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Preparation and Analysis of Nano Thin Films of Titanium Dioxide and Its Applications: A Theoretical review

Graduation Project

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بسم الله الرحمن الرحيم ﴿ اقْرَأْ بِاسْمِ رَبِّكَ الَّذِي خَلَقَ * خَلَقَ الْإِنْسَانِ مِن عَلَقٍ * اقْرَأْ وَرَبُّكَ الْأَكْرَمُ * الَّذِي عَلَّمَ بِالْقَلَمِ * عَلَّمَ الإنسان مَاكَمْ يَعْلَمْ ﴾ صَدَق اللهُ العظيم سورة العلق//آية (1-5)

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بناء على التوصيات المتوافرة ارشح هذا البحث للمناقشة :

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Dedication



To my mother and father To myself And to everyone who helped along the way ...

Thanks and appreciation

In such beautiful moments, let us think before we write letters The letters are scattered and he tries in vain to collect them into lines ...

Many lines in the imagination, and in the end we are left with only a few memories and pictures that bring us together with comrades who were by our side ... We must thank them and bid them farewell as we take our first step into the midst of life we thank everyone who helped us on this long road and gave the outcome of an idea to light our path

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The Aim of Project

The project aims to conduct theoretical studies on titanium oxide and methods for preparing thin films and shed light on the most important manufacturing applications.

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List of Symbols and Abbreviations

Symbol	Description			
AFM	Atomic force microscopy			
Cr	Chromium			
DSSC	Dye-sensitized solar cell			
FSS	flame spray synthesis			
IV	Current–Voltage Characteristics			
NPs	Nano Particles			
NMS	Neuroleptic malignant syndrome			
NIR	Near infrared			
РРҮ	Pure polypyrrole			
РН	Hydrogen ace			
PLD	Pulsed laser deposition			
SEM	Scanning Electron Microscope			
SnO ₂	Tetratin oxide			
TiO ₂	Titanium dioxide			
UV	Ultraviolet Spectrum			
VIS	Visible Light			
XR	X-ray			

hv	Photon Energy	
E	Electron	
<i>h</i> +	Hole	
<i>0</i> ₂	Oxygen	
<i>H</i> ₂ <i>O</i>	Water	

1- Introduction

Semiconductor metal oxide-TiO₂ thin films have become very attractive candidates for numerous fields of industrial, and technological applications including varistors, thin-film solar cells, gas sensors, ultraviolet (UV) detectors, and thin film transistors particularly due to wide band-gap energy at room temperature and high mechanical properties and thermal stability. In modern era of technology, vast deal of consideration being conferred upon the making of low-priced thin films, due to their high varying characteristics like characteristics consist of heat reflective windows, resistivity, and photovoltaic, photo thermal and catalytic properties.[1]

In nano-structured that based TiO_2 thin films deposited on glass substrate they are very effective in optical coatings, electronic devices, catalysis, and gas sensors, (DSSC) .[1]

Titanium dioxide (TiO₂) has attracted many interests as one of the most promising catalyst materials due to its small band gap, superior photocatalytic performance, easy availability, long-term stability, and nontoxicity [2]. However, it is well known that the size and morphology of TiO₂ particles play crucial roles in its properties and applications. Nanostructured TiO₂ is exceptionally versatile and offers unique characteristics such as chemical stability, low cost, earth abundance, biological inertness and electronic properties [3].

To talk about physics of thin films, then it is considered to be an important field of solid state physics. The term of "thin film" is usually used to describe a layer or group of layers of material's atoms with a thickness that is not exceeding 1μ m. There are many uses of thin films in many modern electronic fields, as in the electronic circuit's fields, photodiodes, transparent electrodes, optoelectronics, gas sensors, phototransistors, and solar cells[4]. It has been stimulated the importance of thin films to the emergence and development of different methods for fabricating such films.

One of the materials that has lately attracted much attention is titanium dioxide (TiO_2) [5]. Such interest is related to the unique properties of this material, e.g. nontoxicity, thermal, mechanical and chemical stability, transparency for the light in the wide spectral range, high dielectric constant and photocatalytic activity[6], and one of the most efficient semiconductor photocatalyst for extensive environmental application because of its strong oxidizing power, high photochemical corrosive resistance and cost effectiveness. Due to these

inherent properties, TiO_2 is the most suitable candidate for degradation and complete mineralization of toxic organic pollution in water [7].

