Effect of annealing temperature on structural, optical, and photocatalytic properties of titanium dioxide nanoparticles

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ABSTRACT

Titanium dioxide (TiO₂) nanoparticles (NPs) were produced by simple sol-gel technique and annealed for 2 h in air. The impact of annealing temperature on the physical, morphological, photocatalytic, and optical properties was studied. X-ray diffraction (XRD) measurements proved that a gradual phase change from anatase to rutile occurred with increased annealing temperature. The crystal structures of the NPs were found to change at different temperatures; anatase phase at 450 °C, mixed-phase (anatase/Rutile) at 550–650 °C, and rutile phase at 750 °C. The rise in the annealing temperature improved the crystallinity of the NPs. The crystal and grain size of the NPs increased with the annealing temperature hence reducing the specific surface area due to the condensed boundaries between subunits of the NPs. As per the Kubelka-Munk equation, the bandgap reduced as the temperature elevated. The photocatalytic activity of the NPs was higher in the anatase/rutile mixed-phase than in the single anatase and rutile phase. Experimental results indicated that annealing temperature could effectively change the properties of the TiO₂ NPs.

1. Introduction

Titanium dioxide (TiO₂) is considered an appealing nanomaterial due to its versatility. TiO₂ is polymorphic and exists in three major crystalline structures in nature; anatase with a refractive index of 2.49, brookite with a refractive index of 2.58, and rutile with a refractive index of 2.61 [1,2]. It also exists in mixed phases of rutile-anatase, anatase-brookite, and anatase-brookite-rutile phase [3]. Suggestions show that nanomaterial application depends highly on the crystal phase, morphology, and crystal size [4]. Rutile and anatase phases have been widely reported in literature due to their stability, unlike the metastable anatase and brookite phases. The metastable phases are found to transform to the rutile phase at different conditions, such as high annealing temperatures [11]. A.K.M. Muaz et al. annealed thin films at 300 °C, 500 °C for 1 h and observed a slight transformation from anatase to rutile at 500 °C. A.S Bakri et al. [12] suggested that anatase/rutile phase transformation occurred at temperatures greater than 900 °C after annealing thin films for 1 h. Nanopowders also annealed at 600 °C, 700 °C, and 800 °C for an hour by S. Mathur et al. showed phase transformation at which the rutile phase dominated at 800 °C. According to M.K. Singh et al. [13], there was a total anatase to rutile phase transformation at 800 °C. It is clear that the influence of the annealing temperature on the TiO₂ properties have not been exhaustedly studied. The intention for this study was to examine the effect of annealing for 2 h at (450 °C, 550 °C, 650 °C, 750 °C) on the physical, morphological, and photocatalytic properties of the synthesized TiO₂ NPs by sol-gel technique. The photocatalytic activity was tested by degrading methylene blue using artificial...
UV light. Hence being able to suggest from our study the best photocatalyst for water purification.

2. Methods

2.1. Chemicals

All of the reagents used were of analytical grade purity and were used precisely as supplied by the manufacturer. Tetra isopropyl orthotitanate (C₄H₉O₅Ti) purity >98% from Aldrich was used as titanium precursor, ethanol absolute (C₂H₆O), purity (>99.9%) as a solvent medium, and ethanolamine as a catalyst.

2.2. Synthesis procedure

TiO₂ NPs were prepared by the sol-gel method with 11 mL of tetra isopropyl orthotitanate (TIP) and 62.8 mL of ethanol. After making a solution and vigorously stirring for 2 h (h), 10 mL of (ethanol + deionized (DI) water) was added dropwise under agitation. After that, the sol was aged for 24 h, and the formed gel dried at 130 °C for 2 h. Finally, the samples were annealed at 450 °C, 550 °C, 650 °C, and 750 °C for 2 h to obtain the desired NPs. To allow the reusability of the TiO₂ photocatalyst, the powder was converted into pellets.

2.3. Photocatalytic test

The samples obtained at different thermal treatments were photocatalytically tested using methylene blue dye under UV light radiation. To achieve the adsorption-desorption equilibrium, 2 g/L TiO₂ pellets were suspended in methylene blue solution and continuously stirred for 0.5 h. The UV light was used to irradiate the samples in a normal direction by placing it above the samples. The degradation of MB was carried out for 2 h, with a close track by taking a 5 mL sample every 0.25 h using the Shimadzu-1700 UV-vis spectrophotometer. MB degradation was estimated by adsorption peak at 664 nm [14].

