



Role of Fe doping on crystallographic and functional characteristics of ceramic TiO₂

Mojahid M. Najim¹, Ban A. Yousif², M. Al Nuaimi^{3,*}, Wasan A. Hekmat⁴, Saad Maadh Alhit⁵

¹Nanomaterials Research Center, University of Anbar, Ramadi, Iraq

²Department of Applied Science, University of Technology- Iraq, Baghdad 10066, Iraq

³College of Production Engineering & Metallurgy, University of Technology- Iraq, Baghdad 10066, Iraq

⁴University of Technology, College of Laser and Optoelectronics Engineering, Optoelectronics Engineering Department

⁵Nanomaterials Research Center, University Of Anbar, Ramadi, Iraq

*) Email: msr197575@yahoo.com

Received 4/5/2026, Received in revised form 25/8/2026, Accepted 23/9/2026, Published 15/10/2026

The undoped and Fe doped (0.1, 0.3, 5 wt%) TiO₂ powders (T0, T1, T3, T5) are prepared by hydrothermal synthesis. The goal is to investigate the effect of adding Fe on the crystal structure, particle morphology, elemental distribution, vibrational properties and density of the ceramic. The precursor mixtures are heated at 180 °C for 12 h, the powders are dried at 80 °C for 12 h and calcined at 500 °C for 2 h. The ceramic pellets are then created by uniaxial pressing at 250 MPa and sintering at 900°C for 3 hours. The materials are examined using X-ray diffraction (XRD), field-emission scanning electron microscopy (FESEM), energy-dispersive X-ray spectroscopy (EDS) with elemental mapping, Fourier-transform infrared (FTIR) spectroscopy, Raman spectroscopy, and Archimedes density and porosity measurements. XRD examination revealed that anatase is the most visible crystalline phase in the calcined powders. The observed average crystallite size dropped from 27.9 nm (T0) to 25.2 nm (T1), 21.8 nm (T3), and 19.3 nm (T5). FESEM analysis revealed a drop in average particle size from 82 ± 9 nm for T0 to 68 ± 7 nm, 52 ± 5 nm, and 46 ± 5 nm for T1, T3, and T5, respectively. EDS found Fe in Fe-containing materials, while FTIR showed a change in the major Ti-O absorption band from 612 cm⁻¹ (T0) to 601 cm⁻¹ (T5). The sintered T3 pellets had the highest bulk density (3.87 ± 0.02 g cm⁻³) and lowest apparent porosity (2.35 ± 0.15%), compared to 3.76 ± 0.03 g.cm⁻³ and 4.10 ± 0.20%, respectively, for T0. At 5 wt.% Fe, bulk density decreased to 3.82 ± 0.03 g.cm⁻³, while apparent

porosity increased to $3.18 \pm 0.21\%$. Fe addition affects the structural and morphological properties of TiO_2 , with 3 wt.% nominal Fe resulting in the highest densification under the sintering circumstances tested.

Keywords: TiO_2 ; Fe doping; Hydrothermal; XRD; Ceramic.

1. INTRODUCTION

Titanium dioxide (TiO_2) is a popular oxide semiconductor and ceramic material due to its chemical stability, corrosion resistance, thermal endurance, and versatile structural features. TiO_2 -based materials have been studied for sensor, protective coatings, photocatalysis and functional ceramics applications. Their properties are strongly dependent on their crystal structure, chemical composition, particle shape, and formation history. TiO_2 exists in three different crystalline forms, anatase, rutile and brookite. At room temperature, bulk TiO_2 is thermodynamically stable in the rutile phase, whereas anatase is typically produced at low temperatures. Their number and distribution depend on the precursor chemistry, particle size, synthesis temperature, dopant concentration, and subsequent heat treatment. The formation of phases must be controllable to obtain TiO_2 ceramics with reproducible microstructural and physical properties [1].

Hydrothermal fabrication is a versatile way to make TiO_2 nanoparticles at certain temperatures and pressures. This solution-based approach can lead to crystallization at temperatures lower than those required for the standard solid-state synthesis. It allows you to control the size, shape and chemical composition of the particles. It also allows dopant precursors to be added during particle synthesis, which may help to achieve a more uniform dopant distribution in the final powder. Nevertheless, the hydrothermal products are still dependent on the number of precursors, the pH of the solution, the reaction temperature and the processing time. To investigate the effect of dopant amount, the synthesis conditions must be kept constant [2].

The incorporation of iron can modify the crystal chemistry and microstructure of TiO_2 . Depending on the iron oxidation state, the method of incorporation and the processing conditions, the addition of Fe can change the local lattice distortion, defect populations, crystallite growth or phase transition during heat treatment. At higher concentrations segregation of Fe or secondary phase formation may occur. Note that the addition of Fe precursor does not change the crystal lattice to replace Fe ions for Ti ions. X-ray diffraction (XRD), Raman spectroscopy, Fourier-transform infrared spectroscopy (FTIR) and elemental analysis can be used to understand how phases form, how chemicals bond and how elements distribute. However, these methods do not allow us to determine the crystallographic occupancy or the oxidation state of Fe [3].

Nanoparticles synthesized hydrothermally are processed into ceramic pellets which give them new properties that affect the final material. The microstructure developed during the process of compression and sintering involves particle packing, interparticle diffusion, pore elimination, grain formation and probably phase transitions. Residual porosity and non-uniform grain formation can affect the bulk density and microstructural homogeneity of a material. Therefore, the properties of sintered ceramics are not really represented by the crystallinity and shape of original powders. The phase composition, microstructure, density and porosity should be studied in an effort to establish the influence of Fe concentration on the consolidation of ceramics [4].

Differences in precursor chemistry, synthesis conditions, dopant definitions and thermal treatment techniques may make it difficult to compare Fe-containing TiO₂ materials for different applications. In a targeted ceramic study, it is important to consider the effect of a controlled change in the nominal Fe concentration on the phase formation, grain structure, elemental distribution and densification while all other processing parameters remain constant. The powder structure and the pressed pellets [5] have to be discussed in this problem.

The aim of this study is to examine the effect of Fe concentrations of 0, 1, 3 and 5 wt.% on the structure and microstructure of TiO₂ ceramics produced by heating. The four compositions T0, T1, T3 and T5 are prepared by consistent methods of synthesis, calcination, compaction and sintering. Crystalline phases and structural changes have been studied by XRD and Raman spectroscopy. FTIR however gives more detailed information about vibrational and bonding properties. The morphology of particles and grains are observed by field-emission scanning electron microscopy (FESEM) and the presence and distribution of elements are analyzed by energy-dispersive X-ray spectroscopy (EDX) with elemental mapping. The degree of ceramic consolidation is measured by the bulk density and the apparent porosity. The study aims to investigate the correlations between the nominal Fe content, phase formation, microstructural development and densification by comparing these results.

