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Sustainable Green for the Synthesis of TiO_2 Nanoparticles Using Solanum Melongena Extract and their Application in DNA Sensing

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ABSTRACT: In this study, a sustainable and environmentally benign methodology was developed for the synthesis of titanium dioxide (TiO_2) nanoparticles utilizing Solanum melongena (eggplant) extract as a natural reducing and stabilizing agent. The proposed biosynthetic route was intentionally designed to substitute conventional chemical pathways that commonly involve hazardous precursors and high-energy consumption. The synthesized TiO_2 nanomaterials were subjected to comprehensive structural and morphological characterization through X-ray diffraction (XRD), field-emission scanning electron microscopy (FE-SEM), and Fourier-transform infrared spectroscopy (FTIR). XRD patterns verified the formation of a highly crystalline anatase phase with an estimated mean crystallite size of approximately 13.6 nm. The FE-SEM micrographs revealed nearly spherical to quasi-spherical morphologies with an average particle diameter of about 29 nm, indicating that the bio mediated process enabled controlled nucleation and moderate particle-size homogeneity. The FTIR spectra exhibited characteristic Ti–O–Ti stretching vibrations together with residual phytochemical functional groups originating from the eggplant extract, confirming their involvement as surface-capping species that enhanced nanoparticle stability. In addition to the structural investigation, the electrical sensing performance of the TiO_2 nanomaterials toward DNA molecules was evaluated at ambient temperature. A comparative analysis between uncalcined and calcined (400 °C) TiO_2 samples demonstrated a pronounced improvement in sensitivity following calcination. The thermally treated TiO_2 displayed enhanced electrical conductivity, increased surface activity, and improved crystallinity, achieving DNA-sensing sensitivities in the range of 45–70%, compared with 12–18% for the uncalcined samples. The observed enhancement is attributed to the generation of oxygen vacancies and the phase transformation from amorphous to anatase, which collectively facilitated more efficient charge transfer across the TiO_2–biomolecule interface.

Keywords: Green synthesis; TiO_2 nanoparticles; Solanum melongena; Calcination; Anatase phase; DNA sensing; Oxygen vacancies

1 INTRODUCTION

Nanotechnology is the study and application of materials on the nanoscale of about 1 to 100 nm with a revolution in modern science and engineering that takes advantage of the physicochemical properties that arise with this scale. These are a significantly high surface volume ratio, strong quantum confinement effects, and tunable optical and electronic characteristics which can all be utilized to achieve high-level functional behavior compared to bulk materials. .[1], [2].

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Titanium dioxide (TiO_2) is considered to be one of the most universal and widely studied compounds among other engineered nanomaterials because of its excellent photocatalytic activity, remarkable chemical inertness, environmental friendliness, and optical properties. Notwithstanding these merits, the common synthesis pathways to TiO_2 nanoparticles usually rely on harsh chemical inert and high temperature, which frequently results in the production of by-products that are risky to the environment. Not only do such considerations limit the sustainability of such processes, but also reduce their ability to be compatible with biological and environmentally-friendly applications.[3] , [4].

