International Journal of Nanoelectronics and Materials
Volume 12, No. 3, July 2019 [329-338]

Studying the Effect of Synthesized Nano-Titanium Dioxide via Two Phases on the Pseudomonas Aeruginosa and Portus Bacteria as Antimicrobial Agents

Zainab N. Jameel[1*], Olfat A. Mahmood[2] and Faisal L. Ahmed[3]

[1]*Communication Engineering Department, University of Technology, Baghdad, Iraq.*
[2,3] *College of Science, University of Diyala, Diyala, Iraq.*

Received 12 November 2018, Revised 17 December 2018, Accepted 31 January 2019

ABSTRACT

The Titanium dioxide (TiO_2) Nano-particles (NPs) is synthesized via the Sol -gel method by using titanium tetrachloride ($TiCl_4$) as a starting material while varying the applied temperature in the range of (400-900)°C. The main features of the synthesized NPs have been examined by using X - ray diffraction (XRD), Field Emission Electrons Microscopy FESEM and Transmission Electron Microscopy (TEM). The main test of TiO_2 NPs was carried out as an antimicrobial and antibacterial agent utilizing four types of bacteria strains (Pseudomonas aeruginosa) and Portus. Results of XRD show that the Titanium dioxide structure demonstrate an Anatase phase at (400 and 450)°C as well as the rutile phase at 900°C. The size of particles is found to be in the range of (15-20) nm for Anatase and for the rutile is in the range of (40-60) nm. FESEM results show the form was crystalline and appeared in the form of cauliflower or clusters for Nanoparticles. The antimicrobial agents can be utilized by the two phases of TiO_2 for two types of bacteria (Pseudomonas aeruginosa) and Portus. The obtained results demonstrate that TiO_2 in anatase or rutile phases is effective as anti-bacterial agents.

Keyword: Tio_2 Nanoparticles , $Ticl_4$ Precursor, Sol-Gel Process , Tio_2 Phases, Antibacterial activity, Antimicrobial Agents.

1. INTRODUCTION

In the last decade, nanomaterials play crucial role in various fields of science such as biology, medicine, pharmacology and water industry, making positive attitude toward this research field more than ever. Water borne diseases and water quality are considered essential issues for scientists all over the world [1]. Due to high resistance of microbial agents, scientists believe that the nanotechnology is a valuable agent in multi fields. Properties of nano-materials differ significantly from those of their original molecules, and such differences made have made nanotechnology multidisciplinary [2]. As one in all the foremost sensible technologies, nanotechnology has developed due to distinctive properties of chemical and physical nano-materials, for example Nano-particles of TiO_2, no more than one hundred “nm” in diameter, have turned into a novel origination of the innovative material because of their own vivid and motivating visual properties, dielectric behavior and photo-catalytic characteristic as of quantization of size [3]. TiO_2 is considered as an active photo-catalyst as a self - cleaning and self - sanitizing constituent of the covering for exteriors in many applications [4]. Moreover, it can be used in the purification of environment due to its non-toxicity is photograph persuaded superior-hydrophobicity then anti-fogging influence. Such characteristics are extremely useful in eliminating bacteria in addition to damaging biological material with in air and water,

*Corresponding Author: *11042@uotechnology.edu.iq*

furthermore, it can be utilized as self - cleaning or self - sterilizing exteriors of spaces like centers of medical care [5].

Titanium dioxide has been utilized in many applications due to its extreme stability, low cost, bio-capacity, reused in addition exploitation consequence basically with the case of photo-catalysis, reinforce of catalyst, antibacterial, environmentally friendly remediation, cleansing of air and purification of water [6]. TiO_2 possesses robust influence of oxidizing; hence it can be employed to destroy microorganism under Ultra-violet light luminosity [7,8]. In the recent decades, sinceTiO_2 NPs acts as antibacterial material; it has been used in biology, pharmaceutical and environmental applications. For example, TiO_2 NPs has been utilized in photocatalysts, gas sensors, in addition, it has been used in commercial industrial applications, e.g. solar cell and nutrients technology, ointments, toothpaste, pigment, cosmetics, and paints [9].

Researchers have demonstrated some critical topic about "titanium dioxide" which is the importance of TiO_2 extensive application in self- cleaning and sanitizing materials for several healthful instrumentation of clinical ware , eating – change of state ware and hospital materials beneath consumption. Due to its TiO_2 incontestable by nontoxicity, physical and chemical properties. The organic compounds and microbic organisms decay, like cancer cell, viruses, and therefore the bacterium chiefly related to the victimization of "titanium dioxide" in addition, because the potential applications of medical devices of sterilization [10].

