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**Solution Combustion- Derived Nanocrystalline Anatase TiO₂ as a Recoverable
Multifunctional Platform for Photocatalytic Water Remediation and Electrochemical
Heavy-Metal Sensing**

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Abstract

Titanium dioxide (TiO₂) nanoparticles were synthesized via the solution combustion process and explored for combined photocatalytic and electrochemical applications. The formation of crystalline tetragonal anatase TiO₂ with aggregated nanostructured morphology was confirmed by powder X-ray diffraction (PXRD), diffuse reflectance spectroscopy (DRS), Fourier-transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM) coupled with energy-dispersive X-ray spectroscopy (EDX), and transmission electron microscopy (TEM) analyzes. The material displayed an indirect optical bandgap of 3.16 eV, and the calculated conduction- and valence-band edge potentials were -0.27 and +2.89 V vs. NHE, respectively. The prepared TiO₂ obtained 85.2% degradation of Congo Red (CR) and 57.18% degradation of Fast Orange Red (FOR) during 120 min under UV irradiation. Scavenger experiments showed that hydroxyl ($\cdot\text{OH}$) and superoxide ($\text{O}_2^{\cdot-}$) radicals were the dominant reactive species responsible for dye degradation. To overcome the difficulties regarding the recovery of the nanoparticles, TiO₂ was immobilized on a lightweight floating substrate, thereby creating a floating photocatalytic platform that maintained significant degrading activity and allowed for easy physical recovery of the catalyst. The interfacial charge-transfer capabilities of TiO₂ were also proven by electrochemical characterization via cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS). A TiO₂-modified electrode displayed concentration-dependent responses to Mercury (Hg²⁺) and Lead (Pb²⁺) ions. The results illustrate the possibilities of the combustion-derived TiO₂ as a versatile platform for sustainable environmental applications, including

photocatalytic remediation, recoverable water purification, and electrochemical pollution monitoring.

Keywords: TiO₂; solution combustion synthesis; photocatalysis; buoyon; electrochemical sensing.

1. Introduction:

Water is an essential resource for sustaining life and supporting ecological, agricultural and industrial activities; however, increasing population, urbanization and industrialization have placed growing pressure on freshwater resources [1,2]. The discharge of untreated domestic and industrial effluents has further intensified water-quality deterioration. Among emerging aquatic contaminants, synthetic dyes are of particular concern because of their extensive use in textile, printing, leather, paper, and dye-processing industries. Their chemically stable aromatic structures can impart persistent coloration and resistance to conventional treatment, while even low dye concentrations can reduce light penetration, interfere with aquatic photosynthesis and adversely affect aquatic ecosystems. These challenges have stimulated the development of efficient and sustainable technologies for the remediation of dye-contaminated wastewater [3-5]. Advanced oxidation processes (AOPs), particularly heterogeneous semiconductor photocatalysis, offer an attractive approach because photoexcited charge carriers generated within a semiconductor can initiate surface redox reactions involving dissolved oxygen, water and surface hydroxyl groups, producing reactive oxygen species (ROS) such as hydroxyl ($\cdot\text{OH}$) and superoxide ($\text{O}_2^{\cdot-}$) radicals. The photocatalytic performance is governed by interconnected structural and interfacial parameters, including crystal phase, crystallinity, particle size, morphology, surface characteristics, defects, light absorption and charge-carrier recombination [6–11].

Among semiconductor photocatalysts, TiO₂ has attracted considerable interest because of its chemical stability, resistance to photocorrosion, relatively low cost, low toxicity and strong oxidative capability [12-13]. TiO₂ occurs predominantly in anatase, rutile, and brookite polymorphs, which exhibit distinct structural and electronic characteristics and consequently different photocatalytic properties. Anatase is particularly attractive for environmental

photocatalysis owing to its favorable surface characteristics and photoinduced redox activity. At the nanoscale, the increased surface-to-volume ratio can enhance the availability of active sites; nevertheless, photocatalytic performance is not determined by particle size alone and depends strongly on crystallinity, phase composition, morphology, surface defects, hydroxylation and charge-carrier dynamics [14,15]. The synthesis route is therefore critical for controlling the structural and surface properties of TiO_2 . Conventional approaches, including hydrothermal, precipitation and solvothermal methods, can involve multiple processing steps and extended reaction times [15-17]. In contrast, solution combustion synthesis (SCS) provides a rapid route for producing nanostructured metal oxides through a highly exothermic redox reaction between an oxidizing metal precursor and a suitable fuel. The rapid heat release and evolution of gaseous products can restrict crystal growth and promote the formation of fine, porous and nanocrystalline powders, with the resulting properties dependent on precursor composition, fuel chemistry, fuel-to-oxidizer ratio and post-combustion treatment [18,19].

Beyond photocatalytic remediation, the semiconducting and interfacial charge-transfer properties of TiO_2 make it attractive for electrochemical applications. Techniques such as CV and EIS provide complementary information on redox behaviour, charge transfer, ion transport and electrode–electrolyte interactions [20,21]. These characteristics are particularly relevant to environmental sensing, where toxic heavy-metal ions such as Hg^{2+} and Pb^{2+} represent important water contaminants because of their persistence, toxicity and potential for biological accumulation. Nanostructured TiO_2 can provide an electrochemically accessible surface and facilitate interactions between target ions and the electrode interface, making it a promising material for investigating the electrochemical detection of hazardous metal ions [22,23]. However, the practical deployment of suspended TiO_2 nanoparticles for wastewater treatment remains challenging because of particle aggregation and the difficulty of recovering nanoscale photocatalysts after treatment [24,25]. Immobilization of TiO_2 on a suitable support can address these limitations while retaining photocatalytic functionality. In particular, a buoyant support can maintain the photocatalyst near the water surface, facilitate exposure to irradiation and simplify catalyst retrieval, thereby offering a potentially more practical configuration for recoverable photocatalytic treatment [26].

Here, we report anatase TiO_2 nanoparticles synthesized by solution combustion synthesis and systematically investigate their structural, morphological, optical, photocatalytic and

electrochemical properties. The photocatalytic activity is evaluated using CR and FOR as model dyes under UV irradiation, while scavenger experiments are employed to identify the principal ROS involved in the degradation process. To address the catalyst-recovery limitations associated with suspended nanoparticles, the synthesized TiO_2 is further immobilized on a lightweight buoyant substrate to construct a floating photocatalytic system. The electrochemical behaviour of the synthesized TiO_2 is subsequently evaluated by CV and EIS, followed by its application toward the detection of Hg^{2+} and Pb^{2+} ions. By integrating photocatalytic degradation, catalyst immobilization and electrochemical sensing within a single TiO_2 -based platform.

2. Experimental

2.1: Materials

In this work, all the chemicals were purchased from local suppliers as indicated in the following Table 1.

Table 1. List of chemicals, specifications, and suppliers used.

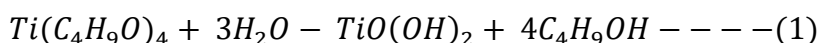
Name	Chemical Formula	Density	Suppliers
Titanium n-butoxide	$\text{C}_{16}\text{H}_{40}\text{O}_4\text{Ti}$	1.00 g/mL at 20 °C (lit.)	Sigma-Aldrich
Isopropyl alcohol (IPA)	$\text{C}_3\text{H}_8\text{O}$	0.785 g/mL at 25 °C (lit.)	Sigma-Aldrich
zNitric acid	HNO_3	1.41 g/mL at 20 °C	Sigma-Aldrich
Urea	NH_2CONH_2	1.335 g/mL at 25 °C(lit.)	Sigma-Aldrich
Epoxy Resin and Hardener	--	1.16 g/cm ³	Zenith Industries, Karnataka, India
Polystyrene (Thermocol)	$(\text{C}_8\text{H}_8)_n$	0.96–1.05 g/cm ³	Local Suppliers, Karnataka, India

2.2: Sol–gel combustion synthesis:

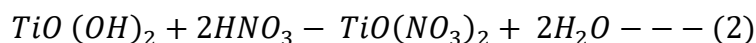
The synthesis of TiO_2 nanoparticles was carried out through a three-step sol–gel combustion process. The first step involved the preparation of titanium oxyhydroxide, the second step

involved its conversion into titanium oxy-nitrate, and the third step involved the combustion of titanium oxy-nitrate with urea as a fuel to obtain TiO₂ nanoparticles.

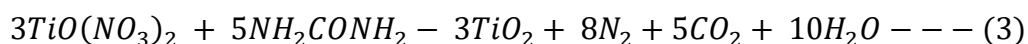
Step 1: Preparation of titanium oxyhydroxide. Titanium n-butoxide was added slowly to isopropyl alcohol (IPA) under continuous stirring to obtain a homogeneous precursor solution, designated as Solution A. Simultaneously, solution B was prepared by mixing IPA and deionized water in a volume ratio of 12:1. Solution B was then added drop wise to Solution A under vigorous and continuous stirring while maintaining the ice-cooled condition by using ice pack. The resulting mixture was stirred continuously for 1 h. During hydrolysis, the solution gradually became turbid, indicating the formation of titanium oxyhydroxide, TiO(OH)₂.



Step 2: Preparation of titanium oxy-nitrate. The titanium oxyhydroxide obtained in the first step was treated with nitric acid (HNO₃). The reaction was carried out using a 1:2 molar ratio of titanium oxyhydroxide to HNO₃. The resulting product, titanium oxy-nitrate [TiO(NO₃)₂], served as the oxidizing precursor for the subsequent combustion reaction.



Step 3: Solution combustion synthesis of TiO₂. In the third step, the required amount of urea (NH₂CONH₂) as the fuel was added to the titanium oxy-nitrate in the ratio 5:1 solution. The resulting precursor solution was heated at 100 °C to evaporate the solvent. As evaporation proceeded, the liquid precursor gradually transformed into a viscous mass. The crucible containing the viscous precursor was transferred in to the preheated muffle furnace maintained temperature at 400 °C, where the precursor underwent rapid combustion. The combustion reaction generated a large quantity of gaseous products of N₂, CO₂ and water molecules, resulting in the formation of a highly porous, foam-like TiO₂ powder (Fig.1). The obtained product appeared as a pale-yellow, foam-like TiO₂ powder [27].



