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## Preparation of Nano-TiO<sub>2</sub> and improve its antibacterial activity

A Thesis

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By

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#### (1.1) INTRODUCTION

The side of discipline specializing in materials style, fabrication, characterization and application moreover as Nano-scale techniques calls Nano-technology. one in every of the foremost important branches data of data of information thought of as sub-category for the technology in mixture knowledge chemistry, physics, and biology though varied scientific ambit enclosed discovering phenomena and doctrinaire of components in Nano-scale[1,2]. Moreover, an unusual role of Nano-technology may be a speedily increasing ratio of extent towards dimensions, exists in many Nano-sized constituents that concentrate on new exterior potential situated continuously in science [3, 4].

Lately, more than a few theoretical examinations have focused on discovering operational package, recommend resolves for case of humidity-instructed each capacity movement of antimicrobial or each. [5].

Above previous decades, inorganics agent of bacterial-static containing Nano-particles dioxided titanium,  $TiO_2$ , necessarily paid a plentiful transaction of consideration. As  $TiO_2$ , considered as comprehensively utilizes in numerous submissions as per of self-disinfectors in addition self-cleaners substantial [6,7].

Aforementioned analysis has once and for all shown that dioxide one common materials in use in several suggestions for the reason that semiconducting, photo-catalytic, transfer of energy, identifying titanium gas and electronic physiognomies. of dioxide of titanium crystals exist in three-polymorphs in natural surroundings in which as like as ,redundancy, anatase, brookite besides rutile[8].

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rutile as well as anatase all at once thought-about as dioxide Titanium of two- sides crystal combinations, thus far, with chemicals alongside anatases course in additionally responsive method. In recent times, researches exposed associate exaggerated attention in metal as nanoparticle and its submissions in numerous disciplines as a photo-catalyst as in water sanitization [9]. Measurement of four-sided NPs counterfeit through "ultraviolet radiation ultraviolet illumination (UV) actinic radiation. actinic ray light, like large energy band gap semiconductors metal dioxide(TiO<sub>2</sub>) very investigated the capability to eliminate organic pollutants of varied media . in addition, TiO<sub>2</sub> could also be practicable in coating, air purification, plastics, paper inks, medicines, pharmaceuticals ,food products, cosmetics, actinic ray protection, photo-catalysis, sensing and dentifrice [10-21]. It will even use as a pigment to colour skim milk [22]. The section of anatase chiefly additional for antibacterial drug implementations by suggests that of the crystal production and a complicated band gap of "3.2 eV unit" comparing with "3 eV in rutile". Promptly obtainable, cheap, and small toxic of entire options occur in NPs. Characteristic of TiO<sub>2</sub> semi-conductive contemplates as considerable of TiO<sub>2</sub> radiation eradicating semi-conductor conservational various organic pollution throughout an energy of light-weight more than similar gap band, by which maybe produce hole-paired electron. Probably going to be defeated in several reduction procedures at the semiconductor or resolve crossing point [23].

Surrounded by supplementary than limited semi-conductor oxide, further supporting roles in cleansing surroundings as of no-toxic, pho tocatalytic purpose, photo influenced superbly-hydrophilcty in addition to anti - confusing significance appears in  $TiO_2$ . The features must be exploited on removing bacteria as well as unhelpful water plus air



materials that are organic, adding to (self-cleaning or self-sterilizing) surfaces in medicinal center[24]. TiO<sub>2</sub>, is commonly utilized by means of every vivacious in coat white besides formation acquisitions, nourishment stain, sun-block plus produces that have cosmetic usage. Bactericidal act of TiO<sub>2</sub> inductees during obliteration oxidative of bacteria walls, decrease in facial energy interior of bacteria bond origins chemical escalation interface in the middle of and carbon diamonded - like coatings and E.coli, in view of additional purpose aiming increases activities of bacteria [25].

Essentially, significance (TiO<sub>2</sub>) has earned according to highly constancy, low charge, bio capacity, re-usability and utilizations effect essentially in photo catalysis, catalyst reinforce, anti-bacterial, conservational re-mediation, air cleansing, water decontamination[26]. TiO2 contains compacted power that oxidizes in the direction of eliminate micro-organism underneath UVlight brilliant[27-28].