It is well known that TiO_2 exists in three crystalline structure: rutile, anatase and brookite. The anatase phase is especially adequate for those application due to its crystal structure and higher band gap of (3.2 eV) compared to the (3 eV) in rutile. Anatase and rutile have properties of interest for sensing applications [8]. In principle, transition metal with proper oxidation state replace some of the Ti (IV) from lattice production an impurity state that reduces the band gap of TiO₂.

Titanium dioxide utilization remains typically confined to (UV) light because of its wide band gap [9]. This limits the efficient utilization of solar energy for TiO₂ because TiO₂ cannot efficiently utilize solar light since (UV) light accounts for only (4–6%) of solar radiation. Therefore, many attempts have been undertaken in recent decades to shift the threshold of the photo-response of TiO₂ into the visible region, which would enhance its potential for chemical solar energy conversion and open possibilities for further applications [10].

 TiO_2 films with specific crystal structure, orientation or morphology exhibit specific characteristics, which makes it important to control the phase structure of TiO_2 films during the growth.

However, extensive research continues to further optimize this technology in order to develop a new visible light driven TiO_2 Nano-based thin film that can use visible light in high efficiency under sunlight irradiation and widen the spectrum of potential application. Development of material engineering in the nanometer scale has generated new photonic materials and systems that could potentially lead to realization of high efficiency and low cost light harvesting material.

The methods of sol-gel, spin coating, anodization, oxygen plasma assisted molecular beams epitaxial and pulsed laser deposition (PLD) have been used to fabricate TiO_2 films.

2- Titanium dioxide TiO₂

Titanium dioxide (TiO_2) has been used for many years in a vast range of industrial and consumer goods including paints, coating, adhesives, paper and paperboard, plastics and rubber, printing inks, coated fabrics and textiles, catalyst system, ceramics, floor coverings, roofing materials, cosmetics and pharmaceuticals, water treatment agents, food colorants, automotive products and many more. Environmental purification using titanium dioxide (TiO_2) photocatalyst has attracted a very great deal of attention with the increasing number of recent environmental problems in the world. TiO₂ photocatalyst thin film has excellent photocatalytic properties as well as high transparency, excellent mechanical and chemical durability in the visible and near infrared region of spectrum. The oxygen deficiency introduces an excess of electrons in the material resulting in an increase of the electrical conductivity. The oxygen vacancies act as electron donors, thus (TiO_{2-x}) is an n-type semiconductor, in contrast with p-type semiconductors which contain electron acceptors and where the charge carriers are holes rather than electrons. Substoichiometric (TiO_{2-x}) is both a poor insulator and a modest semiconductor. Therefore, several attempts have been made either to control the oxygen vacancy concentration or to introduce charge carriers (doping) inside TiO₂ in order to increase or decrease the electrical conductivity, depending on the desired application. During the last 40 years, almost half the atoms of the periodic table have been incorporated into TiO₂.[11]

3- Crystal Structure of TiO₂ and Phase Transformation

Titanium forms four well-defined oxides; monoxide (TiO), sesquioxide (Ti_2O_3) , dioxide or titanic acid (TiO_2) called titania and pentoxide (Ti_3O_5) as shown in fig. (1). From practical standpoint, the dioxide (TiO_2) is the most important oxide[12].



Fig. (1): Phase diagram of the Ti-O system taken from Samsonov. [12].

TiO₂ exists in nature in the form of minerals like anatase, brookite and rutile. Rutile is commonly found in nature; however, anatase and brookite are extremely rare. Generally, TiO₂ exists in an amorphous form at deposition temperature below (350) °C [13]. Above that temperature, anatase phase is formed and at temperatures greater than about (800) °C, the most stable crystalline phase rutile is formed. According to Bokhimi [14], in the most cases of TiO₂ synthesis anatase is the main phase and brookite occurs as a minority phase, depending on synthesis conditions. The crystal structures of the three oxide forms can be discussed further in terms of orientation of the (TiO₂) octahedral [15]. There are many differences in structural and chemical properties between its oxides as shown in table (1).