The study's scope and limits include structural, morphological, compositional, vibrational, and physical characterization. The chosen approaches may identify identifiable crystalline phases, characterize microstructural changes, measure elemental distribution within EDX's spatial resolution, and estimate bulk density and accessible porosity. However, they are unable to detect Fe oxidation states, demonstrate substitutional Fe incorporation, or measure oxygen-vacancy concentrations. Furthermore, the lack of optical, electrical, dielectric, magnetic, mechanical-strength, and photocatalytic studies precludes drawing inferences regarding changes in these functional qualities. Apparent porosity measures may not include all closed pores. As a result, the data are interpreted within the limitations of the methodologies used, with focus on empirically verified correlations between Fe content, phase composition, microstructure, and ceramic densification.

2. MATERIALS AND METHODS

2.1. Materials

All chemicals used in this study are of analytical grade and are used as received without further purification. Titanium(IV) isopropoxide (TTIP, $\text{Ti}[\text{OCH}(\text{CH}_3)_2]_4$, MW = 284.22 g mol⁻¹, purity $\geq 99.9\%$) is employed as the titanium precursor for the synthesis of TiO₂ nanoparticles. Ferric nitrate nonahydrate ($\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, MW = 404.00 g mol⁻¹, purity $\geq 99\%$) is used as the iron dopant source. Absolute ethanol ($\text{C}_2\text{H}_5\text{OH}$, MW = 46.07 g mol⁻¹, purity $\geq 99.8\%$) served as the solvent for precursor preparation, while deionized water (H_2O , MW = 18.02 g mol⁻¹) is used as the reaction medium during hydrothermal synthesis. Nitric acid (HNO_3 , MW = 63.01 g mol⁻¹, 65 wt.%) is employed to modify the precursor solution to the appropriate pH of 3.5, hence regulating the hydrolysis and condensation processes. Polyvinyl alcohol (PVA, MW = 85,000–124,000 g mol⁻¹, 99+% hydrolyzed) is applied as a temporary binder during pellet manufacture. All chemicals are bought from Sigma-Aldrich, St. Louis, MO, USA, assuring excellent purity and repeatability throughout the experimental study.

2.2. Sample preparation

In this study, four kinds of TiO₂ ceramics are synthesized by adding different amounts of Fe. The undoped sample is denoted T0 and the Fe-doped samples with 1%, 3%, and 5% doping are denoted

T1, T3, and T5, respectively. The appropriate amount of ferric nitrate is weighed according to the desired Fe content and mixed in deionized water under constant stirring by magnets. A uniform precursor solution is obtained by mixing titanium(IV) isopropoxide with ethanol. Then, iron precursor solution is added slowly to the titanium precursor solution with very rapid stirring so that Fe ions are well mixed and dispersed in the solution. The solution is slowly acidified with nitric acid to a pH of about 2.2–2.3, which reduced the rate of hydrolysis and prevented premature precipitation. The precursor solution is stirred many times until a clear and uniform mixture is obtained prior to hydrothermal treatment.

2.3. *Hydrothermal synthesis*

The uniform precursor solution is poured into a Teflon-lined autoclave of stainless steel and filled until about 80% of its volume. Using dilute nitric acid (HNO_3) and continuous magnetic stirring, the pH of the solution is slowly loared to 3.5 to get it ready for hydrothermal treatment. The pH of 3.5 during synthesis favors the controlled hydrolysis of the titanium precursor, the mixing of the Fe ions into the TiO_2 lattice, and prevents the premature precipitation of the precursor. The hydrothermal process is performed at 180 °C for 12 h to control the nucleation and crystallization of the TiO_2 nanoparticles. After reaction is finished, the autoclave is cooled down to the room temperature naturally. The precipitates are collected and washed several times with deionized water and 100% ethanol to remove any remaining ions or reaction products. They are centrifuged after each wash cycle. The powders are dried in a laboratory oven at 80°C for 12 h to remove residual water. The dried powders are heated up at 5°C min⁻³ at 500°C for two hours to make them more crystallized, to get rid of any remaining organic matter and to make Fe-doped TiO_2 ceramic nanopowders that are highly crystalline and can be used to make pellets and study their properties. Figure 1 summarizes the experiments used to obtain pure and Fe-doped TiO_2 ceramic nanopowders by hydrothermal methods. The flowchart denotes the stepwise processing procedures: precursor preparation, pH adjustment, hydrothermal treatment, washing, drying, calcination, and the fabrication of TiO_2 powders with different Fe concentrations (0, 1, 3, and 5 wt.%). This systematic synthesis approach ensured good compositional uniformity and repeatability for all synthesized samples.

