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Biological Activity of Titania Nanopowder using Atmospheric Pressure Plasma Jet Technique

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Abstract: *In this work, titanium dixide Nano powder were synthesized by atmospheric pressure plasma jet. The study initiates with the exploitation of atmospheric pressure plasma jet technique recognized for its capability to generate nanoparticles using cold plasma, ensuring high purity and controlled growth. The average particle size and morphology have been examined by FE-SEM and the crystallinity was estimated by XRD analysis and the spectroscopic properties of the powder has neen measured by using FTIR spectroscopy. XRD studies confirm the titanium dioxide have a high degree of crystallinity. Their particle size of TiO_2 was found about 22 nm. The purpose of these study involve the studying of TiO2 nanoparticles characteristic. The outcome considered a new synthesis of TiO_2 nanopowders to employ in many scientific applications using continuous laser diode to increase the oxidization represent as a novel method. It is a simple and cost effective method.*

Keywords: *Tio_2 Nanopowders, Atmospheric Pressure Plasma Jet, Antibacterial Activity.*

Introduction

Titanium dioxides nanoparticles (NP) has shown great promise among transition metal oxide-based nanoparticles due to their interesting properties and applications in the treatment of water, energy generation, soil remedies, coating, and in domestic products (R. R. Remya et. al. 2023). TiO_2 is available in nature and has many properties such as high refractive index, absorption of light, nontoxicity, stability, and low cost (L. Stephen 202). Furthermore, TiO_2 NPs exhibited a notable photocatalytic capacity, biocompatibility, and low toxicity (R. R. Remya et. al. 2023). This has nominated them as a key component for research and applications in nano-biotechnology and nano-medicine, such as: tissue and genetic engineering, healthcare products manufacturing. TiO_2 NPS were also exploited in diagnosis and therapy of cancer.

One of the recently explored uses of TiO_2 is in the field of self-cleaning and self-antiseptic surface materials. These materials are widely used in purification and environmental applications are mainly related to the ability of TiO_2 to induce superhydrophobicity and antifogging activity (S. Banerjee et. al. 2015). These specifications are essential in materials that could provide antibacterial inhibition and air and water purification. This includes the critical task of removing harmful organic compounds and creating self-cleansing surfaces in medical institutes (L. Abdulazeem 2019). TiO_2 NP's can be synthesized by a variety of biological and chemical process that make them cost-effective for production in bulk quantities (M. Aravind 2012).

TiO2 NP's ability to inhibit the activity of microorganisms has attracted a vast interest from research efforts as it can hinder their adverse effect on the food plants (S. A. Mahdy et. al 2017) and lead to the activation of free hydroxyl radicals (M. Chen et. al. 2023). There were many attempts to demonstrate the effect of TiO2 NPs on fungal and bacterial organisms. A recent study (A. Mansoor, et. al 2023) that used a gram-negative (G−ve) and Gram-positive (G +ve) bacteria has highlighted that these nanoparticles can be used to as antimicrobial disinfecting solutions due to their success in impeding their activity.

In this work, TiO2 nanopowder was prepared by cold plasma process. Kato et. al. (K. B. Riad et. al, 2018) reported the preparation of TiO2 powders with an oxygen-deficient anatase using ICP plasma. (D. Chittinan et. al ,2023), on the other hand, prepared pure anatase TiO2 powders via atmospheric dielectric barrier discharge plasmas. Cold plasmas can be more suitable in this aspect. This is particularly due to their success in producing the anatase phase, which has higher activity compared to other TiO2 phases (Z. Liu et. al , 2011). In contrast, jet plasma is not confined by electrodes and its dimension can be adjusted from several centimeters down to sub-millimeter region (L. H. Nie et. al , 2007]. This can enable 3D surfaces to be treated locally. In addition, plasma jet can be regarded as a solution for a compatible and stable high plasma that provides an efficient reaction chemistry in atmospheric plasmas. After igniting the plasma, the reactive species generated in the jet are blown out to a separate region, thus satisfying the requirements (J. Ovenstone 2001).

