

Structure and Morphology of TiO₂ nanoparticles

Farah S. Daabool

¹ College of biotechnology, Al-Qasim Green University, Hilla, Iraq
Email: farah_s_daabool@uoqasim.edu.iq

ABSTRACT

In this paper was synthesis of TiO₂ nanoparticles from TIP in ethanol solution . TiO₂ was prepared is anatase from structure and information product from XRD and SEM . TiO₂ nanoparticles was prepared study structure and morphology for different thickens 50,80 and 130 nm

Keywords:

TiO₂, XRD, SEM

Introduction

Several types of published study have recently focused on the manufacture and application of Titanium dioxide. This is because to its inexpensive cost, good photo-induced corrosion resistance, and chemical stability[1-4]. It's non-toxic, has a high oxidizing power, a 3.2 eV energy bandgap, and maximum light scattering with almost no absorption. Rutile (tetragonal), anatase (tetragonal), and brookite (orthorhombic) are the three distinct crystalline forms of TiO₂[5-10].

The nature of the starting material, sol composition, deposition process, and calcination temperature all influence the creation of each crystalline phase[11-14]. Calcination can change TiO₂ bulk from an amorphous to a crystalline state[15-20].

The surface extension and kind of exposed crystal faces have a big impact on the function of crystalline nanomaterials, especially those with anisotropic forms due to few(er) symmetry structures or synthesis circumstances.

Chemical reactivity, catalysis, rheology, luminescence, and

Photocatalytic activity are only a few examples[21-25].

Because photocatalytic reactions occur at the catalyst-substrate interface, the "golden standard" among photocatalysts, titanium dioxide (TiO₂), has properties that are highly impacted by the structure, surface, and morphology of nanocrystals (NCs) [26-30].

It has a tetragonal crystal structure in comparison to anatase, the most photocatalytically active titania polymorph and surface energy estimates indicate a square bipyramid that is slightly truncated, revealing 101} and {001} facets only[31-33].

Anatase is also the most stable polymorph below 30 nm at the nanoscale, according to research [34-35].

Nonetheless, quantitatively quantifying the morphology and facet area, as well as elucidating their relevance in this size range, is still a work in progress [36-37].

When compared to the most stable facets, NCs with extended 001 facets have been discovered to be particularly desirable, since the greater number of under coordinated Ti atoms (active

sites) present defines a higher reactivity (101 or 100)[38-40].

2. Materials and Methods

The TiO₂ solution was made utilizing Aldrich Company's Titanium (IV) isopropoxide (TTIP) as a precursor material, which had a purity of 97 percent. GCC provided pure ethanol (EtOH) for use as a solvent (99.9% purity).

2.1. Synthesis of TiO₂ nanoparticles

In a beaker, 5 mL acetic acid was dissolved in 250 mL deionized and distilled water for this experiment. The hydrolysis catalyst was the combined solution. Then, in a syringe, 5 mL of the TTIP precursor was added to the aforementioned solution right away. The resultant solution was immediately sonicated at 70 °C for three hours before being dry on a hotplate at 70 °C for around one day. After that, a mortar was used to crush the dried sol-gel product into a fine powder for analysis. The coarsely pulverized sample was heat treated for 3 hours at 200° C in a muffle furnace. It was possible to obtain a yellow white powder [41-42].

2.3. Characterization

XRD investigations were performed in a Bruker D8 Advance diffractometer at room temperature using CuK₁ radiation ((0.154nm) (RT). (The XRD was set up at 40 kV and 30 mA with a scanning angle range of 20-80° and a 0.02° increment. The IR spectra of the samples were obtained using the KBr technique on an FTIR spectrophotometer (IRAffinity-1S, Shimadzu). Compositional studies were performed using a Jeol/JSM-6490 scanning electron microscope (SEM) operating at 20 kV and equipped with an energy dispersive X-ray (EDX) detector. The samples were characterized using a Tecnai F20 FEG-S/TEM operating at 200 kV.

A Chromtech spectrophotometer CT-8600 was used to evaluate the absorbance

measurements. A Jasco Spectrofluorometer FP-8500 was used to measure photoluminescence (PL) at room temperature. A Rayleigh UV-Visible Spectrophotometer, UV1800, was used to determine the concentration of phenol solutions.