Currently, Nano-particles of Titanium dioxide properties are one amongst the most important fascinating analysis subjects because of extremely result accomplishment in biological, pharmacological submissions. environment substance decontamination, electronics arrangement, solar power cells, photo-catalysts, photo-electrodes in addition sensor of aboard American Food gases and Drug Administration(FDA) commendation of persecution in technology of nutrient besides medicines, ointments, paint pigments, cosmetics, also dentifrice [29].

One of most important concern in  $(TiO_2)$ Titanium dioxide research showing TiO<sub>2</sub> significance comprehensively submission of enhancement and materials disinfecting meant for more than a few healthful clinical instrumentality ware, eating – preparation ware, and hospitalists material



below consumptions. pigment  $TiO_2$  characterized via no-toxicity, chemical also physical outdo reliability. Organic combinations plus micro-organism disintegrating, like cells of cancer, virus, and micro-organism usually related to the usage of  $TiO_2$ . Additionally probable submission of medically expedients purification [30].

Classification of self- refinement collected by titanium dioxide(TiO<sub>2</sub>) photo-catalytic accustoms with three vital features simulation through ultraviolet(UV) light:

- strong oxidation power that eliminates odor causing bacteria;
- The breakdown of long chain organic molecules into smaller ones;
- A surface that experiences super-hydrophilicity, which allows these small-chained organic molecules and everyday dirt and stains to be easily washed away with water [20].

The experiment observes the importance of carbon nanotube oxide preparation within the rise of a sol-gel methodology that used as associate degree antibiotic on separated micro-organism Insulates from patients at Baquba General Hospital. Nanoparticle showed high effectivity on insulated bacterium motivation.

#### 1.2 Aims of Study

The aim of this study is to synthesis  $TiO_2$  nanoparticles by sol- gel method using Titanium Tetrachloride (TiCl<sub>4</sub>) as a precursor with different temperatures (400,450,900) °C, study characterization of every structural, morphological, electrical and optical properties of the ready nanomaterials and Evaluating the effect of  $TiO_2$  nanoparticles as antibacterial agents against four type of bacterium; gram-negative bacteria (*Pseudomonas aeruginosa ,E.coli*)and Proteus, and grampositive bacterium (*Staphylococcus aurous*).



#### **1.3 Literature Review**

In Sirimahachai, *et al* (2009), investigated the differential impact of a photo catalysts TiO<sub>2</sub> powders grounding throughout sol-gel process utilizing precursor of TiCl<sub>4</sub>, via transmitting reaction conditions, acids, water contented, and trivalent (Al, B) dopants. (XRD, SEM, and UV-Vis) careful as categorizations of the prepared Titanium dioxide powders . The TiO<sub>2</sub> antibacterial activity discussed through comparing commercial product sand anatase, rutile, and P25 Degussa with five types of bacteria containing *E. coli*, *P. aeruginosa, Bacillus subtilis, Staphylococcus aureus, and methicillin-resistant S. aureus* (MRSA), by an exhaustive evaluation of the agar dilution method usage. All prepared samples have detected throughout varying degree of inactivation activity competence, where two of them revealed a extensive higher action than Degussa P25 [31].

Stoyanova et al (2010), A non-hydrolytic sol-gel technique produces TiO<sub>2</sub> similarly measuring antibacterial structures. Thus, feedback between titanium tetrachloride (TiCl<sub>4</sub>) and benzyl alcohol results synthesis of titanium dioxide in which has been accomplished at moderate temperature, subsequently with calcinations at 500 °C. Prepared TiO<sub>2</sub> nanoparticle construction and morphology categorized through XRD and SEM. Heightened the average of  $TiO_2$  (anatase) particles size of was 10-20 nm. It appears from the aforementioned investigations that numeruos investigation have been conducted the effects of prepared TiO<sub>2</sub> nanoparticles antimicrobial action. However, an attempt was made to Escherichia coli utilization microorganism investigate as test. Nevertheless, the study found providing details revealing the probability of successfully utilized no hydrolytic sol-gel method nanosized TiO<sub>2</sub> (anatase) for sanitization of water on illumination with UV light [32].