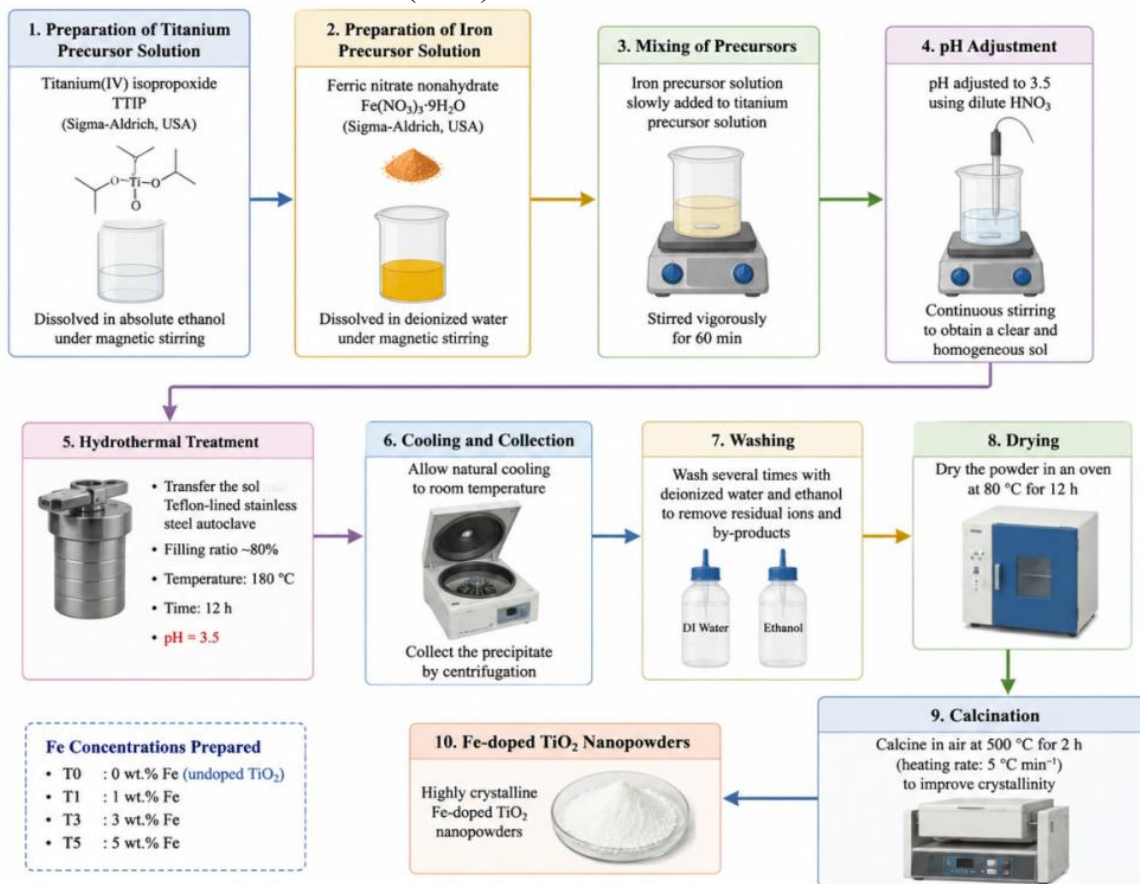


Figure 1. Hydrothermal route for the synthesis of Fe-doped TiO_2 NPs.

2.4. Pellet fabrication and sintering

The resultant nanopowders are pressed into ceramic pellets to investigate their physical, mechanical, dielectric and electrical properties. To make the granules bulkier, 5 wt% polyvinyl alcohol (PVA) solution as a temporary binder is mixed thoroughly with the granules. The powder mixes are crushed in one direction only by a hydraulic press with a pressure of about 250 MPa. This produced cylinder-shaped pellets of 13 mm diameter and 2–3 mm thickness. The green pellets are pressed and dried at 100 °C for two hours to remove moisture and any residual binder. The binder is burned out at 400 °C for 1 h and the final sintering is carried out at 900 °C for 3 h in room temperature air at a heating rate of 5 °C min⁻¹. The sintering pellets are naturally cooled down to room temperature in the furnace, which reduced the thermal stresses and produced dense ceramic samples that could be used to study their structure, mechanics, optics, electricity and photocatalysis.

2.5. Characterization Techniques

The phase composition and crystal structure of the produced $\text{TiO}_2\text{:Fe}$ powders and sintered ceramic pellets are studied using an X-ray diffractometer (XRD, D8 Advance, Bruker AXS GmbH, Karlsruhe, Germany) equipped with Cu $\text{K}\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$), operated at 40 kV and 40 mA. Diffraction

patterns are recorded using a 2θ range of $20\text{--}80^\circ$, a step size of 0.02° , and a scanning speed of 2° min^{-1} . The crystalline phases are recognized by comparing conventional diffraction patterns. Crystallite size, lattice parameters, and microstrain are calculated from diffraction data when applicable, taking into account instrumental peak widening. The morphology of the produced powders and the microstructure of the sintered pellets are investigated using a field-emission scanning electron microscope (FESEM, JSM-7610F Plus, JEOL Ltd., Tokyo, Japan) with an accelerating voltage of 5–20 kV. Representative micrographs are taken at similar magnifications to study particle shape, grain size, grain boundaries, and pore distribution. An energy-dispersive X-ray spectroscopy detector is added to the FESEM to investigate element composition and spatial distribution. EDS spectra and elemental maps of Ti, O, and Fe are obtained to determine the detectable elemental composition and distribution inside the studied materials.

The vibrational properties of the produced materials are studied using Raman and Fourier-transform infrared spectroscopy. Raman spectra are obtained using a confocal Raman microscope (inVia Reflex, Renishaw plc, Gloucestershire, UK) with a 532 nm excitation laser spanning a Raman shift range of $100\text{--}1000 \text{ cm}^{-1}$. The spectra are analyzed to identify typical TiO_2 vibrational modes and evaluate changes in phase composition and structural disorder caused by Fe concentration and thermal processing. Fourier-transform infrared (FTIR) spectra are acquired using an FTIR spectrometer (Nicolet iS50, Thermo Fisher Scientific, Madison, WI, USA) spanning the spectral range of $400\text{--}4000 \text{ cm}^{-1}$ at a resolution of 4 cm^{-1} . The FTIR spectra are studied to explore metal–oxygen vibrations and the existence of identifiable hydroxyl groups or residual chemical species. The green density and apparent porosity of the sintered ceramic pellets are measured by the Archimedes immersion method according to ASTM C373 where applicable. Deionized water is used as the immersion medium. The dry mass, saturated mass and immersed mass of each specimen are recorded using the right saturation method. The bulk density and apparent porosity are calculated from the volumes of the seen masses and the density of the immersion liquid. The relative density is calculated with a theoretical density corresponding to the phase composition found in nature. To explore the differences in densification between T0, T1, T3 and T5, the measurements are performed on three samples.

3. RESULTS AND DISCUSSION

3.1. Structural properties

3.1.1. X-ray diffraction (XRD) analysis

The X-ray diffraction (XRD) patterns of the undoped and Fe-containing TiO_2 powders (T0, T1, T3, and T5), prepared by the hydrothermal method and subsequently calcined at 500°C , are presented in Figure 2. The corresponding structural parameters are summarized in Table 1. The reported diffraction peaks at approximately $25.30\text{--}25.39^\circ$, $37.80\text{--}37.89^\circ$, $48.05\text{--}48.14^\circ$, $54.05\text{--}54.14^\circ$, $55.10\text{--}55.19^\circ$, and $62.70\text{--}62.79^\circ$ can be indexed to the (101), (004), (200), (105), (211), and (204) planes of anatase TiO_2 , respectively, in agreement with the reference pattern (PDF No. 21-1272). Anatase has a tetragonal crystal structure with space group $I4_1/amd$ (No. 141). The (101) reflection is the most intense peak in the reported patterns, consistent with the characteristic diffraction profile of anatase TiO_2 ; its higher intensity alone does not establish a preferred growth orientation [6, 7]. No additional reflections attributable to crystalline iron oxides are reported in the diffraction patterns. This observation suggests that any secondary crystalline phases are absent or present below the detection limit of the

measurement. Nevertheless, the absence of detectable Fe₂O₂ or Fe₂O₄ peaks does not necessarily prove the successful substitution of Fe into the TiO₂ lattice. Small amounts of secondary phases or weakly crystalline Fe containing species might not be detected [8,9].

The anatase (101) peak is reported to shift from around 25.30° at T0 to 25.39° at T5. Bragg's law suggests that the interplanar distance will decrease from ~3.517 Å to ~3.505 Å. The change may be due to the change of anatase lattice with the addition of Fe. However, peak shifts alone cannot determine substitutional doping since residual strain, compositional changes, and equipment alignment may all alter recorded peak locations. Refine the lattice parameters using numerous reflections to offer a more accurate evaluation of changes in the unit cell.

