Thermal-induced effects on the structural and photocatalytic properties of Nickel Oxide nanoparticles for Indigo Carmine dye removal

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Abstract

In this study, NiO nanoparticles were formed using the chemical precipitation method, and the calcination process induced structural parameters and optical bandgap. The research was carried out using a variety of techniques such as XRD, FTIR, and SEM. The XRD analysis reveals that the formed NiO crystallized in an fcc crystal structure, and that the calcination process influences the microstrain, dislocation density, and average surface area. The optical bandgap and crystal structure parameters are significantly affected by increasing the calcination temperature and/or time. The formation of a Ni–O stretching vibration mode is revealed by FTIR, and the broadness of the absorption band confirms that the NiO samples are nanocrystals. The morphology of the prepared NiO reveals the formation of spherical nanoparticles for samples calcined at 700 °C and dodecahedron-like shapes for samples calcined at 800 and 900 °C. Depending on the calcination temperature and time, the optical band gap ranged from 3.33 to 3.71 eV. The photocatalytic performance of NiO nanoparticles as catalysts for the degradation of indigo carmine (IC) dye is investigated under UV-visible irradiation for up to 3 h. The best degradation efficiency was found to be 76% for NiO calcined at 800 °C for 4 h, which revealed the smallest crystallite size of 19.13 nm, and the highest surface area of 47.02 m²/g. A mechanism for the degradation process was proposed, and the reaction rate constants for various NiO catalysts were estimated, with the maximum value of 4.51×10^{−3}/min calculated for NiO calcined at 800 °C for 4 h.

1. Introduction

Nanomaterials made from metal oxide semiconductors have piqued interest due to their unique features and potential applications in a variety of fields, including microelectronics, detection, green medicine, and biomedicine [1–14]. The properties of these resources are typically influenced by their size and structure [1, 2], prompting interest in the development of semiconducting materials with a variety of structures, for example, nanobelts and nanowires, as well as nanospheres, nanoplates, and nanoflowers [3–9].

Nickel oxide (NiO) nanoparticles are mostly used in nanotechnology applications such as UV photodetector, photoluminescence, solar cells, gas sensors, light-emitting diodes, photocatalysts, and so on [8–14]. NiO is a p-type semiconductor with a bandgap of around 3.6 eV. NiO has been identified as an excellent UV-protective material at wavelengths lower than 375 nm. Also, its bandgap and associated energy are quite similar to those of TiO₂. Although colloidal deferral's photocatalytic performance has been researched in various studies, its utility as an ecological solution has not been explored or altered. The literature's conclusions compare NiO's photocatalytic efficiency to TiO₂'s photocatalytic efficiency [15–17]. NiO has the advantage of radiating exceptionally clearly in the visible region. Earlier photochemical experiments looked at the emission properties of NiO nanoparticles based on their size [18–21]. A visible emission of NiO occurs as a result of anionic vacancies, which are sensitive to hole hunters. Quantitatively, the hole foragers, such as iodide ions, quench the emission [22]. The synthesis of a novel catalyst process that can simultaneously sense and destroy harmful pigments is a desirable aspect for decontaminating air and water. The photocatalysis process is activated when the system detects an increase in the presence of aromatic compounds in the environment. NiO is certain to be the best candidate for such biological applications. The sensing properties of NiO have been the focus of a lot of research in recent decades [23–25]. Whereas NiO is a photocatalyst with restricted efficiency due to the wedding band gap, its photocatalytic efficiency in the visible part of the solar spectrum is ineffective [26]. The calcination of the material at various temperatures can improve photocatalytic activity and boost sensing applications due to the increased uniformity.