2.4. Characterization of the samples

Philips Bruker D8 advance X-ray diffractometer, Cu Kα X-ray (λ = 1.5406 Å) was used at 40 kV, 30 mA, at a scanning range of 20–60° to obtain the crystal structure and the phase of the synthesized TiO₂ NPs. Debye-Scherer's equation was used to calculate crystal sizes [15]. Identification of the crystalline phase was made based on the Joint Committee on Powder Diffraction Standards (JCPDS). Morphology and the composition of the NPs were investigated using Tescan Vega 3 Scanning Electron Microscopy (SEM) and Oxford X-Max Energy Dispersive Spectroscopy (EDS), respectively. The diffuse reflectance was done using a Lambda 950 UV-vis spectrophotometer. MB degradation was estimated by adsorption peak at 664 nm [14].

3. Results and discussion

3.1. XRD analysis

The X-ray diffraction (XRD) patterns of all the samples, annealed at different temperatures, were depicted in Figure 1. The peaks were referenced according to #861157 (anatase) and #860147 (rutile) phase. After annealing at 450 °C for 2 h, anatase peaks emerged with a small brookite peak observed. The optimal peak intensities of the anatase phase were observed at 550 °C thermal annealing, after which 650 °C temperature caused a drastic decrease in intensities with an appearance of the peaks from the rutile phase. As the temperature increased, mixed anatase-rutile peaks were observed, with a small peak from brookite detected between 550 °C and 650 °C. Thus, the total transformations to the rutile phase occurred at 650 °C. During this change from (anatase to rutile), there was a phenomenon of polymorphs reconstructing due to the assembling of the atoms and the wide-range reorganization of the bonds of the particles [10]. Therefore, the phase transformation due to varied annealing temperatures was as shown in Eq. (1).

Amorphous →Anatase →anatase/rutile →Rutile

The mole ratios of the anatase and rutile phases were calculated based on the Spurr and Myers Eqs. (2) and (3) using the most intense peak of each phase [5].

\[W_R = \frac{1}{1 + 0.8(I_A/I_R)}\]  

\[W_A = 1 - W_R\]  

Wᵣ and Iᵣ represent the mole fraction and intensity of the rutile phase while Wₐ and Iₐ are the mole fraction and intensity of the anatase phase. The phase content of anatase (101) and rutile (110) was obtained using Eqs. (4) and (5) [16].

\[\text{Anatase}\% = \frac{100 \times I_A}{I_A + 1.265 \times I_R} \tag{4}\]

\[100 - A\% = \text{Rutile}\% \tag{5}\]

Iₐ and Iᵣ represent the intensity of the anatase (101) and rutile (110) phases. When a comparison was made between the two methods, a slight difference was found, associated with each technique's nature. However, the result obtained portrayed a similar trend and was very near, as shown in figure 2(a) and (b) [17].

The effect of thermal treatment was depicted in Figure 2 based on the mole fraction and phase content of anatase and rutile phase. It is observed that both phases are reciprocal to each other. Therefore, we can suggest that anatase content decreases with an increase in annealing temperature while rutile content increases. The Debye-Scherrer equation [15] was used to estimate the average crystal size (D) of the TiO₂ nanoparticles.

\[D = \frac{0.9λ}{βcosθ} \tag{6}\]

β represents the full width at half maximum, λ is the X-ray wavelength (1.506 Å), while θ is the diffraction angle.

According to Figure 3(a), the crystallite sizes are inversely proportional to the FWHM. However, Figure 3(b) shows that the peak intensities of the TiO₂ nanoparticles increase with the rise in annealing temperature, which was an indication of improved crystal quality [18, 19].
A relation was identified between peak intensity, crystallite size, and the FWHM as given by \( \text{Crystallite size} \propto \frac{\text{Peak intensity}}{\text{FWHM}} \). The crystal growth was attributed to thermally promoted crystallite growth. This parameter (annealing temperature) improves the crystalline structure by rearranging the atoms increasing the particle size of the TiO\(_2\) nanoparticles [20]. There was a slow growth rate of particle sizes at lower temperatures, whose growth speed increased at higher temperatures. The rate at which crystal growth took place can be estimated using Eq. (7) [21].

