

pH Değişiminin TiO₂ Nano Taneciklerinin Sol-Jel Sentezi Üzerine Olan Etkisi: Yapısal, Morfolojik ve Yüzey Özellikleri

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ÖZ

Çalışmada, TiO₂ nano taneciklerinin yapısal, morfolojik ve yüzey özelliklerini etkileyen farklı asidik pH değerlerinde hazırlanan çözelti etkisi incelenmiştir. Bu nedenle, titanyum (IV) izopropoksit (TIP) ile sol-jel işlemi kullanılarak üç farklı asidik pH değerinde (pH=2, pH=4 ve pH=6) TiO₂ nano tanecikleri sentezlenmiştir. Üç farklı pH değerinde hazırlanan TiO₂ nanotanecikleri FTIR, Raman spektroskopisi, XRD, SEM, XPS ve BET analitik yöntemleri kullanılarak sistematik bir şekilde karakterize edilmiştir. pH=2'de elde edilen TiO₂ nano tanecikleri (TiO₂-2) başlıca kristal fazı olarak anatazdan ve az miktarda rutilden oluşan çoklu faz içermekte olup, pH=4 (TiO₂-4) ve pH=6 (TiO₂-6) değerlerinde hazırlanan TiO₂ örnekleri ise sadece anataz fazından oluşmaktadır. TiO₂ örneklerinin kristalit tanecik boyutlarının pH değeri arttıkça azaldığı gözlemlenmiştir. TiO₂-4 ve TiO₂-6 numuneleri, TiO₂-2 örneğine kıyasla daha düz ve kümelenmiş taneciklerden oluşmuş bir yüzey morfolojisine sahiptir. Örneklerin izoterm eğrileri Tip IV izotermine uymaktadırlar. TiO₂-2, TiO₂-4 ve TiO₂-6 örneklerinin BET yüzey alanları sırasıyla 50 m²/g, 54 m²/g ve 59 m²/g olarak bulunmuştur.

Influence of pH on the Sol-Gel Synthesis of TiO₂ Nanoparticles: Structural, Morphological, and Surface Properties

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ABSTRACT

This study focused on the effects of acidic pH on the precursor solution, impacting the structural, morphological, and surface features of TiO₂ nanoparticles. Therefore, TiO₂ nanoparticles at three different acidic pH levels (pH=2, pH=4, and pH=6) using a sol-gel process from a titanium (IV) isopropoxide (TIP) precursor were synthesized. The synthesized TiO₂ nanoparticles at three different pH levels were systematically characterized by FTIR, Raman spectroscopy, XRD, SEM, XPS, and BET analytical techniques. The obtained TiO₂ nanoparticles at pH=2 (TiO₂-2) exhibited a multi-phase pattern of dominant anatase and minor rutile, while TiO₂ specimens prepared at pH=4 (TiO₂-4) and pH=6 (TiO₂-6) consisted only of a single anatase phase. The crystallite particle sizes of TiO₂ specimens decreased as the pH level increased. The TiO₂-4 and TiO₂-6 specimens revealed smoother and agglomerated surface morphologies compared to the TiO₂-2 specimen. All the isotherm curves were Type IV, and the BET surface areas of TiO₂-2, TiO₂-4, and TiO₂-6 specimens were 50 m²/g, 54 m²/g, and 59 m²/g.

1. Introduction

In today's world, access to clean water has become a major global concern due to the increased population and industrialization. It is well known that the discharge of insufficiently treated domestic and industrial wastewater causes a threat to the ecosystem. Therefore, the contamination of natural resources by hazardous chemicals associated with untreated wastewater effluents, particularly those containing dyes, pharmaceutically active compounds, and chlorinated solvents from industrial facilities, as well as pollution related to local agriculture, such as the use of pesticides and herbicides, is one of the primary environmental challenges present worldwide. Moreover, the introduction of even small quantities of these toxic and carcinogenic compounds into both the soil and water bodies can lead to significant health problems and genetic mutations. Hence, the adoption of efficient water treatment technologies is a prerequisite for ensuring public and environmental well-being (Shukla et al., 2021; Balakrishnan et al., 2022; Singh et al., 2022; Tuama et al., 2024).