Researchers have been exploring the optical, magnetic, and photocatalytic properties of pure and doped NiO with transition metals such as Co, Cd, Mn, Cu, Cr, V, and Fe, as well as Re elements, for decades. Abo Zeid et al., found that Cd-doped NiO nanoparticles improved the discoloration of methyl orange [27], and Junaid et al. presented a ferromagnetic material made of Fe-doped NiO at ambient temperature [28]. Al Boukhar et al., found a modest blue shift in the optical spectra of Re-doped NiO samples when compared to pure NiO, implying that the optical bandgap has increased. They are investigating the magnetic characteristics of NiO nanoparticles at room temperature and have shown that antiferromagnetic and weak ferromagnetic ordering coexists in both pure and RE³⁺-doped NiO nanoparticles [29]. The addition of Mn to the NiO lattice increases the magnetic characteristics substantially, according to Samar and Verma [30]. Cuong et al., also, improved the catalytic activity of Co-doped highly porous NiO nanorods [31]. The production of NiO nanoparticles and their structural characteristics were shown to be highly dependent on the calcination temperature by Lay et al., [32]. Anita et al., examine the effects of calcined temperatures of 350°C, 450°C, and 550°C for 3 h on phase formation, particle size, and bandgap evolution on Cu-doped NiO powder samples as-prepared. The particle size of the nanoparticles increased from 4 nm to 9 nm as the calcination temperature was increased from 350°C to 550°C for the Cu doped NiO samples [33]. Alshatwi et al., discovered that increasing the calcination temperature improves the morphological and optical features of Fe doped NiO samples and causes them to crystallize. It has also been demonstrated that the volume of the samples has increased, resulting in a modest bandgap [34].

The effects of applying various calcination temperatures 700, 800, and 900°C and various durations 2, 3, and 4 h on the structural characteristics of NiO nanoparticles and photocatalytic efficiency in eliminating indigo carmine (IC) dye from wastewater are investigated in this study. For enhanced photocatalytic performance, the optical band gap is regulated by the calcination temperature and time. After the structural parameters have been researched and the optical band gap has been determined, the photocatalytic degradation mechanisms are investigated based on Langmuir-Hinshelwood kinetics.

2. Experimental Procedures

To make pure NiO nanoparticles, dissolve 0.5 M nickel nitrate, Ni(NO₃)₂·6H₂O, in 100 ml double distilled water for 30 min while stirring. A fresh solution of potassium hydroxide, KOH (1.5 M diluted in 100 ml deionized water) was supplied dropwise to a nickel solution under continuous stirring at 60°C for 2 h as a precipitating agent. The Ni(OH)₂ green precipitate was filtered and washed with distilled water and 98% ethanol many times. The produced sample was

dried overnight in an air oven at 100°C before being pulverized several times. To develop varied crystallite sizes of NiO nanoparticles and increase the crystallinity of NiO, the calcination operation was carried out in a muffle furnace at 700, 800, and 900°C for 2, 3, and 4 h.

To evaluate the structural characteristics of NiO nanoparticles, XRD measurements were done using a Shimadzu X-600 (Cu-K α , $\lambda = 1.54 \text{ \AA}$) from Japan. A scanning electron microscope was used to examine the morphology of the samples (SEM-JSM 6360 LA, Japan). Fourier-transform infrared spectroscopy, or FTIR, spectra were obtained in the 400–4000 cm $^{-1}$ range using Thermo Scientific Nicolet iS50 FTIR equipment to analyze the formed bond in NiO nanoparticles. With an ultraviolet-Visible near-infrared spectrophotometer, the absorption spectra of ultraviolet-visible light through suspension solution of NiO were recorded (model UV-3600).

Due to the high environmental importance of the treatment process of water pollutants, the photochemical decomposition of the IC was chosen to evaluate the photodegradation efficiency using the prepared NiO, and then select the most active sample that achieves the highest degradation rate in a shorter time. Xenon lamp, purchased from Engineering company, limited –ARE, was used as a light source in the procedures of the degradation experiments. The measured received intensity on the catalyst samples was 70 W/cm 2 . The first step in the process is the formation of a suspended solution by addition of 100 mg of the prepared NiO catalyst into 100 ml of IC dye with selected concentration $8 \times 10^{-6} \text{ M}$, then it is stirred for 45 min to achieve complete adsorption equilibrium between NiO nanoparticles sample and the IC dye. The second stage of the work includes the process of irradiating the previously prepared suspension solution for 3 h with continuous stirring to avoid the accumulation of the photocatalyst and thus reduce the chances of any side effects of the destruction process. During the process of exposure to light, 5 ml of the mixture is taken at fixed interval times of 30 min. The efficiency of the photocatalytic activity of the first sample is carried out by following the decrease in the absorption value of the organic dye using spectrophotometer model