\[
V = D \cdot \nu \left[ \exp\left(-\frac{Q}{RT}\right) \right] \left[ 1 - \exp\left(\frac{\Delta f_V}{RT}\right) \right]
\]  

where \( D \) is the crystal size, \( \nu \) is the frequency of the atoms’ mutation, \( Q \) is the atoms’ activation energy that attaches itself to the growing phase after leaving its network, and \( \Delta f_V \) is the molar free energy difference between the two phases. Eq. (7) can be modified in cases of non-crystalline materials to form Eq. (8).

\[
V = D \cdot \nu \left[ \exp\left(-\frac{Q}{RT}\right) \right]
\]  

This is because \( \Delta f_V \) is much greater than \( KT \). At low annealing temperatures, the activation energy is too high hence reducing the growth rate. While at a high annealing temperature, the initiation energy is too low, increasing the growth rate. According to the crystal size obtained using the Debye-Scherer equation, the difference in the anatase and rutile crystal size revealed that the nucleation and growth of the rutile phase would be initiated between 550 °C and 750 °C [21]. Nevertheless, the rate of phase transformation from anatase to rutile is too low for nanosized crystals due to the high surface area energy [22]. It has been mainly reported that the initiation and end temperature that causes the transformation of phases is between 600 °C and 900 °C [8]. It can be assumed that rutile growth starts right after nucleation. At 750 °C there is a complete elimination of the anatase phase and what is left is only the larger rutile crystals [23]. The specific surface area (SSA) was calculated by considering the total area covered by the crystals in unit mass. This was done with the help of Eq. (9), according to [16].

\[\text{SSA} = \frac{6 \times 10^3}{\rho D} \]  

The density (4.23 g cm\(^{-3}\)) of TiO\(_2\) nanoparticles was denoted by \( \rho \), while \( D \) denotes the crystals size obtained by the Debye-Scherrer equation. The crystallographic defects or irregularities formed during crystal formation give rise to dislocations. Therefore, dislocation density \( (\rho) \) is the length of the dislocation lines per unit volume of the material crystal calculated using Eq. (10) [16].

\[\rho = \frac{1}{D} \]  

Crystalline per unit surface area (N) is also an essential structural parameter determined using Eq. (11) [16].

\[N = \frac{d}{D} \]
Figures 4(c) and (d) show that by increasing the annealing temperature on the TiO2 nanoparticles, dislocation density and crystallite per unit surface area decreased. This is an indication that the temperature rise influences a decrease in dislocation density in crystallite sizes. Annealing temperature can produce a subtle effect on the morphology of TiO2 NPs.

Figure 4. Effect of annealing temperature on (a) and (b) crystal size and SSA, (c) and (d) crystals per unit surface area and dislocation density of TiO2 anatase (101) and rutile (110) peaks.

Figure 5. SEM images of samples at (a) 450 °C, (b) 550 °C, (b) 650 °C, and (c) 750 °C annealing temperatures.
3.2. SEM analysis

According to Figure 5, the annealing temperature encourages grain size growth [26]. However, aggregation was evident in the samples and was more pronounced at the annealing temperature of 750 °C. The particle sizes that can be recognized on a nanometer-scale are the primary particles, and their agglomerations are termed secondary particles. The agglomerations might be due to the overlapping of the small and medium particles, which have been caused by various kinds of forces such as weak surface force (soft agglomerates), strong chemical bonds (hard agglomerates), and lack of water on the surface structures [16, 27].

The annealing temperature interferes with the crystal structure and the material's morphology, which affects the specific surface area. These SEM results agree with the results obtained from XRD analysis that the crystal sizes increase with raised annealing temperature.