Since its discovery (T. Matsunaga et. al. 1985], the photocatalytic TiO_2 antibacterial effect reaction investigated the photocatalytic oxidation under UV light against a variety of microorganisms. This microorganisms include green algae (Chlorella vulgaris), yeast (Saccharomyces cerevisiae), Gram-negative bacteria (Escherichia coli), and Gram-positive bacteria (Lactobacillus acidophilus). Numerous studies on photocatalytic disinfection have been carried out considerably on a various microorganism such as: viruses, fungi, and bacterial species (H. A.Foster et.al. 2011). The antibacterial activity of TiO_2 (S. Oh and D. Park 2001) is fueled by OH• radicals and other reactive oxygen species (ROS), which are attributed to the biocidal action of TiO_2 photocatalyst (Z. Huang et.al. 2000).

According to previous reports , reactive photogenerated oxygen species primarily target the cell membrane, resulting in lipid peroxidation (I. Ali et.al 2018). Cell death resulted from a combination of increased oxidative attack on internal components and damage to the cell membrane. Cell respiration suppression and ultimately cell death will

result from the photooxidation of coenzyme A, which is a coenzyme generated from pantothenic acid that is essential for respiration and numerous other metabolic activities (M. Grätzel and F. P. Rotzinger 1985). In general, titanium oxide disinfectants are 1.5 times more potent than ozone and 3 times more potent than chlorine (S. Roberts 1949).

Experimental Setup

Figure 1 displays the atmospheric plasma jet electrochemical production of TiO_2 nanopowder is setup and photographed. The tube is made of stainless steel with a dimension of 7 cm in length and 0.6 mm in inner diameter. This tube serves as the cathode. The titanium foil anode, which measured 6 cm in length and 3 cm in breadth, was positioned 1.5 cm in front of the cathode. There were 4 mm between the needle and the electrolyte medium's surface. A glass flow meter was used to link an argon discharge gas at the syringe and regulate the gas flow, which was determined to be about 60 milliliters per minute. The reaction was conducted in a diameter-measured glass petri dish. Inert gas (argon) was provided so that no reaction occurred with the solution, and the regulator was connected to the gas bottle to control the amount of flow of the gas, here is the cathode, the anode is the plasma electrode, and the foil is titanium.

An inert gas bottle (argon gas was utilized in this instance) serves as the gas source. A regulator controls the bottle's pressure. The tube that joins the electrical circuit with the gas in the bottle will have two cathode and anode tubes, which stand in for an electrolyte solution with titanium and a titanium coil. To produce a uniform plasma beam, the power supply (500V) has an oscillator and a frequency regulator installed.

Figure 1. Experimental setup

In addition to having a bactericidal action against gram-positive and gram-negative bacteria, including those that are resistant to multiple antibiotics, titanium dioxide nanoparticles also have the potential to be an antifungal agent. The antibacterial activity of TiO_2 NPs against harmful bacterial species such as Klpselia spp., Proteus vulgaris, Escherichia coli, and Pseudomonas aeruginosa is investigated in this work.

TiO_2 NPs' antibacterial influence was investigated on agar plates and in liquid nutrient growth media. For both investigations, E. Coli was cultivated in nutritional media (0.5 g NaCl, 1 g yeast extract, and 1 g beef extract, diluted in 100 ml distilled water). To create the inoculum, frozen E. Coli cells were cultured in the nutritional medium for an entire night. The bacterial cultures were cultivated at 200 rpm and 370C in a shaking incubator. TiO_2 NPs were ultrasonically dispersed in autoclaved deionized water. A concentration-appropriate aqueous dispersion of silver nanoparticles was created.

In this experiment, E. coli bacteria (104 cells/ml) were raised with varying concentrations of Ag NPs (0, 5mM, 10mM, and 15mM) in flasks to assess bacterial growth. The nurturing was carried out at 37°C with shaking at 150 rpm, using a total solution volume of 50 ml per flask. A standardized bacterial concentration was injected onto Petri dishes containing growth medium. This was followed by the placement of filter paper disks injected with antimicrobial agents on the agar surface. The next step was to place the dishes under suitable growth conditions. This step is necessary to allow the antimicrobial agent to spread in the agar and react with bacterial growth.