Aashis S. Roy *et al* (2010), deliberated to work out the impact of producing oxide Nano-proportions via citric acid and alpha-dextrose alternative anatomize the atomic number 22, NPs impact on medication action of many anti-biotic counter to MRSA. associated valuation of MRSA development of TiO<sub>2</sub> nanoparticles presence that a lot of vital findings emerged from an too excellent protection profile and nontoxic ascertained once occupied at completely different Nano- sized. selected collected, compound as per of staggeringly benign multifaceted measured in integration treatment distinguished with MRSA in line for latent acting-synergetic through thoughtful anti-biotic for instance lactum-beta besides antibiotic drug [33].

Saowaluk Boonyod *et al* (2011), The analysis has attended target  $TiO_2$  sol fusions from metal tetraisopropoxide (TTIP) as precursor in acidic binary compound solutions via sol-gel technique and once reflection at 80°C for two h through a house kitchen appliance operation. investigation the differential impact of microstructural and therefore the bactericide perspective of ready powders. Analysis of bactericide ready powder action contrasted through the microorganism (E. coli) conjointly compared to the trade P25 Degussa TiO<sub>2</sub> powder [34].

**Vijayalakshmi and Rajendran** *et al* (**2012**), deliberates experiments techniques for facilitating and endorsing  $\text{TiO}_2$  via compination of a nonhydrolytic sol-gel methodology from Ti compound and unit alcohol besides medicinal drug choices exploration. The antimicrobial action of as-prepared TiO<sub>2</sub> mentioned through utilizing *E. coli*, *P. aeruginosa and S. aureus* impact of lightweight. The consideration of organism progress made-to-order with incidence of TiO<sub>2</sub>, by the UV ultraviolet illumination, and every – TiO<sub>2</sub> and UV ultraviolet illumination light presence. The dynamics of antimicrobial accomplishment of the chosen measures that



evaluated by killing curvatures determination. Results analysis that the synthesized  $TiO_2$  (anatase) own solid medicinal drug activity perhaps successfully utlized for medical care above ultraviolet radiation enlightenment [35].

**Bonetta** *et al* (2013), Statistically explored covered exteriors of (Petri - dished in addition ceramics tile)  $TiO_2$  actions of anti- bacteria. Widespread investigation of varied classes for micro- organism contains (*S.aureus, L.innocua , E.coli, and P.putida*), Consequences accessible would probably assist improvements of consummate entitle creased  $TiO_2$  outsides illuminating photo-motivated micro-organism encouraging through completely total bacteria's fledged of  $TiO_2$  emphasizing treatments within lined outsides construction over then construction industries furthermore as ceramic that located in contemplative micro-biological surrounds, as like hospital and nutrition production [36].

**Gupta** *et al* (2013), argued that particular methods that formed "TiO<sub>2</sub> and Ag-doped TiO<sub>2</sub> photo-catalysts by using the method of caustic catalyzed sol–gel in which XRD, UV– vis spectroscopy, TEM, and photoluminescence (PL) regarded as physical characteristics . The TiO<sub>2</sub> sample had all both phases ( anatase and rutile) revealed via outcomes of XRD . Whereas, merely the phase (anatase) stated in Ag-doped TiO<sub>2</sub> nanoparticles. Uv–Vis spectroscopy confirmed that decrease of energy band- gap of Ag-doped TiO<sub>2</sub> Nano-particles compared to TiO<sub>2</sub> Nanoparticles. (*S. aureus*) and (*P. aeruginosa, E. coli*)bacteria have conclusively been shown that evaluation of TiO<sub>2</sub> antimicrobial activity plus Ag-doped, TiO<sub>2</sub> Nano-particles,(3% and 7%). Either in (3% or 7% doping) of TiO<sub>2</sub> nanoparticles Ag-doped concentrations, entirely three microorganisms viability decreased to zero at 60 mg/30 mL culture". At 80 mg / 30 m.L TiO<sub>2</sub> supported models displayed no practicality though

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stabling Ag - TiO<sub>2</sub> 7 % displayed nil expediency at 40 mg / 30 m.L values in the circumstance of *P.aeruginosa* simply [37].