2.4. Degradation of phenol

The photocatalytic activity was conducted in a batch reactor using an Illuminator LuzChem LZC-4V photoreactor with 14 lamps (Sylvania F13T8 13W, Cool White, 412 K Color Temperature) emitting in the visible region between 400 and 700 nm. With a UVX radiometer under continuous illumination, the energy density of the light irradiation on the surface of the sample was 4.5 J/cm².

The synthesized materials were utilized to degrade phenol solution as a catalyst. The phenol was first tested in the absence of the catalyst to see how stable it was. 0.175g/100mL photocatalyst was added to phenol solution to make reaction mixtures (60 ppm). For 1 hour, the mixture (phenol solution and nanoparticles) was stirred and left in the dark pending adsorption equilibrium was reached. The photoreactor was irradiated while being shaken continuously, and samples of the suspension stayed taken at regular intervals, centrifuged, and filtered. A UV-visible spectrophotometer was used to measure the phenol concentration in solution using a calibration curve of the absorbance at 270 nm, which is the maximum absorption of phenol. The results were adjusted to account for the phenol dye's breakdown in the absence of a photocatalyst.

To determine the phenol degradation efficiency, Eq. 1 was employed. $\eta = [1 - C_0/C] * 100$ (1)

where C_0 and C are the solution concentrations at $t = 0$ and t minutes under visible light, respectively.

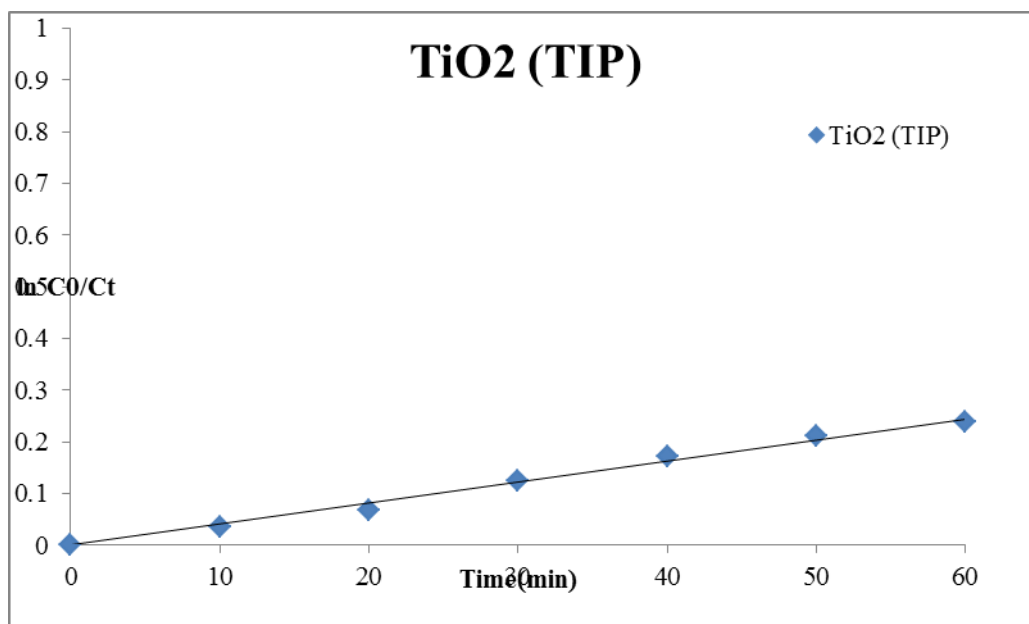


Figure 1
Changes of $\ln C_0/C_t$ according to irradiation times at TiO_2 and the prepared AC/ TiO_2 composite

3. Results and discussion

3.1- XRD

X-ray diffraction is used to measure of graphitization. Figure 1 represented X-ray diffraction of TiO_2 .