3.3. EDS analysis

Figure 6 displays the elemental composition of the synthesized material with some carbon traces [19]. The presence of a carbon trace in the results may indicate contamination from carbon tape during the measurements. The sample annealed at 450 °C has an atomic % of 33.88 (Ti) and 66.12 (O) near Ti and O's theoretical yields, 33.33% and 66.67%, respectively. The EDS results confirm the formation of TiO$_2$ as in XRD and SEM [21]. Figure 7 displays the effect of annealing temperature on the Ti and O elements of TiO$_2$ obtained from the EDS measurements. At about 675 °C, the ratios are stoichiometric, which agrees with XRD findings for the transformation of anatase to rutile phase, which begins at 650 °C. The findings show that the ideal temperature to achieve the TiO$_2$ anatase phase was between 450 °C and 650 °C. It is observed from Table 1 that with an increase in annealing temperature, the titanium proportion increase from 23.52 to 88.53 at% while the oxygen proportion decreased from 76.48 to 11.47 at%. This revealed that oxygen vacancies were driven and moved to Ti$^{3+}$ ions, creating additional Ti$^{3+}$ species, increasing oxygen vacancies' concentration as the calcination temperature raised [28]. Because Ti$^{3+}$ ions are usually produced by the transfer of electrons trapped in oxygen vacancies to neighboring Ti atoms, Ti$^{3+}$ ions in TiO$_2$ also indicate the presence of oxygen vacancies in the material. Using standard Kroger-Vink notation [29], the equilibrium in Eq. (12) can describe the formation of the oxygen vacancies at increased temperatures:

$$O_2 \rightarrow TiO_2 + \frac{1}{2}O_2(g) + 2e$$

The equilibrium constant of the reaction can be expressed as in the equation below [28].

$$k = |V_2| \times P(O_2)^{1/2}$$

According to the transformation of Eq. (13) oxygen vacancy concentration can be stated as a function of, $P(O_2)$ [28] in Eq. (14).

$$[V_2] = Kn^{-2}P(O_2)^{(-1/2)}$$

Figure 6. EDS Spectra for samples annealed (a) 450 °C, (b) 550 °C, (c) 650 °C, and (d)750 °C annealing temperatures.

Figure 7. Elements percentage of Ti and O of samples annealed at 450 °C, 550 °C, 650 °C, and 750 °C temperatures.
where $O_0$ denotes the lattice oxygen, $V_0$ vacancies, $[V_0]$ represents the oxygen vacancy concentration; $P(O_2)$ designates the pressure of the oxygen vacancy. A decrease in the pressure of $O_2$ as seen in Eq. (14), oxygen vacancy concentration increases, such that in conditions with exhausted oxygen, annealing temperature would enable the creation of oxygen vacancies. Due to Eq. (12) reversible condition when TiO$_2$ is exposed in the air at ambient temperature, we consider doping in our subsequent work with ions’ acceptor-type to stabilize the oxygen vacancies.

### 3.4. Optical analysis

Figure 8(a) depicts diffuse reflectance spectra with a wavelength between 600 and 200 nm of all samples synthesized by the sol-gel method. There was an elevation in the percentage reflectance of the nanoparticles from 36.75 % at an annealing temperature of 750 °C to 58.87 % (highest) at 550 °C. The Kubelka-Munk formula $F(R)$ in Eq. (15) was used to estimate the bandgap of the TiO$_2$ [30].

$$F(R) = \frac{(1 - R)^2}{2R}$$

A graph of $(F(R) \cdot h\nu)^2$ vs $h\nu$ was plots to obtain the bandgap of the synthesized NPs as shown in Figure 8(b), where $h\nu$ represents photon energy. Extrapolating a line from the curve’s highest slope to the x-axis yields the bandgap [31]. As the annealing temperature rised, the crystal size increased resulting in a decrease in the bandgap hence approving the XRD analysis. A large crystal size gives a smaller bandgap, and vice versa is true [19]. The estimated bandgap ranges from 3.05 to 3.21 eV.

Higher annealing temperatures yield larger crystal sizes that influence the nanomaterial’s specific surface area, affecting the light scattering. However, crystal sizes and the nature of phases play a serious role in adjusting the bandgap. Reduced interatomic spacing and structural changes have been responsible for lowering bandgap energies as annealing temperature rises. The thermal strains and the quantum size impact experienced in the annealing system greatly influence the bandgap energies. The reduced bandgap energies at raised annealing temperatures confirm the increase in grains as observed in SEM images.