**Tommaso Zuccheri** *et al*(2013), improved dissemination of acrylicwater paint has indicated a pigment in slight content concerning (2 the concerns vol.) for the construction of microorganisms concerned in health sterilization application in health application in hospitals. The NPs and also the coating are studied the application of chemical analysis instruments and TEM traditional their conformable dispersal within the acrylic ester paint. activity the photo-catalytic effective of TiO<sub>2</sub> activated by Associate in Nursing exploitation fluorescent source of illumination . The degenerate paint has been discovered antimicrobial active versus of E- coli, P. aeruginosa. The paint controlled the nano-TiO<sub>2</sub> conserved excellent actinic radiation constancy, strong devotion to the substrate and high hardness. Therefore, paint formulation that invented to aid exteriors cleaning technique in use thought of to be appropriate [38].

Adawiya J. Haider et al(2014), designated nano-particles of  $TiO_2$  that contains anatase manufacture section through sol- gel of TiCl4 methodology as beginning substantial. Additionally, it's potential to expect that nanoparticles of  $TiO_2$  exist in the liquid resolution are additional possible ready out of nano-particle, liquid mixing; one among the foremost appropriate, dynamic and applicable measures for medicinal drug application. As a final purpose the medicinal drug impact of these Nano-particle postponements junction rectifier to qualitative and quantitative clarification, by *Escherichia coli*, a Gram- negative bacteria and *S. auras*, a Gram- positive bacteria.[39]

**Verdier** *et al* (2014), inspected the medicinal drug activity of  $TiO_2$  versus Escherichia coli microorganism, by exploitation of  $TiO_2$  nanoparticles unaccompanied or in clear coatings (varnishes). extensive



medicinal drug activities disclosed from results once two h and four h, proposing that the formulation potency would increase through improvement.[40]

Zainab N. Jameel et al (2015), One of a lot of vital findings emerging of the study is that nano-particles of TiO<sub>2</sub> creation of TiCl4 as a primary element with fermentation solution of ethanol nearly 1:10 quantitative relation in close atmosphere, with no chemical addition. "Synthesized Sol-gel has been dehydrated and calcined at (550-600)°C. The construction, morphology and the constituent size of the nanoparticles have been examined through X-ray Diffraction and Scanning Electron Microscopy (SEM). The visual features deliberated by UV-Visible Spectrophotometer. Results illustrated that anatase phase was only in titanium dioxide powder up to 500. At the range of (3-30) nm, TiO<sub>2</sub> size nanoparticles average grain achieved. Synthesized  $TiO_2$ nanoparticles in 10-5 and 10-3 concentrations presented superiority at antibacterial activity within two forms of bacteria (E-coli) and S.aurous respectively". Nano-particle of TiO<sub>2</sub> of supplementary productivity for each antibacterial features through S. aurous in contrast per E-coli [41].

Neihaya H. Zaki *et al* (2016), Throughout complete analysis, this revision show that antibacterial drug perform as enticing as a brand new agents against unhealthful bacterium thanks to some nanoparticles actions . this analysis illustrated the strategy of antimicrobial effects of metal dioxide-nano-particles against seven of microorganism isolates (E.coli, Enterobacter ,bacteria genus alcaligenes, Aeromonas veronii, Aeromonas hydrophila, bacteria species and *S.aureus*) mentioning to isolation from completely different Baghdad water purification stations. Whereas demonstrating the manner physiochemical characters impact of the consumption water quality for the air and water .The nanoparticles



categorization explained by the usage of Scanning Electrone magnifier, FTIR, and UV-Visible photometer. varied concentration activity of TiO<sub>2</sub> Nps. (50, 100, 150, 200 mg/ ml) measured in liquid medium by mistreatment colorimetry still as evaluating the optical density (OD) for all microorganism species detected. The plate count assay has elaborate to explore the influence of TiO<sub>2</sub> Nps. regarding one model of gramnegative bacterium, (*E. coli*) additionally positive one-gram bacterium (*S. aureus.*) Analysis of analysis reports have shown that discussion sections tend to be notably clarification in nano-TiO<sub>2</sub> potency of antibacterial drug impact in water, that may be used as AN antibacterial drug agent for various resolutions [42].