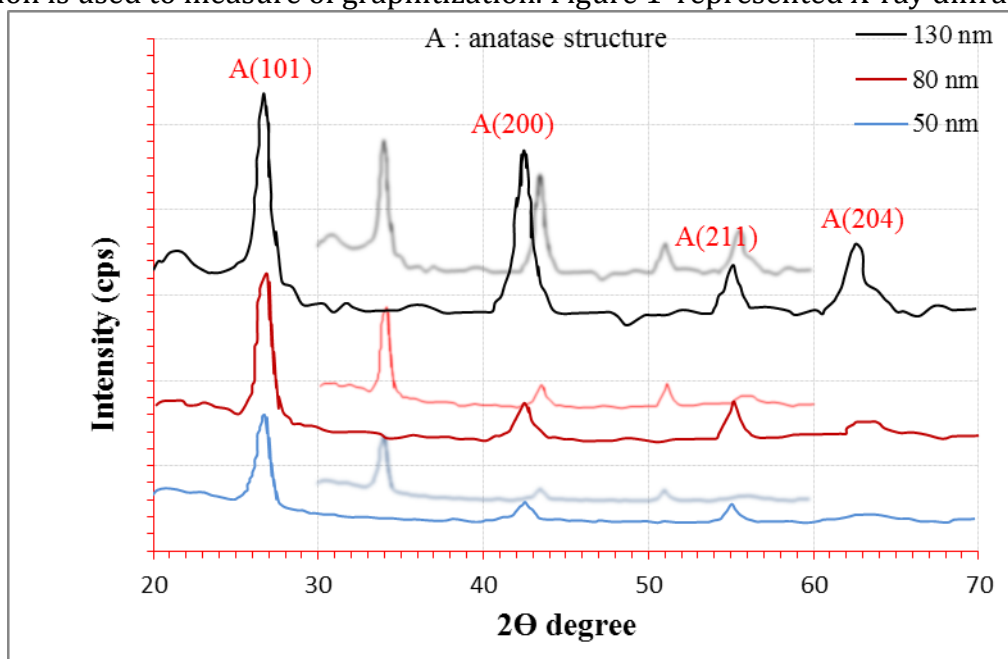


Figure 2: XRD of TiO_2

3.2- SEM

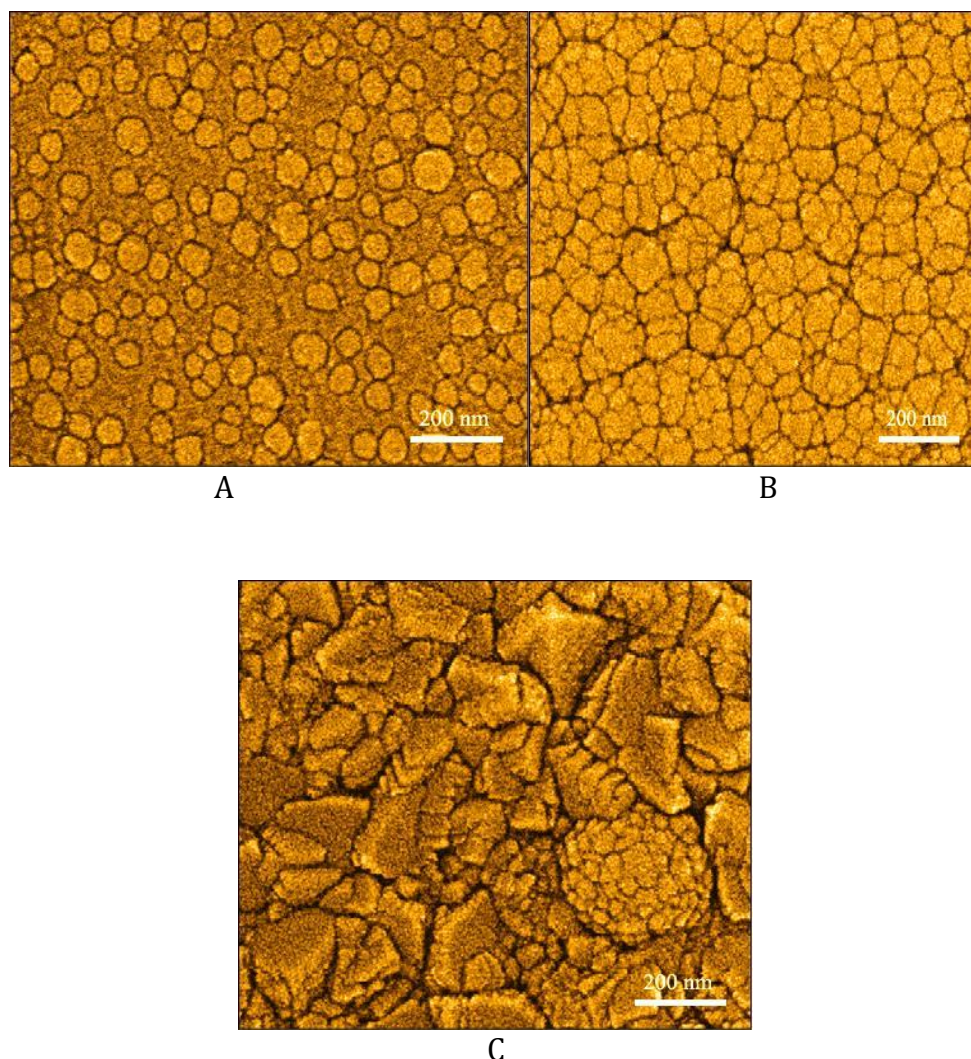


Fig-3 - SEM of TiO₂ for different thicknesses of layer a.50nm,b.80 nm , c. 130 nm

References

- Keito Sano, Fazalurahman Kuttassery, Tetsuya Shimada, Tamao Ishida, Shinsuke Takagi, Bunsho Ohtani, Akira Yamakata, Tetsuo Honma, Hiroshi Tachibana, Haruo Inoue. Optically Transparent Colloidal Dispersion of Titania Nanoparticles Storable for Longer than One Year Prepared by Sol/Gel Progressive Hydrolysis/Condensation. *ACS Applied Materials & Interfaces* **2020**, *12* (40) , 44743-44753. <https://doi.org/10.1021/acsami.0c12951>
- Mangey Ram Nagar, Shahnawaz, Rohit Ashok Kumar Yadav, Jin-Ting Lin, Jwo-Huei Jou. Nanocomposite Electron-Transport Layer Incorporated Highly Efficient OLED. *ACS Applied Electronic Materials* **2020**, *2* (6) , 1545-1553. <https://doi.org/10.1021/acsaelm.0c00166>
- Yu-Hong Hu, Cong-Xue Liu, Jian-Cheng Wang, Xiu-Hui Ren, Xuan Kan, Yu-Bin Dong. TiO₂@UiO-68-CIL: A Metal-Organic-Framework-Based Bifunctional Composite Catalyst for a One-Pot Sequential Asymmetric Morita-Baylis-Hillman Reaction. *Inorganic Chemistry* **2019**, *58* (8) , 4722-4730. <https://doi.org/10.1021/acs.inorgchem.8b02132>
- Nathan A. Reed, Ramesh Raliya, Rui Tang, Baogang Xu, Matthew Mixdorf, Samuel Achilefu, Pratim Biswas. Electro spray Functionalization of Titanium Dioxide Nanoparticles with

