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Facile Green Synthesis of α -Bismuth Oxide Nanoparticles: Its Photocatalytic and Electrochemical Sensing of Glucose and Uric Acid in an Acidic Medium

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Abstract: The nanocrystalline bismuth oxide (Bi₂O₃) was produced utilizing a green combustion process with Mexican Mint gel as the fuel. The powder X-ray diffraction (PXRD) method proved the nanocrystalline nature and Bi₂O₃ nanoparticles (BONPs) in α phase and the average crystalline size of BONPs nanoparticles has been found to be 60 nm. The spherical-shaped structure with bright dot-like spots in the center of the selected area diffraction (SAED) is confirmed by the scanning electron microscopy (SEM) and Energy dispersive X-ray spectroscopy (EDAX) in conjunction with the transmission electron microscopy (TEM) and high-resolution transmission electron microscopy (HRTEM) demonstrating the crystalline behavior of green NPs. The Kubelka-Monk function was used to analyze diffuse reflectance spectra, and the results revealed that BONPs have a band gap of 3.07 eV. When utilized to evaluate the photocatalytic capabilities of NPs, the direct green (DG) and fast orange red (F-OR) dyes were found to be activated at 618 and 503 nm, respectively. After 120 min of exposure to UV radiation, the DG and F-OR dyes' photodegradation rate reduced its hue by up to 88.2% and 94%, respectively. Cyclic voltammetry (CV) and electrochemical impedance techniques in 0.1 N HCl were used to efficiently analyze the electrochemical behavior of the produced BONPs. A carbon paste electrode that had been enhanced with BONPs was used to detect the glucose and uric acid in a 0.1 N HCl solution. The results of the cyclic voltammetry point to the excellent electrochemical qualities of BONPs. Bi_2O_3 electrode material was found to have a proton diffusion coefficient of $1.039 \times 10^{-5} \text{ cm}^2 \text{s}^{-1}$. BONP exhibits significant potential as an electrode material for sensing chemicals like glucose and uric acid, according to the electrochemical behavior.

Keywords: Bi₂O₃ nanoparticles; Mexican Mint; green combustion; cyclic voltammetry; glucose; uric acid

1. Introduction

Bismuth is a p-block transition metal, a non-hazardous naturally occurring environmentally friendly element. When it comes to human health, bismuth has a number of positive impacts. Bismuth salts are used to treat diarrhea, peptic ulcer disease, and gastrointestinal diseases. The unique properties exhibited by α -Bismuth Oxide nanoparticles (α -BONPs) render them highly significant to the field of nanomaterials. α -BONPs are an appealing choice for a range of applications due to their environmental-friendliness and non-toxicity, in contrast to other hazardous materials Additionally, the combination of



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Copyright: © 2024 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). bismuth with other nanomaterials or the development of bismuth-based composites could enhance its performance in some applications. Due to the aforementioned use, researchers are drawn to concentrating emphasis on this component in the field of nanomaterials. It can be incorporated into nanocomposites with other nanomaterials to enhance or impart specific properties due this bismuth oxides may be suitable for photo degradation of dyes and sensing applications.

This may include combinations with other metals, metal oxides, or carbon-based nanomaterials to improve electrical, catalysis, and energy storage devices [1]. The multifunctionality of α -BONPs is underscored by their utilization in nanocomposites, including ZnO/MgO/Fe₂O₃, which demonstrates their efficacy in the process of adsorption to eliminate pollutants. This development has far-reaching implications for the field of nanotechnology, including but not limited to environmental remediation and advanced technological applications. Several nanomaterials such as Zno, TiO₂ and Mn + Cu bimetallic composites have been studied for photocatalytic and electrochemical sensing [2,3].

Bismuth oxide is a versatile material that works effectively for a wide range of significant applications, which include catalysts, energy storage, gas sensors, optical coating, solar cells, antibacterial, anticancer, antioxidant, antifungal, and enzyme inhibitory characteristics. Their outstanding biological applications [4–6] are due to their unique electrical, catalytic, magnetic, and optical capabilities [7–11]. There are six polymorphic forms of bismuth oxide, which are known as Bismuth oxide α -Bi₂O₃ (monoclinic), β -Bi₂O₃ (tetragonal), δ -Bi₂O₃ (face-centered cubic) γ -Bi₂O₃ (body-centered cubic) ω -Bi₂O₃ (orthorhombic) and Bi₂O₃ (triclinic) [12,13].