TiO₂ is a unique metal oxide recognized for its high chemical stability, easy availability, and non-toxic features. In this context, TiO₂ nanoparticles and TiO₂-based materials have been studied for the decomposition of pollutants in water and the neutralization of organic compounds in air (Turkten & Cinar, 2017; Mamaghani et al., 2019; Bakbolat et al., 2020; Turkten et al., 2022; Turkten et al., 2023). Adsorption studies focused on the removal of heavy metals, particularly arsenic, from wastewater and groundwater using the TiO₂ nanoparticles and TiO₂-based materials (Yan et al., 2016; Arora, 2019). Besides, TiO₂-supported adsorbent materials, including activated carbon, zeolites, alumina, and silica, have been utilized to improve photocatalytic performance (MiarAlipour et al., 2018). Research has highlighted that photocatalysis can effectively eliminate dyes and persistent organic pollutants from water by forming electron/hole pairs and facilitating redox reactions on the catalyst surface (Turkten & Cinar, 2017; Gurkan et al., 2017; Khan et al., 2024). Gurkan et al. reported an efficient photocatalytic degradation of 4-nitrophenol using selenium/nitrogen codoped TiO₂ (Gurkan et al., 2017), selenium(IV) ion -doped TiO₂ (Gurkan et al., 2013), nitrogen, carbon, sulfur-doped TiO₂ (Yalçın et al., 2010b), and iron-doped TiO₂ (Yalçın et al., 2010a) photocatalysts.

To date, various synthesis strategies have been employed to prepare TiO₂ nanoparticles, enabling the customization of their crystal structures, sizes, and shapes (Mustapha et al., 2020; Shang et al., 2022). Wet chemical methods are classified into microemulsion, hydrothermal/solvothermal, precipitation, and sol-gel techniques. Among these approaches, sol-gel is known as chemical solution deposition, which is a widely used procedure for synthesizing TiO₂ nanoparticles. This popular process can be categorized into either aqueous or non-aqueous sol-gel methods based on the reaction procedure. In the aqueous sol-gel method, water is only used in the reaction, while non-aqueous solvents such as alcohol, ketones, and ethers are utilized in the non-aqueous sol-gel method. The final product is obtained through five steps: hydrolysis, polycondensation, aging, drying, and calcination. The reaction is initiated by the

hydrolysis of titanium alkoxides, which serve as precursors in a solvent or water reaction medium. This step is followed by a condensation reaction that removes alcohol or water, increasing the viscosity of the solvent and retaining liquid-phase gel. These two steps occur concurrently to obtain a sol or a colloidal suspension. Afterwards, the formed sol follows aging and drying steps, yielding a gel. In the calcination step, the organic residues are finally removed, resulting in the acquisition of the desired crystalline TiO₂ particles. The molar ratio of precursor to water, pH of the solution, and calcination temperature and time are the sol-gel parameters that impact the characteristics of the obtained TiO₂ particles. TIP is primarily used as a precursor, enabling the control of synthesis due to its high reactivity and ability to form a stable colloidal solution. It was reported that pH and the calcination process affected the crystalline structure and phase composition of TiO₂ particles. Furthermore, the chosen type of acid catalyst influences the reaction pathway due to its dissociation constant and coordination ability (Velardi et al., 2019; Mustapha et al., 2020; Shang et al., 2022; Singh et al., 2022; Gatou et al., 2024; Lim et al., 2026).

This study focused on synthesizing and characterizing TiO₂ nanoparticles using the sol-gel method. The pH influence on the sol-gel process for synthesizing TiO₂ nanoparticles was evaluated at three different acidic pH levels of the precursor solution: pH=2, pH=4, and pH=6. A systematic characterization was conducted to analyze the impact of pH variations on structural, morphological, and surface features of the obtained TiO₂ specimens. The synthesized TiO₂ nanoparticles were characterized systematically using FTIR, Raman, XRD, SEM, XPS, and BET spectroscopic analysis.

2. Materials and Methods

2.1. Materials

TIP (Aldrich), acetic acid (Merck), ethanol (Merck), and hydrochloric acid (Merck) were utilized without further purification.

