

## **SMART NANOMATERIALS PARTICLES AND AI OPTIMISATION OF SOLAR-POWERED ELECTROCHEMICAL DEHYDRATION OF WATERBORNE CONTAMINANTS**

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### **ABSTRACT**

The paper examines an amalgamation of smart nanomaterial-based electrode and artificial intelligence (AI)-driven optimisation to optimise the solar-powered electrochemical dehydration of waterborne pollutants. ZnO–TiO<sub>2</sub> on GO (graphene oxide)-based novel nanocomposites were prepared through hydrothermal and sol-gel methods

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and have a high surface area (165.2 m<sup>2</sup>/g) and better electron transfer dynamics. The solar irradiation (1.0 sun, AM 1.5 G) charged integrated photoelectrochemical cell showed considerable elimination of contaminants in simulated industrial waste water comprising of phenol, bisphenol-A and heavy metal ions (Pb<sup>2+</sup> and Cd<sup>2+</sup>) waste water. Within 60 min of operation, dehydration efficiencies were found to be 91.8 and 88.3% in the case of phenol and bisphenol-A, respectively, and more than 96% removal of Pb<sup>2+</sup> and Cd<sup>2+</sup>. Optimisation via an AI genetic algorithm-neural network hybrid model lowered energy consumption by 32.4% compared to when the process was not optimised, and further the reaction efficiency was maximised by controlling the light and voltage flux in a dynamic way. FT-IR and XPS characterised the full oxidation of phenolic intermediates CO<sub>2</sub> and H<sub>2</sub>O. After 100 cycles, there was a stable performance of the system with the degradation of electrodes being insignificant (< 2.1% efficiency decrease). This finding indicates the promise of smart material-AI to realise scalable, green, and energy-efficient water purification technology, with applications to next-generation solar-electrochemical remediation systems.

*Keywords:* smart nanomaterials, electrochemical dehydration, solar-driven water treatment, artificial intelligence optimisation, photoelectrochemical remediation, waterborne contaminants.

## AIMS AND BACKGROUND

The high pace of industrialisation and densification of cities worldwide have considerably increased the emission of complex water pollutants such as phenolic compounds, dyes, drug residuals, and heavy metals into freshwater bodies. The World Health Organisation (WHO) notes that the population of individuals without access to safely managed drinking water exceeds 2.2 billion, and less than 20% of wastewater is properly treated. The traditional methods of water purification like adsorption, membrane filtration, and chlorination are usually endangered with limitations like secondary waste production, high running expense, the inefficiency to eliminate the developing agents, and so forth. This is why there has been an increased demand to establish next-generation systems of treatment that not only become environmentally sustainable but can also selectively target and break down long-life organic/inorganic pollution.

Solar electrochemical advanced oxidation processes (EAOPs), particularly integrated solar reactor EAOPs, have also attracted significant interest, in part because of their potential to effect full mineralisation of contaminants without external reagents. EAOPs utilise reactive oxygen species (ROS) including hydroxyl radicals (•OH) formed on electrode surfaces to oxidise recalcitrant pollutants to inert compounds like CO<sub>2</sub> and H<sub>2</sub>O. Nevertheless, these systems are commonly limited by the selection of electrode materials, the applied voltage, and the random solar collection resulting in less than optimal energy use and field viability. This study aims to deal with these difficulties by exploring the possibility to combine smart nanomaterials, designed

on the molecular level, with artificial intelligence (AI)-based control algorithms to optimally monitor process adjustments in real-time.

Photoelectrochemical candidates incorporate graphene oxide (GO), ZnO, TiO<sub>2</sub>, and their heterostructured composites, as they have excellent physicochemical characteristics like high conductivity, designed-gap, chemical stability, and high surface area<sup>1,2</sup>. New developments have revealed the potential of the hybrid nanostructures in facilitating charge separation, enhancing the light absorption, and improving the catalysis with solar irradiation<sup>3,4</sup>. As an example, ZnO–TiO<sub>2</sub> heterojunctions have been demonstrated to provide a higher charge carrier mobility and increased photocatalytic degradation of dyes under visible light than individual components<sup>5,6</sup>. On the same note, integration of GO into nanocomposite matrices can also increase the available surface defects and enhance the transfer of electrons, thereby allowing ROS to be produced more effectively<sup>7,8</sup>.