The reported average Scherrer crystallite sizes decrease from approximately 27.9 nm for T0 to 25.2, 21.8, and 19.3 nm for T1, T3, and T5, respectively. This trend is consistent with the progressive broadening of the diffraction peaks as nominal Fe concentration increases. It suggests that Fe addition may influence crystallite development during hydrothermal synthesis and calcination. However, Scherrer analysis estimates the size of coherently diffracting domains rather than the particle or grain size observed by FESEM. Furthermore, peak broadening can arise from both finite crystallite size and microstrain; these contributions should be distinguished using an appropriate line-broadening analysis and correction for instrumental broadening [10,11].

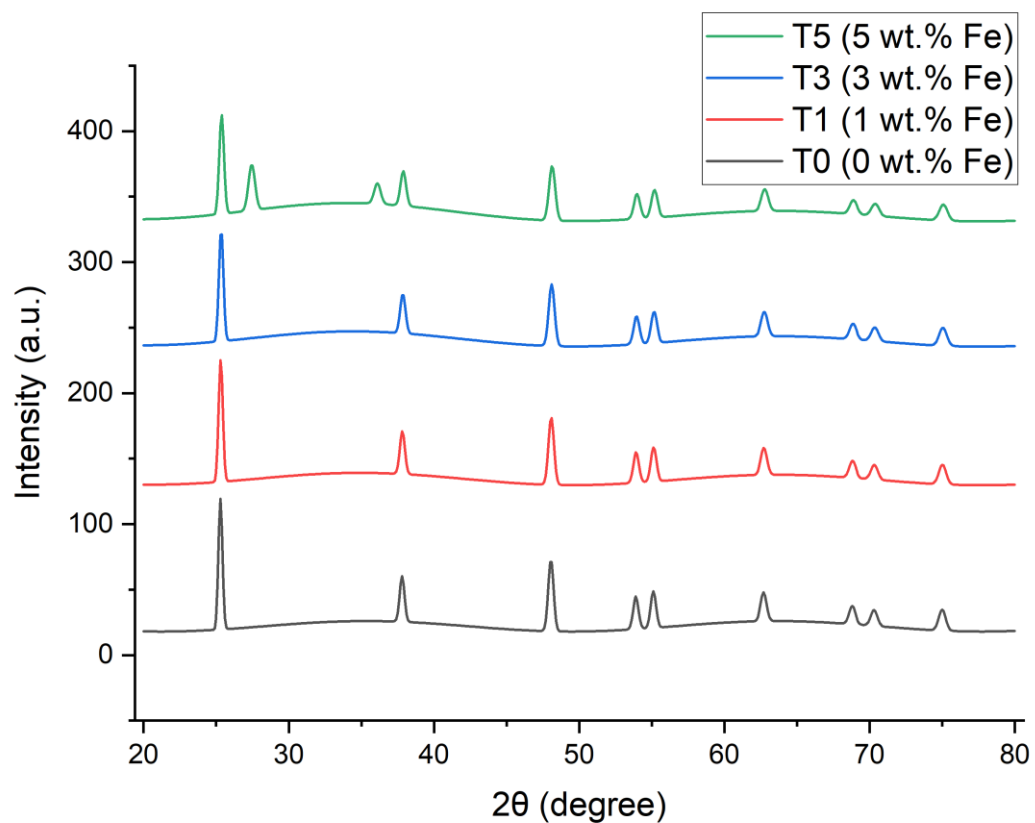


Figure 2 X-ray diffraction (XRD) patterns of hydrothermally synthesized pure and Fe-doped TiO₂ ceramic nanoparticles containing 0 wt.% (T0), 1 wt.% (T1), 3 wt.% (T3), and 5 wt.% (T5) Fe.

Table 1 Structural parameters of hydrothermally synthesized TiO₂:Fe ceramic nanoparticles obtained from XRD analysis.

Sample	2 θ (°)	FWHM (°)	(hkl)	d-Spacing (Å)	Dcs (nm)	Dcs-ave (nm)
T0 (0 wt.% Fe)	25.30	0.28	(101)	3.517	30.2	
	37.80	0.31	(004)	2.378	28.9	
	48.05	0.35	(200)	1.892	27.4	
	54.05	0.38	(105)	1.696	26.8	
	55.10	0.39	(211)	1.667	26.2	27.9
	62.70	0.42	(204)	1.481	27.8	
T1 (1 wt.% Fe)	25.33	0.31	(101)	3.513	27.4	
	37.83	0.34	(004)	2.376	26.3	
	48.08	0.38	(200)	1.891	25.0	
	54.08	0.41	(105)	1.695	24.6	
	55.13	0.43	(211)	1.666	24.0	25.2
	62.73	0.46	(204)	1.480	24.2	
T3 (3 wt.% Fe)	25.36	0.36	(101)	3.509	23.6	
	37.86	0.39	(004)	2.374	22.8	
	48.11	0.43	(200)	1.890	21.5	
	54.11	0.46	(105)	1.694	21.0	
	55.16	0.48	(211)	1.665	20.6	21.8
	62.76	0.51	(204)	1.479	21.3	
T5 (5 wt.% Fe)	25.39	0.41	(101)	3.505	20.8	
	37.89	0.44	(004)	2.372	20.1	
	48.14	0.49	(200)	1.889	19.0	
	54.14	0.52	(105)	1.693	18.6	
	55.19	0.54	(211)	1.664	18.2	19.3
	62.79	0.58	(204)	1.478	19.2	

3.2. Physical properties

3.2.1. Density and porosity

The bulk density, relative density, and apparent porosity of the undoped and Fe-containing TiO₂ ceramic pellets sintered at 900 °C for 3 h are summarized in Table 2. These parameters are evaluated to investigate the influence of nominal Fe concentration on the densification behavior of the ceramics. Bulk density reflects the mass per unit bulk volume of a specimen, whereas apparent porosity represents the fraction of interconnected pores accessible to the immersion liquid during the Archimedes measurement. Relative density is estimated using the theoretical density of anatase TiO₂ as a reference [12, 13]. The undoped sample (T0) has a bulk density of 3.76 ± 0.03 g cm⁻³ and an apparent porosity of 4.10 ± 0.20 percent. Adding 1 wt.% Fe (T1) raises bulk density to 3.81 ± 0.02 g cm⁻³ and reduces apparent porosity to $3.42 \pm 0.18\%$. Increasing nominal Fe content to 3 wt.% (T3) results in the greatest observed bulk density (3.87 ± 0.02 g cm⁻³) and lowest apparent porosity ($2.35 \pm$

0.15%) among the examined compositions. These findings suggest that the T3 pellets achieved a more compact bulk structure with fewer water-accessible holes at the sintering conditions used [14].