2.2. Methods

TiO₂ nanoparticles were synthesized via a sol-gel process at a pH of 2, referred to as TiO₂-2, and characterized as detailed in our previous study (Turkten & Cinar, 2017). A similar synthesis procedure was repeated at pH=4 and pH=6, and the resulting specimens were denoted TiO₂-4 and TiO₂-6, respectively. The representative synthesis of the TiO₂-4 specimen was conducted at a reaction pH of 4, as follows: TIP and acetic acid were used as a precursor and an acid catalyst, respectively. Briefly, Solution A was prepared by adding 0.1 mol of TIP to 100 mL of ethanol in a dropping funnel. Deionized water (10 mL), acetic acid (10 mL), and ethanol (80 mL) were added to a flat-bottomed flask to obtain Solution B. Afterwards, prepared Solution A was added slowly into Solution B under stirring, maintaining a pH of pH=4 using a hydrochloric acid solution. Then, the TiO₂ sol was stirred for 2 h, aged for 48 h, and dried at 105°C. The white powder was washed with extensive water and calcined at 500°C for 5h.

2.3. Characterization

FTIR-ATR analysis was carried out using a Thermo Scientific Nicolet 6700 Spectrometer (32 scans, 4 cm⁻¹ resolution). Raman measurements were attained using a Thermo Scientific DXR Raman Microscope (Ar⁺ laser, 10 mW, $\lambda = 532$ nm). SEM images were obtained on FEI-Philips XL30 Environmental Scanning Electron Microscope. BET analysis was conducted on a Quantachrome Nova 2200e. XPS profiles were performed by a Thermo Scientific K-Alpha X-ray Photoelectron Spectrometer (Al K α radiation, $h\nu = 1486.6$ eV). XRD analysis was recorded on a Rigaku-D/MAX-Ultima diffractometer (Cu K α X ray, $\lambda = 1.54$ Å). Crystallite sizes (D, nm) of TiO₂ nanoparticles were determined employing the Scherrer equation (Eq. (1)) (Scherrer, 1918).

$$D = K\lambda / (\beta \cos\theta) \quad (1)$$

where, K: 0.9, λ : 1.5418 Å, θ : Bragg angle, and β : full width at half maximum intensity in radians.

3. Results and Discussion

FTIR spectra of TiO₂ nanoparticles prepared at three different acidic pH levels are presented in Figure 1 (a). The peak at around 1550 cm⁻¹ was ascribed to the bending mode of adsorbed water. The peaks below 1000 cm⁻¹ corresponded to the Ti–O stretching and O–Ti–O bending vibrations (Turkten et al., 2022).

XRD patterns of TiO₂ specimens at three different acidic pH values are shown in Figure 1 (b). In the diffractogram of the TiO₂-2 specimen, a multi-phase pattern was observed, featuring a dominant anatase phase and a minor rutile phase. The anatase diffraction peaks of TiO₂-2 specimen at $2\theta = 25.53^\circ, 37.92^\circ, 38.05^\circ, 48.25^\circ, 54.21^\circ, 55.29^\circ, 62.89^\circ, 69.26^\circ$ related to the (1 0 1), (0 0 4), (1 1 2), (2 0 0), (1 0 5), (2 1 1), (0 0 2), and (1 1 6) planes (JCPDS No. 73–1764), respectively. The observed rutile peaks at $2\theta = 27.50^\circ, 36.27^\circ, 41.29^\circ, 44.7^\circ, \text{ and } 56.71^\circ$ corresponded to (1 1 0), (1 0 1), (1 1 1), (2 1 0), and (2 0 0) planes (JCPDS No. 99–0090), respectively. The multi-phase transitioned into a single anatase phase, and the rutile diffraction peaks diminished as the pH increased to pH=4 and pH=6. Velardi et al. reported that both anatase and rutile phases were present at a calcination temperature of 450°C and a pH preparation level of pH=2 (Velardi et al., 2019). At higher pH levels (pH=3, pH=5, pH=8, and pH=10), the rutile peaks disappeared completely, and only detectable anatase peaks remained. The reason could be the rapid oxidation, resulting in the formation of the Ti(OH)_{3+x} compound, which facilitated the transformation of the anatase phase (Cassaignon et al., 2007; Yalcin, 2022).