### 3.5. Photoluminescence analysis (PL)

The generated electron-hole pair in the TiO$_2$ nanoparticles get trapped in the defect sites as they move to the surface for a redox reaction, enhancing their lifetime. It has been reported that the shallow traps formed by the defect states in the TiO$_2$ are advantageous in the electron-hole separation by promoting diffusion to the surface, while the deep traps enhance the recombinaction of the carriers [32]. Figure 9(a) shows the intense PL emission around 391 nm from all the synthesized samples excited at a wavelength of 340 nm in ambient temperature. The peak intensities were observed to decrease at raised annealing temperature, attributed to the structural transformation of TiO$_2$ because of the rise in order (in a medium-order range) and the tensile stress experienced by the nanomaterials [33]. Deconvolution was done based on pseudoVoigt fit for the sample at 750 °C annealing temperature yielding 380 nm, 391 nm, and 347 nm emission peaks as shown in Figure 9(b). The peak at 380 nm was credited to direct electron-hole recombinaction from the valence band to the conduction band of the nanomaterial. The other two peaks were ascribed to the defect states attributed to the transfer of charges from the Ti$^{3+}$ to the oxygen anion in the TiO$_2$ associated with the oxygen vacancies. It is somewhat understandable considering that high temperatures give rise to oxygen vacancies that facilitate the formation of the Ti$^{3+}$ when the anatase phase transforms fully to the rutile phase [34]. Charge carrier lifetime is one of the critical factors that highly influence the photo-activity of the TiO$_2$ nanoparticles depending greatly on the defect levels. The charge recombination phenomena are better studied.
using time-resolved photoluminescence (TRPL). The decay curves of all samples were fitted using the single exponential function in Eq. (16)

\[ I = I_0 \exp \left( -\frac{t}{\tau} \right) \]  

where \( I \) and \( I_0 \) are the intensities at time \( t \) and zero milliseconds, respectively, and \( \tau \) is the lifetime. The Eq. (16) suggests carrier trapping sites of varying energy levels, resulting in a carrier transport rate distribution. Although the morphologies and the crystal sizes of the TiO\(_2\) NPs were altered during the annealing process, the observed change of the visible lifetime on the millisecond time scale could not be attributed to particle size changes or particle morphology. According to Figure 9(c), the decay time increased with the annealing temperature and decreased at 750°C as summarized in top-right insets. Low annealing temperature reduces the non-radiative recombination of the carriers due to the defects introduced during the synthesis hence increasing the decay time. The observed decrease at 750°C was caused by the aggregations on the particles and the rutile formation at raised annealing [35], leading to an increased recombination process.

3.7. FTIR analysis

FTIR technique was used to analyze chemical functional groups and chemicals present in the samples. Figure 9(d) depicts the FTIR spectra of TiO\(_2\) NPs obtained at the range of 4000–400 cm\(^{-1}\). The absorption band observed at 1639 cm\(^{-1}\) and 1387 cm\(^{-1}\) are characteristic of O–H of water's bending vibration mode associated with surface adsorbed water [37]. NPs with a large surface area can absorb a large amount of water at specific active sites on the surface, resulting in the formation of NPs Ti\(^{4+}\) OH\(_2\) on the surface. The adsorbed water molecules produce a significant amount of heat, which aids in transforming TiO\(_2\) from anatase to rutile. As the annealing temperature increases, the breakdown of the H–O–H bonds attached to Ti\(^{4+}\) occur thus the negatively charged O\(_2^-\) participates in the formation of new Ti\(^{4+}\) bonds. Therefore, water molecules bound to Ti\(^{4+}\) at the interface and on the surface dissociate, forming new Ti–O bonds at the interface. The stretching vibrations with a shoulder extending from 1045-400 cm\(^{-1}\) are related to Ti–O–Ti and Ti–O stretching bands, which are the characteristic peak of TiO\(_2\) [38]. The fingerprint region around 1500 cm\(^{-1}\) ascribed to the inter-atomic vibration is generally associated with the metal oxide absorption band [39]. The increased surface area of the TiO\(_2\) nanoparticles due to the annealing temperature reduces the surface available for water adsorption. According to Figure 9(d), the intensity of the OH absorption band is found to decrease with increased temperatures. The sharpening of the metal oxide's characteristic peaks shows that the crystalline nature of TiO\(_2\) increases with an increase in annealing temperature.

3.8. Photocatalytic analysis

An evaluation of the samples’ photocatalytic activity at various temperatures was carried out by the degradation of methylene blue (MB) under artificial UV light. Figure 10(a) shows the MB degradation process using TiO\(_2\) photocatalyst annealed at constant temperature and UV radiated at different times. According to the results, it was observed that the concentration of MB decreased with prolonged UV light exposure.
time. This was due to the change in the delivery of oxidation radicals from the irradiated NPs, continuously reducing the concentration of oxidizable MB molecules [40]. The degradation of MB can be understood from the beer-lamberts law, where the absorbance obtained from UV is taken to be directly proportional to the MB concentration. The MB efficiency was shown in Figure 10(b), whereby the anatase-rutile mixed-phase had the highest degradation %. However, the photodegradation of MB was assumed to follow the Langmuir-Hinshelwood kinetic model, and its first-order kinetics were expressed according to [41] Equ. (17)

\[
\ln C_A / C_i = kt
\]

(17)

where \( C_A \) is the initial concentration, \( C_i \) is the degraded MB concentration at time \( t \) (min), and \( k \) is the photocatalytic reaction rate.