Shimadzu-UV260 Japan, and its percentage is calculated from the following formula: $\eta(\%) = \left(\frac{C_o - C_t}{C_o} \right) \times 100$, where η is the degradation rate percent

while C_o and C_t are the concentration or absorption values of IC dye before and after the irradiation process, respectively. For the remaining eight NiO samples, the same steps are repeated, and then we compare the obtained results to reach the ideal sample of the photocatalyst.

3. Results And Discussion

3.1. Structural and electronic parameters

Figure 1 shows the XRD charts for NiO nanoparticles heat-treated at temperatures of 700, 800, and 900°C for 2, 3, and 4 h, respectively, as indicated in Figs. 1a, 1b, and 1c. In our prior research, we investigated the structural parameters of NiO as it was prepared [27]. The NiO nanoparticles are normally crystalline, and their crystallinity varies depending on the calcination temperature and/or duration. For all calcination temperatures or times, the observed diffraction peaks belong to the face-centered cubic (fcc) crystal structure of NiO nanoparticles, which is well-matched with the standard JCPDS Card No: 04-0835 [31]. The diffraction peaks are found at $2\theta = 37.22^\circ, 43.33^\circ, 63.06^\circ, 75.37^\circ$, and 79.38° , respectively, corresponding to the Miller indices of (111), (200), (220), (311), and (222).

The existence of the typical diffraction peaks of NiO in the fcc crystal structure form may be seen in XRD charts of calcinated NiO. The well-known Debye Scherrer equation: $D = \frac{0.9\lambda}{\beta \cos \theta}$, was used to calculate the average crystallite size (D) of the produced phase, where λ is the wavelength of radiation employed in Cu K α ($1.5406 \text{ \AA}$), β is the full width at half-maximum (in rad), and θ is the angle at the maximum peak's position (in rad). The microstrain (ϵ) was also calculated using the Williamson-Hall method [35]: $\beta \cos \theta = \frac{0.9\lambda}{D} + 4\epsilon \sin \theta$. The plot of $\beta \cos \theta$ versus $4\epsilon \sin \theta$ yields straight lines and the value of the microstrain for the NiO phase in the analyzed samples may be calculated from the slope value of the straight line. Furthermore, the presence of dislocation density has a significant impact on crystallographic characteristics. The relationship between crystallite size and dislocation density (δ) is: $\delta = \frac{1}{D^2}$. Table 1 summarized the results for crystallite size, microstrain, and dislocation density for the investigated NiO nanoparticles.

Table 1
Summarize the crystallite size (D), micro-strain (ϵ), dislocation density (δ), equivalent surface area (S), and energy bandgap (E_g) for NiO nanoparticles at various calcination temperatures and times.

Calcination time (h)	2					3					4				
Temp. (°C)	D (nm)	$\epsilon \times 10^{-3}$	$\delta \times 10^{-3}$	S (m 2 .g $^{-1}$)	E_g (eV)	D (nm)	$\epsilon \times 10^{-3}$	$\delta \times 10^{-3}$	S (m 2 .g $^{-1}$)	E_g (eV)	D (nm)	$\epsilon \times 10^{-3}$	$\delta \times 10^{-3}$	S (m 2 .g $^{-1}$)	E_g (eV)
700	25.03	-2.45	1.60	35.94	3.42	19.24	-0.03	2.70	46.75	3.71	25.79	-1.53	1.50	34.88	3.42
800	24.44	-2.45	1.67	36.81	3.33	26.94	0.37	1.38	33.40	3.42	19.13	-3.4	2.73	47.02	3.39
900	27.63	-4.44	1.31	32.56	3.43	26.64	-2.03	1.41	33.77	3.42	23.13	-2.74	1.87	38.90	3.66