Methodology

The methodology used in the study "Biological Activity of Titania Nanopowder using Atmospheric Pressure Plasma Jet Technique" can be summarized as follows:

1. Preparation of TiO_2 Nanopowder
    - Technique: Atmospheric pressure plasma jet technique was utilized to synthesize TiO_2 nanopowder.
    - Experimental Setup:
    - The setup included a stainless steel tube as the cathode and a titanium foil as the anode.
    - Argon gas was used as the discharge medium, regulated to ensure consistent flow at 60 mL/min.
    - A 500V power supply was employed with an oscillator and frequency regulator to generate a uniform plasma beam.
    - Reaction Details:
    - The reaction was performed in a glass petri dish with a titanium electrolyte medium.
    - Synthesis time was varied (20, 30, and 50 minutes), along with the concentration of the catalyst ($TiOSO_4$).

2. Characterization of TiO_2 Nanoparticles
    - XRD Analysis:
    - Used to confirm the crystalline structure and phase composition (anatase and rutile).
    - Analysis revealed particle size, lattice parameters, and the presence of anatase peaks at specific 2θ values.
    - SEM Imaging:

- Scanning electron microscopy (SEM) was used to examine the morphology and size of the nanoparticles synthesized under different conditions.
- FTIR Spectroscopy:
- Spectroscopic properties of the TiO_2 nanopowder were measured to study chemical bonding.

3. Antibacterial Activity Testing
   - Test Organisms:
   - Gram-positive (e.g., Proteus vulgaris) and Gram-negative bacteria (e.g., Escherichia coli, Klebsiella spp.).
   - Procedure:
   - Bacterial cultures were grown in nutrient media.
   - TiO_2 nanoparticles were ultrasonically dispersed in autoclaved deionized water.
   - Disks injected with TiO_2 nanoparticle solutions were placed on agar plates inoculated with bacterial cultures.
   - Conditions:
   - The plates were incubated at 37°C with shaking at 150 rpm to allow the nanoparticles to interact with bacterial cells.
   - Measurement:
   - Inhibition zones were measured to assess the antibacterial activity of the nanoparticles.

4. Analysis of Results
   - Correlation between synthesis conditions (time, catalyst concentration) and nanoparticle properties (size, crystallinity).
   - Evaluation of antibacterial efficacy by comparing inhibition zones for different bacterial species and synthesis conditions.

This methodology effectively combines plasma-based nanoparticle synthesis with advanced characterization and biological testing, enabling a comprehensive evaluation of TiO_2 nanopowders for antibacterial applications.

Result and Discussion

Figures 2 shows the XRD pattern of prepared TiO_2 NP's and table (3-1) includes identification parameters of TiO_2 indexed by the JCPDS 29-1132. The sample was labeled as (1) which synthesized at 20 minutes, while the second sample was listed as (2) at 30 min. and the third sample was (3) at 50 minutes which synthesized without using sulfuric acid and using fixed weight 1gm of ($TiOSO_4$) as catalyst. Finally, the time of reaction was fixed at 50 min. with changing the amount (the weight) of $TiOSO_4$, to obtain the sixth sample labeled as (4) with 1.1gm of ($TiOSO_4$) and the seventh sample as (5) with 1.2gm of ($TiOSO_4$) with fixing the deposition time at 50 min.