The only synthesis pathway that has received much scientific attention is biosynthesis, which is also known as green synthesis as a more environmentally-friendly version of traditional synthesis pathways. Plant-derived extracts containing high levels of phytochemicals including flavonoids, phenolic compounds, terpenoids, and natural antioxidants are utilized as intrinsic reducing, stabilizing, and capping agents, which are used in this technique. Their biochemical activity is useful in the preparation of nanoparticles with mild conditions, which do not use any severe reagents, allowing a more sustainable and less contaminated method of nanomaterial production. .[5]. This green approach not only gets rid of any hard-core chemical lowering agents or artificial surfactants but also reduces the total energy usage significantly, hence making the process more sustainable and less intrusive of the environment. A number of reviews which have been well conducted have noted a huge number of examples of TiO_2 nanoparticle formation using green techniques which have been claimed to exhibit improved biocompatibility, less complicated preparation procedures and significantly low environmental risks in comparison to the conventional chemical synthesis methods. .[6], [7] Some of the latest researches are examples of using various plant extracts to synthesize TiO_2 greenly. [8]. As an example, Ansari et al. (2024) found the green synthesis of TiO_2 nanoparticles using spinach (Spinacia oleracea) leaf extract as a bio-reducing agent successful. They obtained the synthesis of anatase-phase nanoparticles with a size of 10-40 nm by optimizing the synthesis parameters and this clearly shows the environmental benefits and sustainability of the biosynthetic process compared to the conventional chemical process.]9[In another work, Shakeel et al. reported the synthesis of TiO_2 nanoparticles via various plant extracts and discussed how plant-derived biomolecules influence the physicochemical properties of the NPs, In a different article, Shakeel et al. reported the synthesis of TiO_2 nanoparticles by use of different plant extracts and the effects of the biomolecular composition of the plant extracts on the physicochemical properties of the nanomaterials. On this, this paper will use Solanum melongena (eggplant) extract as a green reducing and capping agent to fabricate TiO_2 nanoparticles. Its primary aims include the synthesis of TiO_2 in mild environmentally friendly conditions; the analysis of the crystallinity and morphology of the prepared samples with the help of X-ray diffraction (XRD), Fourier-Transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM); and the comparison of their structural integrity and purity to that of other green and conventional synthesis techniques. This study would add to the current collection of knowledge on green-synthesized nanomaterials and would facilitate the development of sustainable production techniques of TiO_2 nanoparticles.

2 EXPERIMENTAL PART

Titanium dioxide (TiO_2) nanoparticles were synthesized greenly by using an aqueous extract of Solanum melongena (eggplant) peels that were used as a natural reducing and stabilizing system. Peels were washed thoroughly with deionized water to cleanse the surface of the peel, followed by cutting them into small portions and shade drying them to preserve phytoconstituents that are heat sensitive. About 20 g of the dry substance was boiled in 200 mL of deionized water at 80 o C for 30 minutes, after which the mixture was filtered using Whatman No. 1 paper to get a clear extract containing polyphenols, flavonoids, and alkaloids. In order to prepare nanoparticles, a 0.1 M titanium tetrachloride $Ti(OH)_4$ solution was prepared under ice-cold conditions and added drop by drop to 100 mL of the eggplant extract, which was continuously stirred by a magnetic stirrer. The mixture was kept at 50 o C temperature for 2 hours, which favored hydrolysis and nucleation of TiO_2 particles, which was visually represented by a gradual change of color. The ensuing suspension was centrifuged at 4000 rpm over 15 minutes to give the precipitate, which was again washed with deionized water and ethanol to eliminate remaining biomolecules. The purified compound was dried at 80 0 C overnight, then calcinated at (350 -450)/2 h in a muffle furnace to enhance crystallinity and remove organic residues to produce phase-pure TiO_2 nanoparticles.

3 RESULTS AND DISCUSSION

3.1 STRUCTURE ANALYSIS

These were confirmed through the X-ray diffraction (XRD) findings that indicated formation of crystalline titanium dioxide (TiO_2) nanoparticles that had been synthesized using the green route with Solanum melongena extract. A diffraction pattern obtained showed clear peaks with the characteristic reflections of the anatase phase of TiO_2 at 25.26º, 37.77º, 48.05º, 53.92º, 55.04º, 62.65º, 69.6º, and 75.15 2 º. These peaks are related to different crystallographic planes and the values of their Full Width at Half Maximum (FWHM) were used to give an idea of the extent to which the material is crystalline as well as the average size of crystallite domain. The FWHM values in this experiment were ranging between around 0.44º at the 25.26º maximum and 2.26º at the 69.6º maximum which means that the coherent scattering domain dimensions varied between the different lattice orientations. Generally, the sharper and narrower the peaks are the larger the crystallites and the higher the structural order, and the broader the peaks are the smaller the crystalline domains and the lattice strain existing in the nanoparticles.[10].