The community of oxides like ZnO/MgO/Fe₂O₃ nanocomposites used as a remarkable adsorbent material to remove harmful metal particles from polluted water had a significant impact on the environment. These nanocomposites are activated by UV-light [14]. Bi₂O₃ is considered to be of the semiconductor catalysts that demonstrate efficient catalytic characteristics for the breakdown of environmental pollutants due to its distinctive structure and physical properties; Bi₂O₃ offers a high refractive index, less toxicity, wide band-gap, high dielectric properties, and high photoconductivity enzyme inhibition properties [15–17]. The direct optical band gaps of BONPs, which range from 2.96 to 3.96 eV, make them an ideal photocatalyst for the breakdown of pollutant dye when exposed to UV light [18,19]. Bi₂O₃, a heterogeneous semiconductor, has long been recognized as one of the most efficient photocatalysts, playing a pivotal role in modern solid-state technology. Notably, this semiconductor generates highly reactive species that initiate oxidation reactions, leading to the degradation of dyes [20].

 Bi_2O_3 's capacity to oxidize dye and generate superoxide and highly reactive oxide radicals accounts for its photocatalytic activity. Additionally, Bi₂O₃ has distinctive characteristics at the nanoscale that could be targeted during manufacture to improve usage in many fields [21–23]. Several existing analytical techniques are used to detect heterocyclic compounds and antioxidants such as spectrophotometer [24], colorimetry [25], and HPLC [26]. But a low-cost, highly sensitive, and user-friendly analytical method is much needed. The electrochemical method gives high sensitivity and selectivity, compared to other traditional methods. An electrochemical detection technique offers a variety of benefits, one of the industries with the quickest growth sensors technology. Amperometric sensors utilize the process of oxidizing or reducing an electroactive species to measure the voltage difference between a working electrode and a reference electrode [27–30]. An electrode alteration is typically required for a sensitive thorough examination of ascorbic acid and uric acid concentrations in whole blood or urine samples [21,31]. For this purpose, a carbon electrode modified with BONPs has been used. By employing modified electrodes, it is possible to determine the sensitivity and selectivity of ascorbic acid and uric acid substantially superior outcomes [32].

Plenty of researchers have presented several techniques for the preparation of nanoparticles, by epitaxial growth [33], magnetron sputtering [34], chemical precipitation [35], solidstate reaction [36], sonochemical route [37], hydrothermal [38] and green synthesis [39]. In the present work preparation of Bismuth oxide utilizing Mexican Mint gel as a green fuel in solution combustion technique is superior compared to various approaches for the reason that it is simple to process, uses relatively low temperatures, makes it simple to control particle size, which reduces reaction times, produces very pure and crystalline products, and produces nanosized powder with a high specific surface area. Plant extracts are frequently employed as sustainable and environmentally acceptable stabilizing and reducing agents [40]. The impact of Mexican Mint gel on the characteristics of α -Bismuth Oxide nanoparticles (α -BONPs) is an important element that deserves investigation. The gel, when used as a green fuel in the solution combustion approach, ha60s the potential to add distinctive organic components, surface properties, or stabilizing effects during the production of nanoparticles. They can lower the synthesis process's negative environmental effects by taking the place of conventional chemical reagents. Bioactive substances found in plant extracts have the ability to function as reducing agents, assisting in the combustion process of metal ions into nanoparticle-sized metal particles. Furthermore, these substances have the ability to act as stabilizing agents, which avoid agglomeration and guarantee the stability of the resultant nanoparticles.

The widespread utilization of uric acid and glucose as model analytes in the development and evaluation of electrochemical sensors led to their selection as analytes for electrochemical investigations. Glucose and uric acid are frequently selected as diagnostic and medical indicators due to their wide-ranging utility. One application of sensors that integrate these analytes is in the monitoring of blood glucose levels in diabetics, as well as in a wider range of medical diagnostics. In biological fluids, elevated concentrations of uric acid and glucose may be indicative of particular medical conditions. Therefore, in clinical diagnostics, electrochemical sensors for these analytes are indispensable because they enable rapid and sensitive identification and quantification of the substances in blood, serum, or urine.

The objective of this research endeavor is to perform a comprehensive evaluation of the photocatalytic, electrochemical, and sensor characteristics of α -Bi₂O₃. This investigation will yield significant knowledge regarding the potential uses of the material, with a particular focus on its implications in the field of medical diagnostics.

Moreover, the research emphasizes the significance of discussing potential interferences and the electrode's selectivity in the context of uric acid and glucose detection. In order to improve selectivity, emphasis should be placed on the selection of electrode material, surface modifications, and incorporation of particular recognition elements. Potential interferences in practical applications and the strategies employed by the sensor design to address or mitigate these concerns should be explicitly addressed in the study. Enhancing selectivity may be achieved through the incorporation of Bismuth Oxide nanoparticles into the carbon electrode and analyzing the distinct electrochemical signatures of glucose and uric acid. By conducting a comprehensive examination of potential interferences and the electrode's selectivity, the electrochemical sensor's credibility and practicality in detecting glucose and uric acid can be significantly improved.