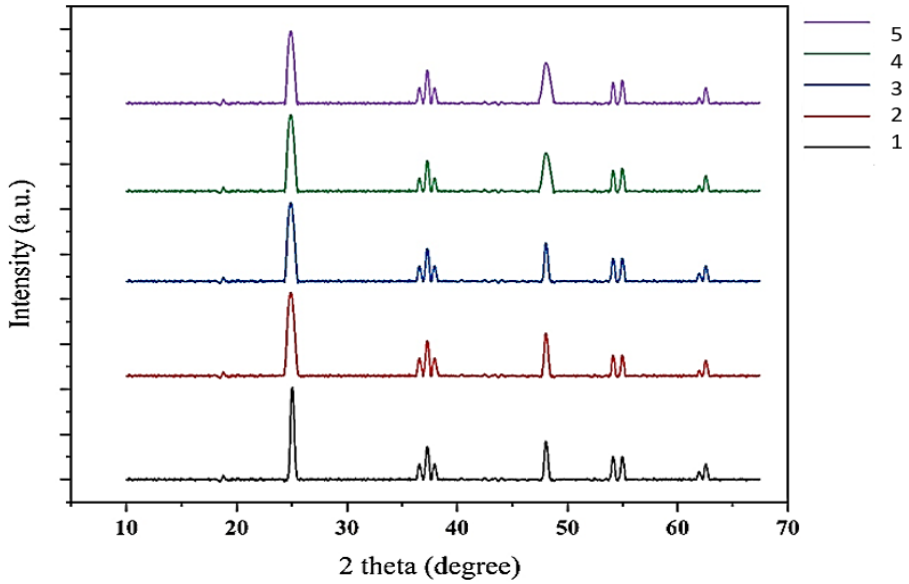


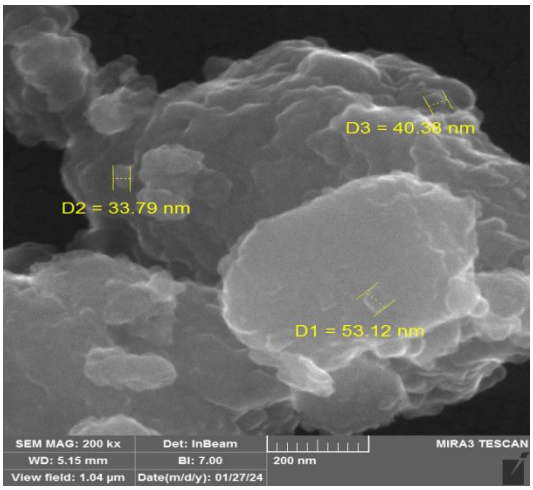
Figure 2. XRD pattern of prepared TiO2 NP's for 1, 2, 3, 4 and 5 samples.

Table 1. Lattice parameters, crystallite size using gauss and W–H plot, strain, and dislocation density of TiO2 NP's

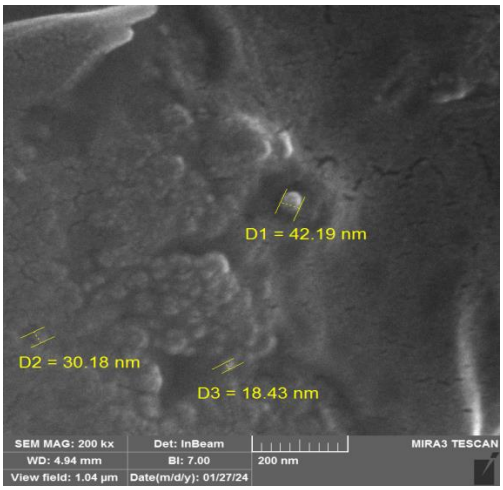
no.	lattice parameter		Gauss crystallite size D	W-H plot crystallite size D_w	Strin, $\varepsilon \times 10^{-3}$	Dislocation density, $\delta \times 10^{-3}$
	a (A)	c (A)				
C	3.782749352	9.636732695	28.67119633	38.30220994	0.61	1.21649
D	3.78277082	9.637091575	29.15234579	35.28091603	1.2	1.176665
E	3.782749352	9.636732695	28.62835802	44.58327974	1.52	1.220133
F	3.780437565	9.63655825	29.12012093	33.25035971	1.86	1.179271
G	3.780331852	9.636710266	28.50362316	31.51227273	1.99	1.230835

It is clear from table (1) that the TiO_2 NP's in as deposited time varying case is amorphous structure. In addition, the as deposited specimens included the times of (20, 30 and 50 min.) have same pattern before fixing the deposition time and varying the catalyst concentration ($TiOSO_4$). On the other hand, it is noticed that the grain size increases with increasing the time of deposition. The 2θ at peaks 25.30, 37.76 and 48.03 confirms the TiO_2 anatase structure. The XRD analysis of TiO_2 nanoparticles revealed a mixture of anatase and rutile phases. The anatase phase peaks were observed at 15 minutes of preparation. However, with longer preparation time (about 25 minutes), rutile phase peaks appeared. The experimental XRD patterns match with JCPDS card no. 29-1132 for anatase and agree with data from other studies (R. G. Breckenridge and W. Hosler 1953). The peak at 2θ = 25.4° confirms the anatase structure of TiO_2. On the other hand, diffraction peaks at 72° provide further confirmation to the anatase phase (P. G. Wahlbeck and P. W. Gilles 1966). The intensity of the XRD peaks supports that the produced nanoparticles have a crystalline nature. Finally, the wide diffraction peaks suggest that crystallites that have a small size exist in these nanoparticles.

