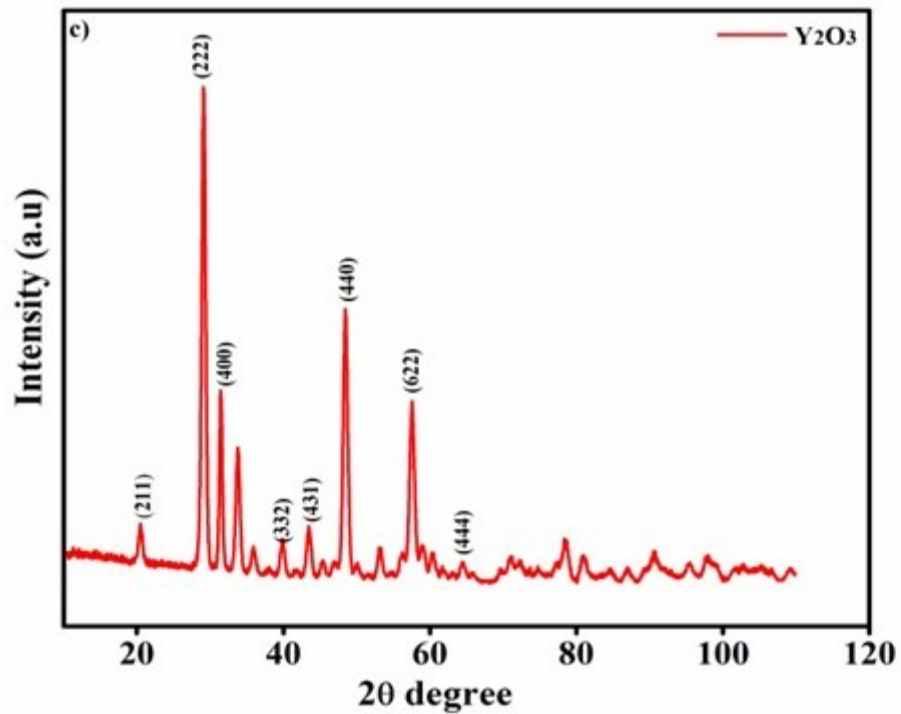
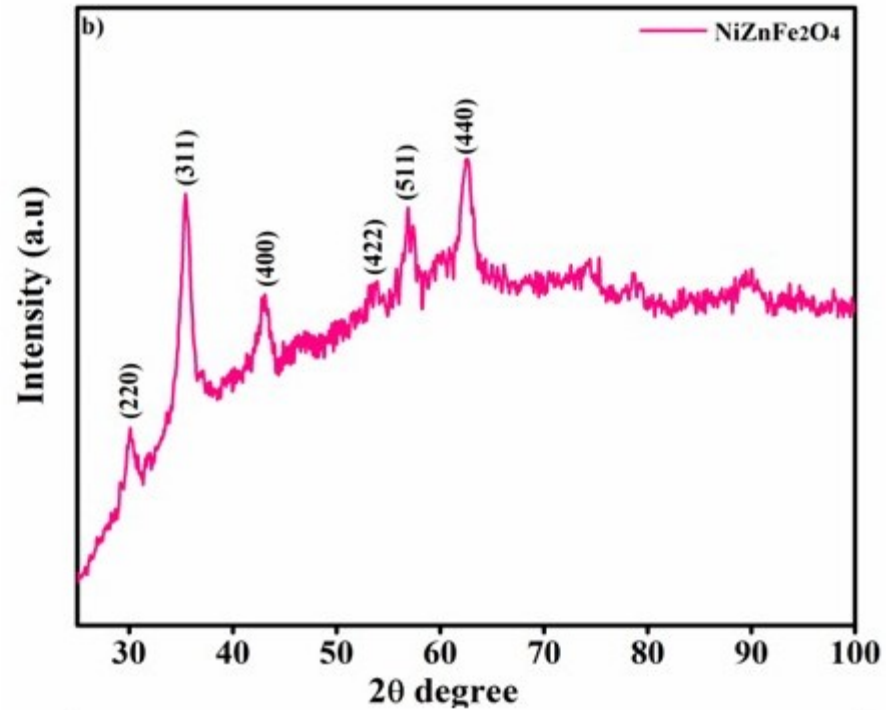