Additionally, an in-depth assessment of the photocatalytic, electrochemical, and sensor properties of α -Bi₂O₃ is made. Lastly, this study looks for ways to improve α -Bismuth Oxide nanoparticles' photocatalytic activity even more. This could include investigating new green synthesis techniques, maximizing the size of nanoparticles, and adding co-catalysts to increase the effectiveness of environmental remediation and pollutant degradation. Expand the use of α -Bismuth Oxide nanoparticles in biomedical sensing even further. Examine their possibilities for physiological glucose and uric acid detection while taking into account aspects like biocompatibility, stability, and repeatability for application in wearable or implantable biosensors.

2. Materials and Methods

2.1. Synthesis of α -BONPs

The synthesis of BONPs by using a green fuel, Mexican Mint latex, was conducted through the combustion method. The Mexican Mint leaves were collected from the nearby nursery, in Saudi Arabia and 5 g of the cleaned and chopped Mexican Mint leaves were used to create the gel, which was then dissolved in 10 mL of double-distilled water and agitated for 30 min to create the solution. The obtained solution was used as Mexican Mint plant-extracted fuel. In these studies, 10 mL of Mexican Mint was used as fuel, and a stoichiometric ratio of bismuth nitrate (Sigma Aldrich St. Louis, MO, USA) was added. For 15 min, the mixture was finely agitated with a magnetic stirrer. The mixture was heated in a muffle furnace to 250 °C for two hours, the gel transformed into white foam, and the foam caught fire. The sample combustion spread throughout the volume quickly, and it generated a yellowish powder residue. The entire process was accomplished within 5 min. To make use of structural and other studies, the resultant product was calcined at 600 °C for 2 h. Graphical representation of synthesis of BONPs is shown in Figure 1.



Figure 1. Graphical representation of synthesis of BONPs.

Fabrication of Working Electrode

The working electrode were fabricated after about 30 min of manual grinding with an agate mortar, graphite powder, silicon oil, and synthesized nanoparticles (α -BONPs) in the ratio of 70:15:15 resulting the graphite in paste form. A handmade Teflon tube is filled with the resultant graphite paste, and the tube's surface is polished by rubbing it against weighing paper [40]. In order to create an electrode that interacts selectively with glucose and uric acid, we picked graphite powder in varying percentages. We then select an optimal amount of graphite powder, which is then modified with Bi₂O₃ NPs.

For the electrochemical investigations, a three-electrode configuration was employed, with a composite material consisting of 70% graphite, 15% silicon oil, and α -BONPs as the working electrode. To measure the cyclic voltametric characteristics the electrodes were fabricated of these composite materials and are subjected to a voltage range of -0.6 V to +0.2 V. The oxidation and reduction potentials linked to the α -BONPs were identified within this range. The voltage at which the oxidation reaction takes place on the surface of the working electrode was determined to be the oxidation potential for the BONPs, which is -0.201 V. This potential is the amount of energy needed to take electrons out of BONPs so

that oxidized species can develop. Likewise, -0.518 V was found to represent the reduction potential for the BONPs. The tendency of a chemical species to acquire electrons and go through reduction at the working electrode is measured by the reduction potential. It stands for the energy needed to give BONPs an additional electron so that reduced species can develop.

2.2. Characterization

The crystal structure of the nanomaterial was determined using a Shimadzu X-ray diffractometer, Nakagyo-ku, Kyoto, Japan operated at 50 kV and 20 mA with CuK radiation. To explore its morphological properties, a JEOL JEM-2100 TEM analyzer Akishima, Tokyo, Japan was employed. The absorption properties were studied using a Shimadzu UV-2600 instrument Nakagyo-ku, Kyoto, Japan. Additionally, the material's optical characteristics were analyzed using a Horiba spectrometer Minami-ku, Kyoto, Japan) at room temperature, with a 450 W Xe-lamp (Horiba Ltd., Minami-ku, Kyoto, Japan) as the light source. Photocatalytic testing was conducted using a mercury vapour lamp UV light source (Horiba Ltd., Minami-ku, Kyoto, Japan) with a pressure of 125 W and a spherical glass reactor (Horiba Ltd., Minami-ku, Kyoto, Japan) with a surface area of 176.6 cm². 60 mg of the photocatalyst sample BONPs was dissolved in 250 mL of a concentrated dye solution containing 20 ppm double-distilled water and agitated during the experiment. In the open air, the reaction mixture was illuminated from a distance of 23 cm. There was no obstruction of alternative light wavelengths by any filters. Following a designated time interval of 15 min, 5 mL of the reaction mixture was withdrawn from the dye mixture and an external magnet was employed to eliminate the catalyst particles [41,42]. The CV tests were conducted using a CHI608E potentiostat (De Zaale 11 5612 AJ Eindhoven The Netherlands) and a three-electrode system consisting of a platinum wire electrode, a carbon paste electrode (De Zaale 11 5612 AJ Eindhoven The Netherlands) (1 mm surface area), and Ag/AgCl as the working, counter, and reference electrodes, respectively, in 0.1 N HCl at scan rates of 10, 20, 30, 40, and 50 mV/s. The potential variation from the platinum wire electrode to the Ag/AgCl electrode ranged from -1.2 to 0.2 V. AC amplitude of 5 mV was utilized throughout the frequency range of 1 Hz to 1 MHz for the EIS investigations.

