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Comparative Study of Sol-Gel and Green Synthesis Technique Using Orange Peel Extract to Prepare TiO_2 Nanoparticles

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Abstract

Plant extracts have been widely explored as a safer alternative to conventional procedures for the manufacture of metal oxide nanoparticles. The present research concentrated on the synthesis and characterization of Titanium dioxide nanoparticles (TiO_2NPs) using an environmentally friendly green synthesis approach (orange peel extract) compared with another technique (Sol-Gel method) at room temperature. The TiO_2 nanoparticles were analyzed using an X-ray Diffraction (XRD) to determine the polycrystalline with Tetragonal structure anatase phase and average crystallite size 30.2 nm and 24.2nm of the sample prepared from Sol-gel and green methods respectively. FTIR spectroscopy was used to determine the functional groups of TiO_2 nanoparticles and their bonding nature. UV-Vis for optical properties shows the band gap energy of 2.85 eV and 3 eV for TiO_2 nanoparticles were prepared using sol-gel and green methods respectively. FESEM analysis identified the morphology of the surface of TiO_2 nanoparticles with the diameters of nanoparticles in the range of 60-80 nm and 75-85 nm were prepared via sol-gel and green methods respectively.

Keywords: FTIR, Green syntheses, Nanoparticles, Orange peel extract, Sol-Gel, TiO_2.

Introduction

Nanotechnology is an interdisciplinary field of science that deals with developing and manipulating things of atomic or molecular size, often less than 100 nm. It is important in a variety of applications because of its capacity to inspect and control at the atomic or molecular level in optics, electronics, optoelectronic devices, and different biomedical applications such as radiation enhancement and gene transfer. Nanostructure materials with intermediate sizes of 1–100 nm can be further altered [1]. The decrease in size to the nanoscale range improved the ratio of surface-to-volume (and hence surface energy), adsorption capacity, and biological efficacy[2,3] Furthermore, Nanomaterials' physical and chemical characteristics, such as permeability, toughness, coloring, and solubility, as well as optic, magnetism, and thermal properties, have improved greatly [4]. Other unique properties of nanoparticles include chemical, mechanical, and kinetic stability, as well as decreased density innovative nanostructured materials and nanocomposites [5], without a doubt, offer a wider variety of applications and superior performance than their macro/bulk counterparts. Nanotechnology is used in variety of disciplines, including healthcare, cosmetics, chemicals, and energy applications [6, 7]. The nanocrystalline of TiO_2 has received a lot of attention in recent years because of its potential use in photovoltaic, gas sensing, and photocatalysis [1]. As a result, studying the structural and electrical characteristics of Nanocrystalline TiO_2, particularly the effect of imperfections on electrical conductivity, is crucial in these applications [8]. At varying temperatures, it may display three distinct phases in the nano range, including anatase, rutile, and brookite. TiO_2 nanoparticles may be synthesized using a variety of processes, including chemical vapor deposition, hydrothermal, solvothemal, sol-gel, and Green synthesis. The sol-gel technique's main purpose is the spontaneous production of a couple of phases of material means (gel) that include a solid structure laden with solvent from (sol) solution, consisting of a chemical reagent or solid clusters like stabilizing agent or an inorganic precursor [9]. The evaporation of the solvent causes the gel phase to change, resulting in the creation of the xerogel phase in bulk solution. This feature will be important and effective when the solution defuses the substrate's surface [10]. However, green synthesis has gained widespread acceptance due to the absence of hazardous consequences generated by other chemical techniques [11].The green synthesis method is an environmentally beneficial technology since it employs plant extracts peels, leaves, blooms, stems, shoots, and pollen, for example [12]. It is one of the finest ways among physical and chemical approaches since it does not necessitate the use of expensive equipment, toxic chemicals, or high temperatures [13]. Different plant components (stem, leaf, flower, peel) are employed to regulate NP aggregation and agglomeration, acting as oxidizing, reducing, and capping agents [14]. Plant extracts have the potential to function as both reducing and capping agents in the creation of nanoparticles. The bioreduction of metal nanoparticles by combinations of biomolecules present in plant extracts (e.g., enzymes, proteins, amino acids, vitamins, polysaccharides, and organic acids such as citrates) is both ecologically and chemically benign. The most widely produced tree fruit in the world is the orange, especially the sweet orange. It is the finest source of Vitamin C and is beneficial to one's health and skin [13]. It has a light yellow to orange skin that completely protects the inner section of the fruit. Because it includes citric acid as its principal source, orange peel works as a reducing agent in the manufacture of TiO_2. Orange peel may be found in bath oils, air fresheners, face lotions, insect repellents, and weight reduction products. In this study, TiO_2 was prepared using sol-gel method and green synthesis of metal Oxide nanoparticles (TiO_2) using plant extract like Orange peel extract have been examined due to the crucial of these methods and the critical functions of plants in bio-based protocols for metal Oxide nanoparticle manufacturing [11]. The main objective of this study is to demonstrate that the green approach produces improved nanomaterial qualities compare with sol-gel method.