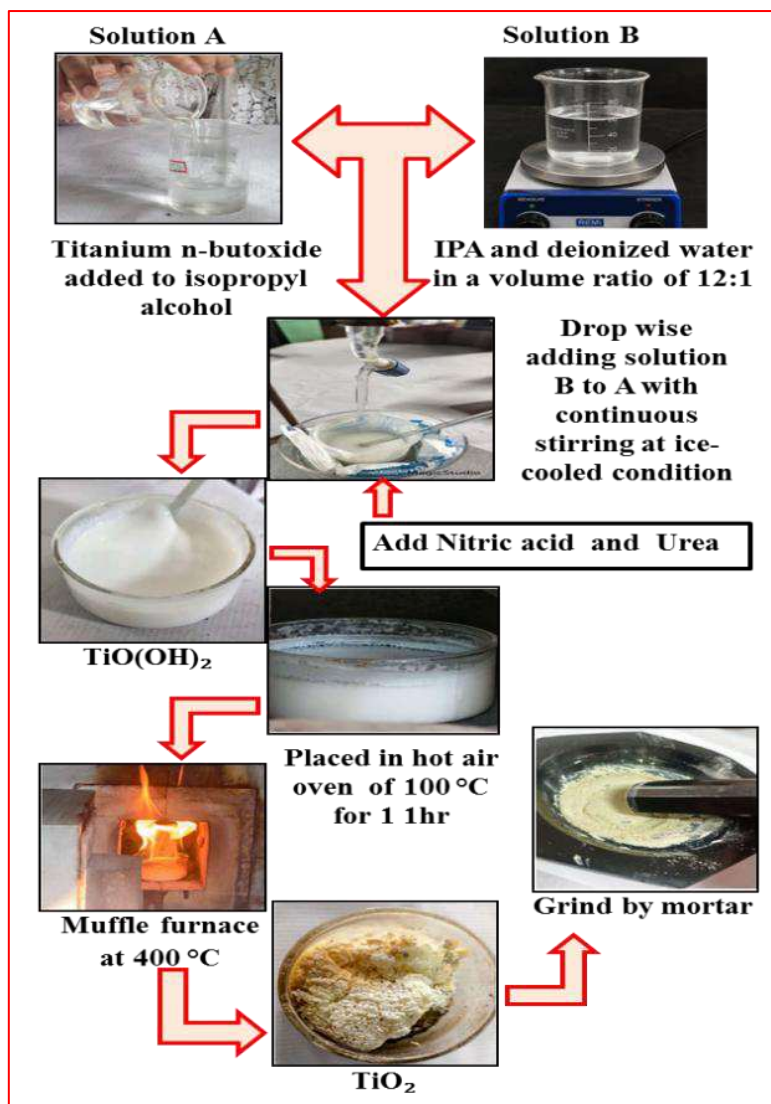


Fig. 1: Schematic diagram of the Sol-gel combustion synthesis process.

2.2: Preparation of TiO_2 -Coated buoyon.

A spherical thermocol of 5 mm diameter was used as the buoyant substrate. Twelve individual thermocol beads of the same diameter were interconnected to form a chain-like floating structure. For preparation of the photocatalytic coating, 2 mg of synthesized TiO_2 nanoparticles was dispersed in a total of 50 mL of epoxy resin-hardener mixture, prepared by mixing the epoxy resin and hardener in a 1:1 ratio. The mixture was thoroughly stirred to obtain a homogeneous TiO_2 -epoxy suspension and to ensure uniform dispersion of the nanoparticles. The thermocol bead chain was then immersed (dip-coated) in the TiO_2 -epoxy suspension, allowing the coating to uniformly adhere to the surface of the thermocol beads. The coated

buoyant chain was subsequently removed and allowed to cure under ambient conditions for 24 h (Fig. 2). After complete curing, the resulting TiO₂-coated floating buoyant system was used for the photocatalytic degradation experiments

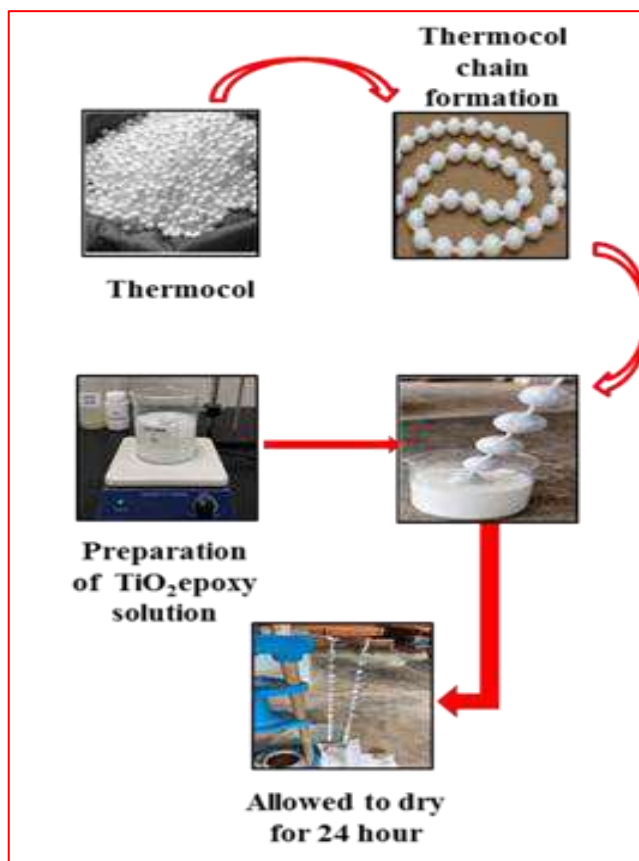


Fig. 2: Schematic diagram of the preparation of TiO₂-coated buoyon.

2.3: Preparation of carbon paste electrode

A carbon paste electrode (CPE) was prepared using graphite powder, synthesized TiO₂ nanoparticles, and silicone oil as the conducting, electroactive, binding components were taken in a (70:15:15) mass ratio and thoroughly mixed manually for 30 min to obtain a homogeneous and uniformly distributed carbon paste was obtained. The resulting TiO₂-modified carbon paste was then carefully packed into a Teflon electrode tube. Prior to each electrochemical measurement, the exposed surface of the electrode was gently polished on weighing paper until a smooth and uniform surface was obtained.[28].

2.3. Characterization

The solution combustion synthesized TiO₂ nanoparticles were investigated using the following characterization techniques:

PXRD: Shimadzu powder X-ray diffractometer by using Cu K α radiation ($\lambda = 1.541 \text{ \AA}$) and a nickel filter at an operating voltage of 50 kV and a tube current of 20 mA. The diffraction patterns were recorded at a scanning rate of 2° min^{-1} .

FTIR: Shimadzu IR Affinity-1S spectrophotometer equipped with the form of KBr pellets prior to spectral measurements range of 400 to 4000 Cm^{-1} .

SEM-EDX : Tescan Vega 3 scanning electron microscope integrated with TESCAN EssenceTM, EDS detector XFlash 7100 oval, with an active area of 100 mm².using magnetic immersion lenses, up to 30 kV.

DRS and Absorbance study in Photachatalysis: Shimadzu UV–Vis spectrophotometer (Model 2600).

TEM: JEOL JEM-2100 instrument operated at approximately 200 kV and equipped with a LaB₆ filament with spatial resolution of 2.5 $\text{\AA}$.

Electrochemical characteristics: CHI608E electrochemical workstation was conducted in 0.1 M KOH electrolyte.

3. Results and discussion

3.1. PXRD:

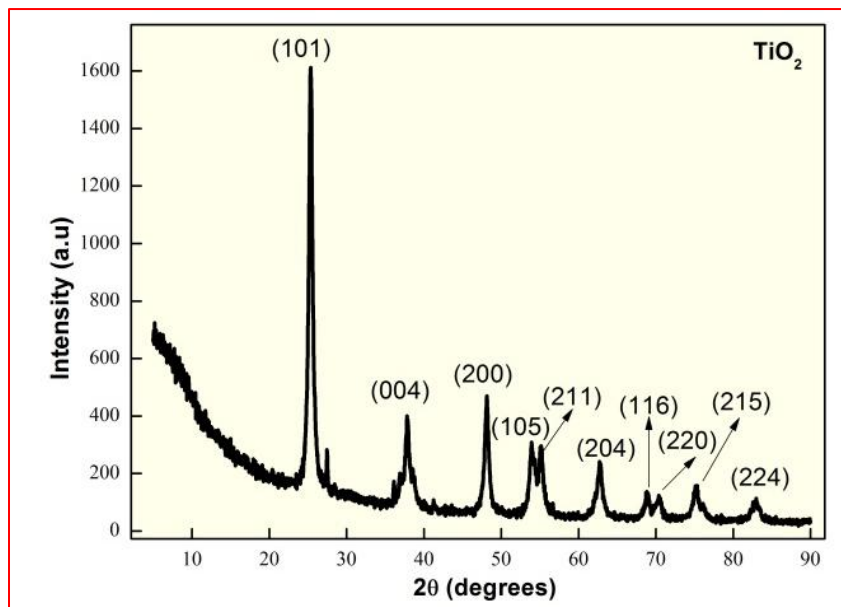


Fig. 3: XRD pattern of TiO₂ nanoparticles.

The PXRD spectra of the synthesized TiO₂ nanoparticles exhibits well-defined peaks are observed in 2θ range shown in Fig.3. The peak position are located at $2\theta = 25.332^\circ$, 37.838° , 48.108° , 53.906° , 55.076° , 62.746° , 68.960° , 70.364° and 75.096° , which can be indexed to the (101), (004), (200), (105), (211), (204), (116), (220) and (215) crystallographic planes, respectively. This peak sequence is characteristic of the tetragonal anatase polymorph of TiO₂ and agrees closely with the reported anatase reference pattern, JCPDS/PDF No. 21-1272 [29]. The most intense reflection occurs at $2\theta = 25.332^\circ$, corresponding to the anatase (101) plane, which is widely recognized as the dominant reflection of crystalline anatase TiO₂. The absence of a discernible reflection near $2\theta = 27.4^\circ$, characteristic of the rutile (110) plane, together with the absence of identifiable additional reflections attributable to brookite or other crystalline phases, indicates that the analyzed pattern is predominantly consistent with the anatase phase [30]. The experimentally determined d-spacing of the (101) peak is 0.3513 nm, which is consistent with the characteristic interplanar spacing of anatase TiO₂. The relatively broadened diffraction reflections indicate a finite coherent diffraction-domain size. The apparent crystallite size was therefore estimated using the Debye–Scherrer relationship of Eq. 4. The

calculated crystallite sizes (8.83 to 21.06 nm) yielded an average of 13.47 nm (Table 2), confirming the formation of highly crystalline, single-phase tetragonal anatase TiO₂ with no detectable secondary phases.

$$D = \frac{K\lambda}{\beta \cos \theta} \text{ --- (4)}$$

Where D is the volume-weighted apparent crystallite size, K - shape factor, λ - wavelength of $Cu K\alpha$ radiation (0.15406 nm) [5], β - full width at half maximum (FWHM) expressed in radians, and θ - Bragg angle. [31].

Table 2. Structural parameters derived from the XRD pattern of TiO₂ nanoparticles.

hkl	2 θ°	θ°	d (nm)	FWHM, β°	β (rad)	D (nm)
(101)	25.332	12.666	0.3513	0.5536	0.00966	14.71
(004)	37.838	18.919	0.2376	0.6760	0.01180	12.42
(200)	48.108	24.054	0.1890	0.6196	0.01081	14.04
(105)	53.906	26.953	0.1699	1.0090	0.01761	8.83
(211)	55.076	27.538	0.1666	0.4255	0.00743	21.06
(204)	62.746	31.373	0.1480	0.7618	0.01329	12.21
(116)	68.960	34.480	0.1361	0.8074	0.01409	11.94
(220)	70.364	35.182	0.1337	0.6435	0.01123	15.11
(215)	75.096	37.548	0.1264	0.9194	0.01605	10.90

3.2. Optical analysis:

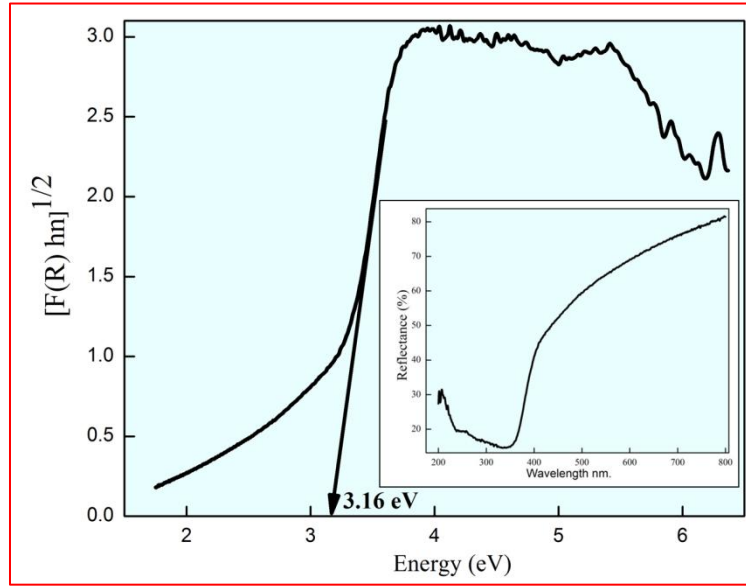


Fig. 4: Energy band gap and reflectance spectra of TiO₂ nanoparticles.