Property	TiO ₂			TiO	Ti ₂ O ₃
	Brookite	Anatase	Rutile		
Color	Dark Brown	White	White	Bronze	Purple-Violet
Melting Point	-	-	1830-1850	1737	2127
Density(25°C) gm/cm ³	4.170	3.900	4.270	4.888	4.486
Crystal Structure	Tetragonal	Tetragonal	Tetragonal	Cubic	Rhombohedral

Table (1): Properties of titanium oxides at various oxidation states [16], [17].

3-1 Anatase

Anatase which refers to the long vertical axis, was named by Hauy in 1801 from the Greek word "anatasis" meaning extension. Anatase has a tetragonal crystalline structure (Fig. (2.a)) and is built up from octahedra that are connected at their edge (Fig. 2.b)[15].



Fig. (2): Crystal structure of anatase[15].

<u>3-2 Rutile</u>

Rutile was discovered by Werner in Spain in 1803. Its name is derived from the Latin "rutilus" meaning red. Rutile is the most stable form of TiO₂ and also the most studied. Rutile is usually the dominant phase in TiO₂ films, and it has a crystal structure tetragonal (Fig.(3.a)) [12][15]. Rutile is built up from octahedra that are connected predominantly at their edges (Fig. (3.b))[15], and it had a band gap of (3.0 eV) and absorbs the ultraviolet rays as well as rays that are slightly closer to visible light. TiO₂ rutile structure is the most common white pigment in paint products due to its extremely high refractive index (n=2.8) at wavelength (4358Å) [18] and it can be used in (UV) protection products[19].



Fig. (3): Crystal structure of rutile[15].

<u>3-3 Brookite</u>

Brookite was discovered by Levy in 1825 at Snowen (Pays de Gales, England) and it was named in honour of the English mineralogist, Brooke. Brookite has an orthorhombic crystalline structure. The crystal structure can be described as distorted octahedra with a titanium atom the center and oxygen atoms in the vertices (Fig. (4.a)). Brookite is built up from the octahedra that are connected at their corner and edges (Fig. (4.b))[15].



Fig. (4): Crystal structure of brookite [15].

In all three forms, titanium (Ti⁴⁺) atoms are coordinated to six oxygen (O⁻²) atoms, forming TiO₆ octahedral. All three forms differ only in the arrangement of these octahedral [14]. The anatase structure, is made up of corner (vertice) sharing octahedral (fig. 5.b) resulting in a tetragonal structure. In rutile, the octahedral share edges to give a tetragonal structure (fig. (5.a)) and in brookite both edges and corners are shared to give an orthorhombic structure (fig. 5.b) [20].



Fig. 5 Unit cells of the titanium dioxide (a) rutile, (b) brookite, (c) anatase[20].

The three crystal structures differ by the distortion of each octahedral and by the assembly patterns of the octahedral chains. Anatase can be regarded to be builtup from octahedrals that are connected by their vertices, in rutile, the edges are connected, and in brookite, both vertices and edges are connected .

4- Structural and morphological measurements

4-1 Spectra of X-Ray Diffraction

One of the most powerful techniques of X-ray power for qualitative and quantitative analysis of crystalline compounds is X-Ray diffraction (XRD). It has long been used the experimental technique in order to define the overall structure of bulk solids, containing lattice constants, identification of unknown materials, orientation of single crystals, orientation of polycrystals, defects, stresses, etc. In this work, X-ray diffractometer type Rigaku MiniflexII, USA, a power diffraction system with Cu-K α X-ray tube at (1.54 Å) with voltage (30.0 kV) and current (15.0 mA) and speed scan (6 deg./min.) is typically used. The scans of X-ray are approximately perform between 2 θ values of 20° and 90°. The crystal structure of the prepared pure TiO₂ thin film and TiO₂ mixed with Ag were investigated. Fig. (6) illustrates the X-Ray diffractometer that is use in current work.



Fig. (6): Rigaku MiniFlex II desktop x-ray diffractometer (XRD).