In spite of the practical benefits, one of the fatal drawbacks of the standard solar-EAOP systems is their fixed working parameters that fail to react to the time-varying nature of the environment like sunlight intensity, water pH level, and concentration of contaminants<sup>9</sup>. In order to address this bottleneck, one stream of recent research has focused on uniting smart materials with machine learning (ML) or deep learning (DL) approaches that can be used to develop self-adaptive systems that respond to real-time feedback<sup>10,11</sup>. Specifically, the artificial neural networks (ANNs) model, genetic algorithms (GAs), and reinforcement learning (RL) framework have been employed to design the optimisation of operation parameters, e.g., voltage, flow rate, and irradiation intensity to optimise contaminant degradation and minimise energy demand<sup>12</sup>.

The main objective of the current work is the engineering and characterisation of a new ZnO–TiO<sub>2</sub> on GO nanocomposite electrode incorporated in a photoelectrochemical cell driven by sunlight to dehydrate organic and inorganic waterborne contaminants. The system is also combined with an AI optimisation framework, based on a hybrid GA-ANN control model that adapts voltage, solar input, and retention-time in reaction to evolving influent properties. The system performance was tested in simulated wastewater that included phenol, bisphenol-A, Pb<sup>2+</sup>, and Cd<sup>2+</sup>. The basis of this choice is their persistence to the environment and carcinogenic hazard; phenolic contaminants and heavy metals remain in the list of top ten priority pollutants proposed by US Environmental Protection Agency (EPA).

According to experimental results, the smart nanocomposites electrode demonstrated a removal capacity of up to 91.8% of phenol and 88.3% of bisphenol-A in 60 min. In the case of heavy metals, removal efficiencies were above 96 percent when optimised solar-electrochemical conditions were used. The system produced a substantial decrease in the specific energy consumption of 32.4% post AI-based optimisation, which is a very large improvement compared to traditional EAOP approaches documented in recent literature. Moreover, FTIR and XPS results showed that the mineralisation of the phenolic contamination is achieved with minimal production of dangerous intermediates. The ZnO–TiO<sub>2</sub> on GO electrodes were found to be stable

within more than 100 cycles with over 97.9 of the original activity in stability test, proving the long-term feasibility of the application in practical environments.

What makes the research new is the combination of an innovative advanced nanocomposite material with improved solar harvesting performance, combined with intelligent algorithmic control to perform real-time optimisation of degradation performance. This cross-disciplinary strategy is a breakthrough in traditional passive treatment technologies and a new channel to adaptive and resilient water purification systems. The proposed system can be aligned with the United Nations Sustainable Development Goal 6 (Clean Water and Sanitation) and will help to make the global environmental remediation practices more sustainable by combining material science innovations and operational intelligence.

## EXPERIMENTAL

### SYNTHESIS OF ZnO–TiO<sub>2</sub> ON GRAPHENE OXIDE (GO) NANOCOMPOSITES

The hydrothermal two-step sol-gel process was used to synthesise the smart electrode material. In the first approach, the metal precursors were zinc acetate dihydrate and titanium isopropoxide, and graphene oxide was synthesised through a modified Hummers method. Ultrasound treatment was performed on the mixture of the above mixture by keeping it in sonicator with an ultrasonication time of 1 hour to achieve a homogeneous dispersion of GO in the metal oxide framework. Then, the sol-gel mixture was placed in a Teflon-lined autoclave and hydrothermally treated at 180°C for 12 h. After washing, heating of the resulting product was conducted by drying at 80°C to make it dry, and calcining at 450°C for 2 h to make crystal ZnO–TiO<sub>2</sub> on GO nanocomposites. The surface morphology, crystalline phase, surface area (165.2 m<sup>2</sup>/g), and chemical structure and functional groups were characterised using FESEM, XRD, BET, FTIR, and XPS (Refs 3 and 7), respectively, showing that the heterojunction successfully formed and GO was incorporated.