Crystallite sizes were calculated using the Debye–Scherrer equation:

$$D = \frac{K\lambda}{\beta \cos\theta}$$

Debye Scherrer equation was used to determine the crystallite size of the produced TiO_2 nanoparticles and the crystallite size is represented by D, shape factor (usually assumed to be 0.94) is given by k, wavelength of incident X-ray (1.54 AA in the case of Cu K a radiation), full width half maximum (FWHM) is represented by B, and Bragg diffraction angle is represented by theta. Using these parameters, the peak of the diffraction at 25.26 o yielded a crystallite f 19.13 nm (estimated). The corresponding crystallites sizes calculated on the peaks of the 37.77, 48.05, 53.92, 55.04, 62.65, 69.6 and 75.15 were 14.05, 18.01, 13.85, 15.62, 12.12, 4.29 and 13.43 nm, respectively. The extremely short dimension of crystallites based on the 69.6 0 peak (4.29 nm) may be ascribed to the strain of lattices or localized changes in the arrangement of crystals towards that plane. An average of all the values obtained in all the reflections gave mean crystallite size of about 13.65 nm which confirmed that the green method of synthesizing TiO_2 nanoparticles using Solanum melongena extract was successful to produce crystallite in a well-defined nanoscale of synthesis. The acuity and the sharpness of the diffraction peaks are also yet another testimony of a high degree of crystallinity, which has been made available in the material prepared. The coincidence of the peak positions with the standard reference data of JCPDS proves the dominance of the anatase phase, which is generally known to be the most effective as a photocatalyst and also with positive characteristics in some biomedical applications. Significance of this phase being pre-eminent is especially important because anatase TiO_2 has a higher capacity of charge separation and nature of transport hence, enhancing its functional capacity.

The Solanum melongena extract in this biosynthetic pathway has a twofold role to play: it reduces the titanium precursor to TiO_2 and also serves as a capping agent that regulates crystal growth and reduces particle aggregation. The active biomolecules affect the kinetics of nucleation and growth giving a well-controlled morphology and crystallite-size of nanoparticles. the XRD pattern gives strong data as shown in Fig.1. that the TiO_2 nanoparticles produced through this green technology are well crystalline in their structure, anatase type mainly, and with an average crystallite dimension of approximately 13.65 nm. These results verify that the biosynthetic approach is effective, reproducible and environmentally friendly which makes the produced nanomaterial a viable candidate in photocatalytic reactions, environmental cleaning technologies and in the development of biomedical applications

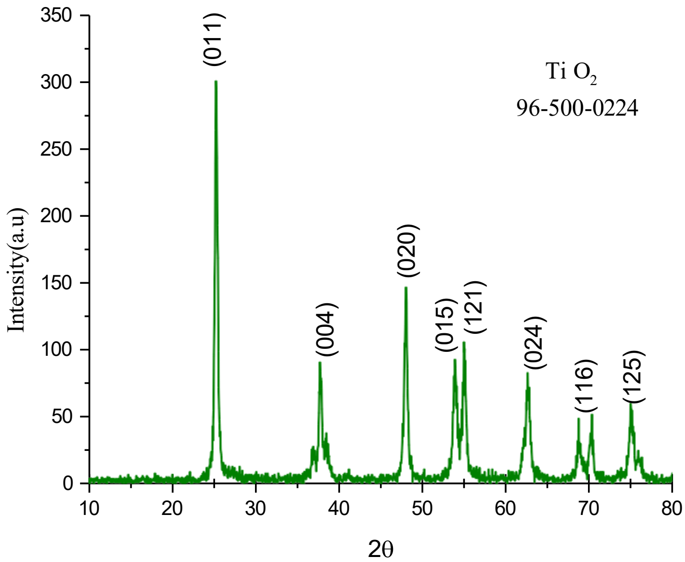


FIGURE 1. XRD pattern of TiO_2 NPs

The table 1. Shown the structural properties of TiO_2 nanoparticles by the green synthesis method.

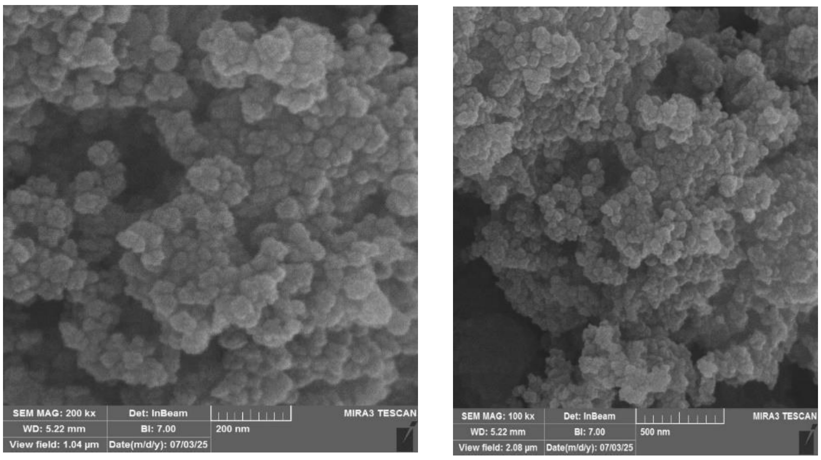
Table 1. - The structural properties of TiO_2 NPs