3. Results and Discussion

3.1. PXRD Analysis

The synthesized α -BONPs were subjected to PXRD analysis at a low scan rate of 2° min⁻¹ to investigate their crystal structure and the purity of the nanomaterial, the outcomes of this analysis are illustrated in Figure 2. The obtained PXRD peaks show 2 θ values of 19.74°, 25.92°, 27.09°, 27.5°, 33.4°, 35.2°, 37.0, 46.45°, 52.57°, 54.9° are correlated to monoclinic α -Bi₂O₃ (020), (002), (111), (120), (200), (212), (121), (041), (-321), (-241) highly matched with the monoclinic structure and in accordance with JCPDS card number. 71-2274 lattice planes [43]. The lattice parameters for the monoclinic structure of α -BONPs were found to be 5:8499A°, 8:1698 A°, 7:5123 A° for a, b c respectively. In addition, α , β , γ were found to be 90°, 112:988°, and γ = 90° respectively [44]. By substituting the values for λ , β and θ in Debye—Scherrer's formula (Equation (1)) [45] the crystallite size for α -BONPs was calculated and it was noted to be ~60 nm.

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

In the equation above, λ is the wavelength of X-rays, β represents the full width at half maximum (FWHM) of XRD peaks.



Figure 2. X-ray powder diffractogram of synthesized α -BONPs.

3.2. FTIR Analysis

FTIR spectral measurements determine the purity of a compound, as well as its physical and chemical properties and several vibrational modes of the functional groups of -BONPs were observed between 400 and 400 cm⁻¹ (Figure 3). Typically, the metal-oxygen (M-O) bending vibrational modes were discovered within a fingerprint area (1500–400 cm⁻¹). Peak 3655 cm⁻¹, which is a medium-broad peak of the group O-H extending, 1132 cm⁻¹ is medium broad peak of group O-H bending [46]. 1359 cm⁻¹ indicating metal-oxygen bonding of (Bi-O) of the strong peak of S=O stretching, 1264 cm⁻¹ and 1104 cm⁻¹ strong peak of C-F stretching, and 832 cm⁻¹ was mainly due to the Bi-O-Bi vibration and 592 cm⁻¹ created are due to the stretched Bi-O vibrations which shows that metal ions were coordinating in the nanoparticles [47].



Figure 3. FTIR-transmittance spectrum of α -BONPs.

3.3. DRS Studies

The optical properties of crystalline and nanostructured α -BONPs were investigated in various spectral regions between 200 and 800 nm using a UV-Vis spectrometer. The spectrometer beam was positioned towards the specimen's surface in a way that the light can be reflected, dispersed, and transmitted through the material to be analyzed, which determines its optical properties. The optical properties of the α -BONPs obtained through UV-Vis DRS are shown in Figure 4a.



Figure 4. (a) UV-Vis Diffuse reflectance spectrum (b) energy band gap of synthesized α -BONPs.

The high amount of light diffusion and high thickness of the powder sample make it difficult to understand the absorption spectra. Applying the Schuster—Kubelka—Munk (SKM) equation to establish a connection with the reflectance spectra, is employed to get the absorption coefficient [48]:

$$F(R) = \frac{(1-R)^2}{2R}$$
(2)

where F(R) indicates the Kubelka—Munk function, the absolute reflectance of the sample was denoted by R and hv represents the light energy.

$$F(R)h\upsilon = A(h\upsilon - Eg)$$
(3)

'A' refers to the absorption coefficient which is given by $4\pi k/\lambda$ (k is the absorption index or absorbance).

It is evident that as the wavelengths were increased, the samples' diffused reflectance also increased. The excitation of electrons from the valence band to the conduction band, which is represented in Tauc relations in Equation (2), is the cause of the optical band gap in the sample. A direct permitted transition results from n = 2, but an indirect allowed transition results from n = 1/2 [49]. The linear portion of the curve was extrapolated to demonstrate that the energy band gap Eg for α -BONPs is (3.07 ev) in Figure 4b, which suits photocatalytic processes that involve irradiation. The electronic band structure influences the sensitivity of a material by utilizing different analytes in sensor applications. A specific band gap can affect the material's responsiveness to certain types of molecules such as glucose and uric acid in an acidic medium [50].

3.4. SEM Analysis

Figure 5a displays SEM micrographs of α -BONPs. The morphological information of NPs reveals non-spherical shapes, porous and agglomerated particles, together with significant voids, which is evidence that the synthesis method employed for the preparation

of nanoparticles was the solution combustion method [33]. The existence of multiple elemental compositions, such as bismuth and oxygen, as shown by SEM EDAX in Figure 5b, serves as evidence for the purity of prepared α -BONPs.