Materials and Methods

Materials

The materials used in this study include chemical materials like Titanium isopropoxide ($C_{12}H_{28}O_4Ti$) (DIREVO Industrial Biotech, Germany). Ethanol (Eth) (99% of purity, Brazil), Nitric Acid (HNO3) (69 %, 14.6 M, CDH, India). Orange peel was obtained from the remnants of consumed orange fruit, and washed all three times using deionized water to remove all impurities.

The Preparation of TiO_2 Nanoparticles Using Sol-Gel Method

As a starting material, TiO_2 Titanium isopropoxide was employed. 10 ml of titanium isopropoxide (TTIP) (96% of purity DIREVO Industrial Biotech) was gradually added to 30 ml of Ethanol, resulting in alkoxide hydrolysis and the precipitation of hydrous titanium oxides. Another solution was produced by mixing 3ml of Nitric Acid (HNO_3) with 150ml of deionized water (DI) and continues stirring for 30min at 40°C. This solution is used for hydrolysis catalysis and it is added drop by drop to the first solution under continuous stirring for 4 hours. In this stage the sol-gel pure solution was in the beaker, then heated this solution for 24 hours at 100°C to remove all residual organic solvent. At the end of this stage, a white TiO_2 nanoparticle precipitate was produced at the bottom of the beaker. The calcinations were then conducted for 4 hours at 450°C.

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Orange Peel Extract as Green Method

To produce Orange peel extract, a small piece of fresh orange peel (50g) was mixed with 150ml of deionized water and boil it for 2hr at 90 ºC. Then the extract was filtered using Whitman filter paper to remove the impurities as shown in Fig 1. The final step was to keep the extract to synthesize TiO_2 nanoparticles.

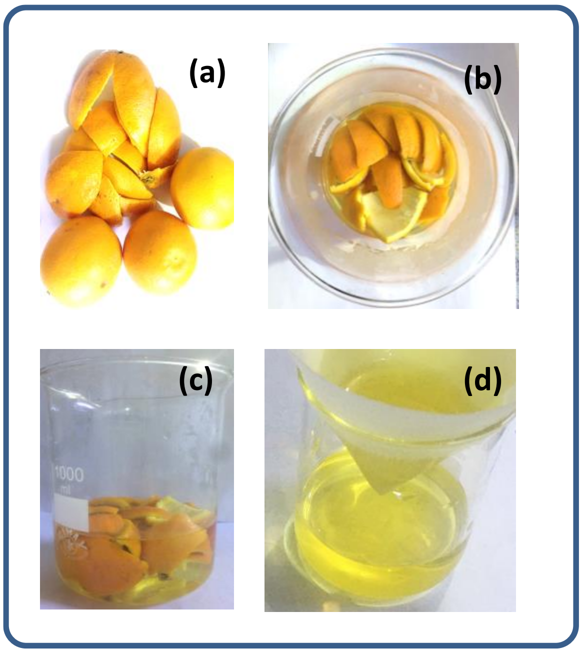

Figure 1. (a Orange fruit with peel, b: Separated Pieces of Orange peel c: In Boiling process, d: Orange peel extract solution.

The preparation of TiO_2 nanoparticles by Orange peel extract

Titanium isopropoxide (TTIP) 8 ml was added dropwise to 15ml of deionized water. This resulted in alkoxide hydrolysis. The solution was stirred for 30min. at room temperature. Then, added dropwise 10 ml of Orange extract to the white solution and stirred for 4 hr at room temperature drop by drop, with continual stirring, the extract was added until the pH of the solution reached 7. Nanoparticles were created during this process. This mixture was centrifuged and washed three times with deionized water and dried at 90ºC for 4hr. The particles were calcined at 450°C for 2 hr to obtain TiO_2 nanoparticles. The creation of dark brown powder suggests the degradation of glucose, furfural, and carboxylic groups and insoluble polysaccharides that are still present in orange peel extract [15]. At the final stage the nanopowder of TiO_2 could be produced and shown in Fig 2.