**B.** Duymaz *et al* (2016), Stated experiment explorations on potency of synthesis of medicine and antifungal TiO<sub>2</sub> Nano-powders achieved arithmetical analysis of recent innovative options and reviewed the offered chances for industrial applications. the present study shows the many of TiO<sub>2</sub> nano particles combination through sol-gel methodology utilizing binary compound and alcoholic solutions of metal tetras prop oxide (TTIP). the results of solvent kind, support material (calcite, talk, metal salt, silica) and aeriation temperature on antibacterial/antifungal and physical properties particularly the regular particles size and expanse of titania particles are examined [43].

Ambika S. Sundrorajan et al (2016), Recent analysis has accompanied that titanium dioxide nano-particles (TiO<sub>2</sub> NPs) inexperienced combination utilize subordinate grade extractor as of Vitex negundo Linn, with or while not ionic liquid (1 - ethyl - 3 - meth limidazolium tetra fluoroborate - [ E M I M ] + B F 4 –) by associate degree eco - friendly technique. Ionics liquid sort of an expensive in the black achieves as a self - assembling arrangement captivating portion in a



energetic part in TiO<sub>2</sub> nano-particles combination. XRD investigation, offerings each one NPs of TiO<sub>2</sub> recognized crystalline nature with the common crystalline size regarding ten nm and nineteen nm within the procedure of anatase part structure. TEM analysis makes confirmation of spherical and rod formed morphologies, whereas the regular particle size is regarding twenty six nm and fifteen nm. The Ti–O stretching bands ar known at 453.47 cm–1–779.32 cm–1 from the FTIR spectrum. Moreover, the TiO<sub>2</sub> NPs bactericide activity in distinction to *Staphylococci aureus* (gram positive) and *Escherichia Coli* (gram negative) organisms are disclosed . Therefore, these inexperienced synthesized TiO<sub>2</sub> nanoparticles used for a comprehensive side of biological applications [44].

M. Naglakshmi et al (2017), Studied the results of titanium (TiO<sub>2</sub>) nanofiber of with success synthesized via electro spinning methodology. The factory-made  $TiO_2$  powder options advanced technologies as diffraction analysis ( XRD) and Scanning lepton microscope(SEM). Texture of synthesized TiO<sub>2</sub> anatase structure exhibited by The XRD pattern, whereas DebyeScherer equation calculated the crystal size. The Nano fibrous morphology of synthesized  $TiO_2$  nanomaterials have displayed by SEM image . modern work proposes AN informative theory concerning with success decomposition of methylthionine chloride dye beneath UV-light irradiation by the synthesized TiO<sub>2</sub> nano-fiber. Higher color elimination potency of eighty seven.36% has earned for the sample calculated at 450°C. what is more, medication influence on the synthesized TiO<sub>2</sub> Nano-fiber has checked by disc diffusion technique utilizing gram positive micro-organism caryophylloid dicot genus and gram negative bacteria enteric pneumoniae . most zone of inhibition(25 mm) in millimeter was known by K. pneumoniae. Thus, all over the



likelihood of acting as gifted constituents of economical photo-catalytic quantitative relation and smart medication agent [45].

Saba A. Mahdy et al (2017), Defines proof primarily based drugs because the aware, express and wise use of the potent antimicrobial effectiveness of nanoparticles compounds within which delineated new answer to microorganism challenges in several infectious diseases. "In the current study highlighting preparation of TiO<sub>2</sub> nanoparticles by sol-gel method with 33nm size in which holding marked antibacterial effect on gram strains of both positive *Staphylococcus aureus* and negative *Escherichia coli*. Smallest attentiveness of embarrassment determination of TiO<sub>2</sub> Nano- particles originate of 30  $\mu$ g/ ml for *S.aureus* besides 40 $\mu$ g/ ml of *E.coli*, by which caused through reduction broth technique with or with no Nano-particles of TiO<sub>2</sub>. Outcomes have determined via agars dispersion evaluate" [46].