Figure 5. (a) SEM and (b) EDAX micrographs of α -BONPs.

3.5. TEM Analysis

The α -BONPs that have large agglomeration and are close to spherical nanocrystals (50 nm) which are confirmed by the TEM images and the same is presented in Figure 6a is evidence for the nanostructure formation of crystalline size range (30-60 nm). The agglomeration is observed for a variety of reasons, including the process of preparing samples for TEM, which requires thinning the specimen to allow electrons to pass through. Particles or components in the sample may cluster together during this process. There are various attraction interactions between particles that cause them to be near together. Particles can stick together due to surface forces. These forces can cause agglomeration, especially in nanoscale materials with more strong surface effects. And the crystallite boundary is clearly discernible from the HRTEM image Figure 6b picture shows a series of crystal gaps spaced (d) the d = 0.43 nm apart, revealing the surface of the densely packed (102) flat crystal surface of α -BONPs [40]. The selective area electron diffraction (SAED) pattern shown in Figure 6c for the synthesized α -BONPs represents the distant rings of different planes that conform polycrystalline nature of α -Bi₂O₃ [51]. Overall, these characteristics provide valuable insights into the nanostructure formation, crystalline size range (30–60 nm), and crystallographic features of the synthesized α -BONPs.



Figure 6. (a) TEM images (b) HR-TEM and (c) SEAD ring pattern of α-BONPs.

3.6. Photocatalytic Studies

The DG and FOR dye degradation experiment was conducted separately in the sunlight, and it was measured by UV absorption spectroscopy. In the procedure, round glass reactors with a surface dimension of 176.6 cm² were filled with 20 ppm of a 250 mL aqueous solution of the DG and FOR dye and 20 mg of photocatalysts. The experiment took place in a sunny environment with continual stirring using a magnetic stirrer around the hours of 11 a.m. to 2 p.m. During these periods, the variations in sunlight intensity were negligible, and no discernible alterations in the sunlight could be observed in the month of May. To prevent errors brought on by variations in sun intensity, the trials were run concurrently. The evaluation was done to determine the absorbance of collecting dye solutions every 15 min up to 120 min of analysis. We use the same experimental procedure as mentioned above when exposing samples to UV light by keeping the distance between the filament and the sample to 15 cm⁻¹ at standard pressure and temperature.

Figure 7a,b shows the absorbance spectrum of DG dye when exposed to sunlight and UV light with a maximum band at 618 and 619 nm. Figure 8a,b is the absorbance spectrum of FOR dye exposed to sunlight and UV light with maximum absorbance band at 503 and 507 nm. The spectra reveal that the intensity of the absorbance spectra reaches its maximum at 0 min and continues to decrease over time [52].



Figure 7. The UV-Vis absorption spectra DG dye (**a**) under sunlight and (**b**) under UV light irradiation using α -BONPs photocatalyst.



Figure 8. UV-Vis absorption spectra of FOR dye (**a**) under sunlight and (**b**) under UV light irradiation using α -BONPs photocatalyst.

Figure 9a,c and Figure 10 illustrate the decay rate of DG and FOR dyes over time when exposed to both sunshine and UV rays. Significantly, DG dye has a half-life of 54.2 min and

76.12 min when exposed to sunlight and UV light, respectively. In comparison, FOR dye has half-lives of 25.87 min and 48.18 min under the same conditions. Figure 8b,d illustrates the percentage deterioration of DG dye and FOR dye concentration after 120 min of exposure to sunlight and UV light. The results show that DG dye experienced a degradation of 66.08% and 88.02%, while FOR dye concentration degraded by 67.95% and 94.20%. Table 1 highlights that the enhanced photo-degradation observed under UV light, as opposed to sunshine, is mainly due to partial size differences and the specific synthesis technique used [53,54]. The subtle variations in degradation performance emphasize the susceptibility of α -Bismuth Oxide nanoparticles (α -BONPs) to different light sources and emphasize the necessity for a thorough examination of the diverse results reported under sunlight and UV light settings.

Table 1. The rate constant (k) and percentage degradation for DG and FOR under sunlight and UV light.

	Rate Constant (k) min $^{-1}$	% Degradation
DG (sunlight)	0.00849	66.08
DG (UV light)	0.02102	88.02
FOR (sunlight)	0.01067	67.95
FOR (UV light)	0.02562	94.20



Figure 9. (**a**,**c**) Half-life of DG dye under sunlight and UV light. (**b**,**d**) Percentage degradation of DG dye under sunlight and UV light using α -BONPs photocatalyst.



Figure 10. (left) Half-life of FOR dye under sunlight and UV light. (right) Percentage degradation of FOR dye under sunlight and UV light.