Calcination at the proposed temperatures and periods reveals a variety of patterns. For example, increasing the calcination temperature from 700 to 800°C for 2 and 3 h results in a minor drop in the crystallite size, which is subsequently marginally increased for a further rise in the calcination temperature from 800 to 900°C. The crystallite size of NiO calcined at 800°C for 4 h has the smallest value that equal to 19.13 nm among the evaluated values. The equivalent surface area, 47.02 m 2 /g, has reached its maximum value. The grain size is increased by agglomeration between neighboring NiO nanoparticles,

whilst the crystallite size is reduced by the particle divider to smaller particles. The change in micro-strain is inversely proportional to the change in the crystallite size. As seen in Table 1, the number of dislocation lines per unit area inside nanoparticles reduces as particle size increases. The estimated values of the equivalent surface area for various NiO nanoparticles are tabulated in Table 1.

The FTIR for various NiO nanoparticles calcined at 700, 800, and 900°C for 2, 3, and 4 h is shown in Figure 2. The goal of the study is to figure out what functional group is present in pure NiO nanoparticles. The band O–H stretching vibrations are responsible for a large absorption band centered at 3440 cm^{-1} [36]. Furthermore, when FTIR sample discs were made in the open air, a faint band about 1647 cm^{-1} ascribed to the mode H–O–H bending vibrations was identified owing to water adsorption in the air [36]. Other detected peaks at 3133–3138 cm^{-1} , 1381 cm^{-1} , 1015 cm^{-1} , 882 cm^{-1} , and 829 cm^{-1} are related to the stretching vibration of the OH group in all NiO samples [37, 38]. The serrated absorption bands in the 1000–1500 cm^{-1} range could correspond to the O–symmetric and asymmetric stretching vibrations, as well as the C–O stretching vibration. Furthermore, the low intensity of this band suggests that the ultrafine powers have a high physical absorption to H_2O and CO_2 . Furthermore, these observed peaks might be generated by water content, asymmetric stretching of C=O, the vibration of adsorbed CO_2 , C–H stretching, and N–H bending mode. The Ni–O stretching also has stretching vibrations at 473 cm^{-1} , and 664 cm^{-1} [39]. These two peaks are due to the Ni–O stretching vibration mode, and the broadness of the absorption band suggests that the NiO particles are nanocrystals. Because the crystallite size of NiO in nanoparticles was significantly smaller than the crystallite size of NiO in bulk form, the IR peak of Ni–O stretching vibration in NiO nanoparticles was moved to the blue direction. Furthermore, due to the quantum size effect and spherical nanostructures, the FTIR absorption of NiO nanoparticles is blue-shifted relative to that of the bulk form.

Optical absorption qualities are known to be related to the optical energy gap (E_g). As a result, we drew the Kubelka-Munk function $F(R)$ using UV-visible diffuse reflectance: $F(R) = \frac{(1-R)^2}{2R}$, here $F(R)$ is the Kubelka-Munk function or the absorption coefficient, and R is reflectivity. The function $F(R)$ can be rewritten in the following expression: $F(R) = \frac{A(h\nu - E_g)^n}{h\nu}$, where A is an arbitrary constant, $h\nu$ is photon energy (E), and n is the probability of transition and equals 2, and $\frac{1}{2}$ for a direct bandgap, and an indirect bandgap, respectively [40]. Figures 3a–c show selected plots of the relation between $[F(R)E]^2$ and E for NiO nanoparticles calcined at 700, 800, and 900°C for 4 h. The tangent lines of the $[F(R)E]^2$ against photon energy (E), and can be used to calculate E_g , as illustrated in Fig. 3. Table 1 summarizes the values of E_g for various NiO nanoparticles that were calcinated at various temperatures and periods. NiO nanoparticles calcined at 800°C for 2 h and NiO calcined at 700°C for 3 h had the lowest (3.33 eV) and highest (3.71 eV) optical bandgap values, respectively. The optical band gap values calculated are agreed with those published by Dhanuskodi et al. [41]. As a result, the bandgap adjusts the volume fractions for a variety of applications, including photovoltaics, photocatalysis, and thermoelectric. The decreasing bandgap values of NiO nanocrystalline are linked to grain size, shape, and structural faults that can be regulated by the calcination process, it may be concluded.