The crystallite particle sizes of TiO₂-2, TiO₂-4, and TiO₂-6 specimens were 33 nm, 16 nm, and 14 nm, respectively. This situation was evident in that the crystallite particle size of TiO₂ nanoparticles was influenced by the pH adjustment during their preparation condition. The results indicated that the crystallite particle size decreased as the pH increased, consistent with the literature (Velardi et al., 2019). Raman spectra of TiO₂ specimens at three different pH values are presented in Figure 1 (c). TiO₂ specimen exhibited five anatase bands at 631 cm⁻¹ (E_g), 508 cm⁻¹ (A_{1g}), 389 cm⁻¹ (B_{1g}), 189 cm⁻¹ (E_g), and 137 cm⁻¹ (E_g), and a low-intensity rutile band at 439 cm⁻¹ (E_g) (Turkten et al., 2019). The rutile band

disappeared at the TiO₂-4 and TiO₂-6 specimens, and these findings were compatible with the XRD data.

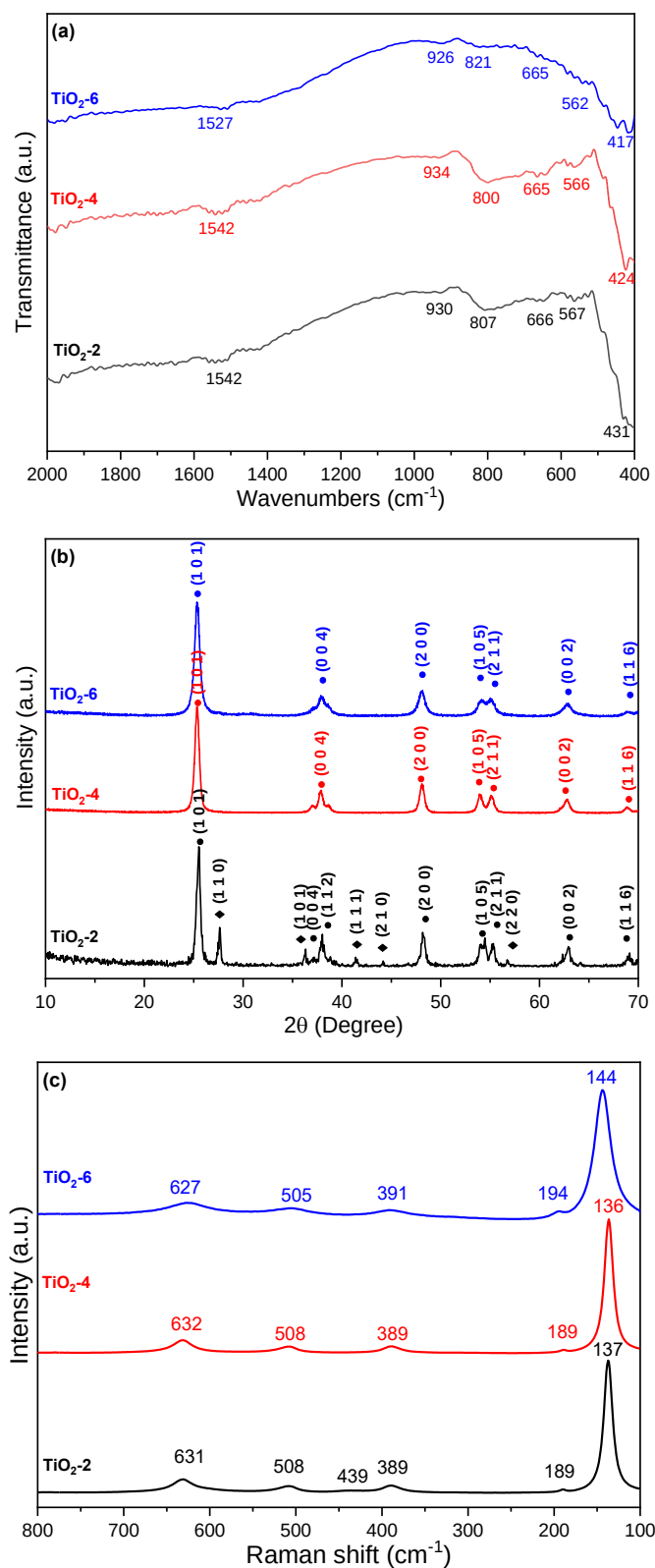


Figure 1. TiO₂ specimens of (a) FTIR spectra, (b) XRD diffractograms (●anatase, ◆rutile), and (c) Raman spectra.