1.1 Synthesis of TiO_2 NPs Powder via the Sol - Gel Method

Tetrachloride of Titanium ($TiCl_4$) (99. 99%) and Ethanol-CH_3CH_2OH, (99. 99%) were used as starting materials. The synthesis process is achieved by adding a sequence of droplets from $TiCl_4$ into the absolute Ethanol with 1:10 ratio [1,2]. The reaction was realized by using magnetic stirrer in the chemical fume hood in order to expel undesired toxic gases i.e. cloud HCl under room temperature. A light yellow solution with PH in the range of 1.4-2 was attained from the formation process. The obtained solution has been converted to gel state by applying 80°C temperature for (24) hours. The calcination process was used to realize the anatase and rutile phases for TiO_2. Anatase phase is obtained under (400-450)°C over (1.5-2) hours; on the other hand, rutile phase is achieved under (900)°C over (1.5-2) hours.

1.2 Characterization of Titanium Dioxide NPs

The structural properties of the realized TiO_2 NPs was analyzed by using X-Ray diffraction; moreover, the surface morphology of TiO_2 NPs was studied by employing Field Emission Scanning Electron Microscopy (FESEM), Transmission Electron Microscope (TEM), and Scanning Electron Microscopy (SEM).

2. RESULT AND DISCUSSION

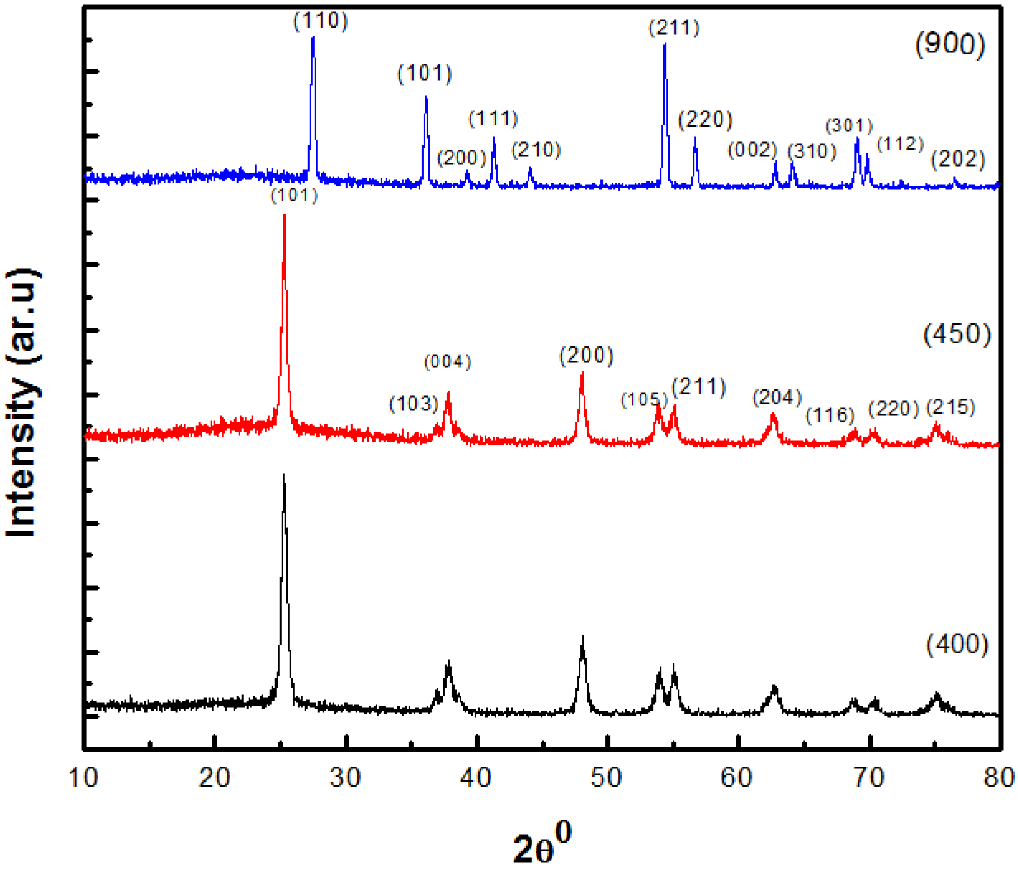

Figure 1. XRD pattern Anatase & Rutile phases at different temperatures (400, 450, and 900)°C.