The synthesized nanoparticle is subjected to optical studies. The inset of Fig.4 reveals the reflectance spectra of TiO₂ nanoparticle, by using Schuster-Kubelka-Munk equation gives absorption coefficient given in Eq. 5, the reflectance spectra validates that the wavelengths is directly related reflectance[32].

$$F(R) = \frac{(1 - R)^2}{2R} = \frac{k}{s} \text{ --- (5)}$$

Where F(R) is the Kubelka–Munk function R is the absolute reflectance of the sample K is the absorption coefficient. S is the scattering coefficient

The band gap value is obtained from Tauc's relation given in Eq.6, by plotting the $[F(R)hv]^{\frac{1}{2}} v/s hv$, the extrapolation to the linear portion of the curve gives the band gap of 3.16 eV reveals in Fig. 4, the value of $n = 2$ this leads to indirect allowed transition [33-35].

$$(F(R)hv)^{\frac{1}{n}} = A(hv - E_g) \text{ --- (6)}$$

Where 'A'-absorption coefficient ($A = 4\pi k/\lambda$; k is the absorbance), ' hv '-photon energy and E_g - energy band gap.

The conduction-band (CB) and valence-band (VB) edge positions of the synthesized anatase TiO₂ nanoparticles were estimated using the Mulliken absolute-electronegativity approach, as described by Eqs. (7) and (8)[36]. The absolute electronegativity of TiO₂ was calculated using Eq. (9) and was found to be 5.81 eV. Considering the experimentally determined indirect optical band gap of 3.16 eV, the corresponding CB and VB edge potentials were estimated to be -0.27 V and +2.89 V vs. NHE, respectively. These calculated band-edge positions provide useful insight into the redox capabilities of the TiO₂ nanoparticles and their potential role in photocatalytic reactions.

$$E_{cb} = \chi - E_e - \frac{1}{2}Eg \text{ --- (7)}$$

$$E_{VB} = E_{CB} + Eg \text{ --- (8)}$$

$$\chi = [\chi_A^a \times \chi_B^b \times \chi_C^c]^{\frac{1}{(a+b+c)}} \text{ --- (9)}$$

Where E_{CB} is conduction-band edge potential, E_{VB} valence-band edge potential, χ is absolute electronegativity of the semiconductor, E_e energy of free electrons on the hydrogen scale and E_g is band gap.

3.3. FTIR studies:

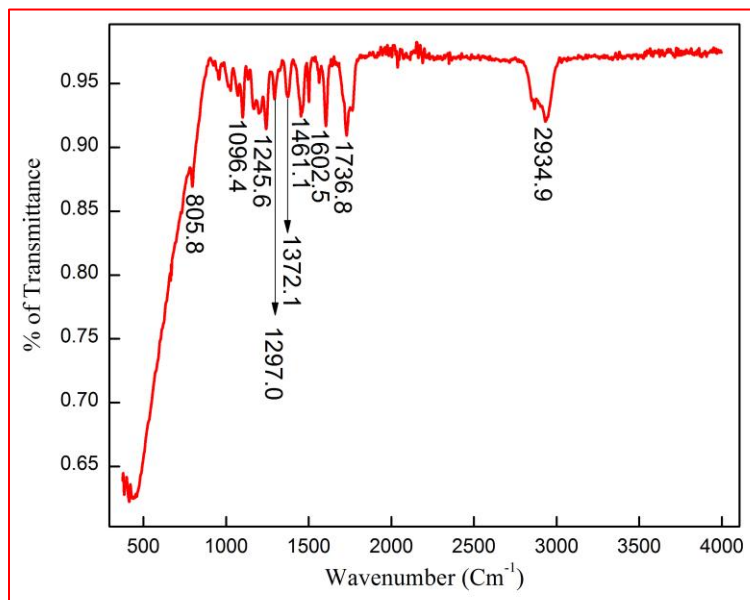


Fig. 5: FTIR analysis of TiO₂ nanoparticles.

The FTIR spectrum of the synthesized TiO₂ nanoparticles shown in Fig 5, was recorded in the range of 400–4000 cm⁻¹ to investigate the characteristic vibrational features and surface functional groups. The chemical bonds absorption bands were observed at 808.5, 1096.4, 1245.6, 1297.0, 1372.1, 1461.1, 1602.5, 1736.8, and 2934.9 cm⁻¹. The band around 2934.9 cm⁻¹ can be attributed mainly to C–H stretching vibrations associated with combustion fuel used during synthesis, whereas the 1736.8 cm⁻¹ may be related to C=O stretching of residual carbon-containing species. It is due to the incomplete burning of urea during the combustion process. The band at 1602.5 cm⁻¹ is associated with the bending vibration of adsorbed hydroxyl species (H–O–H) on the TiO₂ surface. The bands in the fingerprint region 1461.1, 1372.1, and 1297.0 cm⁻¹ arise from surface-associated functional groups and residual precursor species. The low-wavenumber region 1245.6 cm⁻¹ is associated with metal–oxygen lattice vibrations, supporting the formation of a Ti–O based oxide. The low-wavenumber region 808.5 cm⁻¹, is particularly important for TiO₂ because Ti–O and Ti–O–Ti lattice vibrations associated with the metal–oxygen framework. The FTIR observations, together with the XRD results, support the formation of crystalline TiO₂ nanoparticles [37-39].

3.4. Morphological studies

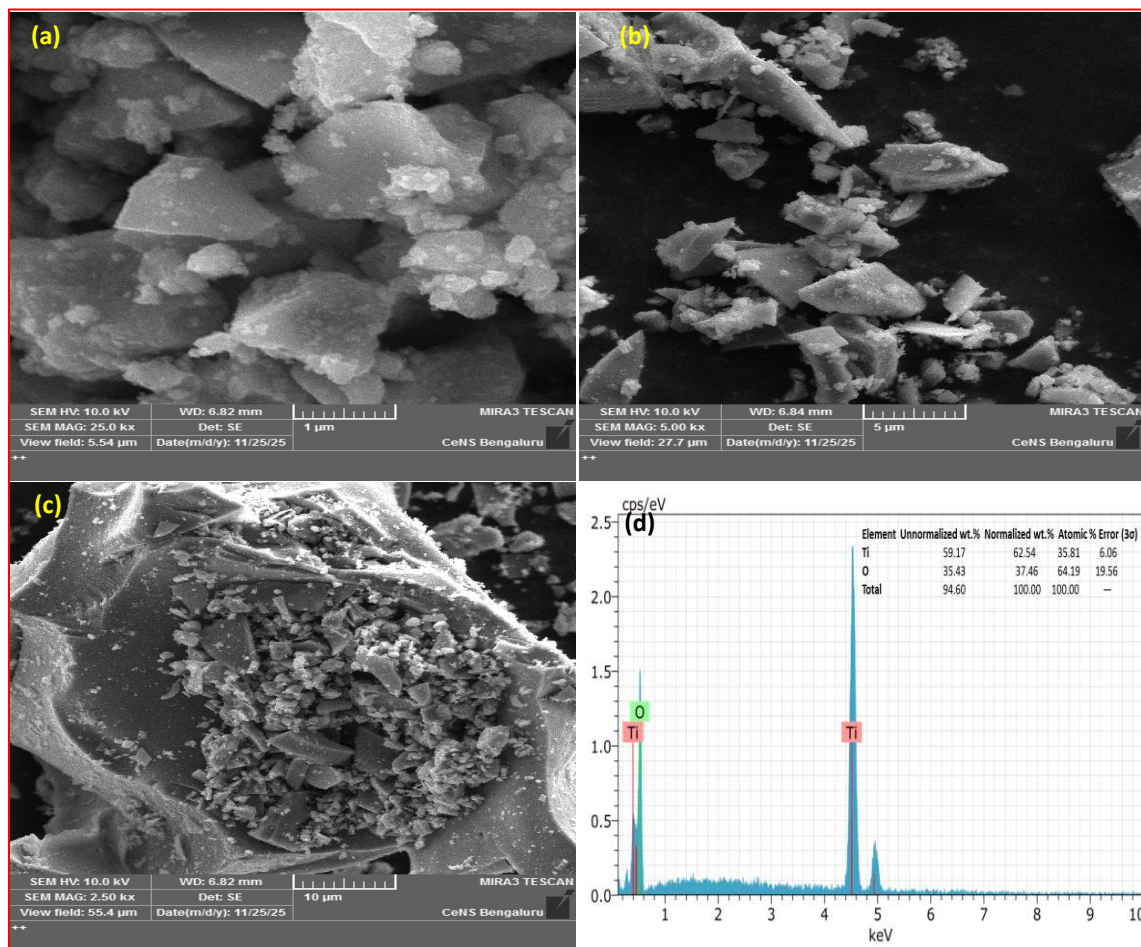


Fig. 6: (a, b and c) SEM micrographs of different magnifications and (d) SEM-EDX of TiO₂ nanoparticles.

The SEM micrographs of different magnifications are presented in Fig. 6 (a–c). The micrographs reveal a highly heterogeneous and agglomerated morphology consisting of irregularly shaped particles with rough and textured surfaces. At higher magnification of figure 5(a), smaller particulate features surround relatively large irregular particles, with secondary multi-particle structures. The lower magnification images of Fig. 6 (b,c) further demonstrate wide aggregation and the coexistence of larger aggregates with finer particulate domains. Such aggregation is observed in TiO₂ nanomaterials because of the high surface energy of nanoscale crystallites and the associated particle–particle interactions. [40,41] The rough and highly heterogeneous surface may provide a large number of interfacial sites. Similar irregular and

strongly aggregated morphologies have been reported for anatase TiO_2 particles, indicating that the sample was generated by solution combustion method [42,43]. The corresponding EDX spectrum of Fig. 6 (d) exhibits prominent signals from titanium and oxygen, with no detectable peaks attributable to other major elemental impurities within the detection limits of the measurement. The quantitative analysis weight and atomic percentage are as shown in inset of Fig. 6 (d).