According to Figure 11(a), the photodegradation rate constants obtained from the slope of the drawn straight line are 0.01266, 0.02434, and 0.01061 for 450 °C, 650 °C, and 750 °C respectively. It shows that the mixed phase’s photo-activity was much higher than that of the single anatase and rutile phase. However, the activation energy of the dye was estimated by Equ. (18) [42].

\[
\log k = -\frac{E_{act}}{2.303RT}
\]

(18)

where \( k \) is the rate constant, \( E_{act} \) the activation energy, \( R \) is the constant \((8.31 \text{ J/mol} \cdot \text{K})\) of the gas, while \( T \) (300K) is the temperature at which the reaction continued. The \( E_{act} \) of 450 °C, 650 °C, and 750 °C were 10,899.89, 9269.20 and 11,340.57 J/mol, respectively. Sample annealed at 650 °C has the highest efficiency, which requires the minimum energy.
of activation. The sample at 750 °C has the lowest efficiency hence requires the highest activation energy [42]. The effect of different TiO2 (mixed-phase) loading on the photocatalytic activity has been investigated, as shown in Figure 11(b). The outcomes show that increasing the amount of TiO2 improves photocatalytic activity. These findings are based on the fact that higher TiO2 concentrations provide more reaction sites for water molecule oxidation and hydroxyl radicals formation, resulting in a higher reaction rate [7]. The tests show that the 10 g/L of TiO2 loading needed further examination. The increase of the surface adsorption site causes a reduction in the time difference during the degradation. Therefore, to facilitate the total breakdown of MB dye, an increase of TiO2 concentration can completely break down various initial quantities of MB. When the amount of TiO2 concentration is increased, the time for degradation decreases since the surface adsorption sites are increased. The photocatalytic efficiency of the samples is known to be influenced by the concentration of MB. Thus, TiO2 photocatalyst has a higher absorbance of the light at low MB concentration. As a result, the photocatalytic activity of TiO2 annealed at 450 °C, 650 °C, and 750 °C were investigated in this study using different concentrations of MB over a 120-minute irradiation period as shown in Figure 11(c). As the MB concentration increased, more adsorption sites on the solid surface become visible, inhabiting the catalyst particles, and raising the amount of desorbed MB when radiated with the light. An increase in TiO2 concentration causes a more significant increase in absorbance of light at low MB concentration. The recyclability of 650-TiO2 was tested for up to four cycles. Figure 11, (d) shows that the prepared nanoparticles are very recyclable. The slight decrease in the photocatalytic activity during each cycle is due to a slight agglomeration of the NPs during the photocatalytic process. Therefore, the photocatalyst can be separated and recycled, making it a promising material for environmental remediation.

4. Conclusion

The calcination temperature is found to have a transformative effect on the TiO2 NPs. The metastable anatase phase is found to transform entirely to rutile at a temperature around 750 °C. EDS data verified the elemental composition of TiO2 NPs and that calcination in the air creates oxygen vacancies. The particle sizes and the agglomeration increase with an increase in calcination temperature. The band-gaps of the TiO2 NPs reduced from 3.21 to 3.05 eV with the rise in calcination temperature from 450 to 750 °C respectively. The PL intensities decreased with an increase in annealing temperatures due to the effect of phase transformation. The methylene blue degradation with the increase in irradiation time was highest in the mixed-phase (anatase/rutile) than in single phases, while anatase was higher than the rutile phase. Therefore, annealing temperature improves the nanomaterial crystallinity, causes phase transformation, and affects photocatalytic activity. Our study suggests that the mixed-phase obtained at 650 °C is the best photocatalyst for water purification due to its performance in the degradation of methylene blue dye.

Declarations

Author contribution statement

Dorah Kawira Muthee: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Wrote the paper; Birhanu Francis Dejene: Conceived and designed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data.

Funding statement

This work was supported by the National Research Fund, NRF S.A.

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