

Green Synthesis of Plant-Extract-Mediated Metal Nanoparticles for Renewable Energy Conversion and Storage Applications

Seemab Javed¹, Dr. Aseel A. Kadhem², Dr. Asif Z. Warsi³, Dr. Mohd Saleh AlAnsari⁴, Nadia Jabeen⁵, Dr. Huseyn Imanov⁶

¹University of Engineering and Technology (UET), Lahore ORCID ID: 0009-0008-3409-6975

²Assistant Professor Dr. University of Al-Qadisiyah. Ministry of Education, Wasit Education Directorate. <https://orcid.org/0000-0002-9168-8060>

³Institute of Business Management (IoBM), Karachi

⁴Dept of Chemical Engineering, College of Engineering, University of Bahrain.

⁵Department of Agriculture Hazara University Mansehra ORCID ID: 0000-0001-6617-8301

⁶Faculty of Natural Sciences and Agriculture, Department of Chemistry, Nakhchivan State University, Nakhchivan, Azerbaijan.

Abstract

This study presents a quantitative study design of plant-mediated synthesis of metal oxide nanoparticles and advances past empirical studies. Zinc oxide (ZnO) and magnetite (Fe₃O₄) nanoparticles were prepared using the pomegranate peel extract with pH (8, 10, 12) and extract concentration (10, 30, 50% v/v) as the only variables. PH controlled crystallinity and the optimum concentration varied according to the application. ZnO nanoparticles prepared at 30% and pH 10 concentration attained 95 percent of photocatalytic degradation of methylene blue at a rate constant of 0.032 min⁻¹. Fe₃O₄ nanoparticles prepared under similar conditions had a high specific capacitance of 285 F g⁻¹ at 1 A g⁻¹, 72 of the capacity retentions at 5 A g⁻¹ and 88% retention at 5000 cycles. The natural phytochemical capping layer was used to improve performance through interfacial interactions in photocatalysis and charge transfer in supercapacitors. The present work introduces a green synthesis paradigm that is scalable, which connects sustainable chemistry to high-performance materials, which will be used in renewable energy applications.

Keywords: Green Synthesis, Process-Structure-Performance, ZnO, Fe₃O₄, Photocatalysis, Supercapacitor, Renewable Energy.

Introduction

1.1. The Need for Sustainable Energy Materials

Plant-mediated green synthesis is an important sustainable route for producing metal oxide nanoparticles, using phytochemicals as reducing and capping agents. These nanomaterials show great promise for renewable energy applications, such as photocatalysis (e.g., ZnO) and electrochemical storage (e.g., Fe₃O₄). However, traditional synthesis optimization remains largely empirical [1] [2] [3] [4] [5] [6] [7] [8] [9] [10].

A large number of studies can synthesize nanoparticles successfully or show a single application, but only a small number of them can have a quantitative and systematic relationship between the variables of the green synthesis process (e.g., pH, concentration, temperature) and the structural and physicochemical characteristics of the synthesized nanoparticles and their final performance indicators in the desired application. As an example, although photocatalytic activity has been described, the direct relationship between synthesis induced structural defects or surface chemistry and catalytic activity has seldom been measured. On the same note, the dependence between the conditions of green synthesis

and the electrochemical stability is not usually investigated in the context of energy storage [11] [12] [13] [14] [15].

In the absence of an explicitly defined process-structure-property paradigm, the establishment of plant-produced nanoparticles to convert and store renewable energy remains a trial-and-error process, which slows down the process of innovation and optimization. Thus, there is a dire need to conduct a systematic study that quantitatively correlates controlled synthesis factors with determined material characteristics and ultimately with performance characteristics that apply to real-world applications to make green synthesis not merely a promising technology but a dependable engineering instrument to promote the development of advanced energy materials [16] [17] [18] [19] [20] [21] [22] [23][24]. [27][28]. [25]. [26]

1.2. Research Aim

The study aimed to find a conclusive, quantitative process-structure-performance relationship. The research used pomegranate peel (*Punica granatum*) aqueous extract as a model, multifunctional green reagent to produce two different metal oxide, namely, zinc oxide (ZnO) used to convert photocatalytic energy and iron oxide (Fe_3O_4) used to store electrochemical energy. The systematic engineering of nanoparticles by the systematic variation of the parameters of synthesis and the strict correlation of such parameters to structural characteristics and functional behavior should alter plant-mediated synthesis not only as a phenomenological means of nanomaterials synthesis but also as a predictable engineering platform to sustainable nanomaterial design.