Hani A. Alhadrami et al (2017), Conceded several strategies that adequately have utilized to organize Nano-structured metal-oxides. However, news synthesis of TiO<sub>2</sub> Nano-particles through superficial hydrothermal method via variable concentration of each precursor and reaction temperature whereas keeping method section continual. Morphological, essential and optical studies performed throughout scanning microscopy ready with energy dissipative spectrographic analysis, X-ray powder diffusion spectrographic analysis and VU-Vis-NIR spectrographic analysis. Morphological besides grammar analysis conduct that ready Nano-particles square measure very pure with a close to traditional particle size of 5-15nm. Crystalline construction and sizes near five nm exposed throughout XRD studies, but the optical captivation researches of gauge boson wavelength vary regarding 300-600 nm illustrating resilient absorbance peak set at around3.5eVnm wherever evident energy is much obvious for materials. As a final purpose, results adequately cowl the TiO<sub>2</sub> nanoparticles antibacterial drug consequence. Lowest concentration that either stop or inhibit growth of microorganism determined by plating technique utilization . Through utilization of TiO<sub>2</sub> nanoparticle technique counter to most typical - wound infection organisms containing: MRSA, *E. coli and genus Pseudomonas aeruginosa*. Altered concentrations of TiO<sub>2</sub> Nano-particles have used one hundred  $\mu$ g/ml, 200  $\mu$ g/ml, 400  $\mu$ g/ml, 600  $\mu$ g/ml and 800  $\mu$ g/ml. completely different concentration of the precursors and synthesis temperature crystal rectifier to different Inhibition of microorganism of ready TiO<sub>2</sub> samples [47].

Kristappagari Manjunath et al (2018), classify many consecrations of the case study to adopt the investigation of with success synthesized anatase section TiO<sub>2</sub> nanoparticles at a hundred thirty °C for two days through ionic liquid-supported hydrothermal technique. A case study approach was chosen to permit the noninheritable merchandise classified to use many analytical techniques. X-ray diffusion information exposes that the sample has anatase TiO<sub>2</sub> nanoparticles. FTIR spectrum displays the incidence of ionic liquid and indicates Ti-O-Ti peak at around 456nm and therefore the bands at 1410 and 1581 cm-1 indicates C-H in-plane vibrations and stretching of imidazolium ring. UV-Vis spectrum illustrates most band at 272nm because of  $TiO_2$  nanoparticles. The  $TiO_2$ Nano-particles unconcealed via SEM pictures nearly spherical in form whereas, TEM pictures expose that the typical diameters of  $TiO_2$ nanoparticles seem within the style of 35nm. TiO<sub>2</sub> nanoparticles given exceptional icon chemical change motion for the degradation of methylthionine chloride organic dye. in addition, the medicine options of



TiO<sub>2</sub> nanoparticles explored contrary to four microorganism strains by agar well diffusion technique.[48]

M. Dhanasekar et al (2018), "The objective of the present work is to investigate Copper doped TiO<sub>2</sub> nanoparticles with reduction of graphene oxide as a firm support presented as fresh ambient light antimicrobial agents. The activity extended doping along with copper in order to improve charge transport during photo catalytic degradation of microorganisms by the visible light and reduced graphene oxide. The antimicrobial motion of the simple in addition to the amended  $TiO_2$ particles tested with four altered microorganisms, explicitly two Gram positive and two Gram negative types. Under visible light conditions, tests of inhibition and minimum inhibitory concentration (MIC) zone executed. The finding highlights Cu2O-TiO<sub>2</sub>/rGO to determine better observable light antibacterial property with higher zone of inhibition area and lower value of minimum inhibitory concentration for both positive micro -organisms Gram matched to the bare TiO<sub>2</sub>." and negative Polymer Nano-composite films organized by usage of Nano-particles with vinyl polymer and therefore the antimicrobial motion that tested over again for potential wrapping functions [49].

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