4-2 Atomic force microscopy (AFM)

Atomic force microscopy (AFM) is a widely used technique for investigating the surface microstructure and quantifies surface topography. The criterion of operation is measuring attractive /repulsive forces between the tip

(cantilever) and constant height sample. The necessary parts are x, y & z that are actuated separately by x, y drives and z-control with extreme precision, so that distances of atomic can be measured. A micro-fabricated cantilever with a sharp tip is deviated by criteria on a sample surface, mostly in a photograph but on a much smaller scale. A beam of laser reflects off the cantilever backside into a set of photodetectors, and then allows the deviation to measured and assembled into an image of the surface. Fig. (7) shows the setup of typical Atomic Force Microscope 2000 Å. The grain size and surface roughness are investigate with a Nanoscope atomic force microscope in tapping mode. In this study, the morphological features of the various films are investigate with Angstrom Advanced AA3000 Scanning Probe Microscope (SPM), which manufacture in USA, as summarized its specification in below table:

Table (2)	: summarized	specific	properties	of Angstrom	Advanced	AA3000	Scanning
Probe Mi	icroscope (SPN	A).					

	Atomic Force Microscope (AFM)					
Functions	Scanning Tunneling Microscope(STM)					
	Lateral Force Microscope (LFM)					
Pasalution	AFM: 0.26nm lateral, 0.1nm vertical					
Resolution	Scanning Tunneling Microscope(STM)					
	X-Y scan scope:~10 micrometer					
Tashrisal	Z distance:~2 micrometer					
Technical	Image Pixels:128X128, 256X256, 512X512, 1024X1024					
Parameters	Scan Angle:0~360 degree					
	Scan Rate: 0.1~100Hz					
	Sample Size: Up to 45mm in diameter, reach 15mm.					
Mechanics	Engagement: Auto engagement with travel distance of 30mm and					
	precision of 50nm;					



Fig. (7): the Atomic Force Microscope setup.

4-3 Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy (SEM) is one of the imaging devises, which is broadly used in nanotechnology. This device provides images of the objects as small as 10nm using bombardment of the sample. Fig. (8) illustrates the beam of highly energetic electrons is produced by the electron gun and this beam is passes vertically through the electromagnetic fields and lenses. As soon as beam hits the sample, it excites electrons inside the sample and they will travel to sample surface [21].



Fig. (8): Electron beam passes through the microscope and hit the sample[21].

Several types of electrons irradiate from the sample surface such as secondary electrons, backscattered electrons, X-rays and more fig. (9). Then, the detector collects the X-rays and primary and secondary electrons and converts them to signal which will be transferred to a display to produce the final image. The brightness of any point of the image is influenced by the intensity of collected signals.



Fig. (9): Secondary electrons are excited by primary electrons and move to sample surface. Afterward they will be collected to get a final image[21].

In this study, the morphological features of the various films are investigated with FEI: QUANTA 450, Czech, equipped with an EDX detector as showed in fig. (10).



Fig. (10): the Scanning Electron Microscope(SEM) and (EDX) setup

5- Optical properties

The optical transmittance spectra of as-deposited thin films of anatase and rutile were measured using Perkin Elmer Lambda 905 (UV–VIS–NIR) Spectrophotometer. These films exhibit good transmission in the spectral range of (250–2,000 nm) wavelength. While the reflectance spectra for both samples were recorded with the same Spectrophotometer in a wavelength range from (300 to 1,100 nm) . The TiO₂ anatase and rutile nanoparticulate thin films increase transmittance with higher substrate temperature in the visible and near-infrared spectra range.

However, there is strong absorption in the ultraviolet (UV) and low visible wavelength regions, which is characterised by a sharp drop in optical transmittance and increase reflectance. This can be ascribed to fundamental absorption of TiO_2 nanoparticulate thin film layer owing to the transition's electron from the valence to conduction bands[22]. This explains why anatase and rutile TiO_2 thin film is attractive for photocatalytic and solar cells applications. Also, the transmittance at a given temperature for both anatase and rutile phase show similar properties. But the transmittance absorption edge is sensitive to change in substrate temperature, which is shifted to long (UV) and low visible wavelengths as the substrate temperature increases. For instance, anatase deposited thin films at various substrate temperatures have absorption edge at ~ 318 (A1), 360 (A2), and 351 (A3) nm. Similarly, the rutile samples R1, R2 and R3 have transmittance absorption edge at ~ 328, 357 and 395 mm.[23]

6- Approaches to the synthesis of nanomaterial

Different deposition methods basics route for the synthesis of nanoparticles allow the control the particle size, particle geometry, doping ratio by different elements, and degree of particle agglomeration. Those particle parameters give the synthesized material new physical and chemical properties for different applications. A deep understanding of the synthetic approach is critical in order to manufacture new structures with unique properties. In this chapter, we are just focusing on those techniques that use pulsed laser techniques. The schematic that is shown in fig. (11) describes simply the difference between both approaches [24].