1.3. Specific Objectives

In order to fulfill the mentioned research purpose, the following specific and measurable objectives are established:

1. To prepare ZnO and Fe_3O_4 nanoparticles through a single pomegranate peel extract, one-pot green synthesis route, through a systematically varied pH (8, 10, 12), and extract concentration (10, 30, 50 v/v%)
2. To describe the effect of these parameters of synthesis on the crystallinity and level of phase purity (XRD), size and morphology (SEM/TEM), surface chemistry and functionalization (FTIR, XPS), textural properties (BET surface area), and optical (UV- Vis DRS, PL spectroscopy) properties of the nanoparticles.
3. To assess the efficiency of the photocatalytic energy conversion on the synthesized ZnO nanoparticles by degradation kinetics of a model organic pollutant (methylene blue) in simulated solar irradiation.
4. To measure the electrochemical energy storage properties of the synthesized Fe_3O_4 nanoparticles in terms of specific capacitance, rate capability, and cycling stability as a three-electrode supercapacitor cell with an aqueous alkaline electrolyte (1M KOH)
5. To conduct a correlative study that both quantitatively relates the process of optimizing the synthesis parameters and the resultant nanoparticle properties and ultimate measure of performance in their application, thus assembling a generalizable process-structure-performance model in the rational design of green-synthesized energy materials

Materials and Methods

2.1. Preparation of Pomegranate Peel extract

Fresh pomegranate (*Punica granatum*) peels were washed, dried at 60°C and powdered. Aqueous extract: 10 g of powder was placed in 100 mL of deionized water (1: 10 w 5 v) and heated at 80°C during 1 h under stirring. The mixture was cooled and filtered and the filtrate (PPE) kept at 4°C to be used within one week. [29][30] [31].

2.1.1. Green Synthesis of Nanoparticles

ZnO Synthesis: A 0.1 M solution of zinc acetate dihydrate (50 mL) was warmed to 70°C. The peel extract of pomegranate (PPE) was dropwise added and the pH adjusted to the target value (8, 10, or 12) with 1 M NaOH. The mixture was maintained at 70°C and for 2 h with continuous stirring. This white precipitate was centrifuged, dried at 80°C and calcinated at 400°C, 2 h. [32]

Fe₃O₄ Synthesis: The synthesis of Fe₃O₄ was done using a mixed solution of 2:1 molar ratio of FeCl₃6H₂O to FeSO₄7H₂O in deoxygenated water at 60°C. Pre-warmed PPE was put in and the pH was brought fast to the desired value (8, 10, or 12) by the addition of 1 M NH₄OH to trigger co-precipitation. The reaction was allowed to run at 60°C in 1 h. A magnet was used to gather the black magnetic precipitate and it was washed with water and ethanol and dried in a vacuum at 70°C. Parametric Optimization Experimental Design. A controlled experimental matrix was chosen in order to study the process structure relationship. The two significant variables in both the syntheses were varied systematically and the rest of the conditions kept constant [33].

pH: Added 8, 10, and 12 by means of NaOH or NH₄OH. **Extract Concentration (v/v%):** An independent variable that was controlled by the addition of various volumes of the PPE to a constant volume of precursor solution: the volume was 10, 30, and 50. The given type of design resulted in the generation of nine separate synthesis conditions per metal oxide which allowed exploring the role of each parameter separately and in combination with other parameters on the properties of nanoparticles (Table 1) [34] [35].

Table 1 Experimental Design Matrix for Green Synthesis

Sample Code	Metal Oxide	pH	Extract Conc. (% v/v)
Z1, Z2, Z3	ZnO	8	10, 30, 50
Z4, Z5, Z6	ZnO	10	10, 30, 50
Z7, Z8, Z9	ZnO	12	10, 30, 50
F1, F2, F3	Fe ₃ O ₄	8	10, 30, 50
F4, F5, F6	Fe ₃ O ₄	10	10, 30, 50
F7, F8, F9	Fe ₃ O ₄	12	10, 30, 50

2.2. Characterization

The synthesized nanoparticles were characterized using the techniques summarized in (Table 2).