SEM images of the TiO_2 specimens under three different pH conditions are presented in Figure 2. At a pH of 2, the spherical particles were nearly uniform, revealing only an insignificant tendency to agglomerate. As the pH increased to pH=4 and pH=6, a smoother surface with a high agglomeration was observed.

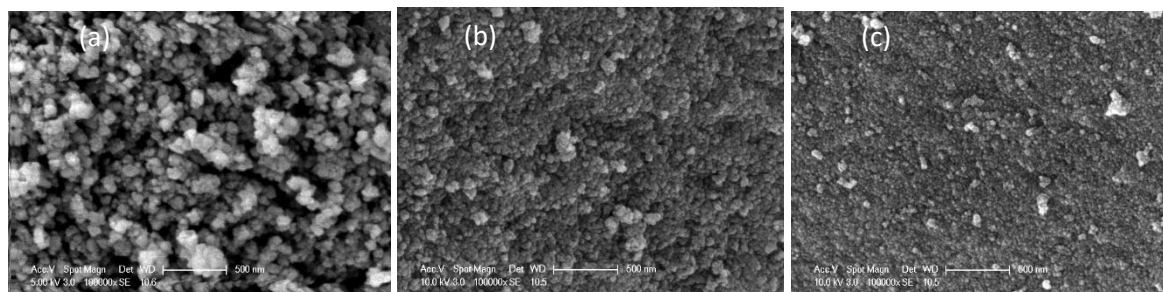
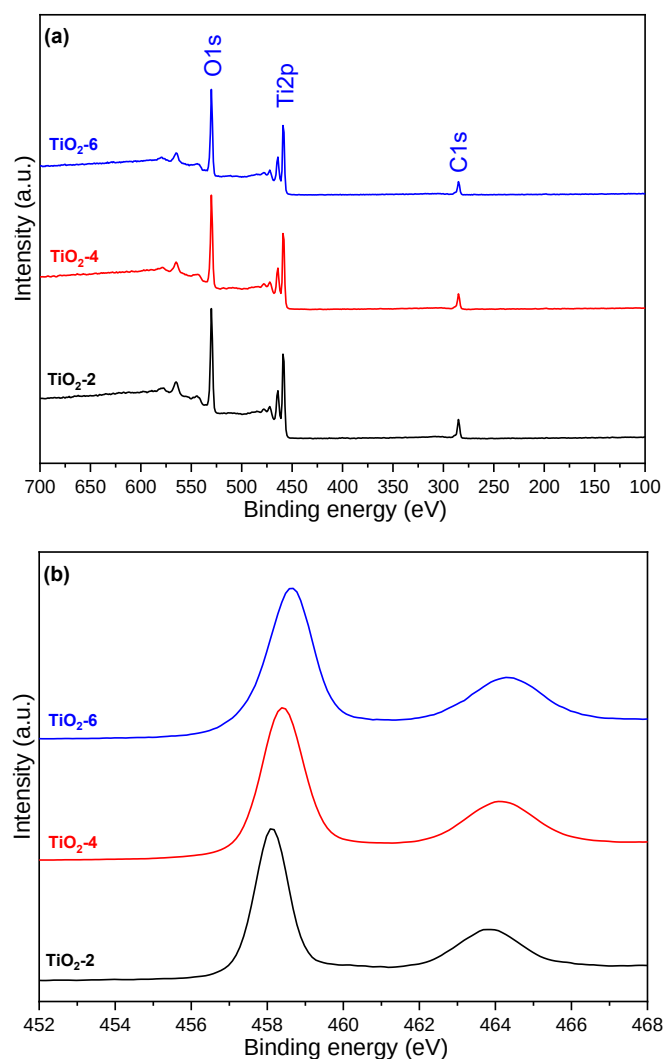


Figure 2. SEM images of TiO_2 specimens (a) pH=2, (b) pH=4, and (c) pH=6.