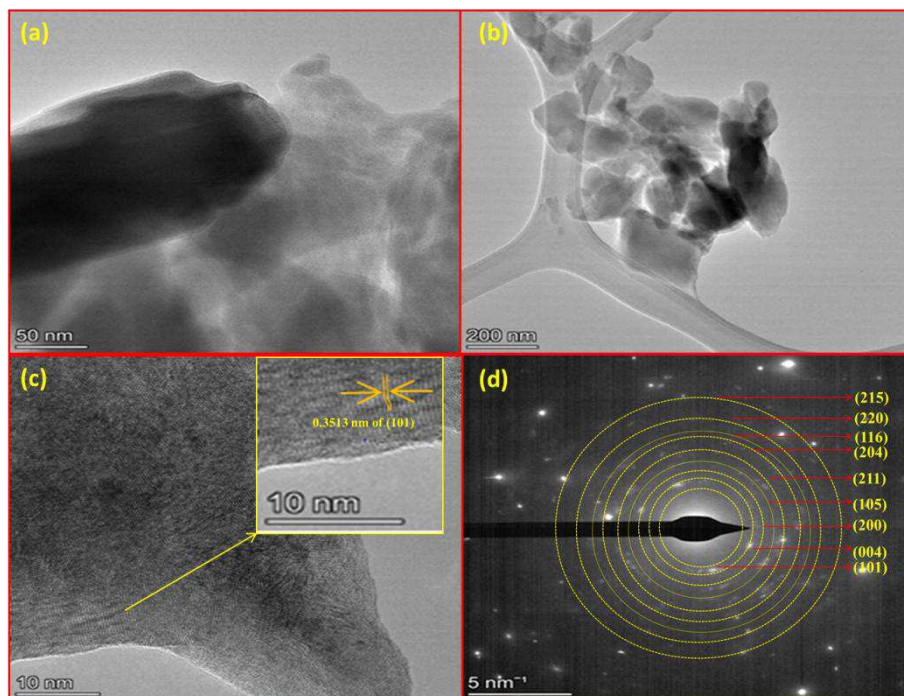


Fig. 7: (a and b) TEM (c) HRTEM (d) SAED of TiO_2 nanoparticles.

The morphology and crystalline structure of the synthesized TiO_2 nanoparticles were investigated by TEM. The TEM images of different magnification is shown in Fig. 7(a - b), reveal irregularly shaped and strongly aggregated particles with non-uniform dimensions, consistent with the agglomerated morphology observed by SEM. The aggregation is attributed to the high surface energy of nanoscale crystallites and their tendency to form secondary aggregates during synthesis processing. [44]

The HRTEM Fig. 7 (c) exhibits ordered lattice features, confirming the presence of crystalline domains within the aggregated structure. The dominant XRD peak at $2\theta = 25.332^\circ$, assigned to the anatase (101) plane, gives an interplanar spacing of 0.3513 nm is shown in inset of image 7(c) [45]. The SAED pattern of Fig. 7(d) displays concentric diffraction rings with

superimposed diffraction spots, characteristic of a polycrystalline structure with multiple crystallographic orientations [46].

4. Photocatalytic studies

The photocatalytic degradation of CR and FOR azo dyes was investigated using the synthesized TiO_2 nanoparticles as the photocatalyst. Separate aqueous solutions of CR and FOR with an initial concentration of 20 ppm were prepared using deionized water. For each experiment, 250 mL of dye solution was taken in a circular glass reactor, and 20 mg of TiO_2 nanoparticles was added as the photocatalyst. The suspension was continuously stirred using a magnetic stirrer to ensure uniform dispersion of the TiO_2 nanoparticles throughout the dye solution. Before UV irradiation, the suspension was stirred in the dark for 15 min to establish adsorption–desorption equilibrium between the dye molecules and the TiO_2 surface [47]. After the dark-equilibration period, the suspension was irradiated directly with a 125 W medium-pressure mercury-vapor UV lamp having a maximum emission wavelength of 370 nm. The distance between the UV lamp and the dye solution was maintained at 25 cm. The experiments were conducted at room temperature under normal atmospheric conditions. The photocatalytic reaction was continued for 120 min under continuous magnetic stirring. At 15 min intervals, approximately 5 mL of the reaction suspension was withdrawn. The collected samples were subjected to centrifugation to separate the TiO_2 nanoparticles from the dye solution and were subsequently analyzed using a UV–Visible spectrophotometer. The absorbance spectra were recorded in the wavelength range of 200–800 nm[48].

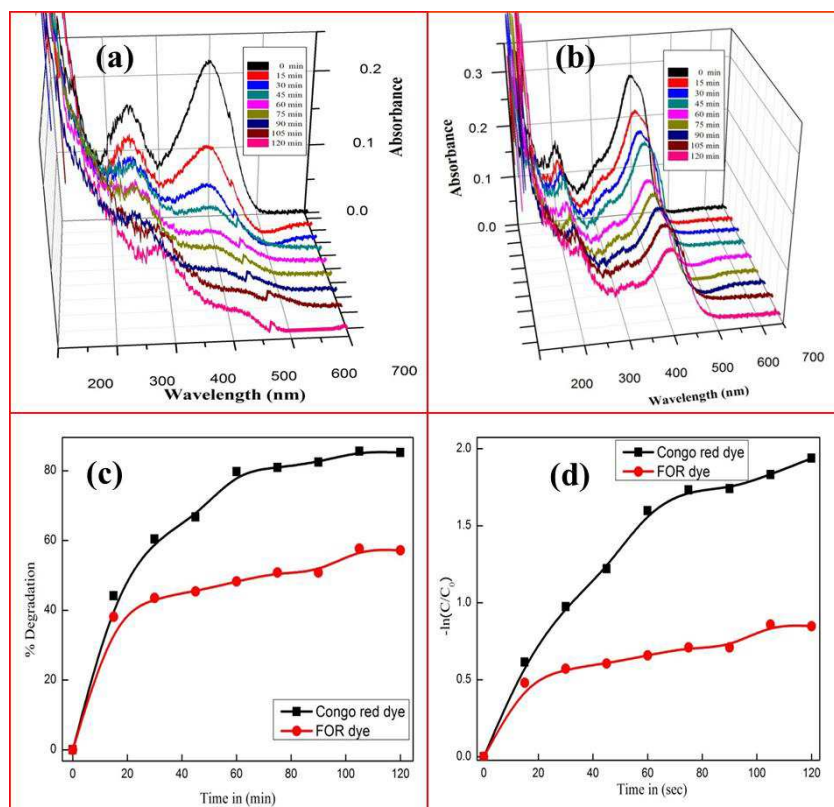


Fig. 8: (a and b) Absorbance spectra of Congo red and Fast Orange Red c) Percentage degradation of dyes d) First order kinematics studys.

The photocatalytic activity of the synthesized TiO_2 nanoparticles was examined using CR and FOR as model anionic azo dyes under UV irradiation. Fig. 8(a - b) show the time-dependent UV-Visible absorption spectra of CR and FOR, respectively. A progressive decrease in the characteristic absorption intensity was observed with increasing irradiation time, confirming the photocatalytic degradation of both dyes. As shown in Fig. 8 (c), CR exhibited degradation efficiencies of 44.0, 60.2, 66.8, 79.2, 80.5, 82.0, 84.8, and 85.2% after 15, 30, 45, 60, 75, 90, 105, and 120 min, respectively. In comparison, FOR showed degradation efficiencies of 38.0, 43.0, 45.0, 48.0, 50.0, 50.0, 57.0, and 57.18% at the corresponding irradiation times. After 120 min, the dye concentration is proportional to its absorbance under the experimental conditions the degradation efficiency was calculated using Eq.10, the maximum degradation efficiencies were 85.2% for CR and 57.18% for FOR. The higher degradation of CR indicates that the synthesized TiO_2 exhibits comparatively better photocatalytic activity toward CR under the employed experimental conditions. The percentage degradation of azo dye by different method

of synthesized TiO₂ is tabulated in Table 3. The degradation kinetics was analyzed using the pseudo-first-order model represent in Eq. 11, and the corresponding kinetic plots are presented in Fig. 8 (d).

$$\text{Degradation (\%)} = \frac{C_0 - C_t}{C_0} \times 100 \text{ --- (10)}$$

$$\ln\left(\frac{C_0}{C_t}\right) = kt \text{ --- (11)}$$

where C_0 is the initial dye concentration and C_t is the dye concentration after irradiation time t . k is first-order rate constant[49-51].

Table 3. Comparison of TiO₂-based photocatalysts for dye degradation.

Parameter	Present work: TiO ₂ nanoparticles	TiO ₂ nanostructure s	TiO ₂ (P25/comm ercial)	TiO ₂ -based composite	TiO ₂ -based modified photocatalyst
TiO₂ synthesis method	Nanoparticle synthesis; anatase phase; subsequently immobilized on floating buoyant support	Hydrotherma l synthesis	Commertia l Degussa P25	Hydrothermal /heterostructur e synthesis	Ultrasonicati on-assisted TiO ₂ synthesis
Light used	UV irradiation	UV irradiation	UV irradiation	UV/visible irradiation	UV irradiation
Dye molecules	Congo Red (CR) and Fast Orange Red (FOR)	CR	CR	CR	CR
% degradation	CR: 85.2% (120 min); FOR: 57.18% (120 min); floating TiO ₂ : CR	CR: 100% (30 min)	Used as reference catalyst	CR: 97% (120 min)	CR: 98.28% (120 min)

	84.9% (120 min)				
References	Present work	[52]	[53]	[54]	[55, 56]

4.1: Scavenger's studies:

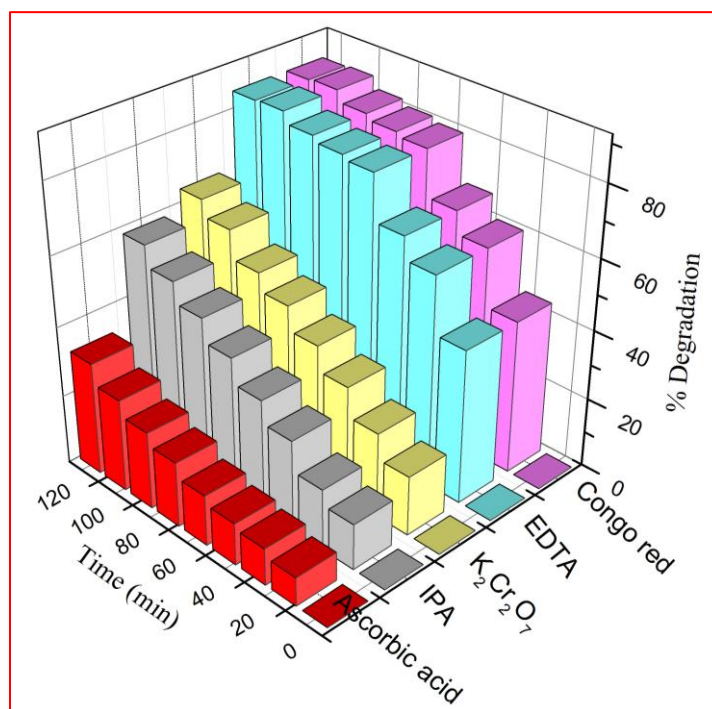


Fig. 9: scavengers for CR Degradation

To explore the contribution of different reactive species to the photocatalytic degradation of CR, a scavenger study was carried out using ascorbic acid, isopropanol (IPA), potassium dichromate (K₂Cr₂O₇), and EDTA. The corresponding degradation results are presented in Fig. 9. Ascorbic acid was employed as a scavenger for superoxide radicals ($O_2^{\cdot-}$), while isopropanol (IPA) was used to quench hydroxyl radicals ($\cdot OH$). K₂Cr₂O₇ was used to investigate the involvement of photogenerated electrons, whereas EDTA was used as a hole (h^+) scavenger [57]. The addition of ascorbic acid and IPA significantly reduced the degradation efficiency of CR compared with

the scavenger experiment it shows 32.4%. The strong suppression of degradation in the presence of ascorbic acid indicates an important contribution from superoxide radicals ($O_2^{\cdot-}$), while the decrease observed with IPA of 58.5% confirms the involvement of hydroxyl radicals ($\cdot OH$) in the degradation process [58]. The addition of $K_2Cr_2O_7$ produced comparatively reduction in the degradation efficiency (64.3%) and EDTA no changes (85.2%). This suggests that the direct participation of photogenerated electrons and holes is less dominant than the reactions involving the generated reactive oxygen species under the present experimental conditions. Therefore, the scavenger study indicates that $O_2^{\cdot-}$ and $\cdot OH$ are the major reactive species responsible for the photocatalytic degradation of CR over the synthesized TiO_2 nanoparticles [59].