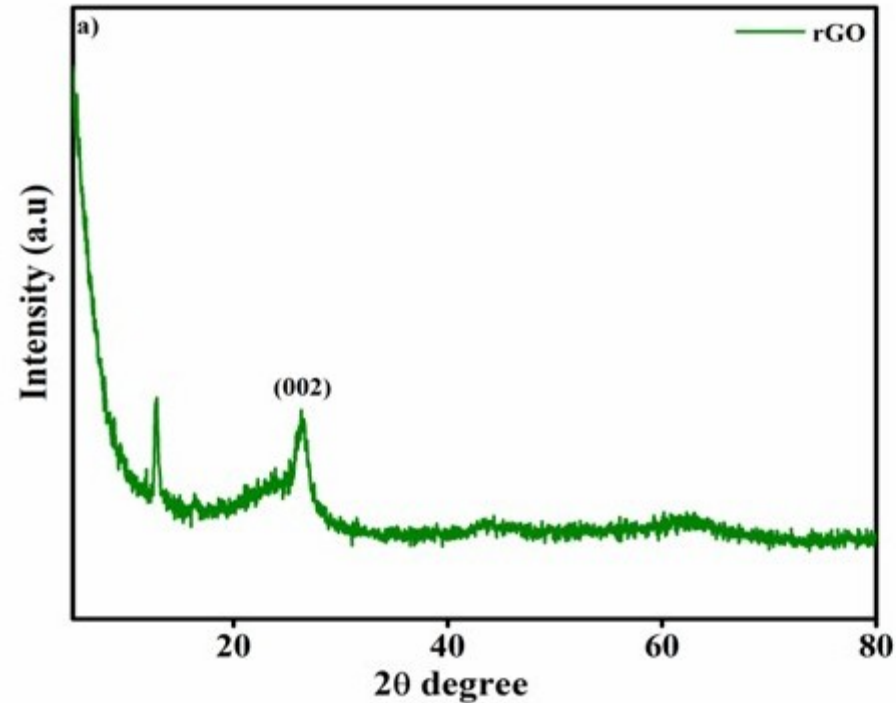


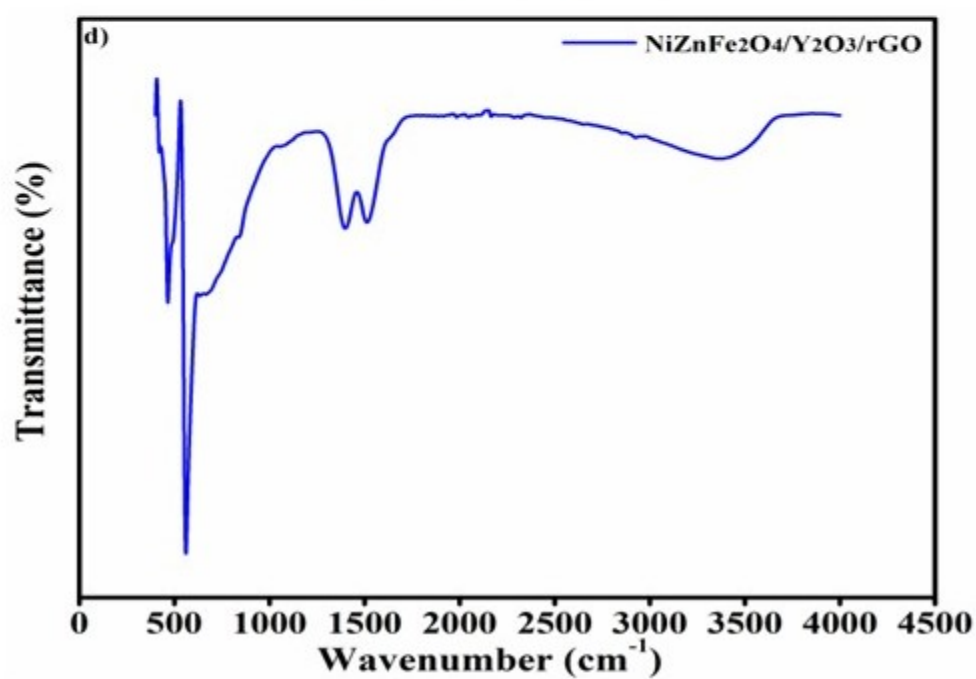