2θ (Deg.)	FWHM (Deg.)	Crystallite Size (nm)	Average Crystallite Size (nm)
25.26	0.44	19.13	13.65
37.77	0.60	14.05	
48.05	0.48	18.01	
53.92	0.64	13.85	
55.04	0.57	15.62	
62.65	0.77	12.12	
69.6	2.26	4.29	
75.15	0.75	13.43	

3.2 FIELD EMISSION SCANNING MICROSCOPY FE-SEM RESULT

The particle size distribution analysis, which is complemented by the field emission scanning electron microscopy (FE-SEM) examination, provides a detailed examination of morphology and dimensional features of the developed TiO_2 nanoparticles, see Figure. 2. The statistical analysis of the data discloses the mean diameter of particles to be about 29.4 nm and the standard deviation of 6.45 nm, which means that the size dispersion is rather low. The sizes of the measured particles are found to be between 18 and 45 nm with a median size of 27.7 nm, which is very close to the size elements of the nanoscale that could be observed in the FE-SEM micrographs. Microstructural images represent mainly spherical to quasi-spherical nanoparticles that will be likely to be in aggregate formations a behavior that is commonly found in TiO 2 systems because of their high surface energy and the strong van der Waals forces between particles. The particle size distribution also supports the fact that the majority of nanoparticles fall under the 20-35 nm range with a smaller portion at larger sizes probably due to the partial agglomeration or overlap of the finer crystallites in synthesis and subsequent calcination.

These findings were confirmed using statistical size analysis and with support of observations confirming that the nanostructured TiO_2 with uniform morphology and well controlled nanoscale dimensions had been successfully fabricated, see Figure 3. The homogenous distribution of particles and the mean size of 30 nm emphasize the efficacy of the green methodology of synthesizing nanoparticles using the Solanum melongena extract in that the nucleation and growth of the nanoparticles are effectively controlled to form homogeneous and stable nanoparticles.



A B

Figure 2. FE-SEM Image at (A) 200nm and (B) 500 nm for TiO_2 Nanoparticles

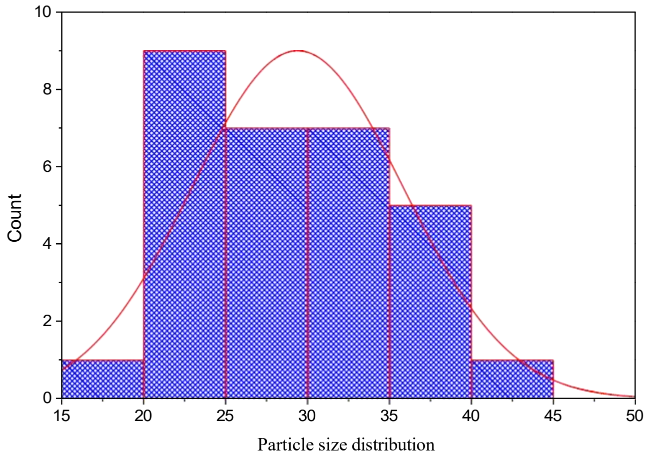


Figure 3. Histogram of Particle Size Distribution (nm) for TiO2 Nanoparticles.