Figure 2. The final white powder of TiO_2 prepared by A) sol-gel method and B) green method.

Characterization Techniques

Different methods were used to analyses the prepared samples. The first technique was X-ray diffraction used to observe the crystal structure and phase identification of the generated samples using (SHIMADZU Japan) XRD 6000 technique under the conditions power diffraction system with target Cu-Ka x-ray tube has a wavelength (λ=1.5406 Å).

And the scan X-ray is performed between 2θ equal to 20° and 100°. The voltage and current are respectively 40 KV, 30 mA, the scan mode is continuous with the speed of 5.0000 (deg. /min) and handled by x-pert software. Another technique was FESEM to determine the surface morphology of specimens with high-resolution images that depend on the density of the powder. Basically, the source of hot filament tungsten wire emitted electrons and accelerated by high voltage (5-20) KV in a high energy electric field. The surface morphology of TiO_2 nanoparticles was investigated using FESEM (ZEISS Supra 55 VP, Germany). The image of the surface powder was created and analyzed by an electron beam. The optical transmittance and absorbance spectrum of TiO_2 nanoparticles were measured using an optical spectrometer. The optical properties were calculated from these optical measurements. The specifications of this device are (Perkin Elmer Lambda 1800 UV-Vis spectrophotometer in a wavelength range between 300 to 1100 nm. This absorbing spectrum is using to calculate the absorbance coefficient and the optical band gap. Fourier Transform Infrared Spectroscopy (FTIR) was used to determine the functional groups of TiO_2 nanoparticles.

Results and Discussion

Fig 3 explains the phase composition and the crystalline size of the two samples that preparation by two different methods (sol-gel and green approach) in the (2Θ) range from 20º to 80º. Seven peaks were identified in the anatase phase detected around 2Θ values. The results indicated that the structure was tetragonal poly-crystalline, and these findings agreed well with (JCPDS card number 01-084- 1285). Furthermore, the results indicated that the detected peaks were responsible for the anatase phase of TiO_2 NPs, and the lack of additional diffraction peaks associated with the rutile or brookite phase of TiO_2 NPs confirmed the purity of the samples. The crystalline size of nanoparticles that were prepared using two different methods was calculated using Debye-Scherer's equation as specified Eq 1 below [16]: -

$$D = \frac{0.9\lambda}{\beta cos\theta} \text{ ------ } 1$$

Where D represents the average crystalline size and 0.9 is denoted by the Debye Scherrer constant. The fill width half maximum (FWHM) is (β) and θ is Bragg's angle, λ is the wavelength of e CuKα-radiation and equal 0.154 nm. From this formula, the crystalline size was equal to 30.2 nm and 24.9 nm using two different methods (Sol-gel and green synthesis), respectively. Table 1 represents the data of TiO_2 (sol-gel) and TiO_2 (green approach) which is used to calculate the crystalline size of TiO_2 nanoparticles and the crystalline size reduces as the breadth of the peak grows. These data agreed well with the research[16].

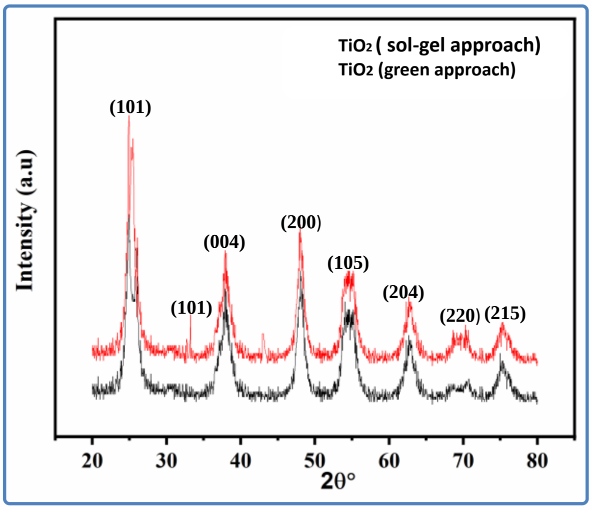

Figure 3. XRD Pattern of TiO_2 NPs prepared by Sol-gel and green approaches.