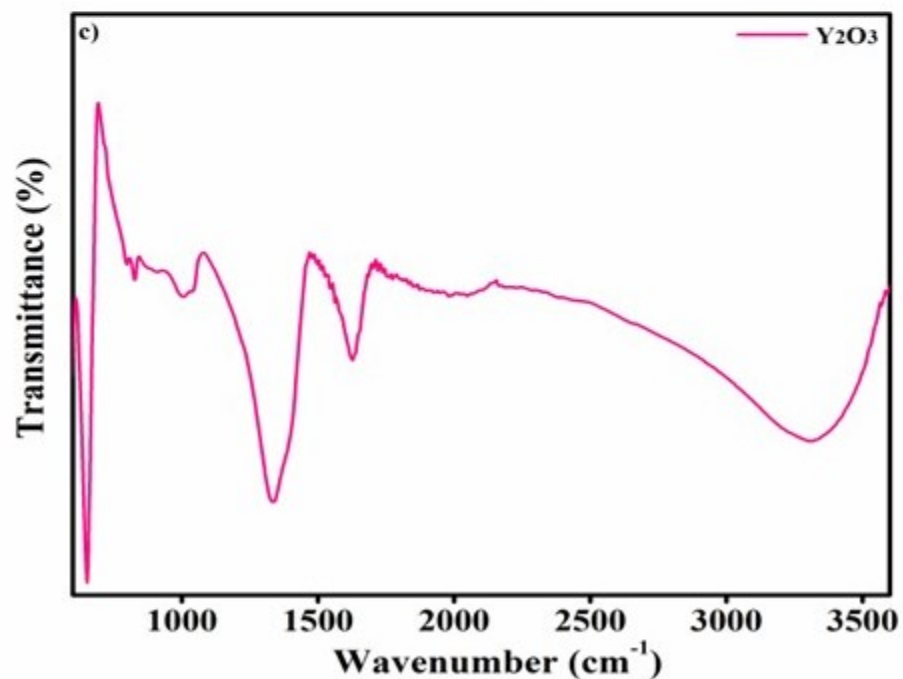
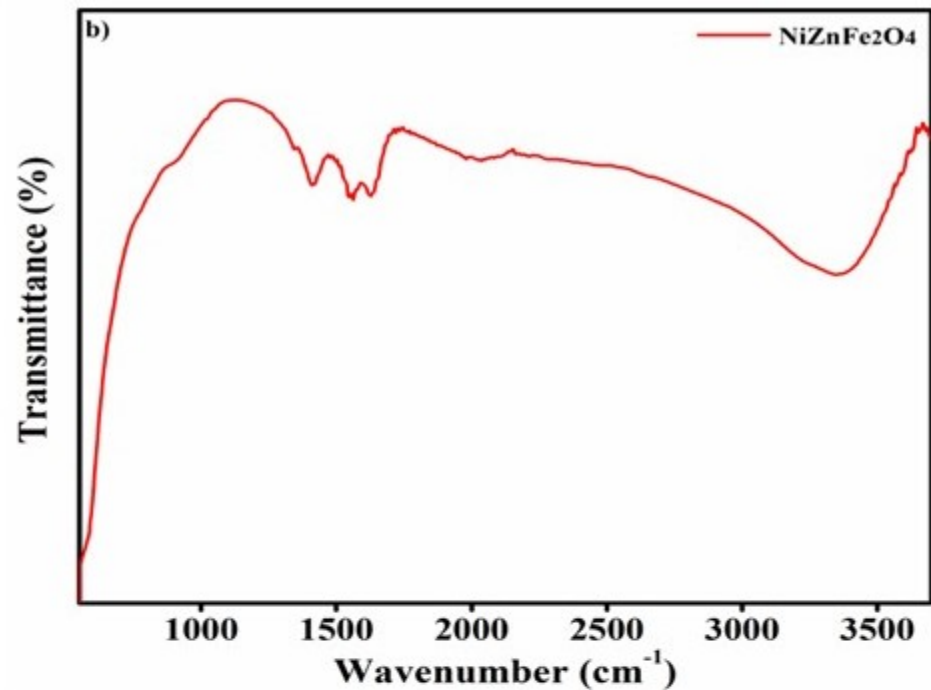
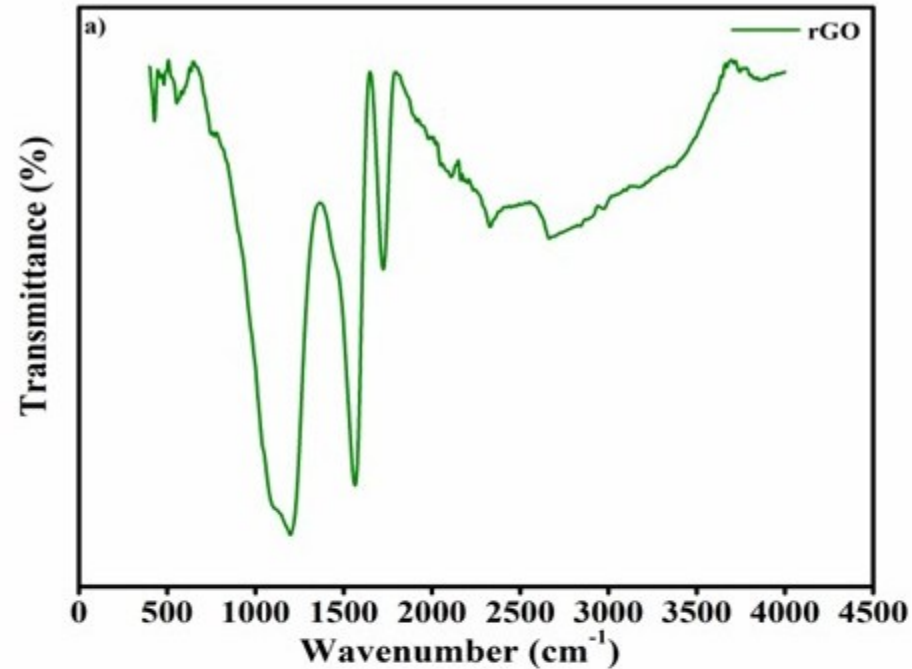