3.3 FOURIER-TRANSFORM INFRARED (FTIR) SPECTRUM RESULT

The Fourier-transform infrared (FTIR) spectrum of the TiO_2 nanoparticles prepared displays various distinct absorption bands which together confirm the effective formation and stabilization of the nanostructure. as shown Feg. 4 . An extensive absorption band at an average of 3400 cm -1 is associated with O -H stretching bands, is attributable to surface-bound water molecules and hydroxyl groups, which are naturally found on TiO_2 surfaces due to their basic hydrophilicity. The absorption band at approximately 2920 cm -1 is attributed to C-H stretching vibrations and it is probably because of the presence of some traces of organic constituents or phytochemicals in the Solanum melongena extract that serve as natural capping and stabilizing reagents during the synthesis. Conspicuous peak at the range of 1630-1650 cm -1 is attributed to C=O stretching vibrations indicating that plant extract carbonyl-containing biomolecules are involved in the stabilization of nanoparticles. Most importantly, the domineering absorption band at the range of less than 800 cm -1 is the Ti -O -Ti stretch modes, which signify the type of fingerprint of the titanium dioxide lattice and indicate that the oxide network has been formed successfully.

The presence of two functional groups; Ti O bond and the remaining organic functional group depict that the eggplant extract successfully mediated bio-reduction and surface stabilization. In spite of the fact the following step of calcification considerably decreased the content of organically remained substances, slight traces of the biological nature of the synthesis path were still observed. All in all, the FTIR results support the importance of Solanum melongena phytochemicals in governing the reduction and stabilization process in addition to the structural identity of the resulting TiO_2 nanoparticles

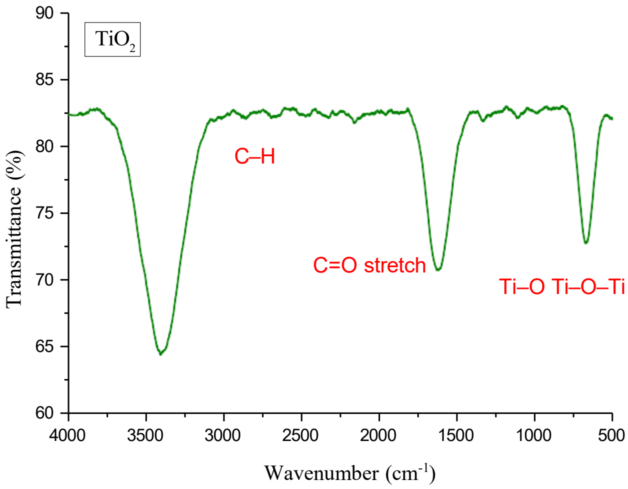


Figure 4. FTIR Spectrum TiO_2

3.4 TiO_2 NPs FOR DNA SENSING

The feasibility of titanium dioxide (TiO_2) nanomaterials in the detection of DNA in the ambient environment. The choice of TiO_2 was informed by its semiconductive nature and surface features, and hence it is a viable biosensing candidate. The electrical resistance of the prepared TiO_2 samples was monitored continuously with time during varying levels of DNA concentration and under different preparation media in order to assess its sensing behavior. The associated resistance profiles of the time series that are shown in Fig. (5), the nature of the TiO_2 nanostructures as a responsive material towards exposure to DNA is clearly demonstrated. The resistance variations which are observed support the ability of the material to interact with biomolecular species and proves that it can be a good transducer in DNA bio-detection.