Rate kinetics are a crucial factor in degradation investigations. The best suitable kinetic model for photocatalytic degradation was pseudo-first-order kinetic model, hence it was used to assess the rate constant of DG and FOR [55]:

$$ln\left(\frac{c}{c_0}\right) = -kt\tag{4}$$

c and c_0 are concentrations of DG and FOR dye at 0 min, and k is pseudo-first-order kinetic. The higher the degradation, the higher the rate constant which is shown in Table 1.

Under UV and sunlight, the concentration of dyes has played a significant impact on photocatalytic degradation. For the dye optimization, the dye concentration was varied from 10 to 30 ppm. Consequently, with an increase in dye concentration, there is a reduction in the number of dye molecules binding to the surface of the photocatalyst the obtained results are depicted in Figure 11a–d. The dye concentration at 20 ppm demonstrates improved photodecolorization activities [56,57].



Figure 11. Estimation of percentage of dye degradation in different ppm dye concentrations with α -BONPs as a photocatalyst treated under Sunlight and UV light irradiation of DG dye (*a*,*b*) and FOR dye (*c*,*d*).

The dye decolorization mechanism examines DG and FOR dyes with exposure to sunlight and UV light as follows and the same is shown in Figure 12. Photosensitization of the DG and FOR dye is demonstrated by the effect of sunlight and UV light on the synthesized catalyst surface. When light energy strikes a dye and catalyst surface, an electron and hole pair are produced, as presented in Equation (5). The number of holes that remain in the valence band is equal to the number of electrons that get excited to the conduction band. Oxygen free radicals, also known as superoxide radicals, are produced when an electron is in the conduction band due to adsorbed oxygen, the same is presented in Equation (6). The left-out holes in the valence band play a crucial role in the breakdown of dyes; these holes interact with water molecules to create hydroxyl radicals, presented in Equation (7). These superoxide radicals and the hydroxyl radical degraded the dyes which is given in Equation (8) [58,59].

$$\alpha Bi_2 O_3 + (h\gamma) \to (h^+ + e^-) \tag{5}$$

$$O_2 + e^- \to O^{2 \bullet -}$$
 (Superoxide radical) (6)

$$OH + h^+ \to OH^{\bullet-}(\text{Hydroxyl radical})$$
 (7)

$$(O^{2\bullet-} \text{ or } OH^{\bullet-}) + \alpha - Bi_2O_3 \to \text{degraded water}$$
 (8)



Figure 12. Mechanism of DG dye and FOR dye decolorization in UV light and sunlight.

The catalyst (α -BONPs) is checked for reusability and stability under light illumination along with degrading the dye. The DG and F-OR dyes concentration of 20 ppm is subjected to 5 cycles run in sunlight and UV light irradiation under the same condition. After the first cycle, the remaining catalyst is recovered and washed with double distilled water, heated for 50°C for 30 min then reused for the next cycle. The synthesized α -Bi₂O₃ nanoparticles show a high photostable nature retaining the same degradation efficacy up to the three consecutive cycles after a negligible change in percentage degradation of less than 5% in all four conditions, as shown in Figure 13a–d [40,60,61].



Figure 13. Reusability of α -BONPs photocatalyst (**a**) DG dye under sunlight. (**b**) DG dye under UV light. (**c**) FOR dye under sunlight, and (**d**) FOR dye under UV light.

3.7. Electrochemical Analysis

Figure 14a displays the Bi_2O_3 nanoparticles' CV curve. It is important to note that the measured capacitance in the CV curve does not resemble an electrical double-layer capacitance in the traditional rectangular form [62]. Rather, a distinct pattern of action suggested by a redox process is indicated by the curve. An anodic peak appears on the CV curve, signifying the oxidation of Bi_2O_3 nanoparticles into Bi^{3+} ions. On the other hand, the reduction of Bi^{3+} ions back into Bi_2O_3 nanoparticles is represented by the cathodic peak. The fact that a quasi-reversible electron transfer process has been seen suggests that there is some degree of reversibility to the electron transfer reaction in question. Put differently, whereas the transfer of electrons during oxidation and reduction reaction is not totally irreversible, it is also not totally reversible [63]. A CV measurement records the current response that occurs when the applied voltage is changed cyclically. The structure and properties of the CV curve reveal important details about the material's electrochemical redox activity. The measurements of CV offer significant understanding of the electrodes' redox reaction, charge-discharge behavior, and charge transfer efficiency.



Figure 14. (a) Cyclic voltammetry of Bi_2O_3 electrode at variable scan rates V/s Ag/AgCl electrode. (b) Nyquist plot & Equivalent circuit (inset) of the Nyquist plot of an impedance measurement of Bi_2O_3 electrode. (c) A plot of Peak current v/s square root of Scan rate (d) Bode diagram of Bi_2O_3 electrode.