450°C
3 hrs



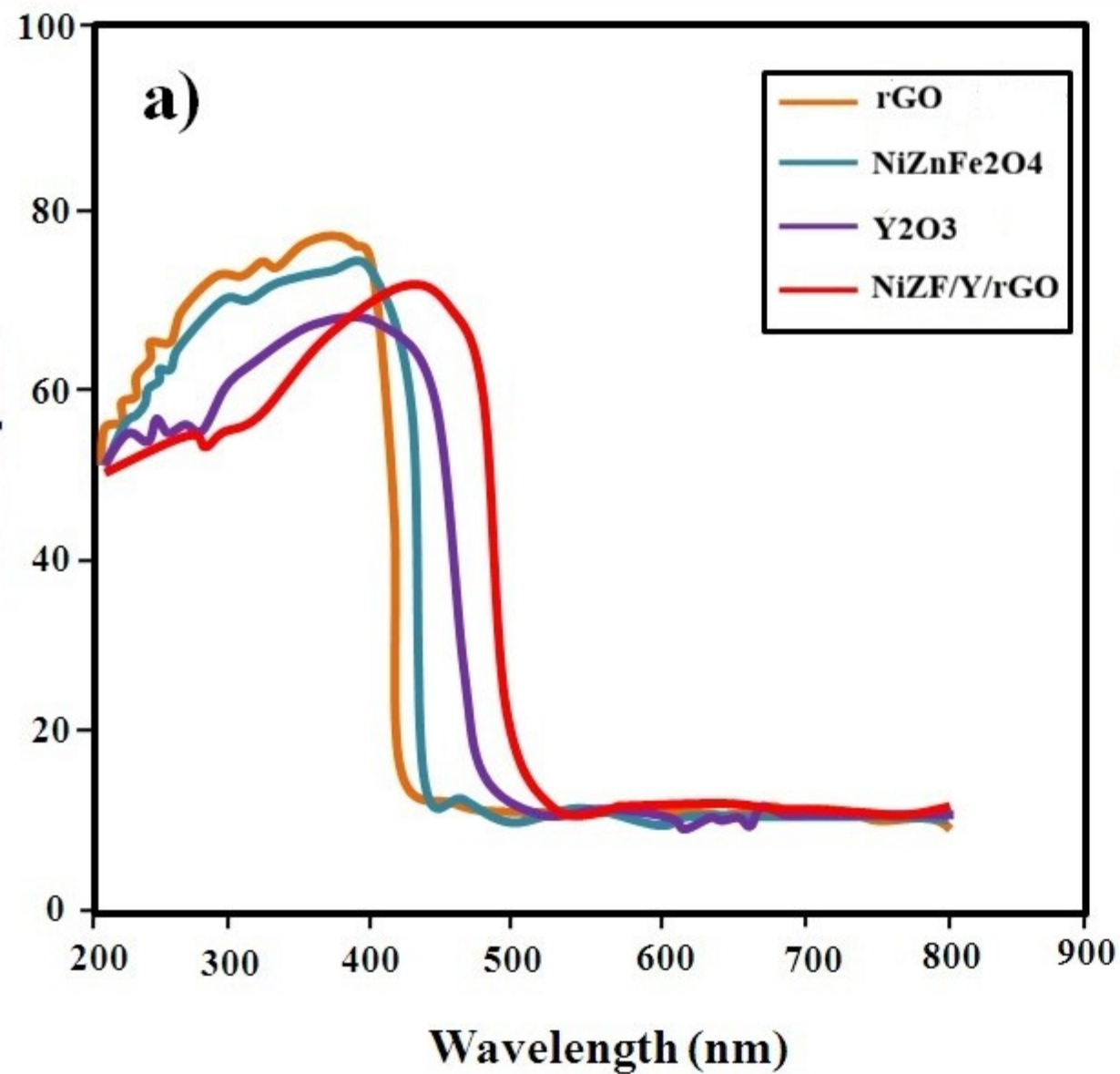
NiZnFe₂O₄ / Y₂O₃/rGO nanocomposite