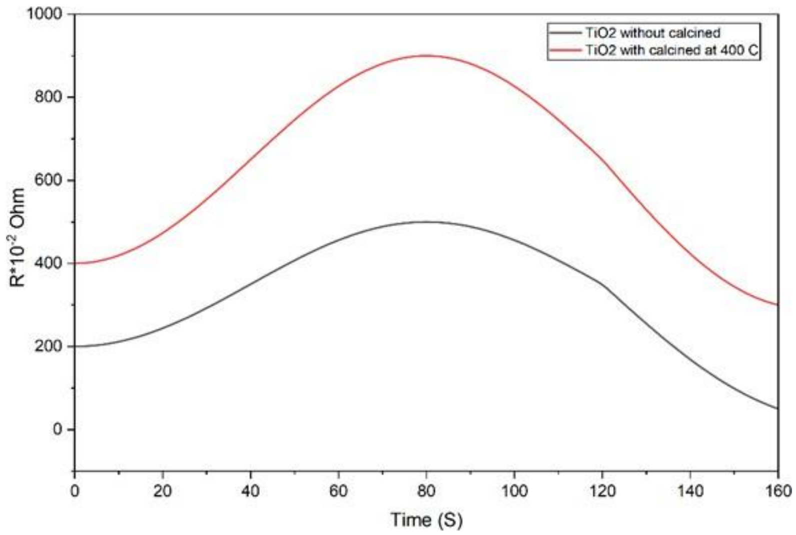


Figure 5 **TiO2 NPs sensing for DNA**

The adsorption of ionic moieties and polar biomolecular groups onto the TiO_2 surface is the main regulator of the sensing mechanism, causing changes in the surface charge density and electron mobility. The interaction influences the net electrical conductance of the nanomaterial in that it promotes or inhibits the transfer of charge between the surface states of the nanomaterial and the adsorbed DNA molecules. It was found that the TiO_2 sample of which the calcination was carried out at 400 C showed significantly better sensing properties and sensitivity to the sample which was not subjected to the calcination treatment. This is due to a better crystallinity, increased surface reactivity and faster charge-carrier transport pathways caused by the ablation of surface-bound organic residues in the course of calcination.

In terms of inorganic and physical chemistry, the difference in the response of resistance between the calcined and uncalcified TiO_2 samples can be explained by the aspects of semiconductor nature of TiO_2 which is an n-type material and is strongly affected by the state of the surface of samples and oxygen vacancies. When in contact with the DNA, the negatively charged phosphate groups and nitrogenous bases are adsorbed onto the TiO_2 surface and it triggers the charge transfer processes which cause changes in the electron density of the conduction band. The extent of such modulation is highly dependent on the structural order, defect density and the electronic configuration of the material. This leads to the more pronounced and stable response of the calcined TiO_2 with lower structural disorder and carrier mobility suitable in biosensing applications.

The thermal calcification process of titanium dioxide (TiO_2) at a temperature of 400 C facilitates a structural transformation of titanium dioxide (TiO_2) between an amorphous form and a distinct anatase crystal form. This method is known to remove residual hydroxyl and organic surface groups formed by the biosynthetic medium and at the same time form-controlled population of oxygen vacancies that act as shallow donor levels in the semiconductor lattice. The overall result of these changes is an improvement in the charge-carrier mobility and stabilization of the active surface sites, thus leading to more efficient charge transfer between the biomolecules adsorbed onto the TiO_2 network and the TiO_2 framework. Consequently, the calcined TiO_2 sample reveals a much better response and a stronger change in resistance when exposed to DNA than the uncalcinated sample, which still retains the effect of structural disorder, higher concentration of trapping centers and lower electrical conductivity. Crystallinity and electrochemical homogeneity are enhanced by the calcination process that leads to a better and more reproducible electrochemical response, which highlights the importance of thermal treatment in the optimization of TiO_2 -based biosensing materials to enable biomolecular detection.

Fig. (6) shows the relationship between S% and calcination temperature of TiO_2 nanomaterials used as detectors of DNA strands. The information shows that the sensing performance highly depends upon the applied thermal conditions and it is clear that calcination has a conclusive effect on the physicochemical and electronic nature of TiO_2, which ultimately determines the efficiency of the latter as a biosensing material.