- Transferrin for Cerenkov Radiation Induced Cancer Therapy. *ACS Applied Bio Materials* **2019**, *2* (3) , 1141-1147. <https://doi.org/10.1021/acsabm.8b00755>
5. Felix Yu. Sharikov, Oleg A. Drozhzhin, Vasiliy D. Sumanov, Andrey N. Baranov, Artem M. Abakumov, and Evgeny V. Antipov . Exploring the Peculiarities of LiFePO₄ Hydrothermal Synthesis Using In Situ Calvet Calorimetry. *Crystal Growth & Design* **2018**, *18* (2) , 879-882. <https://doi.org/10.1021/acs.cgd.7b01366>
6. Ronghua Li, Mickael Boudot, Cédric Boissière, David Grosso, and Marco Faustini . Suppressing Structural Colors of Photocatalytic Optical Coatings on Glass: The Critical Role of SiO₂. *ACS Applied Materials & Interfaces* **2017**, *9* (16) , 14093-14102. <https://doi.org/10.1021/acsami.7b02233>
7. Di Li, Xinyu Li, and Jinlong Gong . Catalytic Reforming of Oxygenates: State of the Art and Future Prospects. *Chemical Reviews* **2016**, *116* (19) , 11529-11653. <https://doi.org/10.1021/acs.chemrev.6b00099>
8. Matteo Cargnello, Thomas R. Gordon, and Christopher B. Murray . Solution-Phase Synthesis of Titanium Dioxide Nanoparticles and Nanocrystals. *Chemical Reviews* **2014**, *114* (19) , 9319-9345. <https://doi.org/10.1021/cr500170p>
9. Sedigheh Abedi and Ali Morsali . Ordered Mesoporous Metal–Organic Frameworks Incorporated with Amorphous TiO₂ As Photocatalyst for Selective Aerobic Oxidation in Sunlight Irradiation. *ACS Catalysis* **2014**, *4* (5) , 1398-1403. <https://doi.org/10.1021/cs500123d>
10. Xiangcun Li, Wenji Zheng, Gaohong He, Rui Zhao, and Dan Liu . Morphology Control of TiO₂ Nanoparticle in Microemulsion and Its Photocatalytic Property. *ACS Sustainable Chemistry & Engineering* **2014**, *2* (2) , 288-295. <https://doi.org/10.1021/sc400328u>
11. Landry Biyoghe Bi Ndong, Murielle Primaelle Ibondou, Xiaogang Gu, Shuguang Lu, Zhaofu Qiu, Qian Sui, and Serge Maurice Mbadinga . Enhanced Photocatalytic Activity of TiO₂ Nanosheets by Doping with Cu for Chlorinated Solvent Pollutants Degradation. *Industrial & Engineering Chemistry Research* **2014**, *53* (4) , 1368-1376. <https://doi.org/10.1021/ie403405z>
12. Chung-Yi Wu Yu-Shiu Lo Chien-Hou Wua . Thickness Dependent Photocatalytic Performance of Nanocrystalline TiO₂ Thin Films. **2014**,,, 85-109. <https://doi.org/10.1021/bk-2014-1184.ch005>
13. Jared T. Wabeke Hazim Al-Zubaidi Clara P. Adams Liyana A. Wajira Ariyadasa Setare Tahmasebi Nick Ali Bolandi Robert Y. Ofoli Sherine O. Obare . Synthesis of Nanoparticles for Biomass Conversion Processes. **2014**,,, 219-246. <https://doi.org/10.1021/bk-2014-1186.ch012>
14. Nitish Roy, Youngku Sohn, and Debabrata Pradhan . Synergy of Low-Energy {101} and High-Energy {001} TiO₂ Crystal Facets for Enhanced Photocatalysis. *ACS Nano* **2013**, *7* (3) , 2532-2540. <https://doi.org/10.1021/nn305877v>
15. Sergey Ishchuk, Dereje Hailu Taffa, Ori Hazut, Niv Kaynan, and Roie Yerushalmi . Transformation of Organic–Inorganic Hybrid Films Obtained by Molecular Layer Deposition to Photocatalytic Layers with Enhanced Activity. *ACS Nano* **2012**, *6* (8) , 7263-7269. <https://doi.org/10.1021/nn302370y>
16. Jian-Chun Wu, Jianwei Zheng, Ping Wu, and Rong Xu . Study of Native Defects and Transition-Metal (Mn, Fe, Co, and Ni) Doping in a Zinc-Blende CdS Photocatalyst by DFT and Hybrid DFT Calculations. *The Journal of Physical Chemistry C* **2011**, *115* (13) , 5675-