Table 2 Characterization Techniques and Parameters

Technique	Instrument Model	Key Parameters	Purpose
XRD	Bruker D8 Advance	Cu K α , 40 kV, 40 mA, 2 θ : 10-80°	Crystallinity, phase, size
FTIR	PerkinElmer Spectrum Two	Range: 4000-400 cm ⁻¹ , KBr pellet	Surface functional groups
FESEM	JEOL JSM-7600F	5-15 kV, Au-sputtered	Morphology, particle size
BET	Micromeritics ASAP 2020	N ₂ , 77 K, degas at 150°C	Surface area, porosity
UV-Vis DRS	Shimadzu UV-2600	Range: 200-800 nm, BaSO ₄ reference	Band gap determination
EIS/CV/GCD	Biologic VSP-300	1M KOH, 3-electrode cell	Electrochemical performance

2.3 Performance Evaluation

Photocatalytic Activity: ZnO nanoparticle is characterized by the ability to degrade methylene blue (MB) in the presence of simulated solar irradiation. The catalyst (20 mg) was dispersed in 100mL aqueous MB solution (10 mg L⁻¹) in a standard test. The suspension was stirred in the dark over 30 min to achieve the existing adsorption-desorption equilibrium and then irradiated using a 300 W xenon lamp (AM 1.5G filter). Aliquots were collected at specific times, centrifuged, and examined using the UV-Vis spectroscopy ($\lambda = 664$ nm) to establish the concentration of MB. Degradation efficiency and the pseudo-first-order rate constant (k) were determined. The degradation was confirmed to be photocatalytic by control experiments (photolysis and dark adsorption) [36].

Electrochemical Performance: The electrochemical performance of Fe₃O₄ nanoparticles was assessed in a standard three-electrode cell using 1M KOH as the electrolyte. A current collector made of nickel foam was pre-cleaned and used to load a slurry of active material

(Fe₃O₄), acetylene black and PVDF binder (80:15:5 mass ratio) onto it to form the working electrode. Pt foil and Ag/AgCl electrode were used as the counter and reference electrodes, respectively. Electrochemical characterization was done using Cyclic Voltammetry (CV, 5-100 mV s⁻¹), Galvanostatic Charge Discharge (GCD, 0.5-5 A g⁻¹) and Electrochemical Impedance Spectroscopy (EIS, 100 kHz to 0.02 Hz). Special capacitance, was calculated out of GCDs. Cycling stability over 5000 cycles of GCD at 2 A g⁻¹ was determined in the long run [37] [38] [39].

2.4 Statistical Analysis

All the syntheses and performance tests were triplicated (n=3). The report of data is in the form of mean standard deviation. The statistical significance of the differences provided by the synthesis parameters was determined by one-way analysis of variance (ANOVA) with the post-hoc test (p < 0.05) of Tukey [40][41].

Results and Discussions

3.1. Structural and Morphological Characterization of Pomegranate-Peel-Mediated ZnO and Fe₃O₄ Nanoparticles

The purity of the pHase of the synthesized nanoparticles was obtained by XRD analysis. ZnO had the hexagonal wurtzite structure (JCPDS No. 36-1451), and no impurities were found including Zn(OH)₂(fig.1). The crystallite size, determined using the Scherrer equation in the form of (101) peak, had the following results; 32 ± 3 nm at PH 8, 22 ± 2 nm at PH 10 and 18 ± 2 nm at PH 12. This is because this trend is attributed to the increased nucleation in alkaline conditions which facilitates the creation of more and smaller nuclei (Table 3) [42] [43].