The structure and the phases of TiO_2 NPs have been determined by X-Ray Diffraction analysis. Figure 1 shows that almost all of the crystal type in spectrum is anatase at (400°C, 450 °C). The intensity peaks of TiO_2 polycrystalline in anatase phase oriented at diffraction angles 2θ at (25.26˚, 36.89˚, 37.78, 48.07˚, 54.03 ˚, 55.07 ˚, 62.67 ˚, 68.79 ˚, 70.43˚and 75.20 ˚ with diffraction planes (101), (103), (200), (004), (200), (105), (211), (204), (116), (220) and (215). The same figure illustrates the peaks of TiO_2 polycrystalline in rutile phase at(900°C)oriented at diffraction angle 2θ (27 .5 ˚, 36 .14 ˚, 39 .27 ˚,41 .36 ˚, 44 .04 ˚, 54 .33 ˚, 56 .71 ˚ , 62.28 ˚, 64 .16 ˚ ,69 .08 ˚,69 .83 ˚ and 76 .54 ˚) with diffraction planes (110), (101), (200), (111), (210), (211), (220), (002), (310), (301), (112) and (202) respectively. The results obtained from X-Ray analysis are consistent with American Society of Testing and Materials (ASTM) and in good agreement with results presented in[1,2].

From the previous results, the intensity of the X-ray diffraction increases with growing temperature because of the agglomeration of particles, and the crystalline phase of TiO_2 converted from anatase phase to rutile phase. In addition, it can be identified that when the crystallite size increases, the peak of diffraction becomes narrow than the TiO_2 calcite at 900°C. In this study the mean particle size for anatase and rutile samples has been evaluated based on "Scherer's equation" in the range of (15 - 20) nm and (40-60) nm for anatase and rutile phases respectively.

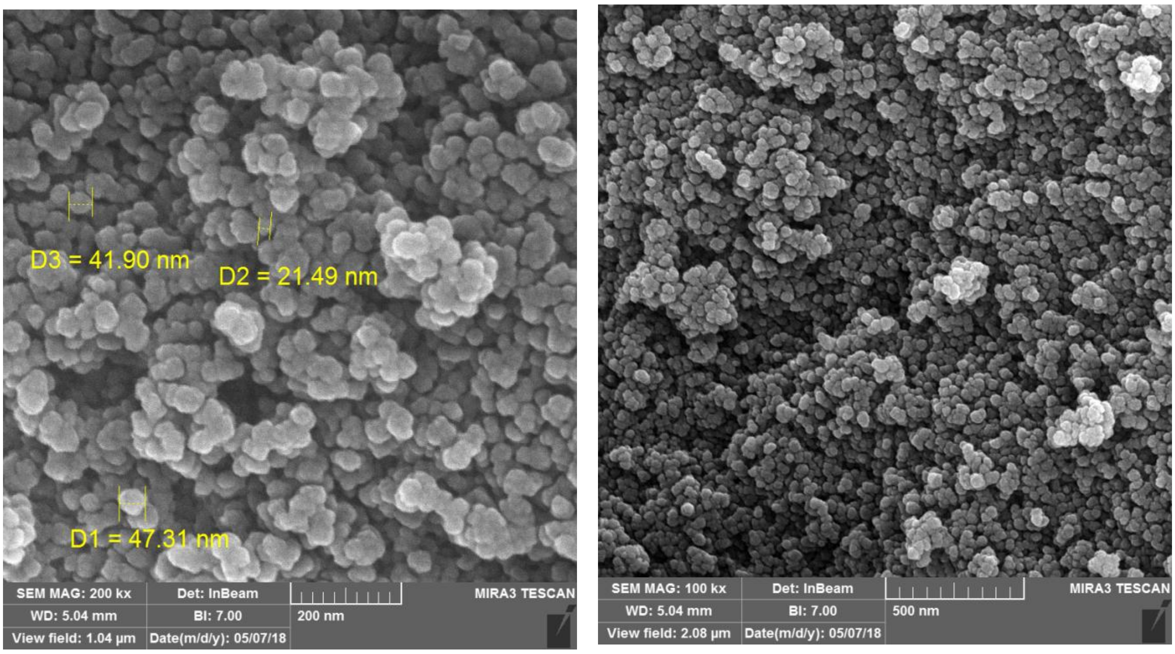

Figure 2. FESEM images of TiO_2 NPs for anatase phase.