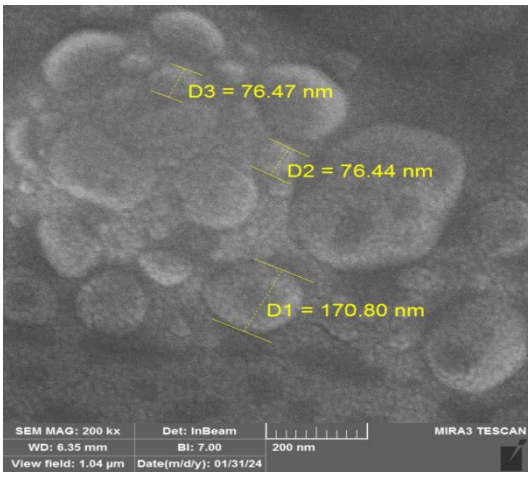
SEM images of prepared TiO_2 NP's are shown in figures (3 a, b, c, d and f), which were synthesized using atmospheric plasma jet rout at different deposition time as (20, 30 and 50 min.) with fixing the weight of ($TiOSO_4$) as 1 gm, and different catalyst concentration ($TiOSO_4$) as (1, 1.1 and 1.2 gm) with fixing the deposition time as 50 min.



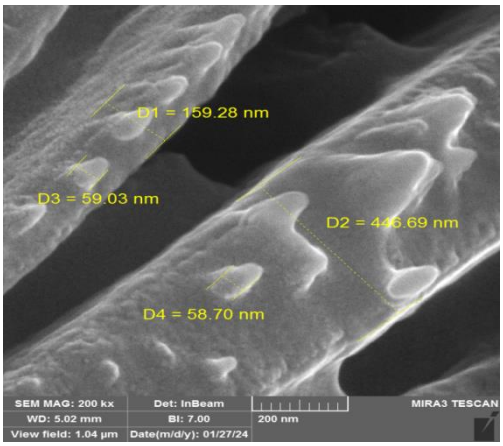
(a)



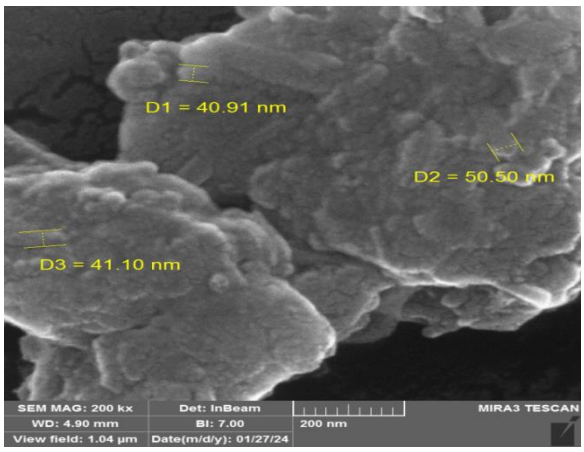
(b)



(c)



(d)



(e)

Figure 3. SEM images of prepared TiO2 NP's for (a) 1, (b) 2, (c) 3, (d) 4 and (e) 5 samples

The antibacterial study of TiO_2 NP's was examined by gram-positive and gram-negative bacteria. For the gram-negative bacteria, the cell walls are composed of thin peptidoglycan. On the other hand, a thick layer of peptidoglycan composes the walls in gram-positive bacteria. The layer of inhibition zone for the TiO_2 NP's was examined against ***Klpselia spp***, ***Proteus vulgaris, Escherichia coli*** and ***Pseudomonas aeruginosa***, which is measured in mm scale and summarized in table (2).