Fig. (11): Show Bottom-up and the top-down approaches in synthesis of carbon-based nanomaterial [24].

7- Thin Film Deposition Processes:

The vast varieties of thin film materials, their deposition processing and fabrication techniques, spectroscopic characterization and optical characterization probes are used to produce the devices. It is possible to classify these techniques into two ways[25]:

- Physical Process
- Chemical Process

Physical method covers the deposition techniques which depends on the evaporation or ejection of the material from a source, i.e. evaporation or sputtering, whereas chemical methods depend on physical properties.

Structure-property relationships are the key features of such devices and basis of thin film technologies. Underlying the performance and economics of thin film components are the manufacturing techniques on a specific chemical reaction[26]. Thus chemical reactions may depend on thermal effects, as in vapor phase deposition and thermal growth. However, in all these cases a definite chemical reaction is required to obtain the final film. Fig.(12), summarized the classification of thin film deposition techniques [27].

Fig. (12): Illustration classification of thin film deposition techniques[27].



Physical and chemical properties

Properties		Properties		
Name	TiO ₂	Melting point	1843 °C, 2116 °K,	
			3349 °F	
The appearance	White steel	The boiling point	2972 °C, 3245 °K,	
			5382 °F	
The smell	Odorless	Solubility in water	Non-atomization	
The molar mass	79.866 g/mol	The range gap	3.05 eV (Rutile)	
Density	$4.23 ext{ g/cm}^3 ext{ (rutile)} ext{ 3.78}$	Absorbable	+5.9·10-6 cm ³ /mol	
	g/cm ³ (anatase)			
Refractive index	2.488 (Anataz)	The size of its	100nm	
(nD)	2.583 (Procket)	molecules		
	2.609 (Rutel)			

Table (3): Physical and chemical properties of TiO₂



Fig. (13) Different forms of TiO₂ .[28]

Among all other semiconductor metal oxides family, $TiO_2(NMS)$ have gained much appreciation and seems to be a distinctive candidate due to their high structural, electronic and optical stability, nontoxicity, corrosion resistance, and low cost[29].

9-<u>Mechanism of photocatalysis</u>

Environmental pollution is becoming a big challenge for the new developing and developed world because of the industrial revolution, which on the one side is facilitating the life but on the other side destroying the environment. The chemical, textile, oil, and gas industries annually send billion of tons of hazardous effluents to the groundwater and upper environment which is affecting badly the marine life, human, and plant kingdom. At present, there are a number of technologies that have been discovered to cater the fastgrowing problem; the most commonly employed method of removing pollutants involves the use of various physical techniques, chemical and biological processes, and thermal techniques to remove commonly found organic and inorganic contaminants. Among many proposed processes being developed for the decontamination of the organic-based pollutants, biodegradation received the greatest attention. However, numerous organic chemicals, especially those which are toxic or refractory, were found very resistant to the biological degradation. Therefore, after the discovery of photocatalytic splitting of water by Fujishima and Honda in 1972, the researchers turned their point of convergence toward semiconductor photocatalysis which proved very effective in the degradation of even those pollutants which are highly difficult to remove by the other means. In comparison with the traditional oxidation process, the photocatalysis has significant advantages, for example, they can be used to degrade and mineralize the dyes and chemicals completely to CO_2 and H_2O and are also helpful in the degradation of very stable compounds which cannot be easily degraded by the other processes, also they can work efficiently at ambient temper ature and pressure conditions, and they do not need any special supply of oxygen. The other advantage of the photocatalysis is that it is a cheap process as compared to the other oxidation process having no waste disposal problems [30]. A large number of semiconductor photocatalysts have been investigated so far for the degradation of different pollutants, such as ZnO, WO₃, Fe₂O₃, CdSe, and SrTiO₃. In general, an ideal photocatalyst should have some basic properties, such as they must be active under UV, visible light, or solar light, they should have property of chemical and biological robustness, as well as they should be

stable toward photocorrosion. The other most important characteristic they must have is that they should be nontoxic and must have low cost and easy availability. These all are key factors for a good photocatalyst.