Increasing the nominal Fe content to 5 wt.% (T5) results in a distinct pattern. The bulk density reduces to $3.82 \pm 0.03 \text{ g cm}^{-3}$, while the apparent porosity rises to $3.18 \pm 0.21\%$. Thus, the observed densification improves from T0 to T3, but does not continue to rise at the highest Fe concentration tested. The non-monotonic tendency indicates that Fe addition affects the sintering reaction of TiO_2 ceramics. Possible contributing factors include changes in initial powder packing, particle agglomeration, grain growth, diffusion behavior, and pore evolution. However, the present density and porosity measurements do not independently identify which mechanism is responsible for the observed trend [15-17].

The apparent porosity values should also be distinguished from total porosity. Because the Archimedes method measures pores accessible to the immersion liquid, closed pores may remain within the ceramic body without contributing to the reported apparent porosity. The FESEM observations of the sintered pellet surfaces and, where available, polished cross-sections can therefore provide complementary evidence of grain packing and residual pore distribution [18,19].

The results in Table 2 show that the T3 composition has the highest measured bulk density and lowest apparent porosity within the investigated series. These findings identify 3 wt.% nominal Fe as the composition associated with the greatest densification under the specific pellet-processing and sintering conditions used in this study. They do not, by themselves, establish an optimum composition for mechanical, electrical, dielectric, or photocatalytic performance, as those properties are not evaluated in the present manuscript.

Table 2 Physical properties of hydrothermally synthesized $\text{TiO}_2\text{:Fe}$ ceramic pellets.

Sample	Bulk Density (g cm^{-3})	Relative Density (%)	Apparent Porosity (%)
T0 (0 wt.% Fe)	3.76 ± 0.03	96.2 ± 0.4	4.10 ± 0.20
T1 (1 wt.% Fe)	3.81 ± 0.02	97.4 ± 0.3	3.42 ± 0.18
T3 (3 wt.% Fe)	3.87 ± 0.02	98.8 ± 0.2	2.35 ± 0.15
T5 (5 wt.% Fe)	3.82 ± 0.03	97.6 ± 0.3	3.18 ± 0.21

3.3. Microstructural and surface properties

3.3.1. FESEM microstructural analysis

The FESEM micrographs of the undoped and Fe-containing TiO_2 samples (T0, T1, T3, and T5) are presented in Figure 3(a–d). The images reveal nearly spherical to irregular particles with varying degrees of agglomeration. The reported average particle sizes decrease progressively with increasing nominal Fe concentration, from $82 \pm 9 \text{ nm}$ for T0 to $68 \pm 7 \text{ nm}$ for T1, $52 \pm 5 \text{ nm}$ for T3, and $46 \pm 5 \text{ nm}$ for T5. This tendency shows that Fe addition affects particle growth during hydrothermal synthesis and subsequent thermal treatment.

The undoped sample (T0) has relatively coarse particles and substantial agglomeration. At 1 wt.% Fe, T1 has smaller particles and a more uniform apparent distribution. Increasing the Fe content to 3 wt.% yields a finer microstructure in T3, with comparably less agglomeration. Although T5 exhibits the smallest reported average particle size, localized particle clusters become more noticeable. These

observations indicate that particle refinement and agglomeration do not necessarily follow the same trend with increasing Fe concentration [20,21].

The reduction in FESEM-derived particle size is broadly consistent with the decrease in XRD-derived crystallite size reported in Section 3.1.1. However, these quantities describe different structural features: FESEM measures visible particles or grains, whereas XRD estimates coherently diffracting domains. The observed refinement alone does not establish a specific grain-growth inhibition mechanism or confirm substitutional incorporation of Fe into the TiO_2 lattice [22,23].

The density and apparent porosity measurements in Table 2 indicate that the T3 pellets achieved the highest bulk density and lowest water-accessible porosity under the applied sintering conditions. This finding may be considered alongside FESEM observations of the sintered pellets, provided that the images in Figure 3 represent those specimens. If Figure 3 instead shows calcined powders, their particle morphology should not be used as direct evidence of pore elimination or grain packing after sintering [24,25].