Table 2. The inhibition zones of TiO_2 NP's against *Klpselia spp*, *Proteus vulgaris, Escherichia coli* and *Pseudomonas aeruginosa*

Sample	Type of bacteria	Inhibition zone (mm)
1	*Escherichia coli*	4
1	*Klpselia spp*	8
1	*Proteus vulgaris*	6
1	*Pseudomonas aeruginosa*	3
2	*Escherichia coli*	3
2	*Klpselia spp*	7
2	*Proteus vulgaris*	9
2	*Pseudomonas aeruginosa*	3
3	*Escherichia coli*	4
3	*Klpselia spp*	2
3	*Proteus vulgaris*	11
3	*Pseudomonas aeruginosa*	0
4	*Escherichia coli*	5
4	*Klpselia spp*	8
4	*Proteus vulgaris*	15
4	*Pseudomonas aeruginosa*	0
5	*Escherichia coli*	7
5	*Klpselia spp*	13
5	*Proteus vulgaris*	14
5	*Pseudomonas aeruginosa*	0

The inhibition zone layer for gram-negative bacteria such as ***Klpselia spp*** are (8, 7, and 2 mm) for the samples (1, 2 and 3) at fixed amount of catalyst $TiOSO_4$ and increasing the time of deposition, respectively and (2, 8 and 13 mm) at fixing deposition time as (50 min.) and changing the weight of catalyst $TiOSO_4$. It's clear that the inhibition zone is decreased as the of deposition increased and its increased as the amount of catalyst

$TiOSO_4$ increased, this may be attributed to the increasing the amount of TiO_2 Np's caused by increasing the amount of catalyst $TiOSO_4$.

The high zone inhibition layer was observed as the increasing the amount of the catalyst $TiOSO_4$ used to prepare the TiO_2 NP's. The zone inhibition layer of pathogenic bacteria ***Escherichia coli*** and ***Proteus vulgaris*** have strong outcomes relative to ***Klpselia spp***. The bacterial cells walls, which have a thin nature, suffered from a fast disruption by the TiO_2 ions with a positive charge. This can be regarded as a result of the electrostatic interaction between positive charge (of TiO_2) and negative charges (of the *E.coli* and *proteus vulgaris* cell walls). This has led to an observable inhibition zone. For the bacterial inhibition, bacterial cell walls are commonly affected by reactive Oxygen species (ROS). These species include hydroxyl radicals and superoxide. These species cause a rupture in the cell wall. Since nanoparticles have a high surface area, the contact area of these species with bacteria increases, which create a higher probability of damaging bacterial cell walls and cytoplasmic membranes.

This study demonstrated that gram-negative bacteria are highly susceptible to TiO_2 nanoparticles compared to gram-negative ones. The observable variation in the diameters of the inhibition zone is related to differences in the susceptibility of bacteria. Other factors that can also be critical to effective inhibition are nanoparticles' morphology, composition, size and shape. These factors are mainly related to the processing conditions. NPs of TiO_2 exhibit a strong antibacterial activity against gram -positive and -negative bacteria. The size of the inhibition zone revealed a strong performance for the nanoparticles. These findings highlight the potential of TiO_2 NPs for biomedical and health

Conclusion

In conclusion, this study contributes to the evolving landscape of nanomaterials to prepared TiO_2 nanopowder. The outcomes of this study hold promise for biological activity application, the properties of the TiO_2 nanopowder is poised to unlock new possibilities in materials engineering and technology development.

References

Abdulazeem, L., Al-Amiedi, B. H., Alrubaei, H. A., & AL-Mawlah, Y. H. (2019). Titanium dioxide nanoparticles as antibacterial agents against some pathogenic bacteria. Drug Invention Today, 12(5), 963-967.

Ali, I., Suhail, M., Alothman, Z. A., & Alwarthan, A. (2018). Recent advances in syntheses, properties and applications of TiO 2 nanostructures. RSC advances, 8(53), 30125-30147.

Banerjee, S., Dionysiou, D. D., & Pillai, S. C. (2015). Self-cleaning applications of TiO2 by photo-induced hydrophilicity and photocatalysis. Applied Catalysis B: Environmental, 176, 396-428.

Breckenridge, R. G., & Hosler, W. R. (1953). Electrical properties of titanium dioxide semiconductors. Physical Review, 91(4), 793.

Chen, M., Zhang, J., Yang, T., Mao, S., & Zhao, H. (2023). Peroxymonosulfate activation for preferential generation of hydroxyl radical with atomic Mn anchored TiO2 in photoelectrochemical process. Environmental Functional Materials, 2(1), 13-24.