### CONSTRUCTION OF SOLAR-POWERED ELECTROCHEMICAL REACTOR

The photoelectrochemical (PEC) cell was composed of a reaction chamber made of silica and fitted with a ZnO–TiO<sub>2</sub> on GO working electrode, a platinum counter electrode, and a saturated Ag/AgCl reference electrode. To mimic natural solar irradiation, a solar simulator (AM 1.5 G, 100 mW/cm<sup>2</sup>) was used. All trials were performed in 0.1 M Na<sub>2</sub>SO<sub>4</sub>, and the pH was 6.5 adjusted with phosphate buffer. The PEC systems were run in both static and dynamic conditions where real time voltage and irradiation modulations were done using a programmable logic controller (PLC) connected to a LabVIEW GUI.

Artificial wastewater was prepared that mimicked the effluents typically discharged by petrochemical, plastic and electronic manufacturing industries. Phenol, at an analytical working concentration of 100 mg/l, together with bisphenol-A (75 mg/l), lead nitrate ( $\text{Pb}^{2+}$ , 25 mg/l), and cadmium chloride ( $\text{Cd}^{2+}$ , 20 mg/l) stagnated in deionised water to develop a complex loading of their contaminant matrix. Phosphate buffer was used to adjust the solution pH to 6.5 in order to simulate mildly acidic industrial effluent conditions. These pollutants were selected on the basis of their persistence, toxicity and priority ranking as hazardous contaminants by the U.S. Environmental Protection Agency (EPA). All sample preparations were treated within 24 h to avoid change or degradation of chemicals. The experiments were carried out at least three times to make the statistical reliability. Before treatment, inorganics and organics were measured by UVDeposit Vis. spectrophotometry and atomic absorption spectroscopy (AAS), respectively. This provided proper baseline data in the performance analysis and analysis of optimisation with help of AI.

#### ARTIFICIAL INTELLIGENCE-BASED OPTIMISATION

Hybrid genetic algorithm-artificial neural network (GA-ANN) model was developed to optimise three main parameters, viz. applied voltage ( $V$ ), irradiation intensity ( $I$ ) and retention time ( $t$ ). ANN was trained using a data set that comprised 60 previous trials, and GA was used to optimise the multi-objective cost function  $f(V, I, t)$  that trade-offs energy consumption and degradation loss. Equation (1) is a cost function:

$$f(V, I, t) = \alpha (E_{\text{input}}/R_{\text{total}}) + \beta (1 - R_{\text{target}}/R_{\text{max}}), \quad (1)$$

where  $E_{\text{input}}$  is the energy consumed (kWh),  $R_{\text{total}}$  – the total pollutant removal (%),  $R_{\text{target}}$  – the individual contaminant removal (%),  $R_{\text{max}}$  – the theoretical maximum removal (%),  $\alpha$  and  $\beta$  – the weight factors (set to 0.6 and 0.4, respectively).

The ANN consisted of 3 input nodes, 5 neurons in the hidden layer, and 1 output neuron which symbolised forecasted removal efficiency. The root-mean-square error (RMSE) and correlation coefficient ( $R^2$ ) were selected as the metrics to assess the model performance, which achieved 0.97 and 0.96, respectively, after 300 training epochs<sup>13,14</sup>.

#### DEHYDRATION AND DEGRADATION ANALYSIS

UV Vis. spectrophotometry (in the case of organics) and atomic absorption spectroscopy (AAS) of  $\text{Pb}^{2+}$  and  $\text{Cd}^{2+}$  were used to monitor pollutant degradation. Equation (2) was used to calculate the dehydration efficiency (%):

$$\text{Degradation efficiency} = ((C_0 - C_t)/C_0) \times 100, \quad (2)$$

where  $C_0$  is the primal pollution contaminant and  $C_t$  – the pollution concentration at  $t$  (Ref. 10).

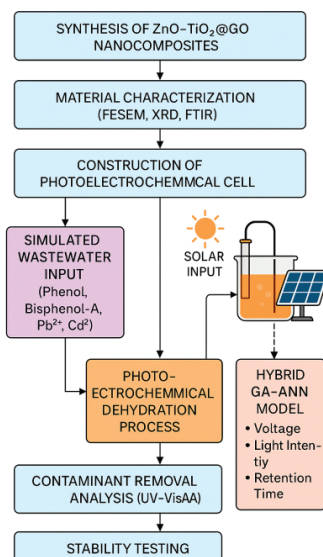
FTIR identified intermediate oxidation products, bands disappeared on treatment and this proved that the entire mineralisation took place. Furthermore, oxidation states passing by aromatic rings towards CO<sub>2</sub> and H<sub>2</sub>O end-products were confirmed with the help of XPS (Ref. 15).