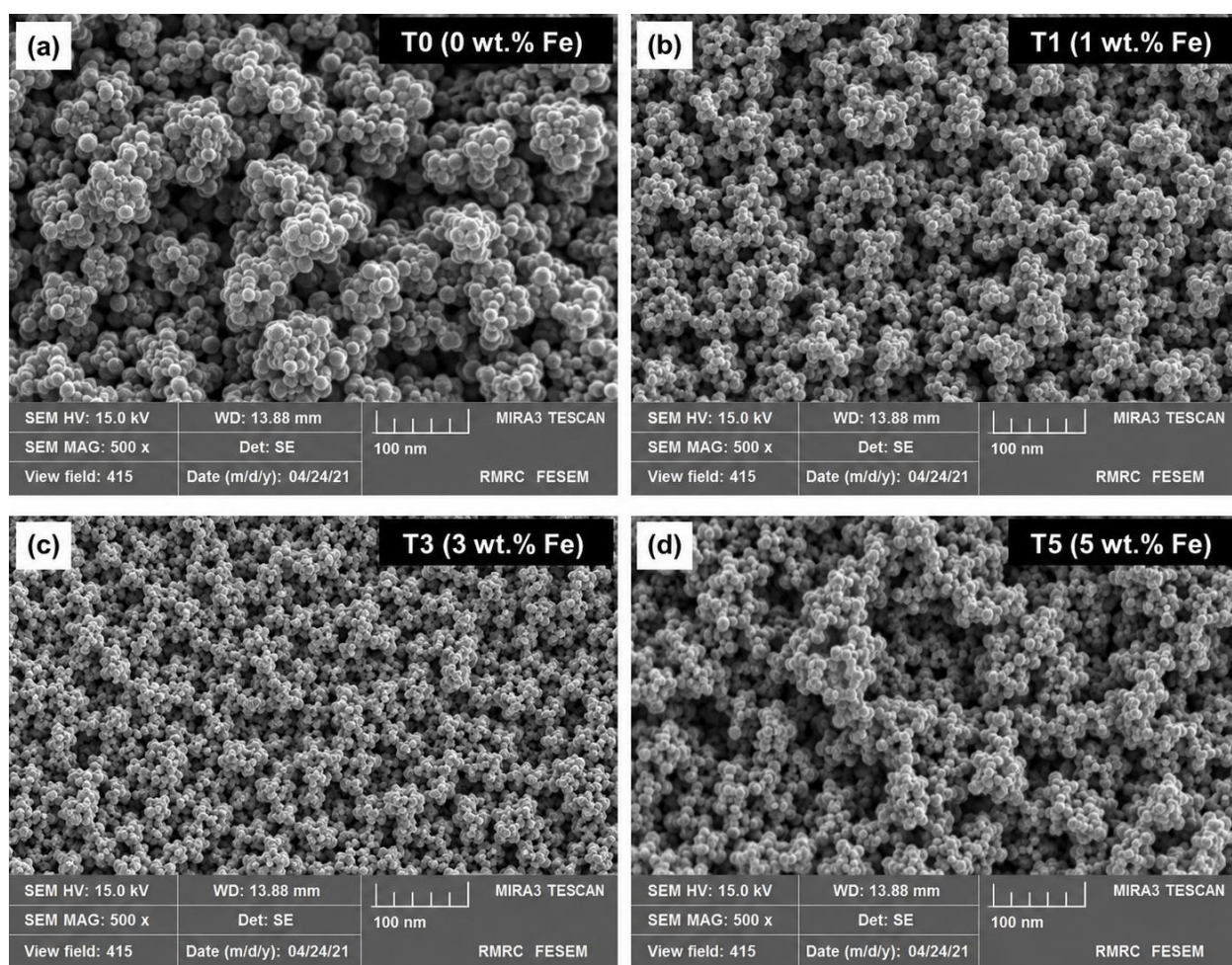


Figure 3 FESEM micrographs of hydrothermally synthesized TiO_2 ceramic nanoparticles containing (a) T0 (0 wt.% Fe), (b) T1 (1 wt.% Fe), (c) T3 (3 wt.% Fe), and (d) T5 (5 wt.% Fe).

3.3.2. EDS and Elemental Mapping

The elemental composition and the spatial distribution of Ti, O and Fe in the TiO₂ samples synthesized by hydrothermal method are studied by energy-dispersive X-ray spectroscopy (EDS) and elemental mapping, respectively. The EDS spectra and mapping of T0, T1, T3 and T5 are shown in Figure 4 (a–d) and the numbers of what is found are presented in Table 3. The undoped sample (T0) exhibits the usual EDS signals for Ti and O and no visible Fe signal. In the Fe-containing samples, the Fe signal becomes progressively more pronounced as the nominal Fe concentration increases from 1 to 5 wt.%. The quantitative results in Table 3 should be used to assess how closely the measured elemental compositions correspond to the nominal formulations, taking into account the uncertainty of EDS measurements, particularly for oxygen and low Fe concentrations [26,27].

The elemental maps indicate the spatial distribution of Ti, O, and Fe within the examined regions. For T1 and T3, the reported Fe distribution appears relatively uniform at the spatial resolution of the measurement. In T5, localized regions of higher Fe signal may indicate compositional enrichment; however, variations in particle thickness, surface geometry, and X-ray counting statistics should also be considered when interpreting map intensity [28].

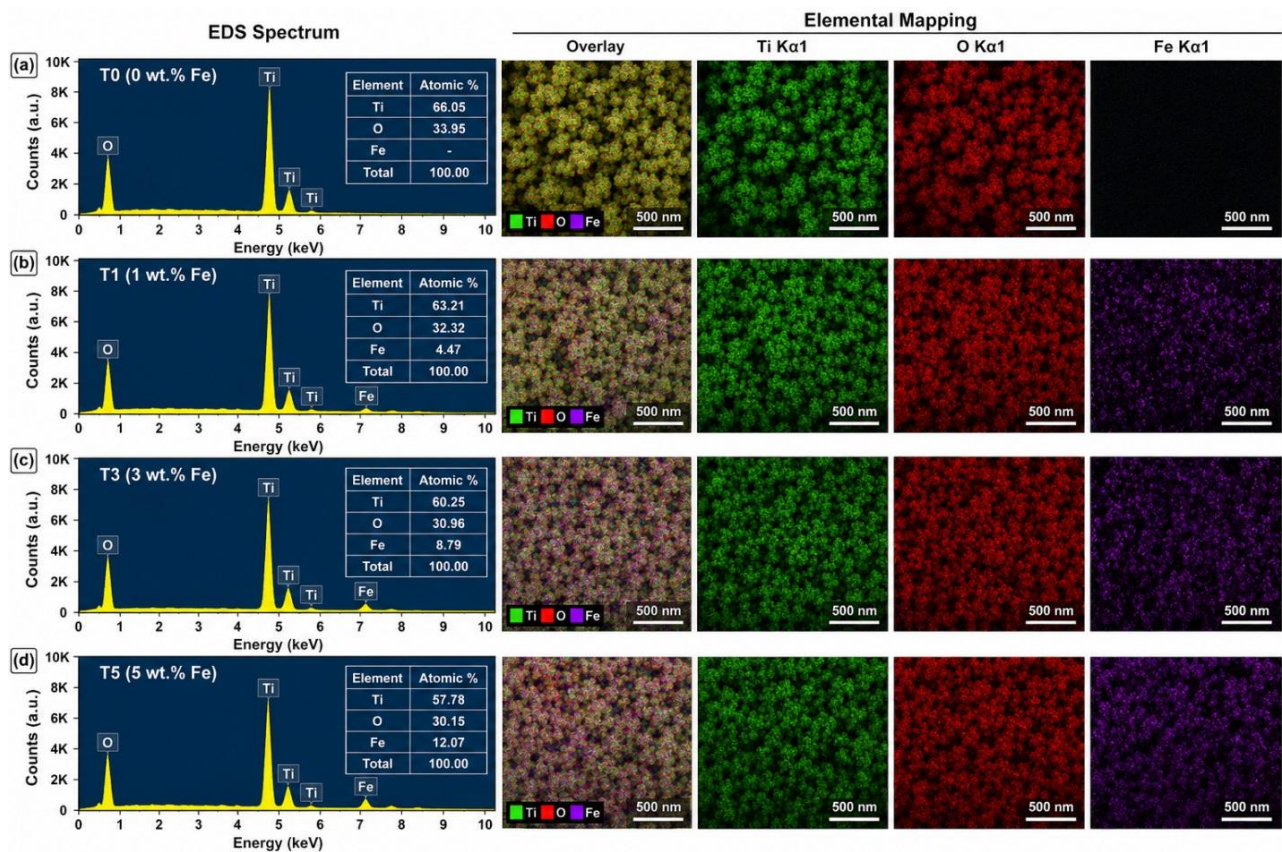


Figure 4 EDS spectra and corresponding elemental mapping images of hydrothermally synthesized TiO₂ ceramic nanoparticles containing (a) T0 (0 wt.% Fe), (b) T1 (1 wt.% Fe), (c) T3 (3 wt.% Fe), and (d) T5 (5 wt.% Fe).

Table 3. Quantitative EDS elemental composition of hydrothermally synthesized TiO₂:Fe ceramic nanoparticles.