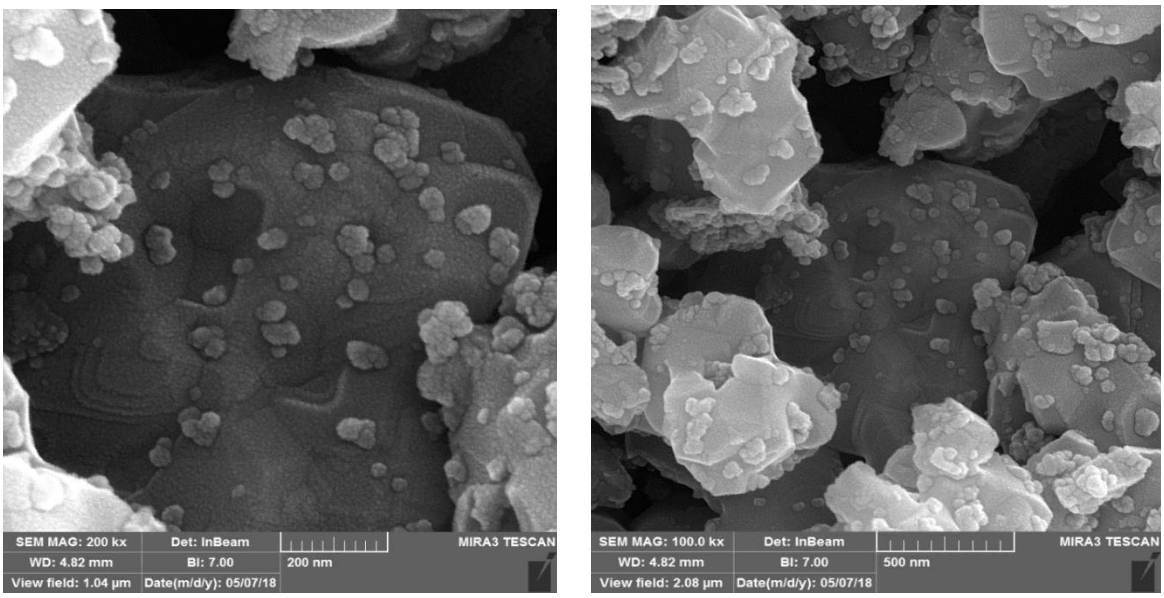

Figure 3. SEM images for TiO_2 NPs for rutile phase**.**

The morphological study of the synthesized TiO_2 NPs at different calcination temperatures has been characterized utilizing the Field Emission Scanning Electron Microscope (FESEM) to measure the grain size and shape of the TiO_2 nano-powder. The morphology of calcined TiO_2 NPs powder at (400°C and 450°C) observed by The Field Emission SEM as shown in Figure 2. TiO_2 NPs have spherical form of nanoparticles in the range of (20- 50) nm. When the temperature increases to 900°C, the size of TiO_2 NPS has been increased to be larger and accumulation comes to be substantial, as illustrated in Figure 3. TiO_2 NPs have been exhibited non-uniformly of the particle shape because of the initial particles accumulation of aggregating is within the same size of crystalline. The accumulation in calcination temperatures may expressively accumulates the particles cause the little growth. The sizes of the calcination temperatures at 400, 450 and 900°Care in the range of (36.9 and 58 nm) respectively and these results are in covenant through informed standards [11]. Such results have been covenanted through X-Ray diffraction outcomes by which displayed size of particles for anatase phase lesser than the rutile phase.