The direction and spontaneity of redox reactions in electrochemical cells are mostly determined by the potential difference between the oxidation and reduction potentials $(E_O - E_R)$. Electrons move from the lower oxidation potential species to the bigger reduction potential species when the potential difference is positive $(E_O > E_R)$, indicating that oxidation is more likely to occur in this reaction. On the other hand, if the potential difference is negative $(E_O < E_R)$, electrons flow from species with a higher oxidation potential to those with a lower reduction potential, making the reaction more favorable for reduction. A larger degree of reversibility is suggested by a smaller difference [64] and it also confirms that it is a photocatalytically active material.

It is clear from Figure 14a that the sample obtained by the combustion approach has a larger difference across both oxidation and reduction peaks when it comes to Bi_2O_3

electrodes. The difference between E_O and E_R was calculated, and the result was -0.317 V, respectively. The evolution of oxygen during the oxidation process gets simpler when the gap between E_O and E_R is less. As a result, the electrode's electrochemical reaction is more reversible, the active material is better utilized, and charge efficiency is raised [65].

An Ag/AgCl electrode was used to measure the stable condition of electrochemical impedance measurements within the frequency ranging from 1 Hz to 1 MHz having 5 mV AC amplitude. Nyquist plots of Bi_2O_3 electrodes are shown in Figure 14b.

In an electrochemical system, an electrode's total impedance (Z) can be written as follows:

$$Z(w) = Z' + jZ'' = ZRe + jZIm = R + jX$$
(9)

$$\mathbf{j} = \sqrt{-1} \tag{10}$$

The real and imaginary components of the impedance were denoted as Z' or ZRe and ZC' or ZIm, respectively [65].

The Bi_2O_3 electrode's impedance spectra show higher impedance values, which denote more resistance to the flow of current. This higher impedance is caused, among other things, by the Bi_2O_3 layer and the electrochemical processes occurring at the electrode-electrolyte interface.

The presence of a capacitive element in the system is indicated by the observation of a depressed semicircle in the high-frequency portion of the EIS spectrum. A more realistic description of the electrochemical system can be produced by adding the constant phase element (Q_1) to the equivalent circuit. This will enable a better fit of the experimental data collected via methods like electrochemical impedance spectroscopy (EIS).

$$Z_{CPE} = \frac{1}{Y(j\omega)^n} \tag{11}$$

The angular frequency (rad s⁻¹) is represented by the symbol ω in the equation, and the adjustable parameters Y and *n* are connected to the constant phase element (Q₁). In fact, the constant phase element (Q1)'s value of n can be 1, 0, or 0.5, which correspond to several electrochemical phenomena: When '*n*' is set to 1, double-layer capacitance (C_{d1}) behavior is present. If '*n*' is equal to 0, then resistance (R) is present in the system. In the case when '*n*' takes a value of 0.5, Warburg diffusion (W) behavior is present.

An equivalent circuit used to show the Nyquist plots obtained from impedance measurements on the α -Bi₂O₃ electrode is included in the inset of Figure 13b. The highfrequency area where the real axis and the semicircle of the Nyquist spectrum intersect is represented by the solution resistance (Rs) in this circuit, which is typically correlated with resistance at the electrode-electrolyte interface. Plotting shows semicircular patterns due to either polarization resistance (Rp) or interfacial charge transfer resistance (Rct), which are connected in parallel to the double-layer capacitance (C). Overall, the presence of a Warburg element is indicated by the observation of a straight line in the low-frequency area of the Nyquist plot, highlighting the significance of the diffusion processes investigated in the electrochemical system. The presence of a plane line in the lower frequency range of the Nyquist plot indicates that bismuth ions and electrons are dispersed into tiny pores in the outermost layer of the α - Bi₂O₃ electrode, and that this behavior has a significant influence on the electrochemical behavior of the system as a whole [66]. The electrical double layer's capacitive activity and the charge transfer process' resistive behavior interact to produce the semicircle shape. The charge transfer resistance (Rct) and semicircle diameter are correlated; larger semicircles indicate greater resistance levels. The charge-transfer resistance (Rct), leakage resistance (Rl), and constant phase element (Q) are connected in parallel within the equivalent circuit [67].

The relationship between the square root of the scan rate (v1/2) and the peak cathodic current (ip) of an α -Bi₂O₃ electrode is shown in Figure 13c. According to [67], the more linear relationship between ip and v1/2 indicates that hydrogen diffusion limits the Bi₂O₃

electrode's electrode reactivity. The value of 1.039×10^{-5} cm² s⁻¹ is the hydrogen diffusion coefficient of the α -Bi₂O₃ electrode, as calculated by sloping the fitted line in Figure 13c.

The Bode diagram in Figure 13d explains the phase angle and frequency relationship. It is evident that at -53° , the phase angle is nearly at the ideal value of -90° for a capacitor [68].