The XPS spectra of TiO_2 specimens prepared at three different pH values are presented in Figure 3.



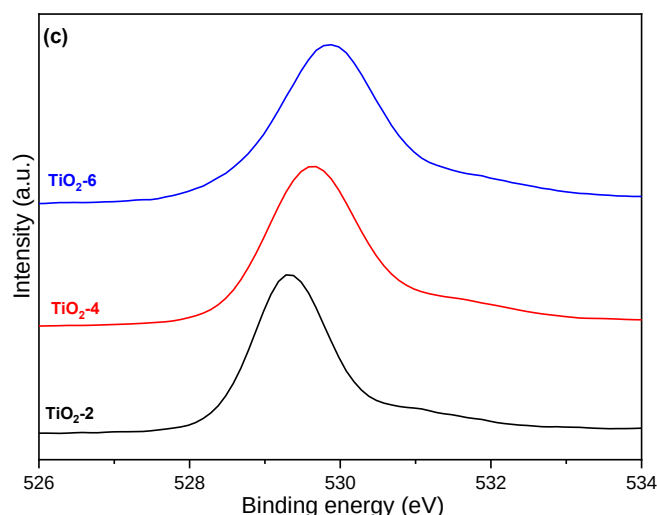


Figure 3. XPS spectra of TiO₂ specimens (a) all survey, (b) Ti 2p, and (c) O 1s.

The survey XPS spectra mainly consisted of Ti 2p and O 1s regions (Figure 3(a)). The peak at 285 eV could be related to the C 1s level of the adventitious carbon sourced from environmental contamination. Ti 2p_{1/2} and Ti 2p_{3/2} core levels appeared at binding energies of ~458 eV and ~464 eV, respectively, verifying Ti⁴⁺ state. (Figure 3 (b)). The core level of O 1s peaks at ~529 eV could belong to the metallic oxides (Figure 3 (c)) (Turkten et al., 2022). The peaks corresponding to Ti 2p and O 1s were shifted to slightly higher binding energies at pH levels of pH=4 and pH=6.

N₂ adsorption-desorption isotherms of TiO₂ specimens at three different pH levels are presented in Figure 4.