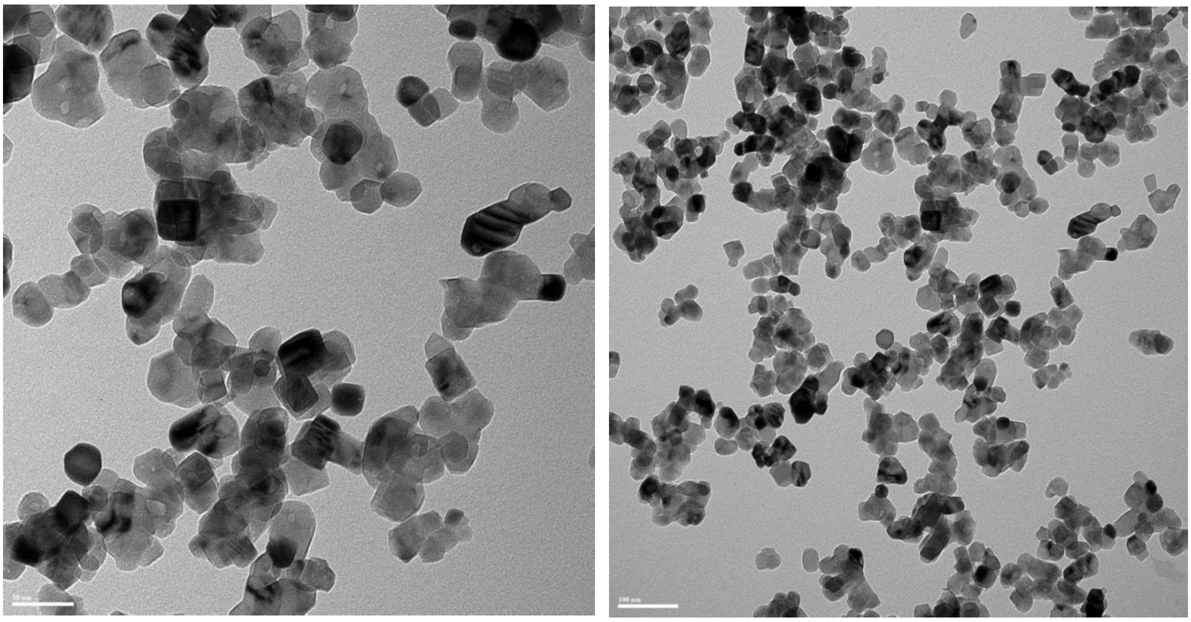

Figure 4. TEM images of anatase phase for TiO_2 NPs.

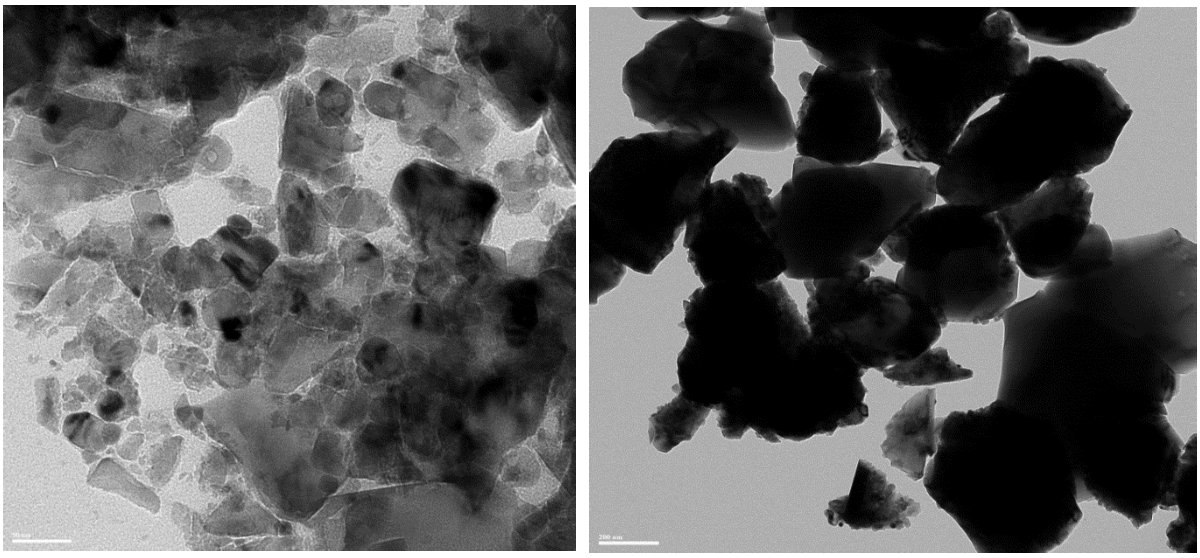

Figure 5. TEM images of rutile phase for TiO_2 NPs.

Transmission Electron Microscopy (TEM) Results introduce both the size and the shape of the particles from anatase and rutile phases of TiO_2 nanoparticles synthesized by using the $TiCl_4$ as starting material via the Sol - Gel procedure. TEM for TiO_2 NPs in Anatase phase have been measured the size and the shape in the range of (20-100) nm with "hexagonal uniform" also spherical homogenous particles, these results agreed with the reference[12]. The homogenies of TiO_2 NPs allow for such particles to effect on to the local paint. The anatase phase will be utilized in paint due to its photo-catalytic features that is used in self-cleaning surfaces and antibacterial agents. Figure 4 and Figure 5 demonstrate the TEM images for the rutile phase of TiO_2 NPs at different sizes in the range of (50- 200) nm with elliptical shape and can be employed in paint application due to their characteristics.