5682.
<https://doi.org/10.1021/jp109567c>
17. Nichola M. Kinsinger, Ashley Wong, Dongsheng Li, Fabian Villalobos, and David Kisailus. Nucleation and Crystal Growth of Nanocrystalline Anatase and Rutile Phase TiO₂ from a Water-Soluble Precursor. *Crystal Growth & Design* **2010**, *10* (12), 5254-5261. <https://doi.org/10.1021/cg101105t>
18. Xiaobo Chen, Shaohua Shen, Liejin Guo, and Samuel S. Mao. Semiconductor-based Photocatalytic Hydrogen Generation. *Chemical Reviews* **2010**, *110* (11), 6503-6570. <https://doi.org/10.1021/cr1001645>
19. Ujwal Shreenag Meda, Khushi Vora, Yash Athreya, Ujwal Arun Mandi. Titanium dioxide based heterogeneous and heterojunction photocatalysts for pollution control applications in the construction industry. *Process Safety and Environmental Protection* **2022**, *161*, 771-787. <https://doi.org/10.1016/j.psep.2022.03.066>
20. Muhammad Bilal Tahir, Sohail Farman, Muhammad Sagir. Advances in Photocatalytic Materials for Waste Water Treatment Applications. **2022**,, 759-767. <https://doi.org/10.1016/B978-0-12-815732-9.00032-2>
21. Yunhong Shi, Awatef Abidi, Yacine Khetib, Long Zhang, Mohsen Sharifpur, Goshtasp Cheraghian. The computational study of nanoparticles shape effects on thermal behavior of H₂O-Fe nanofluid: A molecular dynamics approach. *Journal of Molecular Liquids* **2022**, *346*, 117093. <https://doi.org/10.1016/j.molliq.2021.117093>
22. Andrey A. Rempel, Albina A. Valeeva, Alexander S. Vokhmintsev, Ilya A. Weinstein. Titanium dioxide nanotubes: synthesis, structure, properties and applications. *Russian Chemical Reviews* **2021**, *90* (11), 1397-1414. <https://doi.org/10.1070/RCR4991>
23. Golnoush Zamiri, Samira Bagheri, Arman Amani Babadi, Seyedehmaryam Moosavi, M.S. Naghavi. The impact of immersion time and thickness of TiO₂ photoanode on power conversion efficiency of dye-sensitized solar cells using graphene quantum dots as photosensitizer. *Optical Materials* **2021**, *122*, 111720. <https://doi.org/10.1016/j.optmat.2021.111720>
24. Yunhong Shi, Seyedmahmoodreza allahyari, S. Mohammad Sajadi, Mashhour A. Alazwari, Payam Firouzi, Nidal H. Abu-Hamdeh, Ferial Ghaemi, Dumitru Baleanu, Arash Karimipour. The Molecular dynamics study of atomic Management and thermal behavior of Al-Water Nanofluid: A two phase unsteady simulation. *Journal of Molecular Liquids* **2021**, *340*, 117286. <https://doi.org/10.1016/j.molliq.2021.117286>
25. Büşra BULUT, Şeyma DUMAN. EFFECTS OF CALCINATION TEMPERATURE ON HYDROTHERMALLY SYNTHESIZED TITANIUM DIOXIDE SUBMICRON POWDERS. *Konya Journal of Engineering Sciences* **2021**,, 703-712. <https://doi.org/10.36306/konjes.915062>
26. Damiano Cani, Jan C. van der Waal, Paolo P. Pescarmona. Highly-accessible, doped TiO₂ nanoparticles embedded at the surface of SiO₂ as photocatalysts for the degradation of pollutants under visible and UV radiation. *Applied Catalysis A: General* **2021**, *621*, 118179. <https://doi.org/10.1016/j.apcata.2021.118179>
27. Katarzyna Siuzdak, Łukasz Haryński, Jakub Wawrzyniak, Katarzyna Grochowska. Review on robust laser light interaction with titania – Patterning, crystallisation and ablation processes. *Progress in Solid State Chemistry* **2021**, *62*, 100297. <https://doi.org/10.1016/j.progsolidstchem.2020.100297>