The BONPs used to detect glucose and uric acid are presented in cyclic voltammograms of Figure 15a–d. The shifting of oxidation and reduction peaks implies, indicates that the BONPs combined carbon paste electrode is a very good material to sense the concentration of glucose and uric acid ranging from 1 to 5 mM. The current response reaches the steady state within 3 s, after the injection of 1 mM glucose or uric acid it takes over 50 s of sampling time to attain a steady state. The anodic oxidation shifts from -0.2 V to -0.135 V. It was also observed that an extra oxidation peak was obtained at 0.1511 V. It was found that the cathodic reduction peak shifted from 0.815 V to 0.348 V and an extra reduction peak obtained at -0.471 V for the detection of glucose sensor shown in Figure 14a, confirms the prepared electrode is a better material for the detection of glucose. Similarly, oxidation peaks at 0.2 V are shifted to -0.185 V and the reduction peak at -0.815 V splits in to two extra peak at -0.225 V and -0.345 V during uric acid sensor shown in Figure 14b reveals that the material capability for the uric acid detection. The CV of the BONPs electrode in Figure 15c,d for glucose and uric acid sensor of 50 mV/s, demonstrating that adding glucose or uric acid to the electrolyte causes a noticeable shift in the peak locations.



Figure 15. Cyclic voltammogram of α -Bi₂O₃ electrode during detection of (**a**) Glucose and (**a**) Uric acid, concentration range 1–5 mM. Cyclic voltammogram of Bi₂O₃ electrode with and without (**c**) Glucose sensor and (**d**) Uric acid sensor.

Figure 16a,b show amperometric i-t curves for glucose and uric acid biomolecule sensing at -0.6 V to 0.2 V potential sweep. The current response of the Bi₂O₃ electrode at 0 mM concentrations of uric acid and glucose is shown in Figure 16c,d. Additionally, following repeated injections of 1 mM glucose and uric acid at 50-s sampling intervals, the

current response increased and achieved steady-state current in less than 3 s. This kind of activity clearly shows the sensor's quick amperometric response to the oxidation of glucose and uric acid. It has been discovered that the currently manufactured sensor has a sensitivity of 0.0102 amps for glucose and 0.0103 amps for uric acid [69].



Figure 16. Amperometric–t curve Bi₂O₃ electrode for the sensing of (**a**) Glucose and (**b**) Uric acid molecule. (**c**) Peak current vs. concentration of Glucose (**d**) Peak current vs. concentration of Uric acid.

Figure 16c,d shows the amperometric current responses in relation to glucose and uric acid concentration from 0 to 5 mM with fitted line (inset) with a correlation coefficient of 0.998 and 0.999, respectively. The α -Bi₂O₃ electrode sensor shows an almost linear trend with a rise of glucose and uric acid concentrations. This emphasizes even more how sensitive the modified Bi₂O₃ electrode is to the electrochemical activity of the biomolecules being investigated.

4. Conclusions

In this study, Bismuth Oxide Nanoparticles (BONPs) were synthesized using an environmentally friendly combustion process, employing Mexican Mint gel as the fuel source. Various analytical techniques were employed to characterize the synthesized nanoparticles. X-ray Diffraction (PXRD) analysis revealed a crystallite size of approximately 55 nm for the nanoparticles. This size was further corroborated by Transmission Electron Microscopy (TEM) analysis, which indicated a size of less than 55 nm. Through Diffuse Reflectance Spectroscopy (DRS) analysis, the nanoparticles' bandgap was determined to be 3.07 eV. The catalytic properties of the nanoparticles were explored through two avenues: photocatalysis and electrochemical sensing. Photocatalytic degradation experiments were conducted under both sunlight and UV light for a duration of 120 min. Significantly higher degradation rates were observed under UV light, reaching 88.2% and 94% for the DG and

FOR dyes, respectively. The nanoparticles were also assessed for their electrochemical sensing capabilities. Notably, they exhibited excellent performance in detecting glucose and uric acid in alkaline medium, as evidenced by cyclic voltammetry (CV) measurements. Particularly striking was their rapid response to glucose and uric acid at concentrations as low as 1 mM. Furthermore, the study highlighted the ease of manufacturing reliable α -Bi₂O₃ electrode materials. Based on these compelling outcomes, the synthesized nanoparticles emerge as candidates that are cost-effective and well-suited for photocatalytic and sensor applications.

Further research directions may be explored in light of this study, such as refining environmentally friendly synthesis methods for α -Bismuth Oxide nanoparticles (α -BONPs) through the examination of precursor concentrations and reaction conditions. In order to mitigate the observed agglomeration tendencies, subsequent research may investigate potential approaches such as surface modifications or the application of stabilizing agents. Further analysis focused on specific applications, such as photocatalysis and electrochemical sensing, may yield valuable knowledge regarding the enhancement of α -BONPs' functionality.

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