Sample	Ti (at.%)	O (at.%)	Fe (at.%)	Total (at.%)
T0 (0 wt.% Fe)	66.05 ± 0.52	33.95 ± 0.48	—	100.00
T1 (1 wt.% Fe)	63.21 ± 0.46	32.32 ± 0.41	4.47 ± 0.18	100.00
T3 (3 wt.% Fe)	60.25 ± 0.44	30.96 ± 0.39	8.79 ± 0.25	100.00
T5 (5 wt.% Fe)	57.78 ± 0.48	30.15 ± 0.36	12.07 ± 0.31	100.00

3.3.3. Fourier transform infrared (FTIR) spectroscopy

Figure 5(a) shows the FTIR spectra of undoped and Fe-containing TiO₂ powders (T0, T1, T3, and T5) recorded across 400-4000 cm⁻¹. All samples show a wide band at 3423-3438 cm⁻¹ due to O-H stretching vibrations of surface hydroxyl groups and adsorbed water. The thinner band at 1628-1634 cm⁻¹ corresponds to H-O-H bending vibrations. A low-intensity feature at 1379-1386 cm⁻¹ may be due to residual nitrate or carbonate species; its assignment depends on the precursor chemistry and measurement circumstances (Table 4) [29,30].

The absorption band between 430 and 700 cm⁻¹ is linked to Ti-O and Ti-O-Ti lattice vibrations. The stated primary band gradually moves from 612 cm⁻¹ for T0 to 608, 604, and 601 cm⁻¹ for T1, T3, and T5. The alterations indicate that Fe addition alters the local bonding environment of TiO₂. However, band shifts alone cannot reveal substitutional Fe incorporation or the concentration of oxygen vacancies.

Fe-O vibrations may contribute to a weak shoulder seen at 486-476 cm⁻¹ in Fe-containing materials, which coincides with TiO₂ lattice absorption. Consequently, the shoulder cannot independently validate Fe-O bond formation inside the TiO₂ lattice or eliminate distinct iron-containing entities [31–32].

Table 4 FTIR absorption bands of hydrothermally produced TiO₂ ceramic NPs.

Sample	–OH Stretch (cm ⁻¹)	H–O–H Bending (cm ⁻¹)	Carbonate (cm ⁻¹)	Ti–O (cm ⁻¹)	Fe–O (cm ⁻¹)
T0 (0 wt.% Fe)	3438	1634	1386	612	—
T1 (1 wt.% Fe)	3432	1632	1384	608	486
T3 (3 wt.% Fe)	3427	1630	1381	604	481
T5 (5 wt.% Fe)	3423	1628	1379	601	476

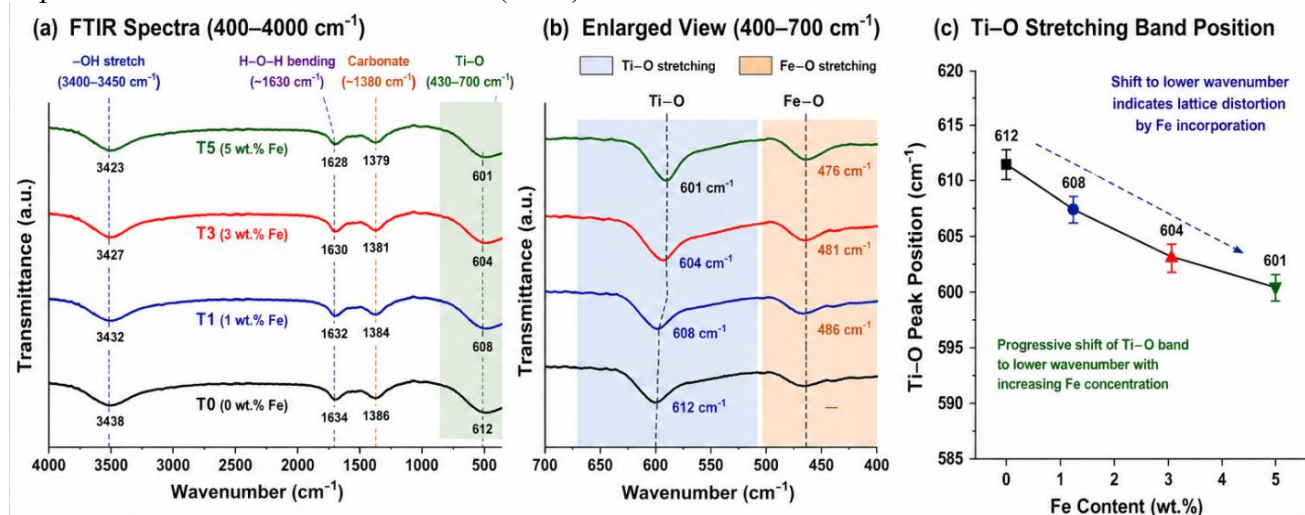


Figure 5 (a) The FTIR spectrum of hydrothermally produced TiO₂ ceramic NPs including Fe at 0% (T0), 1% (T1), 3% (T3), and 5% (T5) concentrations.

4. CONCLUSIONS

Hydrothermal reaction was used to create undoped and Fe-containing TiO₂ powders with nominal Fe concentrations of 0, 1, 3, and 5 wt.% (T0, T1, T3, and T5). These powders were then calcined and formed into ceramic pellets. The effects of Fe concentration on phase composition, particle shape, elemental distribution, vibrational properties, and densification were studied by XRD, FESEM, EDS elemental mapping, FTIR, Raman spectroscopy, and Archimedes measurements. The XRD data showed that anatase was the most identifiable crystalline phase in the calcined powders. As Fe content increased, diffraction peak locations changed, and the reported average crystallite size decreased from 27.9 nm for T0 to 19.3 nm for T5. The FESEM analysis confirmed the reduction of the size of the particles with time and the average size was reduced from 82 ± 9 nm to 46 ± 5 nm. EDS confirmed the presence of Fe in the samples that contained Fe and showed where it was located. The FTIR data showed only small variations in the main absorption band width increase in Fe content with the normal TiO₂ lattice vibrations still present. These results imply modification of structure and local bonding properties in TiO₂ by Fe addition, but do not provide evidence for Fe substitution or oxygen vacancy formation. There was no clear trend in the densification of the ceramic pellets. The T3 sample showed the lowest apparent porosity ($2.35 \pm 0.15\%$) and the highest measured bulk density (3.87 ± 0.02 g cm⁻³). In contrast, the increase of the nominal Fe content up to 5 wt.% led to lighter and more porous material. Therefore, the highest density was obtained for 3 wt.% Fe among the mixtures studied under the sintering conditions used. The results show that the nominal Fe content modifies the structure, shape and physical characteristics of TiO₂ ceramics prepared using high temperatures.