4.2. Photocatalytic mechanism

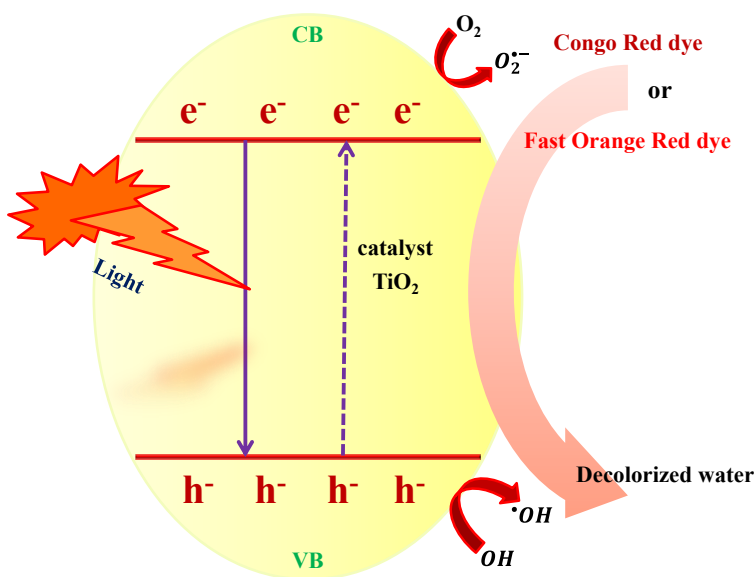
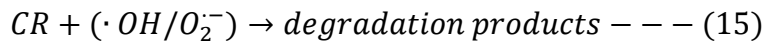
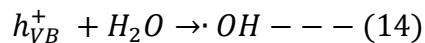
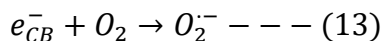
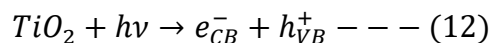


Fig. 10: Mechanism of dye degradation

The proposed photocatalytic degradation mechanism for CR and FOR dyes over the synthesized anatase TiO_2 nanoparticles under UV irradiation is illustrated in Fig.10. Subsequently both dyes are treated using the same TiO_2 photocatalyst and UV source, the fundamental photocatalytic mechanism is similar for both dyes. When TiO_2 is irradiated with UV light having photon

energy equal to or higher than its band gap of 3.16 eV, electrons in the valence band (VB) are excited to the conduction band (CB). The conduction- and valence-band edge potentials of the synthesized TiO₂ nanoparticles were estimated to be -0.27 and +2.89 V vs. NHE, respectively. These band-edge positions provide the redox ability of the photogenerated charge carriers [60]. The generating electron-hole pairs presented in Eq.12. The photogenerated electrons in the conduction band react with dissolved oxygen molecules to generate superoxide radical presented in Eq. 13 [61]. The photogenerated holes in the valence band can react with adsorbed water molecules at the TiO₂ surface to produce highly reactive hydroxyl radicals presented in Eq. 14 [62]. The generated superoxide $O_2^{\cdot-}$ and hydroxyl $\cdot OH$ radicals act the adsorbed CR and FOR dye molecules presented in Eq. 15. The azo bonds and other chromophoric structures of the dyes are progressively broken, producing smaller organic intermediates [63].



4.2. Photocatalytic activity on buoyon:

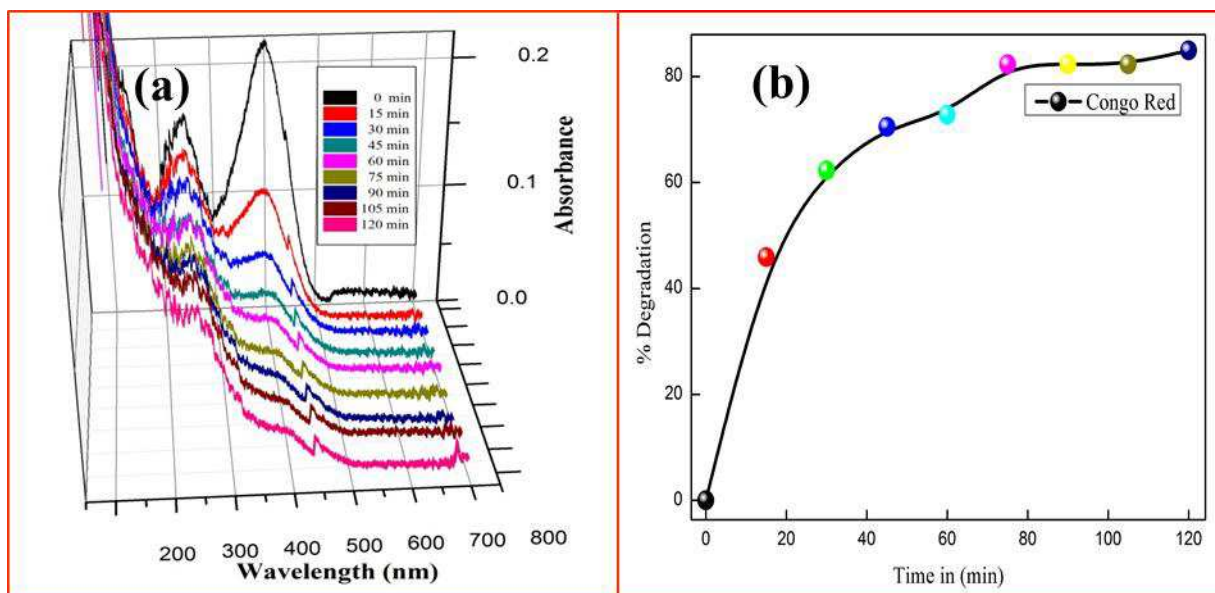


Fig. 11: a) Absorbance spectra by TiO₂-Coated buoyon of CR dye. b) Percentage of degradation.

Fig. 11a presents the photocatalytic performance of the fabricated TiO₂-coated floating buoyant system for CR dye under the same experimental conditions used for the buoyant TiO₂ photocatalyst. In this experiment, the TiO₂ nanoparticles were immobilized on the surface of the buoyant substrate using an epoxy matrix, allowing the photocatalyst to remain floating at the water surface during UV irradiation. The TiO₂ coated buoy was exposed to UV light while the dye solution was maintained under the same irradiation conditions as mentioned above. A gradual decrease in the characteristic absorbance of CR with irradiation time indicates the progressive degradation of the dyes. The photocatalytic activity of the coated buoy can be attributed to the generation of electron-hole pairs in TiO₂ under UV irradiation, followed by the formation of reactive oxygen species such as $\cdot OH$ and $O_2^{\cdot -}$, which participate in the oxidative degradation of the dye molecules[64]. The percentage of degradation of CR dye is 84.9 % is shown in Fig. 11b. This indicated that the TiO₂ coated buoyant is also a very good tool for photodegradation. The major advantage of the TiO₂-coated buoy is that it immobilizes the photocatalyst on a floating support, thereby avoiding the direct dispersion of TiO₂ nanoparticles throughout the treated water [65]. Conventional slurry photocatalysis requires the separation of

nanosized TiO₂ after treatment, which can be difficult and may result in secondary nanoparticle contamination if the catalyst is not completely recovered [66]. After completion of the treatment, the buoy can be physically removed from the water without requiring an additional filtration or centrifugation step for catalyst recovery. The floating configuration also allows the TiO₂-coated surface to remain exposed to incident UV or solar radiation, making the system potentially useful for surface-water treatment.

5. Electrochemical studies:

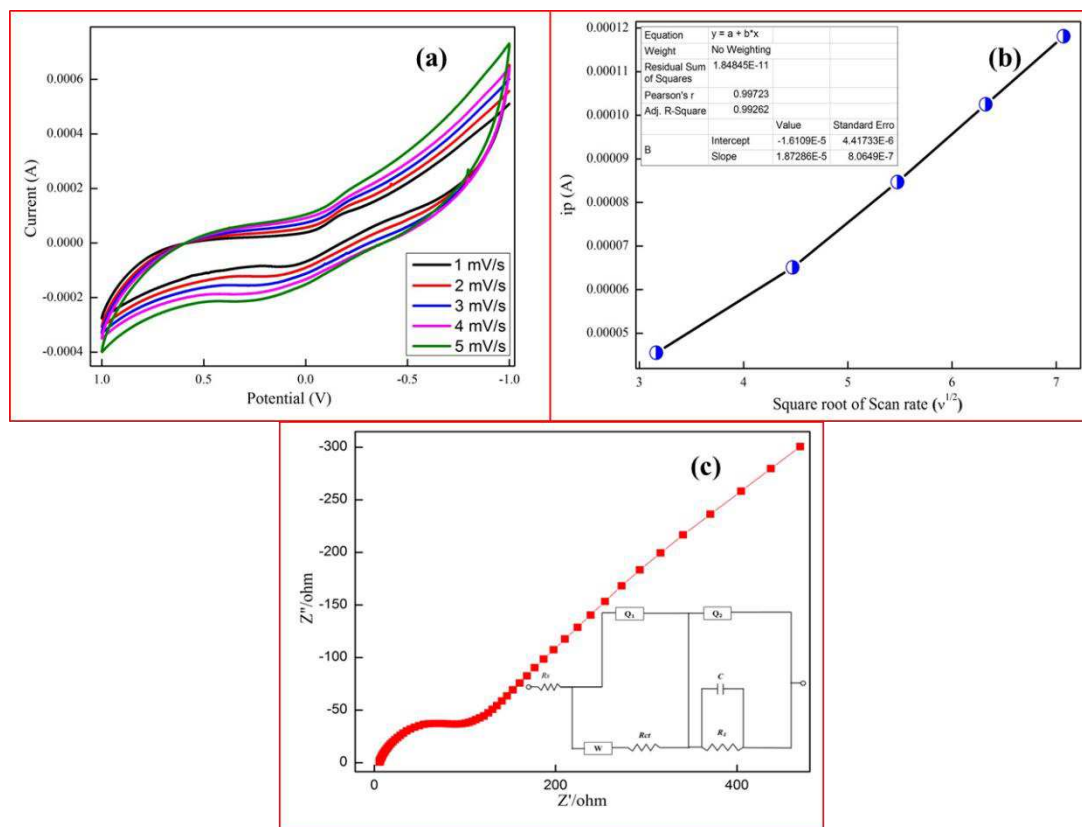


Fig.12: (a) Cyclic voltammogram at various scan rate (b) Plot of peak current (I_p) vs the square root of the scan rate ($v^{1/2}$) and (c) Nyquist plot of TiO₂ NPS in 0.1 M KOH electrolyte.