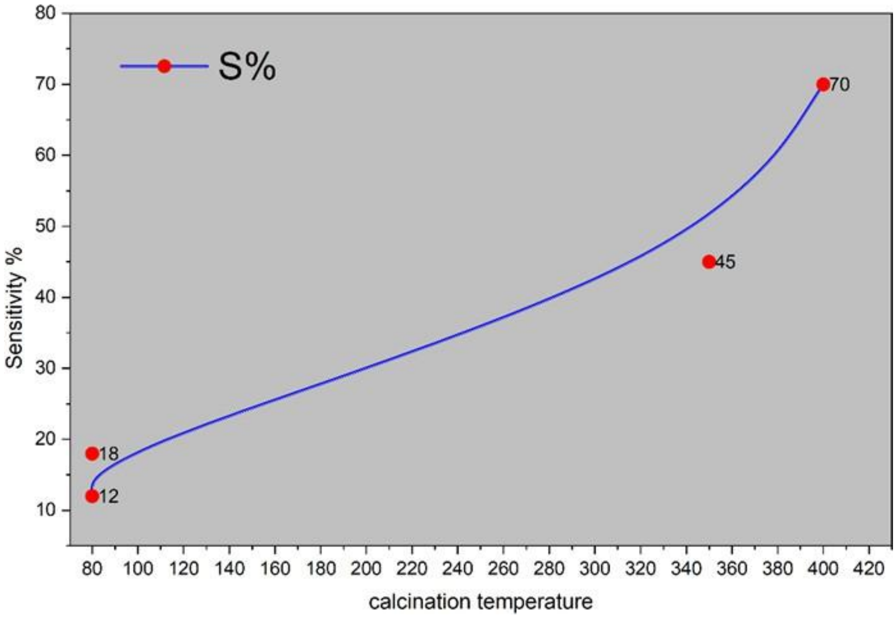


Figure 6. **Relationship Between S% and Calcination Temperature of Tio_2**

The uncalcinated TiO $_2$ samples had relatively low sensitivity values of about 12 and 18 as shown on the left side of the respective plot. This low sensing performance is also mainly caused by amorphous and poor crystallinity of TiO 2, before heat treatment. The presence of residual hydroxyl surface, interred adsorbed organics and irregular grain boundary surfaces leads to an inefficient charge transport across the nanomaterial. These structural flaws present many electron-storing sites that limit the mobility of carriers and hinder the proper interaction of the sensing surface and the target biomolecules. Consequently, the uncalcium TiO $_2$ samples have low electrical conductivity and their response to resistance is weak when the adsorbent is exposed to an analyte, resulting in a general low sensitivity with that of its thermally treated counterparts.

Sample TiO_2 which had undergone calcification at 400 ℃ showed a significant improvement in sensing, and the values of sensitivity rose to about 45 percent and 70 percent. The strong enhancement of this can be directly ascribed to the thermally driven structural change of an amorphous to a well-ordered anatase phase. The transformation enhances the optimization of TiO_2Ti linkages, reduction of lattice defects and creation of oxygen vacancies, which serve as active sites in the adsorption and cation exchange of molecules. In addition, the increase in crystallinity leads to a significant increase in electrical conductivity and a higher level of electron movement across the nanoparticle interface and therefore charge transfer across the adsorbed biomolecular species and TiO $_2$ conduction band.

Physically speaking, it can be concluded that because the sensitivity increases gradually as the temperature of calcification increases, the thermal treatment is effective in optimizing the surface reactivity of TiO_2 as well as the electronic band structure of the material. These enhancements minimize the possible impediment of charge transfer to the process of charge transfer in sensing, hence increasing the efficiency of electron exchange between the semiconductor and the adsorbed biomolecules. The sensitivity curve suggests exponentially in nature, further suggesting that there is an activation condition, which in the case of TiO $_2$ is usually 300 -400 ℃, when the material becomes crystalline enough and has an appropriate population of surface defects. These aspects combine in a way that there is a significant enhancement in its sensing response and overall performance

4 CONCLUSION

Synthesis of titanium dioxide (TiO $_{2)}$ nanoparticles by using an eco-friendly, sustainable approach of synthesizing nanoparticles as intrinsic reducing/stabilizing media by Solanum melongena (eggplant) extract. The developed biosynthetic strategy was efficient in substituting the traditional chemical procedures that require the use of toxic substances and energy-demanding conditions yet resulted in nanomaterials of high crystallinity, structural consistency, and compositional purity. In-depth structural characterisation established the successful generation of anatase-phase TiO_2 with nanoscale characteristics. Analysis with the X-ray diffraction (XRD) indicated an average crystallite size of approximately 13.65 nm, but FE-SEM micrographs depicted almost homogeneous and well dispersed particles whose average diameter was approximately 29 nm. The FTIR results supported the presence of bioactive phytochemicals in the eggplant extract, and as a consequence, it played a key role in reducing, stabilizing the surface, and inhibiting agglomeration of nanoparticles. These electrical sensing researches also demonstrated that there is a direct relationship between the temperature of calcification and the behavior of the biosensor. The sensitivity of samples that were calcined at 400 C to DNA molecules was significantly higher than that of the uncalcified samples as high as 70 percent towards DNA molecules. This is due to the fact that it is improved as a result of transition to crystalline anatase structure, surface oxygen vacancies enrichment and carrier mobility in general that enable more efficient charge transfer between species of biomolecules and TiO_2 surface.

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