Table 1. The structure properties of TiO_2 nanoparticles prepared by Sol-gel and green approaches.

Sample	2Θ (Deg.)	d_{hkl} (A°)	FWHM (Deg.)	hkl	C.S. (nm)	Avarage C.S.(nm)	Phase
TiO_2 (sol-gel)	25.35	3.507	0.1951	(101)	31.333	30.2	Anatase
	38.12	2.484	0.21	(101)	32.385		
	48.32	2.370	0.359	(004)	30.631		
	54.33	1.892	0.47	(200)	37.928		
	63.08	1.689	0.219	(105)	18.986		
	70.01	1.479	0.244	(204)	30.026		
TiO_2 (green approach)	25.37	3.507	0.18251	(101)	28.323	24.9	Anatase
	34.04	2.484	0.6922	(101)	24.049		
	37.92	2.370	0.1852	(004)	28.037		
	48.04	1.892	0.412	(200)	29.922		
	54.26	1.689	0.45	(105)	21.216		
	62.76	1.479	0.219	(204)	19.093		
	68.94	1.360	0.234	(220)	18.836		
	76.29	1.247	0.29	(215)	30.435		

Fig 4 represents FESEM images in 100kx and 700x magnification power for two powders of TiO_2 nanoparticles that were prepared via two different methods, where a) and b) represent the Sol-Gel method and c) and d) represent the green method using orange peel extract. The photos show hexagonal and irregular in shape nanoparticle crystals of varying sizes from each sample aggregating with one another. The diameter of these particles was in the range of 60-80 nm and 75-85 nm which were prepared via the sol-gel and the green method, respectively. So, the spherical shape could be assigned to TiO_2 nanoparticles and this is a good agreement with XRD analysis. As a result, we may assume that the orange peel extract covers the surface of the TiO_2 nanoparticles[15].

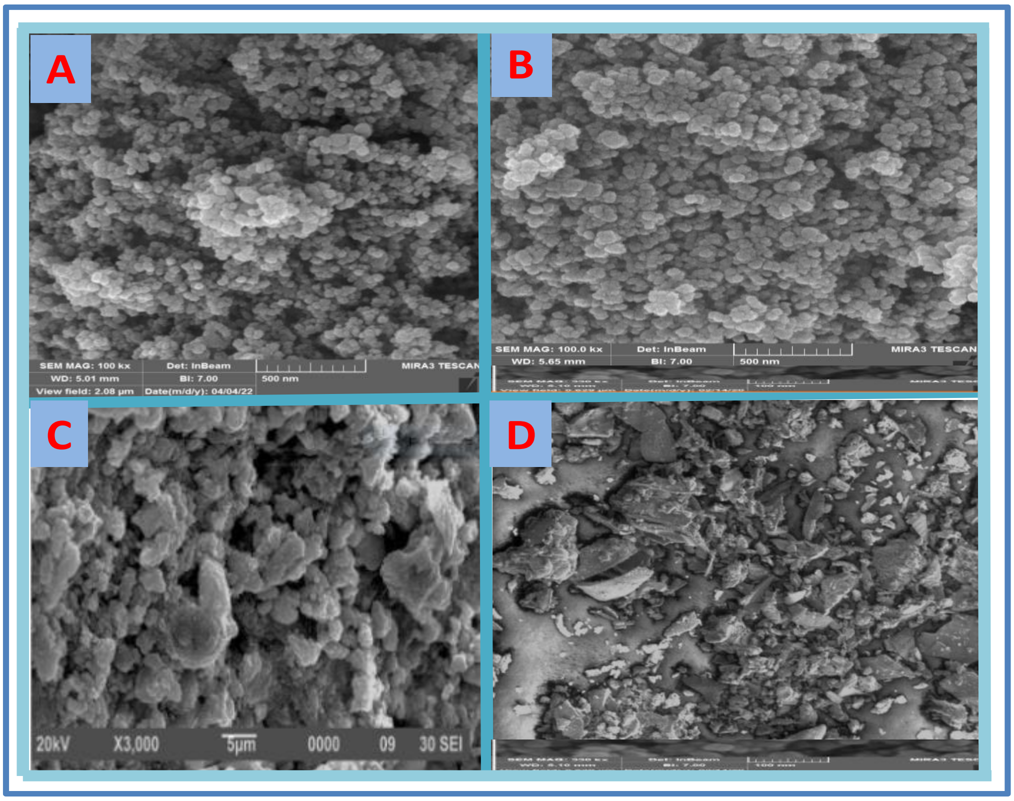

Figure 4. FESEM of the particles that were prepared via two different methods and two different magnifications; a) and b) represent the Sol-Gel method: c) and d) represent the Green method.