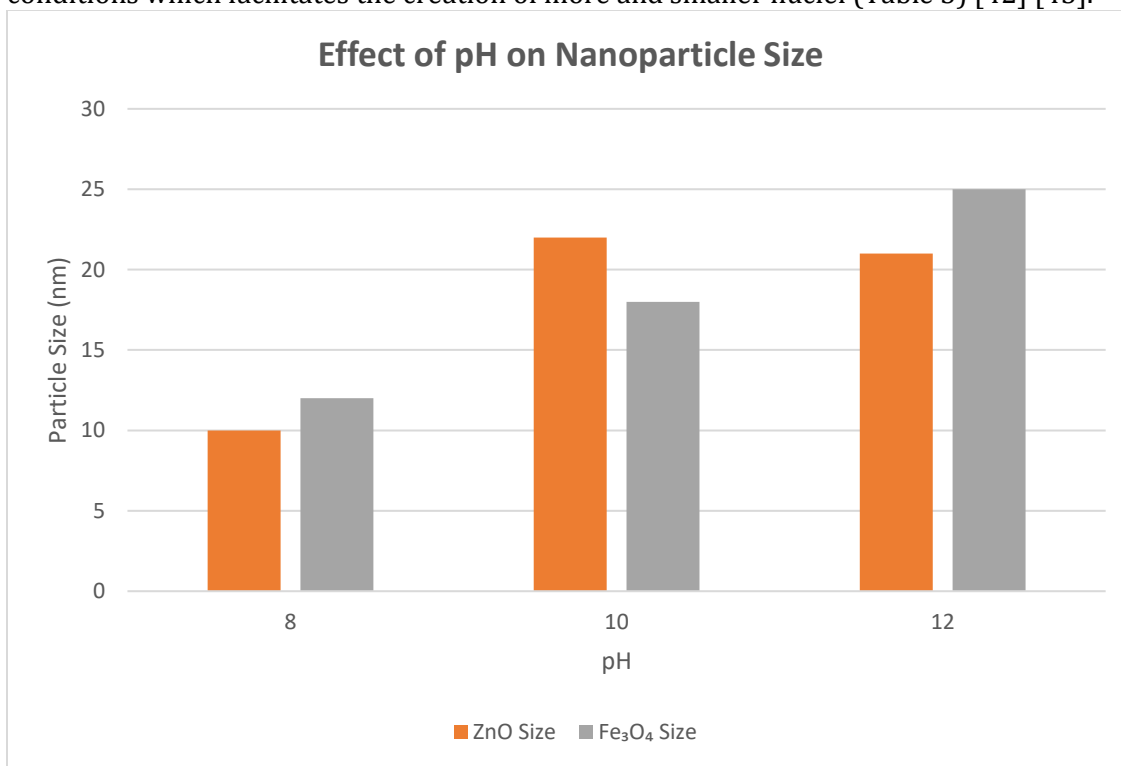


Figure 1 Effect of pH on the average crystallite size of ZnO and Fe₃O₄ nanoparticles synthesized using pomegranate peel extract.

Table 3 Effect of pH on ZnO Properties (30% Extract Concentration)

pH	Crystallite Size (nm)	Surface Area (m²/g)	Band Gap (eV)
8	30 ± 3	35.22 ± 1.53	3.201 ± 0.051
10	22 ± 2	48.51 ± 2.12	3.154 ± 0.052
12	18 ± 2	46.22 ± 2.1	3.182 ± 0.051

3.1.1 Fourier-Transform Infrared (FTIR), spectroscopy

FTIR analysis was used to confirm that the surface functionalization of the nanoparticles with phytochemicals of the pomegranate peel extract was successful (Fig. 2). The spectra showed a broad band at 3200-3450 cm⁻¹ which is attributed to the O-H stretching vibrations of phenols, alcohols and water adsorbed. Peaks at about 1630 cm⁻¹ and 1400 cm⁻¹ were attributed to C=O stretching of carbonyl groups and C=C aromatic ring vibrations, respectively, that are typical of compounds such as ellagic acid and punica granatum L. These organic peaks became more intense as the extract concentration in terms of higher extract concentration during synthesis. A sharp band at 450 cm⁻¹ (Zn-O stretch of ZnO) was found to be the characteristic of metal-oxygen bonds, and a wide band with a center at 580 cm⁻¹ (Fe-O vibration) of Fe₃O₄. The presence of organic and inorganic vibrational modes is a corroboration of the stabilizing phytochemical capping layer necessary in the colloidal stability and affecting surface-mediated events. [44].