Fig. 12(a) presents the CV plots of the synthesized TiO₂ electrode recorded in 0.1 M KOH electrolyte at scan rates ranging from 10 to 50 mV/s over a potential window of +1.0 to -1.0 V. The CV curves exhibit a scan-rate-dependent current response, with the current increasing as the scan rate increases. The curves show a broad and relatively smooth current response over the

investigated potential range. This behavior suggests that the charge-storage response is predominantly associated with interface processes and electric-double-layer-type charging, rather than a highly pronounced reversible faradaic redox reaction indicates electrochemical activity at the TiO₂/electrolyte interface [67].

Furthermore, the peak current for a reversible process is described by the Randles-Sevcik Eq. 16.

$$i_p = 2.69 \times 10^5 \times n^{\frac{3}{2}} \times A \times D^{\frac{1}{2}} \times C_0 \times v^{\frac{1}{2}} \text{ --- (16)}$$

The letters n, A, D, v, and Co. represent the number of electrons transported in the reaction, electrode area, diffusion coefficient, scanning rate, and chemical starting concentration, respectively [68]. In this case, Eq. 17 can be used to get the initial concentration of the TiO₂ electrode.

$$C_0 = \frac{\rho}{M} \text{ --- (17)-}$$

Where, M -molar mass of TiO₂ and ρ - theoretical density. The scan rate ($v^{1/2}$) and cathodic peak current (i_p) is reveal in Fig. 12 b. Its linear behavior suggests that hydrogen diffusion limits the electrode response of TiO₂ [69]. The hydrogen diffusion coefficient (D) of the TiO₂ electrode was calculated using Eq. (16) and (17) and, accounting for the slope of the fitted line in Fig. 12 (b), was found to be $1.872 \times 10^{-5} \text{ cm}^2\text{s}^{-1}$.

Fig.12(c) shows the Nyquist plot obtained from EIS. The spectrum consists of a semicircular region followed by a low-frequency tail, indicating the contributions of interfacial charge-transfer and ion-diffusion processes, respectively. The semicircular feature is associated mainly with the charge-transfer resistance (R_{ct}), while the low-frequency tail is related to diffusion/transport processes in the electrode–electrolyte interface [70]. To obtain a more quantitative understanding of the impedance response, the experimental data were fitted using the equivalent electrical circuit shown in inset of Fig. 12(c). The circuit contains the series resistance R_s, constant phase elements Q₁ and Q₂, charge-transfer resistance R_{ct} Warburg impedance W, and the R1–C combination. Here, R_s represents the uncompensated solution and contact resistance, whereas R_{ct} represents the resistance associated with charge transfer across the TiO₂/electrolyte interface. The constant phase elements Q₁ and Q₂ account for the non-ideal capacitive behavior of the TiO₂ electrode surface. The Warburg element W represents the diffusion-controlled contribution at lower frequencies [71]. The R1-C component accounts for an

additional resistive–capacitive relaxation process observed in the impedance response. The fitted charge-transfer resistance was determined to be $5\ \Omega$. The relatively low R_{ct} indicates comparatively facile charge transfer at the TiO_2 /electrolyte interface, which is favorable for electrochemical charge-storage and related applications [72].

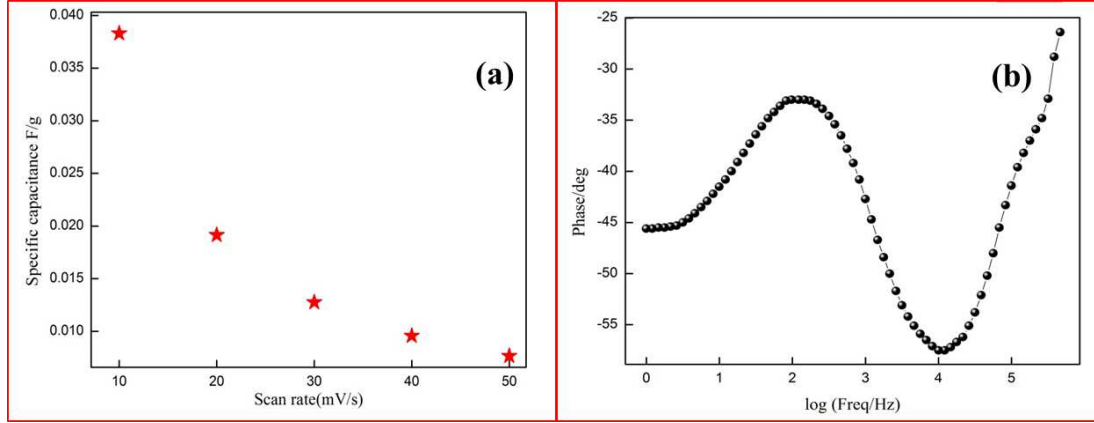


Fig.13: a) Specific capacitance v/s scan rate. b) Relation between the phase angle and frequency.

Fig. 13(a) illustrates the variation of specific capacitance with scan rate for the TiO_2 electrode. The specific capacitance decreased from $0.038\ \text{F g}^{-1}$ at $10\ \text{mV s}^{-1}$ to $0.0078\ \text{F g}^{-1}$ at $50\ \text{mV s}^{-1}$. The decrease in capacitance with increasing scan rate can be attributed to the limited time available for electrolyte ions to access the electrochemically active regions of the TiO_2 electrode. At lower scan rates, enhanced ion diffusion allows a larger fraction of the active surface to participate in charge storage, whereas at higher scan rates the electrochemical response is increasingly restricted to readily accessible surface sites [73]. Fig. 13(b) shows the frequency-dependent phase-angle response obtained from EIS. The phase angle varies from -45° to -25° , with an intermediate minimum of about -58° , demonstrating non-ideal capacitive behavior and the contribution of resistive, charge-transfer, and diffusion-related processes [74]. The deviation from ideal capacitive behavior can be associated with the TiO_2 electrode/electrolyte interface. The decrease in specific capacitance with scan rate, together with the frequency-dependent phase response, indicates that ion transport and interfacial processes play an important role in the charge-storage behavior of the synthesized TiO_2 electrode.

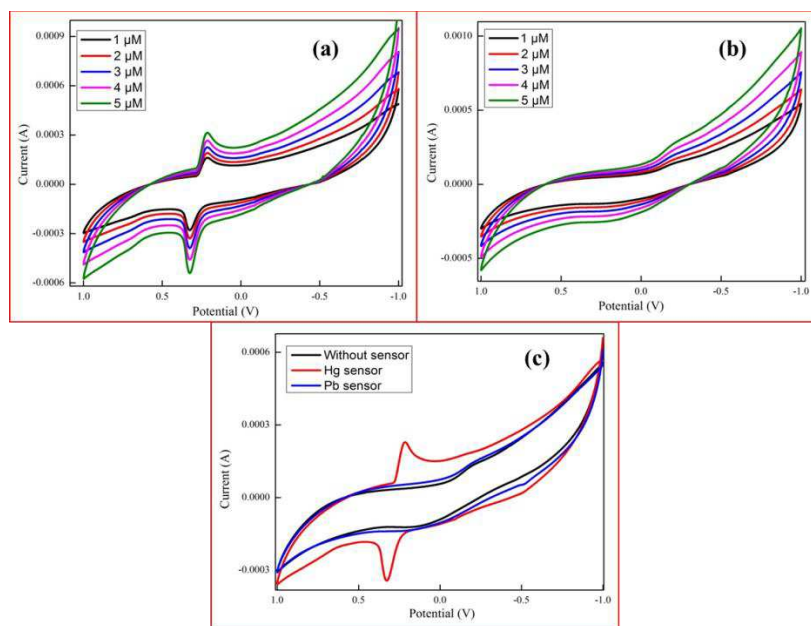
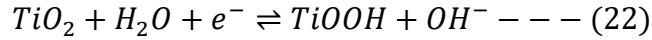
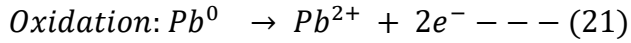
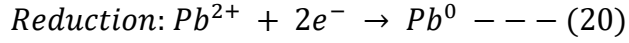
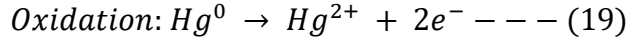
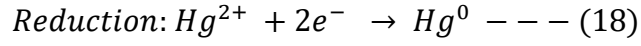


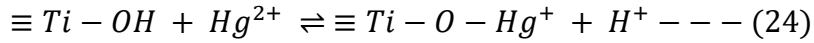
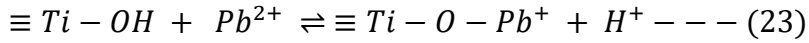
Fig. 14: (a, b) CV curves for the electrochemical detection of mercury and lead at different concentrations using the TiO₂-based sensor within the potential window of -1.0 to $+1.0$ V. c) comparison of the CV responses of the TiO₂ electrode in the absence and presence of Hg²⁺ and Pb²⁺ ions.

The electrochemical sensing performance of the synthesized TiO₂ electrode toward Hg²⁺ and Pb²⁺ ions was investigated by CV. The Fig. 14(a) illustrates the CV responses obtained for Hg²⁺ concentrations ranging from 1 to 5 μM. A progressive variation in the current response was observed with increasing Hg²⁺ concentration, indicating concentration-dependent electrochemical interaction between Hg²⁺ ions and the TiO₂ electrode surface, resulting in measurable changes in the current response is given Eq. (18 & 19). Similarly, Fig. 14(b) presents the CV responses toward Pb²⁺ at different micro molar concentrations, where systematic changes in the current response were observed upon increasing Pb²⁺ concentration mentioned in Eq. (20 & 21). Fig. 14(c) compares the CV response of the TiO₂ electrode with Hg²⁺ and Pb²⁺ ions. The introduction of the metal ions produced noticeable changes in the anodic and cathodic current responses, confirming their interaction with the TiO₂/electrolyte interface. The observed concentration-dependent changes can be attributed to the adsorption/interfacial interaction of the metal ions with active sites on TiO₂ and the associated charge-transfer processes [75]. The

reversible electrochemical reaction is given in Eq. 22, these results indicate that the synthesized TiO₂ electrode has potential for the electrochemical detection of Hg²⁺ and Pb²⁺ ions.



Role of TiO₂ electrode is mainly acts as a sensing/adsorption and electron-transfer surface. Surface hydroxyl groups can interact with metal ions, which can be represented schematically as (Eq. 20 and 21). These interactions facilitate the accumulation of Hg²⁺ and Pb²⁺ at the TiO₂-modified electrode surface. During stripping voltammetry, the accumulated metals are oxidized, producing analytical peaks proportional to their concentrations [76].