A photocatalysis reaction refers to a process in which the photocatalyst itself does not change when irradiated with light; rather, it converts light energy into chemical energy to promote the production or decomposition of compounds. While photosynthesis is the most representative example of photocatalysis in nature[31]

 $TiO_{2} + hv(light energy) \rightarrow e^{-}(electron) + h^{+}(hole)$ $e^{-} + O_{2}(dissolved oxygen) \rightarrow O_{2} . -(Superoxide radical)$ $h^{+} + H_{2}O \rightarrow H^{+} + .0H$ $O_{2i}^{-} + HO \rightarrow HO_{2} \cdot [32]$

10- Application of TiO₂

When pure polypyrrole (PPy) and TiO_2 nanoparticles polypyrrole (TiO_2 NPs/PPy) form composite thin films on an alumina substrate, characterization shows that, it can be used as humidity sensor. The sensor made of TiO_2 (NPs/PPy) composite thin films, using the added amount of TiO_2 (NPs) as (0.0012g) showed the highest sensitivity, smaller hysteresis and best linearity. Moreover, other sensing properties, such as effects of applied frequency, ambient temperature, response and recovery time and long-term stability were also investigated[33].

A new type of solar cell (called of the third generation) based on the use of a TiO_2 layer sensitized with a dye (dye-sensitized solar cell [DSSC]). This cell differs from the massive (single crystal or polycrystalline) silicon cells based on a p-n junction. TiO_2 nanotubes are typically one-dimensional material, which has a wealth of physical and chemical properties and low production cost, and therefore, it bears a broad application prospect In particular, recent studies show that, due to large specific surface area and nanosize effect, compared with other forms of nanostructures, TiO_2 nanotubes show great potential for development in photo catalysis sensors, solar cells M and other areas. Undoped TiO_2 , chromium-doped (TiO_2 :Cr) and (TiO_2 :SnO_2) synthesized by flame spray synthesis (FSS).technique acts as a hydrogen sensing (Gas sensor) . TiO_2 has excellent physical and chemical properties. It is low price, large available, non-

toxic material. The different properties of TiO_2 like structural, electrical, optical etc..., can be obtained by synthesis parameters, deposition conditions and dopants [33].

Titanium dioxide (TiO₂) nanoparticles are used in a variety of applications due to their unique properties, including high surface area, good chemical stability, and high refractive index. Some of the most common applications of TiO_2 nanoparticles include [34].

1.Optical filters

 TiO_2 nanoparticles are commonly used in sunscreens because they can absorb and scatter ultraviolet (UV) light, helping to protect the skin from harmful (UV) radiation.[34]

2. Biological

TiO₂ nanoparticles are used in coating for various applications, including to improve the durability and appearance of paints and to provide antimicrobial properties to surfaces.[34]

3.Catalysts

 TiO_2 nanoparticles are used as catalysts in chemical reactions, such as in the production of plastics, resins, and chemicals.[34]

4.Water treatment

TiO₂ nanoparticles are used in water treatment systems to remove contaminants and improve water quality.[34]

5. Food and cosmetics

 TiO_2 nanoparticles are used in food products as a whitening agent and to improve the texture of certain products. They are also used in cosmetics like foundation creams to provide a matte finish and to improve the opacity of products.[34]

6.Energy production

 TiO_2 nanoparticles are used in solar cells and other energy production technologies to improve their efficiency. Other than the above-stated applications they are used as carriers in cancer therapy for the proper delivery of drugs at the tumor site. Titanium dioxide is extensively used as an opacifier and colourant in medicines due to its multiple functionalities. It is widely used in confectionery, cosmetics and foods, in the plastic industry. It is used in topical and oral pharmaceutical formulations as a white pigment. In pharmaceutical formulations, titanium dioxide is used as a white pigment in film coating suspensions, sugar – coated tablets and gelatin capsules. TiO_2 may be admixed with other pigments. TiO_2 is also used in dermatological preparations and cosmetics, such as sunscreens. TiO_2 is having light scattering properties that may be exploited in its use as a white pigment and opacifier.[34]

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تحضير وتحليل أغشية ثنائي أكسيد التيتانيوم النانوية وتطبيقاته : مراجعة نضريه

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