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Fourier Transform Infrared Spectroscopy (FTIR) was used to investigate the bond analysis of the pristine TiO_2 Nps after calcination as shown in Fig 5 and sol-gel process as shown in Fig 6 in the range of 500 $cm^{-1}$to 4000 cm^{-1}. The water molecules representing TiO_2 nanoparticles' surface caused the O-H stretching and bending vibration bond in the range of 3500 cm^{-1} and 1500 cm^{-1}. The N-H and carbohydrates and protein [17]. Orange peel extract contains aromatic, and lignocellulose group characteristics bands and its centered at 1415 cm^{-1},1163 cm^{-1} and 922 cm^{-1} indicate to vibration bands of (C-C) and (C-O) groups [18]. On the other hand, C=O groups appeared in 2779 cm^{-1} in pristine TiO_2 Nps and 2969 cm^{-1}, 2359 cm^{-1} and, 2800 cm^{-1} for TiO_2 nanoparticles which were prepared through the sol-gel method while this group would have appeared in the range of 2975 cm^{-1}, 2368 cm-^{1} and 2808 cm^{-1} for TiO_2 nanoparticles which prepared through the green method as shown in Fig 7. These groups suggest the presence of fatty acids, All peaks that appear under 800 cm^{-1}represent (TOT) vibration bonds groups as a fingerprint of TiO_2 nanoparticles [13]. All these peaks showed in Table 2.

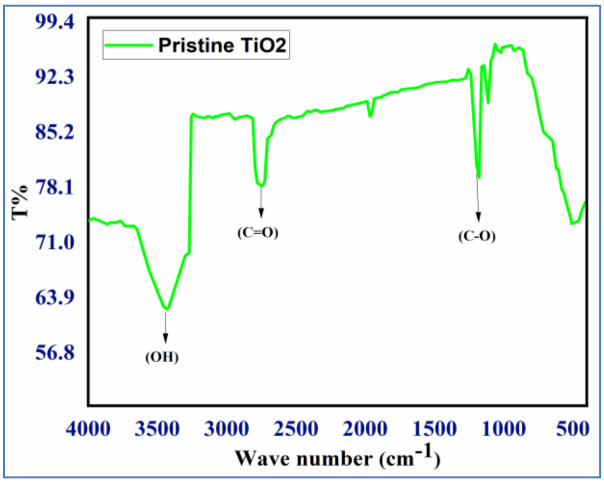

Figure 5. FTIR spectrum of pristine TiO_2 NPs.

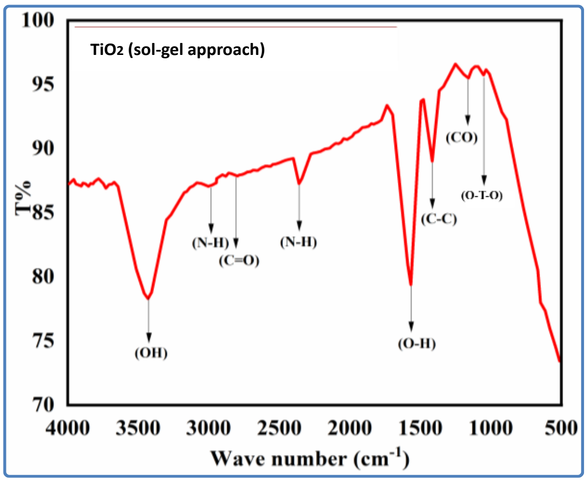

Figure 6. FTIR spectrum of TiO_2 NPs prepared using sol-gel approach.