Figure 4 depicts the surface morphology of the NiO nanoparticles were examined by the SEM. The morphology shape of NiO nanoparticles calcined at 700°C for 4 h revealed sphere-shaped nanoparticles composed of unevenly shaped pieces clustered together, as shown in Fig. 4a. Micrographs of NiO samples calcined at higher temperatures, such as 800°C and 900°C for 4 h, show distinct grains, which differ from the spherical nanoparticles, with increased particle size, as shown in Figs. 4b and 4c, respectively. In the case of 800°C for a 4 h sample, a dodecahedron-like shape appeared, which was attributed to the optimal temperature and time for good dispersion and agglomeration of the prepared nanostructures. These dodecahedron-like shapes, as well as some nanoparticles, are concentrated at the surface. As the number of nanoparticles increases, so does agglomeration, and as the temperature rises, so does the accumulation of grains, which reduces the dispersity of the particles at the surface. The average particle size estimated using ImageJ equal 0.25±0.05 μm , 0.75±0.1 μm , and 0.25±0.02 μm for NiO nanoparticles calcinated at 700, 800, and 900°C for 4 h, respectively.

3.2. The photocatalytic performance of NiO nanoparticles

Another goal of this research is to use it as a catalyst for treating wastewater from IC dye. The IC dye is considered one of the most famous dyes of the indigo family, which is a source of water pollution and consequently leads to many diseases such as diarrhea, nausea, and vomiting. In this study, nine different NiO nanoparticle samples are used to examine the photocatalytic performance of removing the IC dye from wastewater. For convenience, these samples are labeled A, B, C, D, E, F, G, H, and I for NiO calcinated at calcination temperatures and times of 700°C for 2 h, 700°C for 3 h, 700°C for 4 h, 800°C for 2 h, 800°C for 3 h, 800°C for 4 h, 900°C for 2 h, 900°C for 3 h, and 900°C for 4 h, respectively. Figures 5a–c show the relationship between the absorbance spectra and the incident photon wavelength through IC dye that contains NiO nanoparticles for various UV-visible irradiation times. The purpose of this examination is to evaluate the efficiency of the photocatalytic properties of selected NiO samples were prepared at calcination temperatures of 700, 800, and 900°C, and at calcination times 2, 3, and 4 h, respectively, by photodestruction process of IC dye. It is commonly observed that a peak at $\lambda = 609 \text{ nm}$ characterizes the absorbance in IC dye, and the intensity of this peak decreases as the UV-visible irradiation time increases up to 3 h. Or by other words, by fixing the light source, intensity, and UV-visible irradiation time) Xenon lamp and for 3 h), as well as the concentration of the selected dye solution, the results clearly show that the degradation rate in the presence of NiO was prepared at 700°C equal 56%, 60%, and 66% for 2 h, 3 h, and 4 h, respectively. In the next stage of the work, when the calcination temperature was increased to 800°C, a rise in the decomposition rate was observed, where the calculated values increased to 68%, 71%, and 76% for 2 h, 3 h, and 4 h, respectively. By using NiO nanoparticles with the highest calcination temperature of 900°C in the last step of the process, a significant decrease was found in the degradation rate of the dye solution, where the values are decreased to 47%, 50%, and 53% for 2 h, 3 h, and 4 h, at UV-visible irradiation time of 3 h, respectively. The NiO samples with 800°C have a superior efficiency compared to samples with 700°C due to the distinct improvement that occurs in the crystalline properties of the catalyst when the calcination temperature and time increase. For example, the crystallite size was reduced, while the active surface area was increased. This observation was confirmed by the specific energy gap values, which are given the lowest values for the photocatalyst samples that were prepared at 800°C. Also, at the excellent calcination temperature, the catalytic behavior of the catalyst develops, and its efficiency increases due to the improvement of the charge separation process, which leads to an increase in the time required to electron-hole recombination, which subsequently leads to the increased possibility of formation of effective oxidizing radicals ($\text{OH}^\cdot$ and