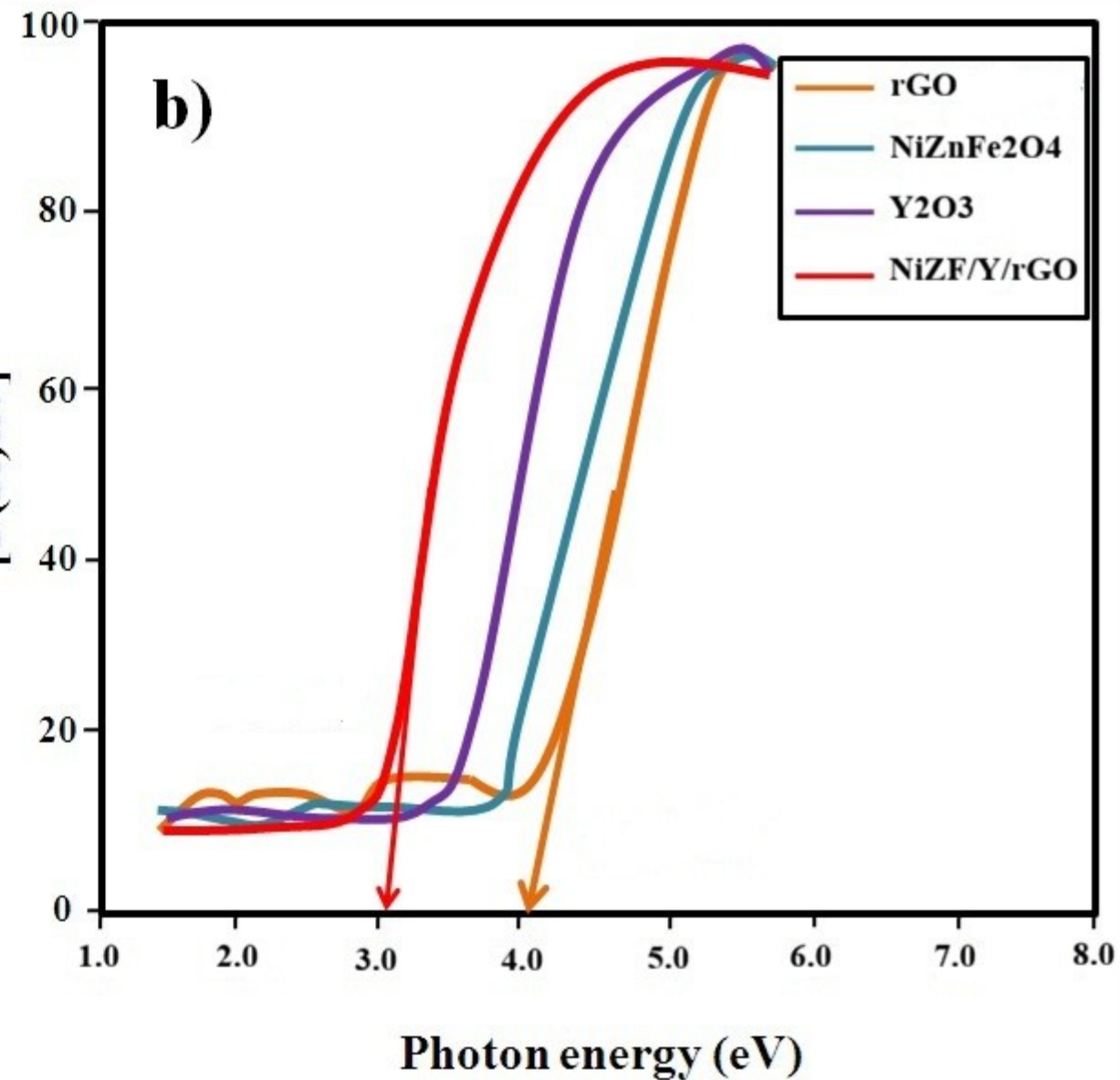




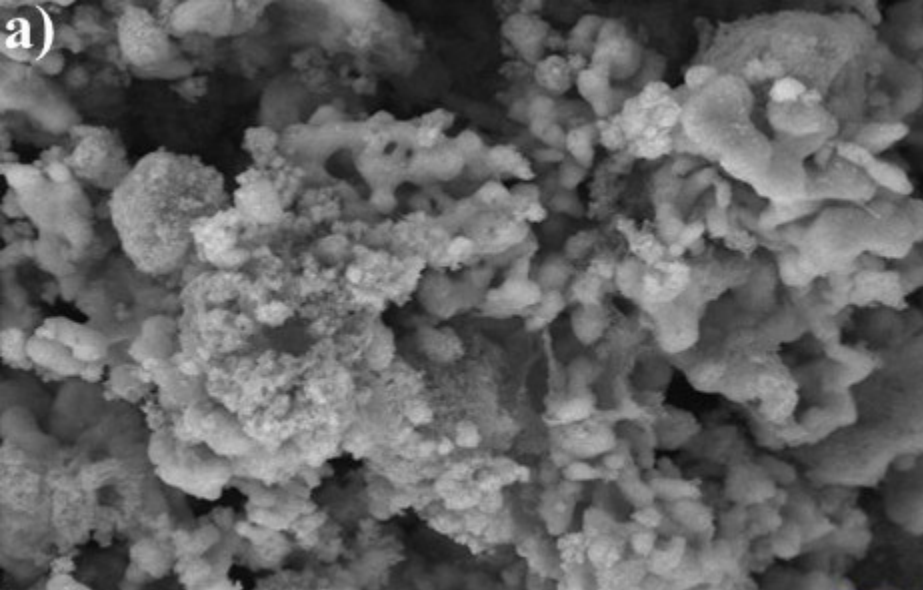