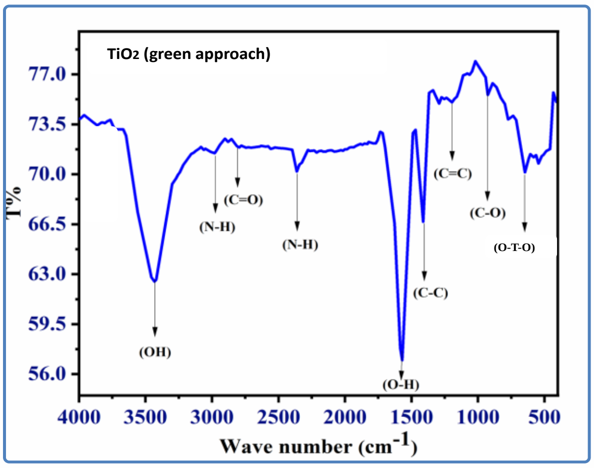

Figure 7. FTIR spectrum of TiO_2 NPs prepared using green approach.

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Table 2. The functional groups which appear in TiO_2 nanoparticles prepared using Sol-gel and green method

Functional group	Wave number of TiO_2 (sol-gel) (cm^{-1})	Wave number of TiO_2 (green approach) (cm^{-1})	Wave number from literature review (cm^{-1})	No. of Reference
O-H	3500	3515	3400-3600	19
	1500	1520	1400-1600	13
N-H	2969	2975	2930	17
	2359	2368	2349	
C=O	2800	2808	1698	20
C-C	1415	1415	1437	21
C-O	1163	922	1100	19
T-O-T	780	750	700	22
		636		

The optical properties of TiO_2 nanoparticles using two different methods were determined by the UV-vis spectrum. The tauc plot for the direct bandgap was created using the absorption spectra of TiO_2 NPs. shown in Fig. 9. It illustrates high absorption at 350nm. It is worth that in the anatase phase of TiO_2, an electronic transition occurs between the valance band of oxygen 2p and the conduction band of (Ti) 3d[23]. It is obvious that the spectrum depicted by the curve (TiO_2 with orange extract) shows absorption from TiO_2 nanoparticle aggregates. This might be owing to the precipitative effects caused by the use of ethanol as the connecting network to produce the oxide and the effect of the orange extract on the surface of TiO_2 nanoparticles. The band gap of TiO_2 nanoparticles was estimated by graphing $(hv)^2$ by projecting the linear portion of the curve to zero absorption as a function of photon energy, as shown in Fig.5. The band gap of TiO_2 nanoparticles prepared by sol-gel and green methods were equal to 2.85 eV and 3.25 eV, respectively, are shown in Fig.9, which is in excellent agreement with the band gap of the anatase phase[17]. The band energy of the TiO_2 sample produced using the green technique was lowered, making it appropriate for use in various applications.

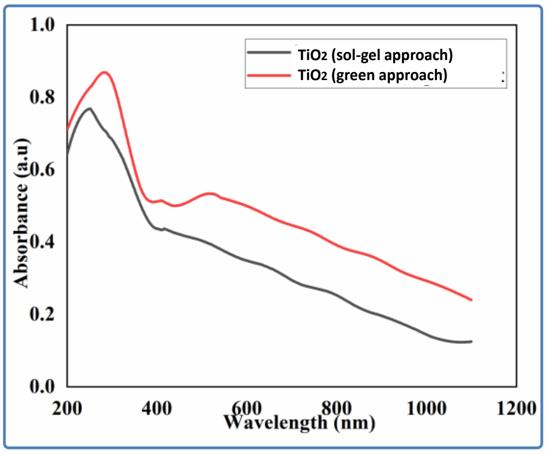

Figure 8. UV-Vis absorption spectra of TiO_2 NPs prepared by two different methods (sol-gel and green method).

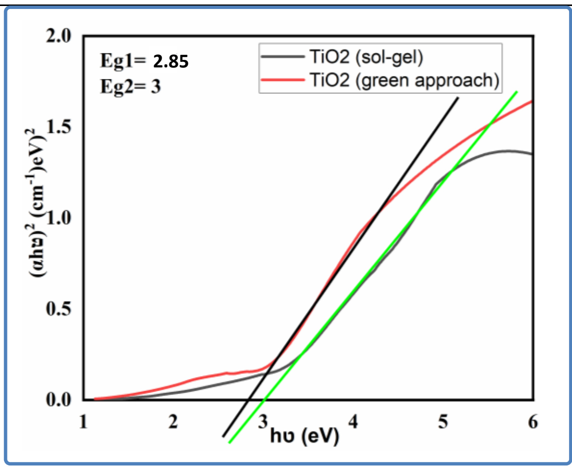

Figure 9. The Tauc plot for TiO_2 NPs.