O₂⁻²), which is a key species in the decomposition process of environmental organic pollutants [42]. The deterioration of the catalytic efficiency of the prepared samples with 900°C compared to the samples with 800°C can be attributed to the phenomenon of the reverse effect, which occurs when the calcining temperature is raised to a certain degree, where the increase in the temperature and time of the calcination processes leads to the accumulation of nanoparticles and a decrease in the effective surface area and photocatalytic activity [43, 44].

Figure 6 depicts the photodegradation rate of IC dyes ($\frac{C_t}{C_0}$) versus UV-visible irradiation time (t) for NiO nanoparticles. The NiO nanoparticles that were calcined at 800°C for 4 h demonstrated a higher degradation rate of the IC dye. Similarly, Figure 7 depicts the removal effectiveness of IC dye as a function of UV-visible irradiation time in the instance of NiO nanoparticles. The effect of the prepared nanocompounds on the absorbance change of organic dye and the values of degradation rates at various time intervals with a time step of 30 min for each run. Figure 8 depicts capture experiments of active species in the photocatalytic degradation of IC dye using NiO catalysts after 3 h of UV-visible irradiation. The sample designated F or calcinated at 800°C for 4 h showed the most deterioration. To examine the catalytic activity of NiO nanoparticles further, the degradation rate constants of IC dye were obtained using Langmuir-Hinshelwood kinetics. The formula that can be used to compute the disintegration rate of a pseudo-first-order response is:

$$\ln \left(\frac{C_t}{C_0} \right) = -kKt = -k_{app}t \text{ [45], where } k \text{ denotes the degradation rate constant, } K \text{ is the adsorption equilibrium constant, and } k_{app} \text{ is the apparent rate}$$

kinetic constant, also known as the pseudo-first-order reaction constant. The response rate was calculated using $k_{app} = -\ln \left(\frac{C_t}{C_0} \right) / t$. Table 2

summarizes the k_{app} values determined for various NiO nanoparticles. The greater catalytic performance of the optimal sample of NiO (Sample F) was explained by achieving the highest values of reaction rate constants of $4.51 \times 10^{-3} \text{ min}^{-1}$, and the lowest values for the half-life compared with other prepared catalysts as presented in Table 2.

Table 2
The reaction rate constants (k_{app}) for several NiO catalysts for IC dye removal.

Sample No.	A	B	C	D	E	F	G	H	I
Calcination temperature (°C)	700		800			900			
Calcination time (h)	2	3	4	2	3	4	2	3	4
Degradation (%)	56	60	66	68	71	76	47	50	53
$k_{app} \times 10^{-3} \text{ (min}^{-1}\text{)}$	3.01	3.40	3.52	3.55	3.93	4.51	2.53	2.55	2.77
$E_g \text{ (eV)}$	3.42	3.71	3.42	3.33	3.42	3.39	3.43	3.42	3.66