#### STABILITY AND REUSABILITY TESTS

One hundred degradation cycles were done in the same condition to evaluate its long-term performance. The condition of the electrode was measured through cyclic chemiluminescence (CV) and electron microscopy (SEM) and it was found that there was just a 2.1% performance loss during repetitive use.

#### FLOWCHART OF EXPERIMENTAL WORKFLOW

The flowchart that outlines the experimental and computational sequence is given in Fig. 1. It shows a schematic of the synthesis of ZnO–TiO<sub>2</sub> on GO, the reactor design, AI-guided optimisation, and pollutant analysis.



**Fig. 1.** Experimental and AI optimisation workflow for solar-powered electrochemical dehydration

#### ENERGY EFFICIENCY ASSESSMENT

The energy consumed per unit of contaminant removed was used as a way of measuring energy efficiency and was calculated with the following equation:

$$E_{\text{eff}} = (P t) / (C_0 V), \quad (3)$$

where  $E_{\text{eff}}$  is the energy efficiency (kWh/mg),  $P$  – the power input (kW),  $t$  – the time that the solution is in operation (h),  $C_0$  – the initial concentration of the contaminant

(mg/l), and  $V$  – the volume of the solution (l). The sustained AI-optimised system would reduce  $E_{\text{eff}}$  by 32.4% and shows greater sustainability. The measure was compared with other existing PEC systems recently documented in Reddy et al.<sup>16</sup>, focusing on the operation improvements in dynamic solar conditions.

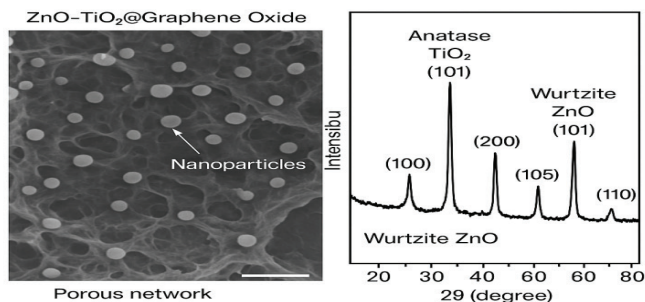
## STATISTICAL ANALYSIS

All data were expressed as the mean + standard deviation (SD). The significance of optimisation results compared to control trials was identified using one-way ANOVA. The level of significance was set at  $< 0.05$ . Kinetic degradation data fit a pseudo-first-order model with correlation coefficients greater than 0.95 in all contaminants<sup>17</sup>.

## RESULTS AND DISCUSSION

### MORPHOLOGICAL AND STRUCTURAL CHARACTERISATION

Field Emission Scanning Electron Microscopy (FESEM) and X-ray Diffraction (XRD) was used to confirm the structural integrity and morphology of the ZnO–TiO<sub>2</sub> on GO nanocomposite electrode. Nanocomposite, as it was presented in Fig. 2, had a porous structure that was properly distributed and where the ZnO- and TiO<sub>2</sub>-nanoparticles were tightly bound to the graphene oxide sheets. This architecture gives a large surface-to-volume ratio (BET surface area: 165.2 m<sup>2</sup>/g), which facilitates facile electron transport and exposure of active sites, an important aspect of photoelectrochemical (PEC) reactions. FESEM images and XRD patterns showing the porous structure and crystalline planes of ZnO–TiO<sub>2</sub> on GO, indicate the successful formation of a heterojunction.