28. Jinyi Sui, Xifan Chen, Yang Li, Wenchao Peng, Fengbao Zhang, Xiaobin Fan. MXene derivatives: synthesis and applications in energy conversion and storage. *RSC Advances* **2021**, *11* (26) , 16065-16082. <https://doi.org/10.1039/D0RA10018H>
29. Gertrude Kignelman, Wim Thielemans. Meta-analysis of TiO₂ nanoparticle synthesis strategies to assess the impact of key reaction parameters on their crystallinity. *Journal of Materials Science* **2021**, *56* (10) , 5975-5994. <https://doi.org/10.1007/s10853-020-05607-1>
30. Norah Alonizan. Effects of CdS Nanoparticles on the Physical Properties of T-CdS Nanocomposite Materials. *Journal of Inorganic and Organometallic Polymers and Materials* **2021**, *31* (3) , 1086-1094. <https://doi.org/10.1007/s10904-020-01722-3>
31. Komaraiah Durgam, Radha Eppa, Ramana Reddy M.V., Sivakumar J, Sayanna R. Effect of metal ions doping on structural, optical properties and photocatalytic activity of anatase TiO₂ thin films. *Surface and Interface Analysis* **2021**, *53* (2) , 194-205. <https://doi.org/10.1002/sia.6901>
32. Sonia Bahrani, Mehrorang Ghaedi, Rasikh Tariq, Ziba Zalipour, Fardin Sadeghfar. Fundamental developments in the zeolite process. **2021**, 499-556. <https://doi.org/10.1016/B978-0-12-818806-4.00003-6>
33. Şana Sungur. Titanium Dioxide Nanoparticles. **2021**, 713-730. https://doi.org/10.1007/978-3-030-36268-3_9
34. Nadya I. Politova-Brinkova, Sonya R. Tsibranska-Gyoreva, Slavka S. Tcholakova, Nikolai D. Denkov, Thomas Danner. Preparation of TiO₂ Nanoparticle Aggregates and Capsules by the 'Two-Emulsion Method'. *Colloids and Interfaces* **2020**, *4* (4) , 57. <https://doi.org/10.3390/colloids4040057>
35. Klaus D. Jandt, David C. Watts. Nanotechnology in dentistry: Present and future perspectives on dental nanomaterials. *Dental Materials* **2020**, *36* (11) , 1365-1378. <https://doi.org/10.1016/j.dental.2020.08.006>
36. M. Davide Cappelluti, Emina Hadzifejzovic, John S. Foord, Duncan H. Gregory. Flash microwave-assisted solvothermal (FMS) synthesis of photoactive anatase sub-microspheres with hierarchical porosity. *RSC Advances* **2020**, *10* (61) , 37233-37245. <https://doi.org/10.1039/D0RA05796G>
37. Eka Cahya Prima, Harbi Setyo Nugroho, Nugraha, Gema Refantero, Camelia Panatarani, Brian Yulianto. Performance of the dye-sensitized quasi-solid state solar cell with combined anthocyanin-ruthenium photosensitizer. *RSC Advances* **2020**, *10* (60) , 36873-36886. <https://doi.org/10.1039/D0RA06550A>
38. Sijia Sun, Hao Ding, Jie Wang, Wei Li, Qiang Hao. Preparation of a microsphere SiO₂/TiO₂ composite pigment: The mechanism of improving pigment properties by SiO₂. *Ceramics International* **2020**, *46* (14) , 22944-22953. <https://doi.org/10.1016/j.ceramint.2020.06.068>
39. Yoongook Park, Jun Hyup Lee. Wide viewing optically clear adhesives using PMMA/TiO₂ core-shell micro/nanoparticles. *Molecular Crystals and Liquid Crystals* **2020**, *705* (1) , 135-140. <https://doi.org/10.1080/15421406.2020.1743430>
40. Daniel D. Lane, Kvar C. L. Black, Ramesh Raliya, Nathan Reed, Nalinikanth Kotagiri, Rebecca Gilson, Rui Tang, Pratim Biswas, Samuel Achilefu. Effects of core titanium crystal dimension and crystal phase on ROS generation and tumour accumulation of transferrin coated titanium dioxide nanoaggregates. *RSC Advances* **2020**, *10* (40) , 23759-

23766.

<https://doi.org/10.1039/D0RA01878C>

41. Moumita Chandra, Debabrata Pradhan. Engineering the Morphology and Crystal Phase of 3 D Hierarchical TiO₂ with Excellent Photochemical and Photoelectrochemical Solar Water Splitting. *ChemSusChem* **2020**, *13* (11) , 3005-3016.

<https://doi.org/10.1002/cssc.202000308>

42. Ozioma U. Akakuru, Zubair M. Iqbal, Aiguo Wu. TiO₂ Nanoparticles. **2020**,, 1-66.

<https://doi.org/10.1002/9783527825431.ch1>