Figure 10 presents the mechanism of NiO nanoparticles action as a photocatalyst and its effect on the photodecomposition process of organic dye. Crystal properties, surface area, energy bandgap, and particle size are the basic components of the photodegradation process that can effectively contribute to the increase in photocatalytic efficiency [46]. The photocatalytic activity is directly proportional to the surface area, which leads to more adsorption of the reactant materials [47]. We can present the degradation process in the presence of the prepared NiO samples in the form of a set of stages. The first step is to use the light source to irradiate the surface of the catalyst. As a result of the previous step, the electrons in the valence band are excited and then transferred to the conduction band. The process of electron transfer is accompanied by the formation of positive holes equal to the number of excited electrons, which gives an ideal opportunity to form a pair of positive holes- negative exciting electrons [48]. This step shows the significant effect of the energy gap value on increasing or decreasing the efficiency of the catalyst, as it is noted that the most active samples are the least in the energy gap value and thus it is the one that achieves more time for the process of re-formation of the resulting free radicals. At this stage, the most important process occurs, which is the formation of hydroxyl and oxygen radicals at the positive holes and the excited electrons, respectively. These radicals represent the basic elements in the process of photo-destruction of the organic dye indigo cochineal through their superior ability to convert the organic pollutant into environmentally friendly compounds, namely carbon dioxide and non-polluting water [42, 49]. We can express reactions and processes that occur during the photo detoxification process of IC dye as follows:

Prepared sample (NiO nanoparticles) + light source (hv) → photocatalyst with positive holes (h⁺) and excited electrons (e⁻) (1)

Excited electrons + Oxygen molecule (O₂) → Oxygen radicals (O₂^{•-}) (2)

Positive holes + water molecule (H₂O) → hydroxyl radical (OH[•]) (3)

Organic dye (IC) + two produced active radicals → environmentally friendly compounds (CO₂ and non-polluting water) (4)

4. Conclusion

The chemical precipitation method was successfully used to synthesize NiO nanoparticles in this study, and then their structural parameters and optical bandgap are controlled by the calcination process. The XRD analysis reveals that the formed NiO crystallized in an fcc crystal. The calcination temperature and/or time has a significant effect on the optical bandgap and crystal structure parameters such as the microstrain, dislocation density, and average

surface area. FTIR reveals the formation of a Ni–O stretching vibration mode, and the broadness of the absorption band confirmed that formed NiO powders are nanocrystals. The morphology of the prepared NiO reveals the formation of spherical NiO nanoparticles at 700°C, meanwhile dodecahedron-like shapes at 800 and 900°C. The optical band gap varied from 3.33 to 3.71 eV depending on the calcination temperature and time. Under UV-visible irradiation for up to 3 h, the photocatalytic performance of NiO nanoparticles as catalysts for the degradation of IC dye was investigated. The best degradation efficiency was discovered to be 76% for NiO nanoparticles that were calcined at 800°C for 4 h, because of its smallest crystallite size (19.13 nm) and the highest surface area (47.02 m²/g). A mechanism for the degradation process was proposed, and the rate of degradation for various catalysts was estimated.

Declarations

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Figures

Figure 1

XRD charts for NiO nanoparticles calcined at (a) 700 °C, (b) 800 °C, and (c) 900 °C for 2, 3, and 4 h.

Figure 2

FTIR charts for NiO nanoparticles calcined at (a) 700 °C, (b) 800 °C, and (c) 900 °C for 2, 3, and 4 h.

Figure 3

$[F(R)E]^2$ versus E for NiO nanoparticles calcined at (a) 700 °C, (b) 800 °C, and (c) 900 °C for 4 h.

Figure 4

SEM images top-view of NiO nanoparticles calcinated at (a) 700 °C, (b) 800 °C, and (c) 900 °C for 4 h.

Figure 5

UV-visible absorption spectra of IC dye as a function of λ after photodegradation of the IC dye for various times using NiO catalysts calcinated at (a) 700 °C for 2 h, (b) 800 °C for 3 h and (c) 900 °C for 4 h.

Figure 6

Plot of concentration ratio (C_t/C_0) at various irradiation times (t) of removing IC dye using NiO nanoparticles.

Figure 7

Plot of photocatalytic efficiency (η) at various irradiation times (t) of removing IC dye using NiO nanoparticles as a catalyst.

Figure 8

Capture tests of active species in the photocatalytic degradation process of IC dye using NiO nanoparticles after irradiation for 3 h.

Figure 9

Kinetic curve of IC for various irradiation times using NiO nanoparticles.

Figure 10

Schematic illustration of a photocatalytic mechanism for the degradation process of IC under UV-visible irradiation towards NiO nanoparticles.