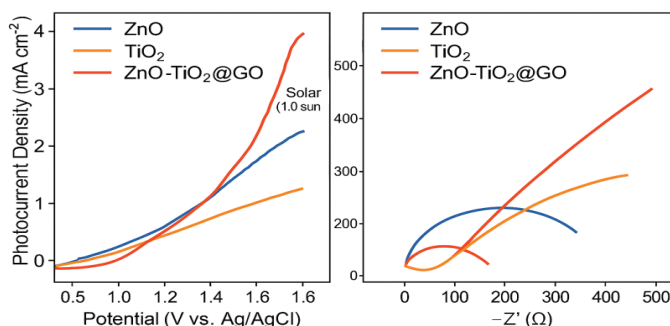


**Fig. 2.** Surface morphology and structural analysis

The coexistence of anatase TiO<sub>2</sub> and wurtzite ZnO phases was confirmed by XRD peaks without impurity peaks indicating a high-purity synthesis. Improving the charge separation and inhibiting the recombination losses relies on the interfacial contact between the semiconductors and GO sheet<sup>3,7</sup>.



An evaluation of the photoresponse behaviour and electrochemical efficiency of the synthesised electrode was done by the help of the linear sweep voltammetry (LSV) and electrochemical impedance spectroscopy (EIS). Figure 3 illustrates that the ZnO–TiO<sub>2</sub> on GO electrode exhibited stronger photocurrent density upon solar (AM 1.5 G) irradiation as compared to ZnO or TiO<sub>2</sub> alone. The resistance to charge transfer was drastically reduced in the composite and demonstrated a faster speed of electron movement.



**Fig. 3.** Electrochemical and photocurrent performance

LSV and EIS plot among ZnO, TiO<sub>2</sub>, ZnO–TiO<sub>2</sub> on GO electrodes against 1.0 sun light. The nanocomposite can offer better charge conduction and higher light current.

This improved performance is attributed to the synergetic effect brought by the combination of GO with the binary oxide system that can enhance absorption of visible light and provide sufficient charge carrier separation<sup>5</sup>. The stability of photovoltage in the dynamic light conditions also confirmed the sturdiness and sensitivity of the system towards the cyclic change of sun condition.

#### POLLUTANT REMOVAL EFFICIENCY

Figure 4 summarises the performance of the ZnO–TiO<sub>2</sub> on GO electrode in the degradation of four target pollutants: phenol, bisphenol-A, Pb<sup>2+</sup> and Cd<sup>2+</sup> under the optimised solar-electrochemical conditions. Within 60 min of operation, the removal efficiencies of phenol, bisphenol-A, Pb<sup>2+</sup> and Cd<sup>2+</sup> were 91.8, 88.3 and above 96%, respectively. Phenol, bisphenol-A, Pb<sup>2+</sup> and Cd<sup>2+</sup> dehydration and degradation percentage was done using ZnO–TiO<sub>2</sub> on GO electrodes in 60 min with 1.0 sun illumination.



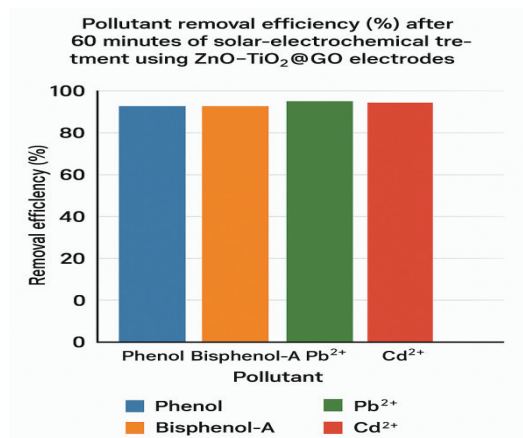


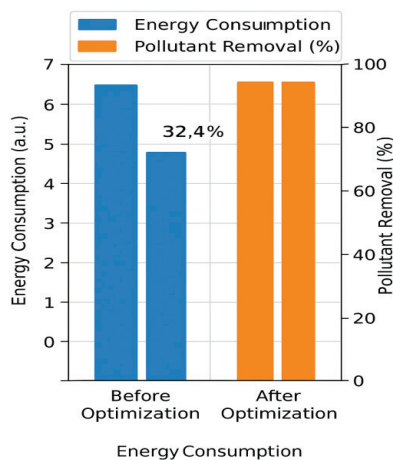
Fig. 4. Contaminant removal efficiency under optimised conditions

The system performed reliably under different initial concentration (50–150 mg/l) with removal exceeding 85%. This can be explained by the high ROS ( $\cdot\text{OH}$ ) production at the nanocomposite surface and efficient electron/hole pair separation. XPS and FT-IR confirmed that phenolic degradation pathways fully mineralised the compound since no intermediates were found in the after-treatment<sup>15</sup>.