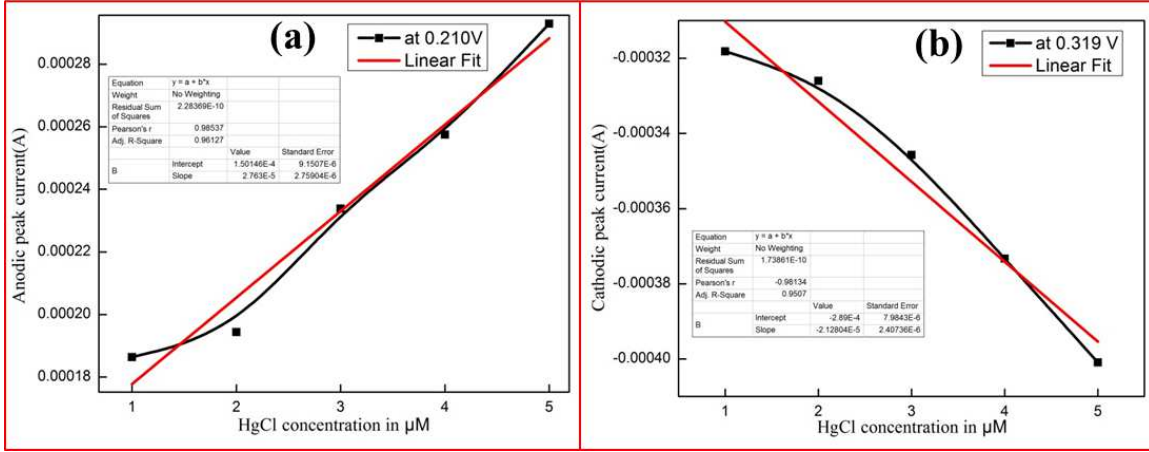


Fig. 15: (a) Anodic and (b) Cathodic peak currents with Hg^{2+} concentration in the range of 1–5 μM .

The calibration characteristics of the synthesized TiO_2 electrode toward Hg^{2+} were investigated by correlating the anodic and cathodic peak currents with Hg^{2+} concentration in the range of 1–5 μM , as shown in Fig. 15(a) and (b). The anodic peak current increased progressively with increasing Hg^{2+} concentration and exhibited a linear relationship described by $I_{pa} = 2.7635 \times 10^{-5} C + 1.50464 \times 10^{-4}$, with an R^2 value of 0.96127. The corresponding apparent sensitivity was $27.64 \mu\text{A} \mu\text{M}^{-1}$. In contrast, the cathodic peak current decreased with increasing Hg^{2+} concentration and followed the relationship $I_{pc} = -2.1280 \times 10^{-5} C - 2.89 \times 10^{-4}$ with $R^2 = 0.9507$, corresponding to an absolute sensitivity of $21.28 \mu\text{A} \mu\text{M}^{-1}$. The concentration-dependent variation in both anodic and cathodic responses confirms the electrochemical responsiveness of the TiO_2 electrode toward Hg^{2+} . Among the two responses, the anodic peak current exhibited the higher sensitivity within the investigated concentration range [77–79].

6. Conclusions

The present work successfully demonstrated the synthesis of nanocrystalline anatase TiO_2 nanoparticles through a solution combustion route. The synthesized TiO_2 exhibited an indirect band gap of 3.16 eV, with calculated CB and VB potentials of -0.27 and $+2.89$ V vs. NHE, respectively. The nanoparticles showed effective photocatalytic activity toward CR and FOR, achieving 85.2% and 57.18% degradation, respectively, after 120 min of UV irradiation. The scavenger study confirmed the important involvement of $\text{O}_2^{\cdot-}$ and $\cdot\text{OH}$ reactive species in the

degradation process. A TiO₂-coated floating buoyant system was successfully fabricated, providing a recoverable platform for photocatalytic water treatment. The electrochemical studies further demonstrated the charge-transfer and capacitive characteristics of TiO₂, while its concentration-dependent response toward Hg²⁺ and Pb²⁺ demonstrated potential for heavy-metal sensing. Overall, the results highlight the multifunctional potential of anatase TiO₂ for photocatalytic water purification, floating environmental remediation, and electrochemical sensing applications.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

References

1. Das A, Mishra S (2025) Smart and sustainable water management: policy, technology, and innovation pathways for resource resilience. *GRN TECH RES SUSTAIN* 5:5.
2. Saxena V (2025) Water Quality, Air Pollution, and Climate Change: Investigating the Environmental Impacts of Industrialization and Urbanization. *Water Air Soil Pollut* 236:73.
3. Han F, Kambala VSR, Srinivasan M, Rajarathnam D, Naidu R (2009) Tailored titanium dioxide photocatalysts for the degradation of organic dyes in wastewater treatment: a review. *Applied Catalysis A: General* 359:25-40.
4. Reza KM, Kurny ASW, Gulshan F (2017) Parameters affecting the photocatalytic degradation of dyes using TiO₂: a review. *Applied Water Science* 7:1569-1578.
5. Das A, Adak MK, Mahata N, Biswas B (2021) Wastewater treatment with the advent of TiO₂ endowed photocatalysts and their reaction kinetics with scavenger effect. *Journal of Molecular Liquids* 2021:116479.
6. Ishwarya S, Nagaswarupa HP, Naik YV et al. (2025) Multifunctional applications of barium zinc vanadate nanoparticles for photocatalytic dye degradation, energy storage and sensing applications. *Int J Miner Metall Mater* 32:3052-3071.
7. Bhoomika V, Monalisa KT, TMS, Basavaraju N, Khasim S, ShashiShekhar TR et al. (2026) Biogenic MgO/CuO-MgO nanoparticles from lemon extract for enhanced

electrochemical and photocatalytic performance. *Journal of Dispersion Science and Technology* 1-17.

8. Sagadevan et al. (2022) Photocatalytic Efficiency of Titanium Dioxide for Dyes and Heavy Metals Removal from Wastewater. *Bulletin of Chemical Reaction Engineering & Catalysis* 17.
9. A Review on Photocatalysis Used for Wastewater Treatment: Dye Degradation (2023) Journal article/review.
10. Photocatalytic activity improvement and application of UV-TiO₂ photocatalysis in textile wastewater treatment: A review (2019). *Journal of Environmental Chemical Engineering* 7:103248.
11. Environmental risks of textile dyes and photocatalytic materials for sustainable treatment: current status and future directions (2025). *Discover Environment*.
12. Nasr M, Eid C, Habchi R, Miele P, Bechelany M (2018) Recent Progress on Titanium Dioxide Nanomaterials for Photocatalytic Applications. *ChemSusChem*.
13. Ziental D, Czarczynska-Goslinska B, Mlynarczyk DT, Glowacka-Sobotta A, Stanis B, Goslinski T, Sobotta L (2020) Titanium Dioxide Nanoparticles: Prospects and Applications in Medicine. *Nanomaterials* 10:387.
14. Triebold S, Luvizotto GL, Tolosana-Delgado R et al. (2011) Discrimination of TiO₂ polymorphs in sedimentary and metamorphic rocks. *Contrib Mineral Petrol* 161:581-596.
15. Morais ME, Griz JCZ, Brackmann R, Rocha RDCD, Sikora MDS (2026) Hydrothermal tuning of anatase-brookite TiO₂ nanostructures: Influence of synthesis parameters on surface charge, crystallite size, and photocatalytic activity. *Catalysis Today* 469:115753.
16. Guo R, Bao Y, Kang Q, Liu C, Zhang W, Zhu Q (2022) Solvent-controlled synthesis and photocatalytic activity of hollow TiO₂ microspheres prepared by the solvothermal method. *Colloids and Surfaces A: Physicochemical and Engineering Aspects* 633:127931.
17. Uthiravel V, Narayanamurthi K, Raja V, Anandhabasker S, Kuppusamy K (2024) Green synthesis and characterization of TiO₂ and Ag-doped TiO₂ nanoparticles for photocatalytic and antimicrobial applications. *Inorganic Chemistry Communications* 170:113327.
18. Varma A, Mukasyan AS (2016) Solution Combustion Synthesis of Nanoscale Materials. *Chemical Reviews* 116:14493-14586.

19. Wen W, Wu JM (2014) Nanomaterials via solution combustion synthesis: a step nearer to controllability. *RSC Advances* 4:58090-58100.
20. Recent review on TiO₂-based electrochemical sensing and photocatalytic environmental applications, including Hg²⁺ and Pb²⁺ detection (2026). *Next Sustainability* 8:100430.
21. Das et al. TiO₂ wastewater review for photocatalytic mechanism, reaction kinetics and scavenger effects.
22. Ullah MA, Robel FN, Bahadur NM, Tabassum S, Bashar MS, Ahmed S, Hossain MS (2025) Organic modifier-assisted structural tuning of nano-TiO₂ for optimized electrochemical sensing of lead (Pb) in aqueous system. *Surfaces and Interfaces* 77:108009.
23. Soundarya TL, Manukumar KN, Babu N et al. (2025) Facile synthesis of heterojunction CdS-TiO₂ nanocomposites for energy storage supercapacitors, heavy metal detection, photochemical applications, and forensic science advancements: a comparative study with pure CdS and TiO₂ nanoparticles. *Environ Dev Sustain*.
24. Spasojević L, Ivanišević I, Sikirić MD (2025) Stability and application of TiO₂ nanomaterials in aqueous suspensions: a review. *RSC Adv* 15:21341-21368.
25. K A, Devarajan Y (2025) Nanomaterials-Based Wastewater Treatment: Addressing Challenges and Advancing Sustainable Solutions. *BioNanoSci* 15:149.
26. Recent advances in the development of effective TiO₂-based photocatalysts immobilized on floating substrates: A mini review (2024). *Environmental Nanotechnology, Monitoring & Management* 22:101021.
27. Chung SL, Wang CM (2011) A sol-gel combustion synthesis method for TiO₂ powders with enhanced photocatalytic activity. *J Sol-Gel Sci Technol* 57:76-85.
28. Basavaraju N, Shwetha HN, Ravikumar CR, Khasim S, Tan KB, Kelele KG, Chan KY, Murthy HCA (2025) Aloe Vera-Assisted Synthesis of Dy-Doped MgNb₂O₆ for Sustainable Sensing and Photocatalytic Applications. *Journal of Nanotechnology* 2025:6674176.
29. Shirke BS, Korake PV, Hankare PP et al. (2011) Synthesis and characterization of pure anatase TiO₂ nanoparticles. *J Mater Sci: Mater Electron* 22:821-824.
30. Ba-Abbad MM, Kadhum AAH, Mohamad AB, Takriff MS, Sopian K (2012) Synthesis and Catalytic Activity of TiO₂ Nanoparticles for Photochemical Oxidation of

Concentrated Chlorophenols under Direct Solar Radiation. *International Journal of Electrochemical Science* 7:4871-4888.