Foster, H. A., Ditta, I. B., Varghese, S., & Steele, A. (2011). Photocatalytic disinfection using titanium dioxide: spectrum and mechanism of antimicrobial activity. Applied microbiology and biotechnology, 90, 1847-1868.

Grätzel, M., & Rotzinger, F. P. (1985). The influence of the crystal lattice structure on the conduction band energy of oxides of titanium (IV). Chemical Physics Letters, 118(5), 474-477.

Hammoodi, R. A., Abbas, A. K., & Elttayef, A. K. (2014). Structural and optical properties of CuO thin films prepared via RF magnetron sputtering. Int. J. Appl. Innov. Eng. Manag, 3(7), 1-7.

Hammoodi, R., Khalaf, M., & Abbas, A. (2018). Construction of the Non-Thermal Helium Plasma Jet using Two Electrodes and studying its Characteristics. Int. J. Sci. Res. IJSR, 8, 1372-1378.

Hammudi, R. A., Hammudi, Y. A., Salim, A. A., Alsadoon, Z., Radh, R. S., Abbas, I. M., & Hammad, A. T. A. (2023). Plasma in Dentistry. Malaysian Journal of Fundamental and Applied Sciences, 19(3), 332-336.

Hammudi, R. A., & Mahmood, M. A. (2025). Cold Atmospheric Plasma in Chronic Diabetic Foot Ulcers. Int. J. Thin. Fil. Sci. Tec, 14(1), 15-20.. doi:10.18576/ijtfst/140103

Hammudi, R. A., Alsadoon, Z., Mahmood, M. A., Allawi, R. A., Lafta, H. A., Al Hasani, M. H., & Sharhan, S. I. The antimicrobial activity of (DBD) homemade plasma jet against Staphylococcus aureus using different gases.

Huang, Z., Maness, P. C., Blake, D. M., Wolfrum, E. J., Smolinski, S. L., & Jacoby, W. A. (2000). Bactericidal mode of titanium dioxide photocatalysis. Journal of Photochemistry and photobiology A: Chemistry, 130(2-3), 163-170.

Lafta, H. A., Allawi, R. A., Mahmood, M. A., Hammudi, R. A., Al Hasani, M. H., Sharhan, S. I., & Abbas, I. M. (2023, February). Effect of radiation exposure on function human body by CBC parameter for workers in nuclear medicine field. In Journal of Physics: Conference Series (Vol. 2432, No. 1, p. 012002). IOP Publishing.

Liu, Z., Chen, Q., Wang, Z., Yang, L., & Wang, C. (2011). Production of titanium dioxide powders by atmospheric pressure plasma jet. Physics Procedia, 18, 168-173.

Mahdy, S. A., Mohammed, W. H., Emad, H., Kareem, H. A., Shamel, R., & Mahdi, S. (2017). The antibacterial activity of TiO2 nanoparticles. J. Univ. Babylon, 25(3), 955-961.

Mahmood, M. A., & Hammudi, R. A. (2024). Spectroscopic, Diagnostics, and Surface Modification of Stainless Steel Doped with Ti Plasma Produced by DC Magnetron Sputtering and Used in Biomedical Applications. Tribology in Industry, 46(4), 736.DOI: 10.24874/ti.1787.11.24.11

Matsunaga, T., Tomoda, R., Nakajima, T., & Wake, H. (1985). Photoelectrochemical sterilization of microbial cells by semiconductor powders. FEMS Microbiology letters, 29(1-2), 211-214.

Renuka, R. R., Jayaraman, N., Julius, A., Palanivel, V., Ramachandran, V., Pandian, R., ... & Subbiah, S. K. (2023). Synthesis, Characterization and Applications of Titanium Dioxide Nanoparticles. In Biomedical Applications and Toxicity of Nanomaterials (pp. 339-361). Singapore: Springer Nature Singapore.

Riad, K. B., Wood-Adams, P. M., & Wegner, K. (2018). Flame-made TiO2 (B). Materials Research Bulletin, 106, 276-281.

Stephen, L. (2020). Titanium dioxide versatile solid crystalline: An overview. Assorted dimensional reconfigurable materials, 1, 92056.