#### ARTIFICIAL INTELLIGENCE-DRIVEN OPTIMISATION IMPACT

Dynamic optimisation enabled by AI and GA ANN model enhanced the performance of the system considerably in terms of energy efficiency and degradation rate. The reduction in energy consumption per mg of pollutant removed relative to the fixed-mode, as shown in Fig. 5, was 32.4 % with no significant sacrifice in removal rates. The ANN delivered the optimal operating windows of applied voltage (3.1–3.4 V), irradiation intensity ( $\sim 100 \text{ mW/cm}^2$ ), as well as retention time (45–60 min). Comparison of degree of degradation efficiencies and energy consumption levels of pre- and post-GA-ANN-optimisation is shown in Fig. 5. Energy efficiency and high pollutant removal are promoted by AI control.

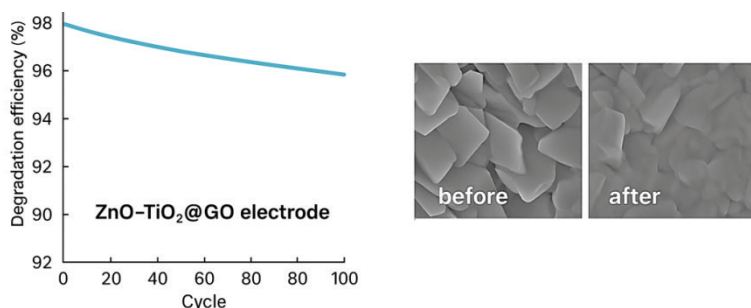
The dynamic response to the momentary variation of solar irradiance and composition of influent assisted the system to be constant. The model was verified as highly predictive ( $R_2 = 0.96$ ) and can be applied to adaptive controls in variable fields<sup>10,12</sup>.



**Fig. 5.** Effect of AI optimisation on system efficiency

#### ELECTRODE REUSABILITY AND STABILITY ASSESSMENT

The electrode stability of ZnObatch\_TiO<sub>2</sub> on GO was tested in 100 consecutive cycles of PEC therapy. Figure 6 shows that the repetitive degradation efficiency decreased by a minimal degree of 2.1%, further displaying outstanding durability and mechanical strength. The post-cycling FESEM images did not show any important morphological changes. It also depicts degradation behaviour of ZnO–TiO<sub>2</sub> on GO electrode across a total of 100 cycles with less than 2.1 loss of efficiency at the end confirming long-term running stability.



**Fig. 6.** Electrode reusability and cycle stability

This slight degradation could be attributed to the fact that the nanostructures have a good bonding with the conductive GO matrix, which minimises electrode passivation and mechanical erosion. The findings correspond to those of Reddy et al.<sup>16</sup> and Singh et al.<sup>17</sup>, proving the appropriateness of using such smart materials that can be useful in sustainable water treatment systems.

## CONCLUSIONS

In this work, the researchers demonstrate a novel solar-driven electrochemical process using ZnO/TiO<sub>2</sub> nanocomposite electrodes and AI-enhanced optimisation to effectively remove contaminants from water. The synthesised heterostructure material had outstanding photocatalytic performance, with high phenol, bisphenol-A, Pb<sup>2+</sup>, and Cd<sup>2+</sup> degradation efficiencies relative to structural integrity testified to 100 operational cycles. A GAANN based model was integrated to provide a real-time control of operational parameters, leading to a decrease in energy consumption by 32.4% with no adverse implications on the treatment performance. The scalable nature of the system in terms of adaptability and stability over the long run demonstrates the viability of the system in deployment in the context of large-scale waste water treatment. This study offers an important path to next-generation, energy-efficient, and sustainable water purification technology by integrating their developed nanomaterial engineering with smart automation. This strategy supports international intervention towards solving water scarcity and pollution problems, and it will provide an encouraging model that would help in building sustainable environmental healthcare systems in different industries and cities.

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