Conclusion

TiO_2 nanoparticles were created utilizing an environmentally benign method of synthesis known as green synthesis. The exciting successful production of TiO_2 nanoparticles is extended by employing orange peel extract in a green manner. From XRD analysis, it is observed that a Tetragonal structure with an anatase phase was formed and the average crystallite size of the sample prepared from sol-gel and green method was obtained 30.2 nm and 24.2 nm, respectively. The presence of several functional biomolecules that worked as reducing and capping agents for the conversion of TiO_2 nanoparticles was shown by FT-IR findings. The maximum UV-Visible absorption appears to be approximately 350 nm, with a band gap energy of 2.85 eV and 3 eV for TiO_2 nanoparticles prepared by Sol-gel and green methods, respectively. The hexagonal and irregular in shape particles were confirmed through the FESEM analysis. The diameters of TiO_2 nanoparticles were in the range of 60-80 nm and 75-85 nm which were prepared via sol-gel and green methods, respectively.

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Authors' Declaration

- Conflicts of Interest: None.
- We hereby confirm that all the Figures and Tables in the manuscript are ours. Furthermore, any Figures and images, that are not ours, have been included with the necessary permission for re-publication, which is attached to the manuscript.
- Ethical Clearance: The project was approved by the local ethical committee in University of Middle Technical University.

Authors' Contribution Statement

All authors of this work have participated directly in the planning, execution, and analysis of this study. H. F. O. and A. J. R. designed and conducted the experiments, analyzed data, and co-wrote the manuscript. S. I. S. and A. A. B. assisted in device characterizations and gave conceptual advice in data analysis, overall manuscript editing, and writing.

Journal Declaration:

H. F. O. is an Editor for the journal but did not participate in the peer review process other than as an author. The authors declare no other conflict of interest.

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دراسة مقارنة بين تقنية Sol-gel والطريقة الخضراء باستخدام قشر البرتقال لتحضير جزيئات اوكسيد التيتانيوم (TiO_2) النانوية

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[4]الكلية التقنية الهندسية، الجامعة التقنية الوسطى، بغداد، العراق.

الخلاصة

تم استكشاف المستخلصات النباتية على نطاق واسع كبديل أكثر أمانًا للإجراءات التقليدية لتصنيع الجسيمات النانوية لأكاسيد المعادن. ركز البحث الحالي على تصنيع وتوصيف جزيئات ثاني أكسيد التيتانيوم النانوية (TiO_2NPs) باستخدام نهج صديق للبيئة (مستخلص قشر البرتقال) مقارنة بتقنية أخرى (طريقة Sol-Gel) في درجة حرارة الغرفة. تم تحليل جزيئات TiO_2 النانوية باستخدام حيود الأشعة السينية (XRD) لتحديد متعدد البلورات مع طور anatase لهيكل رباعي الزوايا ومتوسط حجم بلوري 30.2 نانومتر و 24.2 نانومتر من العينة المحضرة من Sol-gel والطريقة الخضراء على التوالي. تحليل مطيافية فوريرللاشعة تحت الحمراء (FTIR) لمعرفة طبيعة الاواصر وتحديد المجاميع الوظيفية لجسيمات اوكسيد التيتانيوم النانوية. تُظهر الأشعة فوق البنفسجية المرئية للخصائص البصرية(UV-Vis) أن طاقة فجوة النطاق كامت بين 2.85 الكترون- فولت و 3 الكترون- فولت للجسيمات النانوية TiO_2 تم تحضيرها باستخدام طرق sol-gel والطريقة الخضراء على التوالي. حدد تحليل FESEM الشكل المورفولوجي لسطح جسيمات TiO_2 النانوية بأقطار الجسيمات النانوية في حدود 60-80 نانومتر و 75-85 نانومتر تم تحضيرها عبر طرق sol-gel والطريقة الخضراء على التوالي.

الكلمات المفتاحية: تحليل فورير للاشعة تحت الحمراء، الطريقة الخضراء، الجسيمات النانوية، مستخلص قشور البرتقال ، سول-جل، ثنائي اوكسيد التيتانيوم.