References

- [1] T. Sugiyama, A. H. A. Dabwan, H. Katsumata, T. Suzuki, S. Kaneco, Open Journal of Inorganic Non-metallic Materials, 4 (2014) 28. <https://doi.org/10.4236/ojinm.2014.43005>

- [2] N. A. Quiroz, D. J. R. Gutierrez, S. S. Martínez, C. L. Bahena, *International Journal of Geosciences*, 2 (2011) 669. <https://doi.org/10.4236/ijg.2011.24068>
- [3] C. Kaewtip, K. Accanit, N. Chaowai, K. Areerat, P. Reanjaruan, V. Boonumnauyvitaya, *Advances in Materials Physics and Chemistry*, 2 (2012) 40. <https://doi.org/10.4236/ampc.2012.24B012>
- [4] P. Ctibor, V. Stengl, Z. Pala, *Chemical Science International Journal*, 3 (2013) 387. <https://doi.org/10.9734/ACSJ/2013/4745>
- [5] E. Alzahrani, *American Journal of Analytical Chemistry*, 8 (2017) 95. <https://doi.org/10.4236/ajac.2017.81008>
- [6] A. M. Hamid, H. W. Hassan, F. A. Osman, *Asian Journal of Physical and Chemical Sciences*, 7 (2019) 1. <https://doi.org/10.9734/ajopacs/2019/v7i330094>
- [7] A. Hajjaji, A. Atyaoui, K. Trabelsi, M. Amlouk, L. Bousselmi, B. Bessais, M. A. El Khakani, M. Gaidi, *American Journal of Analytical Chemistry*, 5 (2014) 473. <https://doi.org/10.4236/ajac.2014.58056>
- [8] J. I. Arishi, J. Ozuomba, *Chemical Science International Journal*, 24 (2018) 1. <https://doi.org/10.9734/CSJI/2018/43904>
- [9] T. E. Amakoromo, *Chemical Science International Journal*, 31 (2022) 21. <https://doi.org/10.9734/CSJI/2022/v31i4816>
- [10] F. Jabli, R. Alshammari, *Journal of Materials Science and Chemical Engineering*, 13 (2025) 12. <https://doi.org/10.4236/msce.2025.139002>
- [11] K. Nishizawa, M. Okada, E. Watanabe, *Materials Sciences and Applications*, 5 (2014) 112. <https://doi.org/10.4236/msa.2014.53016>
- [12] V. Guzmán-Velderrain, Y. Ortega López, J. Salinas Gutiérrez, A. López Ortiz, V. H. Collins-Martínez, *Green and Sustainable Chemistry*, 4 (2014) 120. <https://doi.org/10.4236/gsc.2014.43017>
- [13] J. C. Morales Mejia, L. Angeles, R. Almanza, *Computational Water, Energy, and Environmental Engineering*, 3 (2014) 36. <https://doi.org/10.4236/cweee.2014.31005>
- [14] M. J. AlSultani, M. F. A. Alias, *Experimental and Theoretical NANOTECHNOLOGY*, 9 (2025), 103 <https://doi.org/10.56053/9.S.103>
- [15] A. Nilchi, S. Janitabar-Darzi, S. Rasouli-Garmarodi, *Materials Sciences and Applications*, 2 (2011) 476. <https://doi.org/10.4236/msa.2011.25064>
- [16] K. K. Kasem, A. Finley, J. Folberth, M. Syed, E. Kirkpatrick, *Materials Sciences and Applications*, 3 (2012) 265. <https://doi.org/10.4236/msa.2012.35039>
- [17] K. K. Kasem, H. Ramey, V. Ahmed, *Materials Sciences and Applications*, 4 (2013) 637. <https://doi.org/10.4236/msa.2013.410078>
- [18] P. Paschalidou, C. R. Theocharis, *Materials Sciences and Applications*, 11 (2020) 715. <https://doi.org/10.4236/msa.2020.1111048>
- [19] S. Tanigawa, T. Takashima, H. Irie, *Journal of Materials Science and Chemical Engineering*, 5 (2017) 129. <https://doi.org/10.4236/msce.2017.51017>
- [20] Hala Majed Mohi, Enas Ajil Jasim, Aya Adnan Mousa Abd, *Experimental and Theoretical NANOTECHNOLOGY*, 9 (2025), 115 <https://doi.org/10.56053/9.S.115>
- [21] S. Yamauchi, S. Saiki, K. Ishibashi, A. Nakagawa, S. Hatakeyama, *Journal of Crystallization Process and Technology*, 4 (2014) 79. <https://doi.org/10.4236/jcpt.2014.42011>
- [22] M. R. Meghana, H. A. Deepa, R. Ravishankar, *International Journal of Engineering Research & Technology*, 8 (2019) 33 <https://doi.org/10.17577/IJERTV8IS080277>
- [23] Athraa Ali Salman, Fouad K. Mashee Al Ramahi, Inbethaq M-Ali Abdulameer, Duaa Haidar M. Ali, *Experimental and Theoretical NANOTECHNOLOGY*, 9 (2025), 123 <https://doi.org/10.56053/9.S.123>
- [24] M. Z. Sahdan, N. Nayan, S. H. Dahlan, M. E. Mahmoud, U. Hashim, *Advances in Materials Physics and Chemistry*, 2 (2012) 16. <https://doi.org/10.4236/ampc.2012.24B005>

- [25] M. Kaneko, Asian Journal of Physical and Chemical Sciences, 11 (2023) 42. <https://doi.org/10.9734/ajopacs/2023/v11i1195>
- [26] N. A. Al-Omair, S. M. Reda, F. M. Al-Hajri, Advances in Materials Physics and Chemistry, 4 (2014) 29. <https://doi.org/10.4236/ampc.2014.42005>
- [27] J. Bandy, Q. Zhang, G. Cao, Materials Sciences and Applications, 2 (2011) 1427. <https://doi.org/10.4236/msa.2011.210193>
- [28] U. Soni, V. N. Shukla, R. C. Prajapati, S. Surve, International Journal of Engineering Research & Technology, 9 (2020). <https://doi.org/10.17577/IJERTV9IS040442>
- [29] H. I. Ahmed, AIP Conference Proceedings, 2235 (2020) 020038. <https://doi.org/10.1063/5.0008913>
- [30] H. Hasan, A. Nooruldeen, H. I. Ahmed, and M. J. Jaafer, Periodicals of Engineering and Natural Sciences (PEN), 8 (2020) 2 727. <https://doi.org/10.21533/pen.v8.i2.1100>
- [31] H. I. Ahmed, AIP Conference Proceedings, 2235 (2020) 020038. <https://doi.org/10.1063/5.0008913>
- [32] Eman M. Al-zoubaidi, Essa A.F., Hayder S. Musa, Experimental and Theoretical NANOTECHNOLOGY, 9 (2025), 135 <https://doi.org/10.56053/9.S.135>