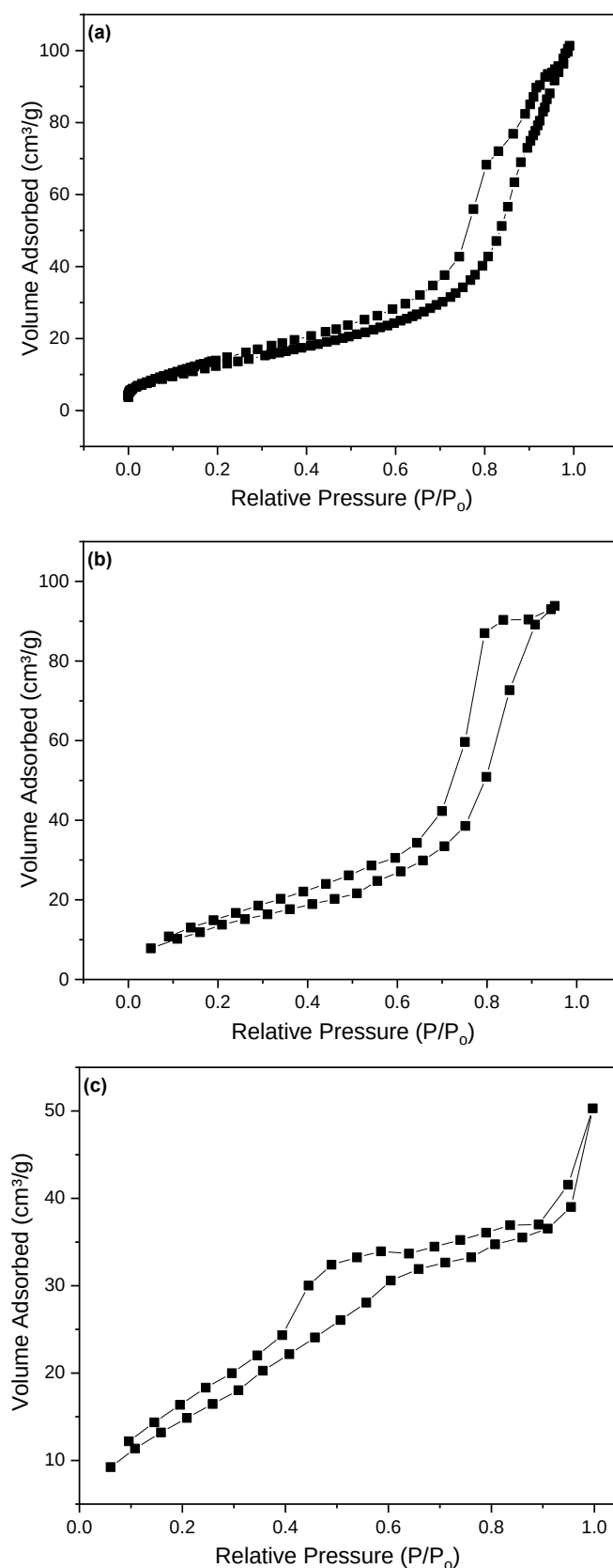


Figure 4. N₂ adsorption-desorption isotherms of TiO₂ specimens (a) pH=2, (b) pH=4, and (c) pH=6.

All the isotherms belonged to Type IV according to IUPAC classification (Sing, 1985). BET surface areas of TiO₂-2, TiO₂-4, and TiO₂-6 specimens were 50 m²/g, 54 m²/g, and 59 m²/g, respectively. Tryba

et al. prepared TiO₂ nanoparticles using the sol-gel method at pH=3 and pH=10, resulting in surface areas of 179 m²/g and 406 m²/g, respectively (Tryba et al., 2016). In another sol-gel study, the influence of pH and the titanium precursor on the preparation of TiO₂ nanoparticles was investigated (Simonsen & Sogaard, 2010). It was reported that the surface area of TiO₂ nanoparticles under acidic conditions (pH=3) from TIP precursor was very low (6 m²/g), while the surface areas of TiO₂ nanoparticles at pH=7 and pH=10 were found to be 130 m²/g and 196 m²/g, respectively. The surface areas of TiO₂ nanoparticles prepared at pH values of pH=5, pH=6, pH=8, and pH=9 were reported as 125 m²/g, 157 m²/g, 146 m²/g, 142 m²/g, and 140 m²/g, respectively (Khan, 2017).

4. Conclusion

TiO₂ nanoparticles at three different acidic pH levels from the precursor solution were synthesized via a sol-gel procedure. The impact of pH in the sol-gel method on TiO₂ nanoparticles was investigated. It was observed that the pH variation influenced the structural, morphological, and surface properties of the TiO₂ specimens. The TiO₂-2 specimen was composed of a multi-component system containing both anatase and rutile phases, while the TiO₂-4 and TiO₂-6 specimens were a single phase of anatase. Raman results confirmed the XRD analysis. A notable alteration was noted, especially in the crystallite particle size of the TiO₂-2 (33 nm) specimen, whereas the crystallite particle sizes of the TiO₂-4 and TiO₂-6 specimens reduced to 16 nm and 14 nm, respectively. In comparison to the TiO₂-2 specimen, the TiO₂-4 and TiO₂-6 specimens exhibited smoother and more agglomerated morphology. BET surface areas of TiO₂ specimens were slightly increased with rising pH levels. It was found that the surface areas of TiO₂-2, TiO₂-4, and TiO₂-6 specimens were 50 m²/g, 54 m²/g, and 59 m²/g, respectively. The designed TiO₂ specimens can be further applied in various areas such as water treatment, self-cleaning surfaces, and sterilization.

Declarations

Ethical Approval Certificate

Not applicable.

Author Contribution Statement

Nazli Turkten: Conceptualization, investigation, formal analysis, writing the original draft, review, and editing

Yunus Karatas: Supervision, Conceptualization, Methodology, writing the original draft, review, and editing

Yelda Yalcin Gurkan: Methodology, writing the original draft, review, and editing

Simal Kurumoglu: Methodology, investigation, writing the original draft

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Conflict of Interest

The authors declare that they have no conflict of interest.

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