2.1 The Study of Antibacterial Activity

It is believed that the charges are carried by the metal oxides whereas the negative charges are carried by microorganisms. This leads to the conclusion that the magnetic force attraction between microorganisms and therefore the metal oxides cause the chemical reaction and eventually death of microorganisms. They end up in pits or holes in the wall of microorganism cell and can be connected with internalized particles, resulting in increasing porousness and therefore the death of the cell. The TiO_2 NPs effect due to their little size and high surface to the volume quantitative relation, endure the next level of interaction with the microorganism cells surface rather than the larger particles, leading to a high antibacterial drug activity. It has been detected that NPs "TiO_2" arranged by two dissimilar present attentions displayed square of anti-bacterial activity. TiO_2 NPs Antibacterial activities have appeared via "agar" fit dispersion technique in contradiction of "Portus and pseudomonas" aimed at TiO_2 NPs of three different concentrations at (0.002, 0.004 and 0.006) g/ml. all samples of Anti-bacterial circle-photos. If the diameter of antibacterial circle of one sample is larger than 7mm, it means that sample has improved antibacterial activities, nevertheless, if antibacterial circle diameter equals to or less than 7 mm, it means that the sample has poorer antibacterial activity. From the results in Table 1 below it can be seen that all samples have better antibacterial activity because their antibacterial circle diameter is much larger than 7 mm .The TiO_2 nanoparticles are proved to be exact actives on confirmed strains of Gram - positive, the differential sensitivity of gram-negative and gram-positive bacterium toward nanoparticles can be explained by the very fact that the liquid medium may be affirmative. The shut interaction between the suspended nanoparticles and therefore the gram-positive microbic cells, that might be highly attached and anchor to the surface of the microbic cell, inflicting structural changes and damages resulting in death [12]. Bacteria of Gram-positive have a comparatively thick wall composed of "many layers of peptidoglycan" chemical compound, and just 'one membrane' (plasma membrane). Bacteria of Gram-negative have merely a skinny layer of 'peptidoglycan' and a lot of advanced cyto-membrane with 'two' cell membranes, an exterior membrane, besides a 'plasma membrane' [14]. Adding of the outer membrane of the bacterium of gram - negative influences the cells porousness of the many molecules. Under bounded conditions, the gram-negative bacterium is much immune to several chemical agents than gram-positive cells. Additionally, the cell walls of gram-negative bacterium are more liable to mechanical breakage due to the low quantity of peptidoglycan [14.15,16]. It seems that antibacterial activities of the nano-materials increase with escalation of surface –to-volume proportion. Thus, reduction occurs in nano-particles size[17] .

Table 1 The antibacterial activity of TiO_2 (for two phases (Anatase & Rutile) by the Wall - Diffusion procedure

Temperature °C	Concentration g/ml	Zone of inhibition mm	
		Proteus	Pseudomonas
400	0.002	26	17
	0.004	28	19
	0.006	30	21
450			
	0.002	23	14
	0.004	24	15
	0.006	26	17
900	0.002	12	11
	0.004	13	13
	0.006	15	14