% Absorption



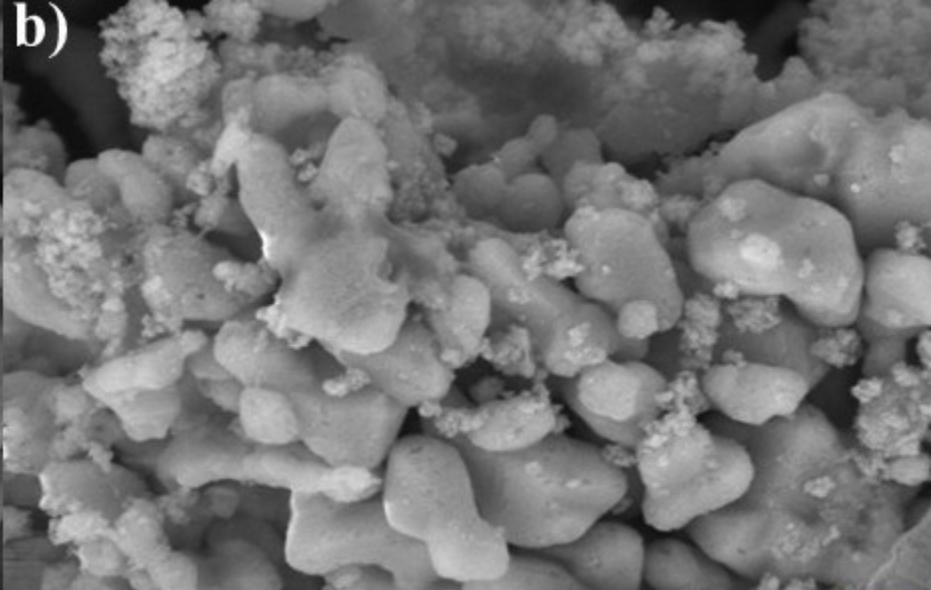
$[F(R)hv]^2$



a)



b)



c)