31. Klug P, Alexander LE (1954) *X-Ray Diffraction Procedure*. Wiley, New York.
32. Kubelka P, Munk F (1931) Ein beitrag zur optik der farbanstriche. *Z Tech Phys* 12:593-601.
33. Tauc J, Grigorovici R, Vancu A (1966) Optical Properties and Electronic Structure of Amorphous Germanium. *Physica Status Solidi (b)* 15:627-637.
34. Kardile AV, Sawant PV, Parekar MA et al. (2025) Eco-friendly synthesis of TiO₂ nanoparticles using citrus peel extract for enhanced photocatalytic degradation of textile dyes. *Interactions* 246:86.
35. Basavaraju N, Prashantha SC, Nagabhushana H, Chandrasekhar M, Kumar AN, Shashi Shekhar TR, Ashwini S, Anantharaju KS (2021) A benign approach for novel synthesis of Eu³⁺ doped MgNb₂O₆: Its photoluminescence and photocatalytic studies. *Ceramics International* 47:14899-14906.
36. Mulliken RS (1934) Electronic Structures of Molecules. IX. A New Electronegativity Scale, Together with Valence Board Energies and Polarity. *J Chem Phys* 2:782.
37. Liu J, Zhang F, Dou S, Zhu M, Ding L, Yang Y (2022) Adsorption of serine at the anatase TiO₂/water interface: A combined ATR-FTIR and DFT study. *Science of The Total Environment* 807:150839.
38. Sa'aya NSN, Halim NA, Tajuddin HA, Norrrahim MNF, Demon SZN, Che Ros F, Aziz S, Hamsan MH, Azmi AFM, Abidin NHZ (2025) Exploring the effective parameters on the photocatalytic activity of TiO₂ nanoparticles. *South African Journal of Chemical Engineering* 53:127-141.
39. Moges TY (2023) Effect of Temperature Sintering on Grain Growth and Optical Properties of TiO₂ Nanoparticles. *Journal of Nanomaterials* 2023:3098452.
40. Sentis MPL, Feltin N, Lambeng N et al. (2024) Investigation of nanoparticle dispersibility and stability based on TiO₂ analysis by SMLS, DLS, and SEM. *J Nanopart Res* 26:55.
41. Rachmaniar S, Nugraha DA, Santjojo DJDH et al. (2024) Prevention of particle agglomeration in sol-gel synthesis of TiO₂ nanoparticles via addition of surfactant. *J Nanopart Res* 26:45.

42. Gaber AA, Khattab RM, Sadek HEH, Hessien MA (2025) Characterization of anatase TiO_2 nanoparticle: Comparative synthesis via microwave combustion and sol-gel techniques. *Main Group Chemistry* 24:82-93.
43. Balamurugan M, Silambarasan M, Saravanan S, Soga T (2022) Synthesis of anatase and rutile mixed phase titanium dioxide nanoparticles using simple solution combustion method. *Physica B: Condensed Matter* 638:413843.
44. Alam MA, Bishwas RK, Mostofa S, Jahan SA (2024) Low-temperature synthesis and crystal growth behavior of nanocrystal anatase- TiO_2 . *Materials Letters* 354:135396.
45. Ahmed S, Shishir MKH, Islam MT, Rahaman MA, Aman S, Aidid AR, Sadia SI, Rana MM, Alam MA (2025) Crystallinity integration of anatase (TiO_2) nanocrystal by whole powder pattern fitting (WPPF) method: A Rietveld refinement study. *Results in Materials* 26:100673.
46. Swathi KS, Naik KG (2026) Calcination effect on the properties of sol-gel synthesized TiO_2 nanomaterials. *Discov Ind Chem Mater* 1:3.
47. Raghavendra N, Nagaswarupa HP, Shashi Shekhar TR, Mylarappa M, Basavaraju N, Surendra BS (2024) Electrochemical Sensor and Photocatalytic Studies of Ferrite Nanoparticle Using Different Fuels via Green Approach. *Sensing Technology* 2:1.
48. Lakshmana Naik R, Bala Narsaiah T, Justin P, Naveen Kumar A, Somashekar MN, Raghavendra N, Ravikumar CR, Khan AA, Santosh MS (2023) Hydrothermally synthesized cobalt vanadate nanoparticles for photocatalytic degradation of Fast Orange Red dye and supercapacitor applications. *Materials Science and Engineering: B* 298:116861.
49. Raghavendra N, Nagaswarupa HP, Shashi Shekhar TR, Mylarappa M, Surendra BS, Prashantha SC, Ravikumar CR, Anil Kumar MR, Basavaraju N (2021) Development of clay ferrite nanocomposite: Electrochemical, sensors and photocatalytic studies. *Applied Surface Science Advances* 5:100103.
50. Basavaraju N, Shwetha HN, Ravikumar CR, Khasim S, Tan KB, Kelele KG, Chan KY, Murthy HCA (2025) Aloe Vera-Assisted Synthesis of Dy-Doped MgNb_2O_6 for Sustainable Sensing and Photocatalytic Applications. *Journal of Nanotechnology* 2025:6674176.

51. HMD, DMT, HVH, HHPN, Naik R, NB (2024) Multifunctional applications of hybrid $\text{CaMgTi}_2\text{O}_4$ nanoparticles for electrochemical sensors and photocatalysis. *Sensing Technology* 2:1.
52. Erdemoglu S, Aksu SK, Sayılkan F, İzgi B, Asilturk M, Sayılkan H, Frimmel F, Güçer Ş (2008) Photocatalytic degradation of Congo Red by hydrothermally synthesized nanocrystalline TiO_2 and identification of degradation products by LC-MS. *J Hazard Mater* 155:469-476.
53. Harun NS (2017) Photocatalytic degradation of Congo red dye based on titanium dioxide using solar and UV lamp. Student Project, Faculty of Applied Sciences, Universiti Teknologi MARA, Pahang.
54. Gopal R, Chinnapan MM, Bojarajan AK, Rotte NK, Ponraj JS, Ganesan R, Atanas I, Nadarajah M, Manavalan RK, Gaspar J (2020) Facile synthesis and defect optimization of 2D-layered MoS_2 on TiO_2 heterostructure for industrial effluent, wastewater treatments. *Sci Rep* 10:21625.
55. Turcu E, Coromelci CG, Harabagiu V, Ignat M (2023) Enhancing the photocatalytic activity of TiO_2 for the degradation of Congo Red dye by adjusting the ultrasonication regime applied in its synthesis procedure. *Catalysts* 13:345.
56. Quintanilla-Villanueva GE, Sicardi-Segade A, Luna-Moreno D, Núñez-Salas RE, Villarreal-Chiu JF, Rodríguez-Delgado MM (2025) Recent advances in Congo Red degradation by TiO_2 -based photocatalysts under visible light. *Catalysts* 15:84.
57. Dubey V, Parikh KD, Jadav RJ et al. (2025) Eco-Friendly Fabrication of $\text{Cr}_2\text{V}_4\text{O}_{13}$ Nanoparticles: A Promising Material for Photocatalysis, Electrochemical Sensing, and Supercapacitor Applications. *J Clust Sci* 36:9.
58. Manjunatha A (2020) Synthesis of Citrus Limon mediated SnO_2 - WO_3 nanocomposite: Applications to photocatalytic activity and electrochemical sensor. *J Environ Chem Eng* 8:104500.
59. Basavaraju N, Prashantha SC, Nagabhushana H, Pratapkumar C, Ravikumar CR, Anil Kumar MR, Surendra BS, Shashi Shekhar TR, Premkumar HB, Nagaswarupa HP (2021) $\text{MgNb}_2\text{O}_6:\text{Dy}^{3+}$ nanophosphor: A facile preparation, down conversion photoluminescence and UV driven photocatalytic properties. *Ceramics International* 47:10370-10380.

60. Fujishima A, Rao TN, Tryk DA (2000) Titanium dioxide photocatalysis. *J Photochem Photobiol C: Photochem Rev* 1:1-21.
61. Nakata K, Fujishima A (2012) TiO_2 photocatalysis: Design and applications. *J Photochem Photobiol C: Photochem Rev* 13:169-189.
62. Yamakata Y, Vequizo JJM (2019) Curious behaviors of photogenerated electrons and holes at the defects on anatase, rutile, and brookite TiO_2 powders: A review. *J Photochem Photobiol C: Photochem Rev* 40:234-243.
63. Basavaraju N, Raghavendra N, Hamdalla TA, Ravikumar C, Nagaswarupa H, Shekhar TS, Surendra B, Murthy HA (2024) A Novel Synthesis of ZnNb_2O_6 Nanoparticles via Combustion Method: Supercapacitor and Photocatalytic Properties. *Part Part Syst Charact* 41:2400061.
64. Guo MY, Ng AMC, Liu F, Djurišić AB, Chan WK (2011) Photocatalytic activity of metal oxides—The role of holes and OH radicals. *Applied Catalysis B: Environmental* 107:150-157.
65. Davari N, Falletta E, Bianchi CL, Yargeau V, Boffito DC (2024) TiO_2 nanotubes immobilized on polyurethane foam as a floating photocatalyst for water treatment. *Catalysis Today* 436:114725.
66. Chung WJ, Nguyen DD, Bui XT, An SW, Banu JR, Lee SM, Kim SS, Moon DH, Jeon BH, Chang SW (2018) A magnetically separable and recyclable Ag-supported magnetic TiO_2 composite catalyst: Fabrication, characterization, and photocatalytic activity. *J Environ Manage* 213:541-548.
67. Krishnan SG, Archana PS, Vidyadharan B, Misnon II, Vijayan BLK, Nair VM, Gupta A, Jose R (2016) Modification of capacitive charge storage of TiO_2 with nickel doping. *J Alloys Compd* 684:328-334.
68. Katz E (2023) Electrochemical contributions: John Edward Brough Randles (1912-1998). *Electrochem Sci Adv* 3:e2300005.
69. Mamatha KM, Srinivasa Murthy V, Thammanna BM, Naveen Kumar T, Jahagirdar AA, Naveen Kumar A, Muniyappa M, Ravikumar CR, Ananda Murthy HC (2023) Electrochemical sensor of carboxymethyl cellulose and photocatalytic degradation of NavyBlue dye by sonochemically synthesized Titanium oxide nanoparticles. *Sensors International* 4:100239.

70. Zhu J, Wei L, Hu J, Xue C (2017) Anchoring iron oxide nanoparticles on polypyrrole/rGO derived nitrogen-doped carbon as lithium-ion battery anode. *Journal of Alloys and Compounds* 723:729-735.
71. Jorcin JB, Orazem ME, Pébère N, Tribollet B (2006) CPE analysis by local electrochemical impedance spectroscopy. *Electrochim Acta* 51:1473-1479.
72. Yadav P, Pandey K, Bhatt P, Tripathi B, Pandey MK, Kumar M (2016) Probing the electrochemical properties of TiO₂/graphene composite by cyclic voltammetry and impedance spectroscopy. *Materials Science and Engineering: B* 206:22-29.
73. Al-Hazmi MAM (2025) Tuning diffusion coefficient, ionic conductivity, and transference number in rGO/BaCoO₃ electrode material for optimized supercapacitor energy storage. *J Energy Chem*.
74. Aguiar dos Santos JP, Rufino FC, Ota JIY, Fernandes RC, Vicentini R, Pagan CJB, Da Silva LM, Zanin H (2023) Best practices for electrochemical characterization of supercapacitors. *J Energy Chem* 80:265-283.
75. Ludwig C, Schindler PW (1995) Surface complexation on TiO₂: I. Adsorption of H⁺ and Cu²⁺ ions onto TiO₂ (anatase). *J Colloid Interface Sci* 169:284-290.
76. Jovanovski V, Hrastnik NI, Hočevár SB (2015) Copper film electrode for anodic stripping voltammetric determination of trace mercury and lead. *Electrochem Commun* 57:1-4.
77. Gao F, Gao N, Nishitani A, Tanaka H (2016) Rod-like hydroxyapatite and Nafion nanocomposite as an electrochemical matrix for simultaneous and sensitive detection of Hg²⁺, Cu²⁺, Pb²⁺ and Cd²⁺. *J Electroanal Chem* 775:212-218.
78. Lai Y, Ma Y, Sun L, Jia J, Weng J, Hu N, Yang W, Zhang Q (2011) A highly selective electrochemical biosensor for Hg²⁺ using hemin as a redox indicator. *Electrochim Acta* 56:3153-3158.