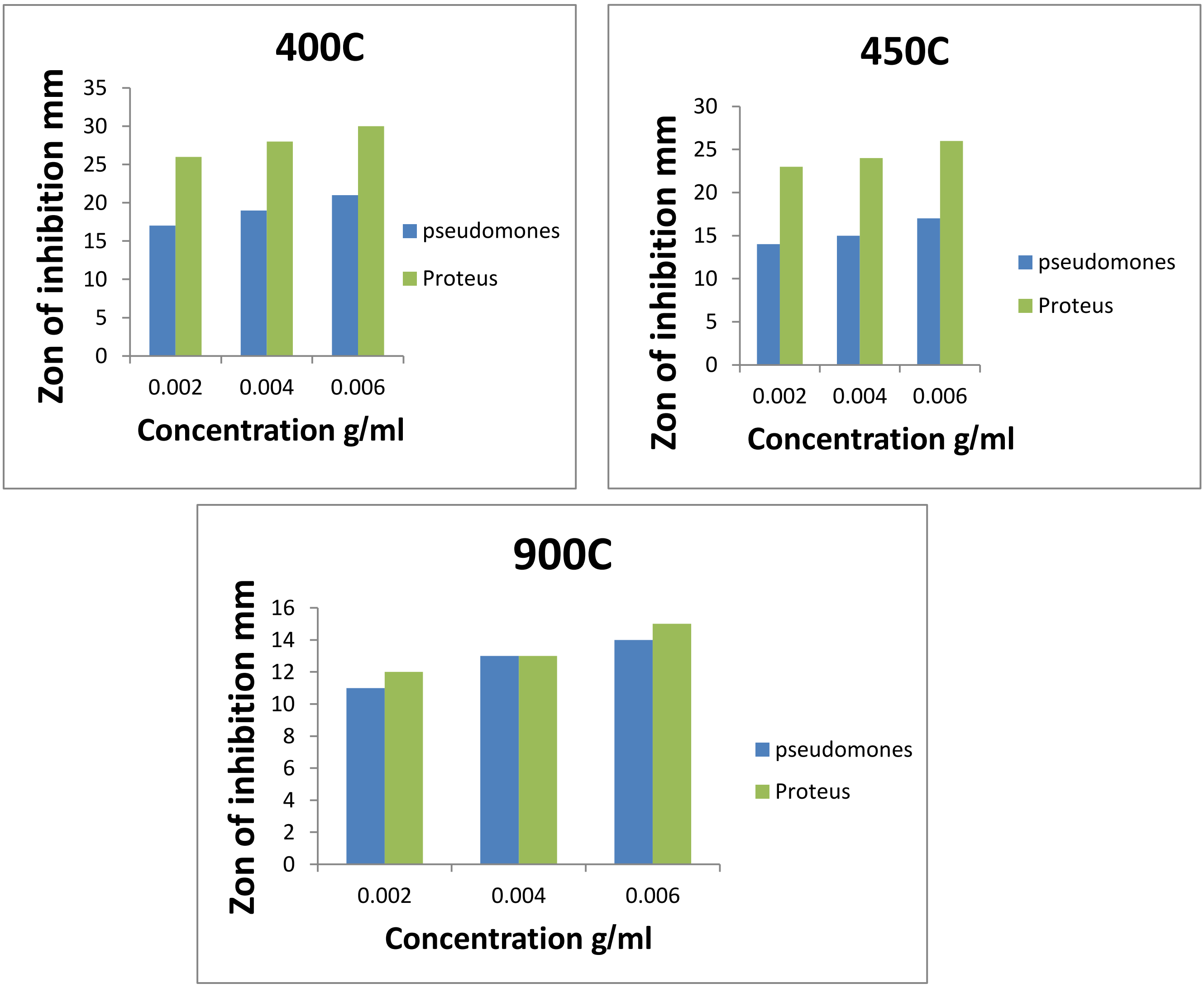

Figure 6. Antibacterial activity of anatase phase (Portus and Pseudomonas) at concentration (0.002, 0.004 and 0.006) g/ml by using Will-Diffusion.

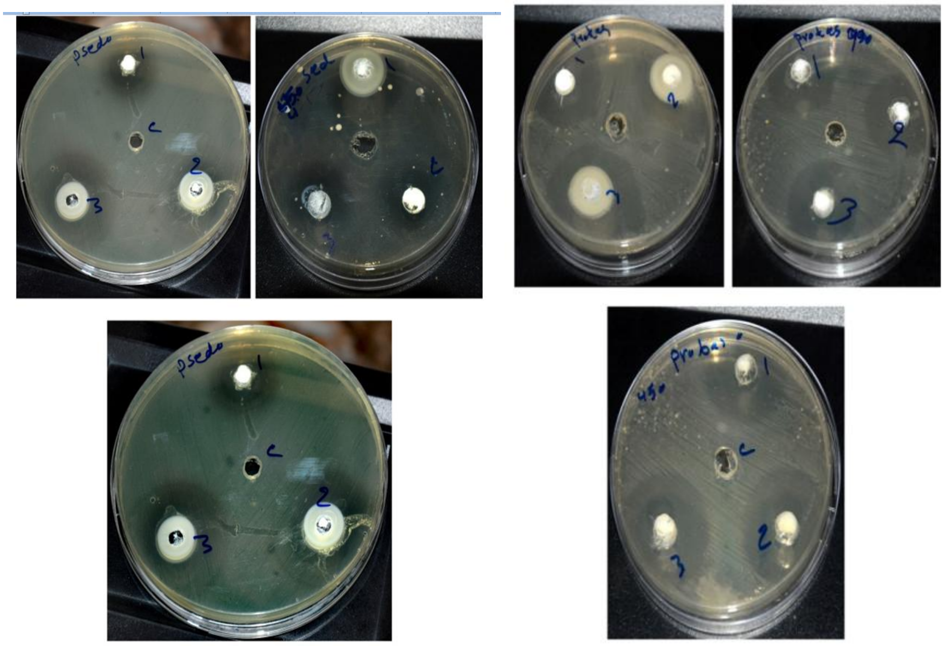

Figure 7. Photograph of antibacterial activity of Tio_2; Portus & Pseudomonas.

3. CONCLUSION

We obtain the TiO_2 NPs at different temperatures in the range of (400,450 and 900) ^oC will be managed on the scale of the particle size for the anatase phase of TiO_2 NPs in the range of (15-20) nm dissolved in water appropriate for the testing of medicinal drug agents and the large-size for the rutile phase of TiO_2 NPs in the range of (40-60) nm liquefied in 'ethanol' using as a self - cleaning tests. The typical size of particles of TiO_2 NPs increases with the temperature due to the crystallites accumulation which is very important to the creation of large-particles. In Rutile phase a 'correlation' occurs concerning the results of (XRD, FESEM & TEM) have been represented the crystal dimension for the fabric is to be regarded as 50 nm. The results of the FESEM tested of the anatase phase displaying that the procedure within the kind of Nano-structures spherical clusters and incompletely the formula of cauliflower. The NPs of anatase arisen by extra efficacy of Nano-particles rutile using as a 'Gramothic antagonist' contrary to bacteria in addition through concentration of ,' 0.002, 0.002 and 0.006' g/ml.

ACKNOWLEDGMENT

The authors gratefully acknowledge the technical support provided by College of Science, University of Diyala, Diyala, Iraq, also we would like to thank the center of Nanotechnology and Advanced Materials Research, University of Technology, Baghdad, Iraq for supporting in experimental research.

REFERENCES

[1] Zainab N. Jameel , Adawiya J. Haider, Samar Y. Taha "Synthesis of TiO_2 Nanoparticles by Using Sol-Gel Method and Its Applications as Antibacterial Agents" Eng. & Tech. Journal **32**, part B No.3 (2014).
[2] Zainab N. Jameel, Adawiya J. Haider, Samar Y. Taha, Shubhra Gangopadhyay, Sangho Bok "Evaluation of Hybrid Sol-gel Incorporated with Nanoparticles as Nano Paint," (AIP-Scitation) **1750**, (2016).
[3] Adawiya J. Haider, Zainab N. Jameel, Samar Y. Taha "Synthesis and Characterization of TiO2Nanoparticles via Sol-Gel Method by Pulse Laser Ablation," Eng. & Tech. Journal **33,** part B No.5 (2015).
[4] Greenberg, A. E, Clesceri, L. S. & Andrew, D. E. Standard Methods for examination of water and wastewater. American Public Health Association, 18th Edition, (1992).
[5] Zhang, W. X., Nanotechnology for Water Purification and Waste Treatment. Frontiers in Nanotechnology US EPA Millennium Lecture Series. Washington D. C (2005).
[6] Phong, N. T, Thanh, N. V. & Phuong, P. H. Fabrication of antibacterial water filter by coating silver nanoparticles on flexible polyurethane foams. Journal of Physics: Conference Series 187, (2009).
[7] Sadiq, M., Dalai, S., Chandrasekaran, N. & Mukherjee, A. Ecotoxicity study of (TiO2) NPson two microalgae species: Scenedesmus sp. and Chlorella sp. Ecotoxicology and Environmental Safety **74** (2011) 1180–1187.
[8] K. Venkatasubramanian, S. Sundaraj, Antibacterial activity of Zinc Oxide and Ag Doped Zinc Oxide Nanoparticles against E. coli, Chem. Sci. Rev. Lett. **3** (2014) 40-44.
[9] K. Gupta, R. P. Singh, A. Pandey, Photocatalytic antibacterial performance of TiO2 and Ag-doped TiO2 against S. aureus, P. aeruginosa and E. coli, Beilstein J. Nanotechnol. **4** (2013) 345–351.
[10] J. Liu, Y. Ge, X.Z. Zhi, Study of antibacterial effect of polymethyl methacrylate resin base containing Ag-TiO_2 against Streptococcus mutans and Saccharomyces albicans in vitro, West China J. Stomatology **30** (2012) 201–205.
[11] Awati, P. S., Awate, S. V., Shah, P. P., Ramaswamy, V., "Photocatalytic decomposition of methylene blue using nanocrystalline anatase titania prepared by ultrasonic technique", Catalysis Communications **4**, 8 (2003) 393-400,.
[12] R. M. Abdullah, "A Study the Effect of TiO2 Nanoparticles Combination with Antibiotics and Plant extracts Against Some Gram Negative Bacteria**.**
[13] Tianyou. P, De. Z, Ke. D, Wei. Sand. K. H, "Synthesis of Titanium Dioxide Nanoparticles with Mesoporous Anatase Wall and High Photocatalytic Activity"J. Phys. Chem. B **109** (2005) 4947-4952.
[14] R. Vijayyalakshmi & V. Rajendran, "Synthesis and characterization of nano-TiO_2 via different methods," Archives of Applied Science Research **4**, 2 (2012) 1183-1190.
[15] Chwalibog, E. Sawosw, A. Hotowy, J. Szeliga, K. Mitura, M. Grodzik, P. Orlowski, A. Sokolowka Visualization of interaction between inorganic nanoparticles and bacteria or fungiInt. J. Nanomed. **5** (2012) 1085-1094,
[16] G. Tortora, R. B. Funke, L. C. Case "Microbiology: An Introduction" Addison-Wesley Longman Inc., New York, (2001).
[17] Priyanka. A, Ganesh. K, Suresh. G, Anjali. R "Synthesis of TiO_2 nanoparticles by electrochemical method and their antibacterial application," Arabian Journal of Chemistry, (2005).