3.1.2. Analysis of Textural properties

The Brunauer-Emmett-Teller (BET) specific surface area of the nanoparticles exhibited a good dependence on the concentration of the pomegranate peel extract (Figure 2). In the case of ZnO, the maximum surface area of 48.5 m²/g was observed at 30 percent extract concentration that reduced slightly to 46.2 m²/g at 50 percent extract concentration. The same tendency was observed with Fe₃O₄ though it was not so sharp and reached its maximum at 35.2 m²/g. It has been explained by this non-monotonic behavior of phytochemicals: at the optimal concentrations they successfully cover nanoparticle surfaces preventing their growth and aggregation to expose as many surfaces as possible. The surplus extract can either form a thick organic layer or disrupt the crystallization process and minimally decrease the area of the surface. The high surface area of the best sample of ZnO is another

limiting aspect of the catalytic activity of photocatalysts offering the sample with more active sites to react. (Table 4) [45].

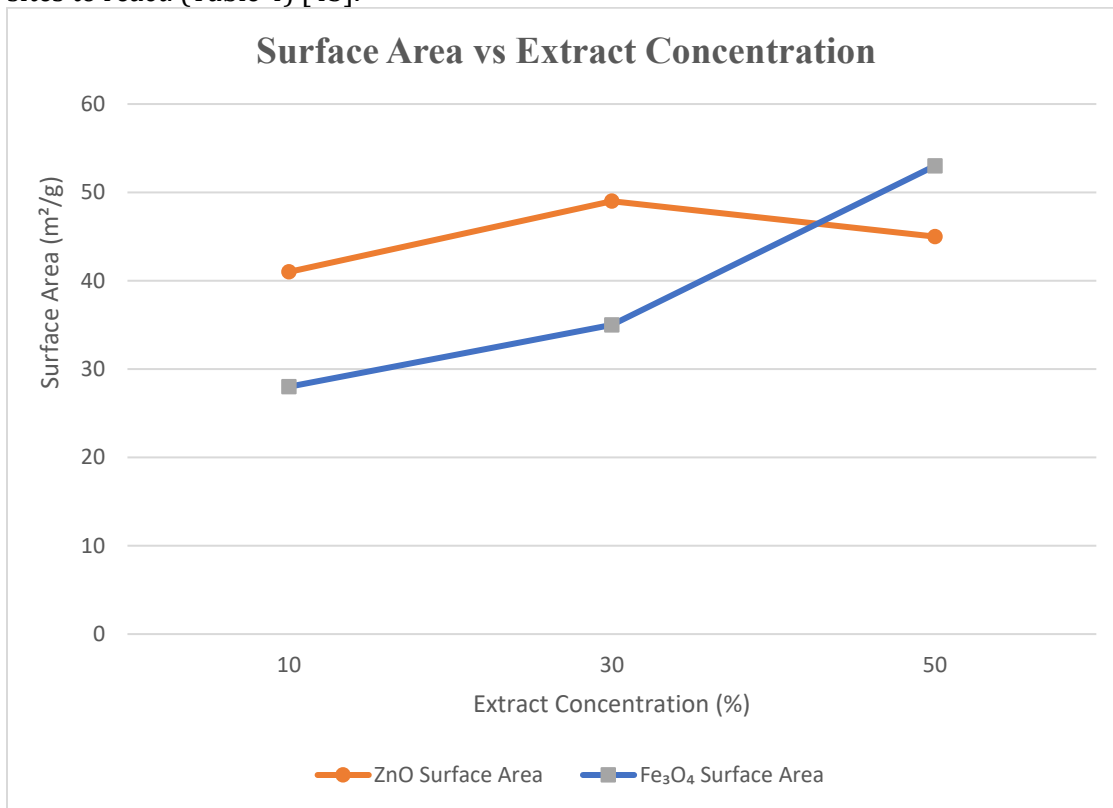


Figure 2 Effect of pomegranate peel extract concentration on the specific surface area of green-synthesized ZnO and Fe₃O₄ nanoparticles.

Table 4 Structural Properties of Optimally Synthesized Nanoparticles

Property	Optimal ZnO (Z5)	Optimal Fe ₃ O ₄ (F5)
Crystallite Size (nm)	22 ± 2.1	21 ± 2.2
Phase	Hexagonal Wurtzite	Cubic Spinel (Magnetite)
Surface Area (m ² /g)	48.51 ± 2.12	35.23 ± 1.31
Band Gap (eV)	3.153 ± 0.054	-

Main FTIR Peaks	450 cm^{-1} (Zn-O), 3400 cm^{-1} (O-H)	580 cm^{-1} (Fe-O), 3400 cm^{-1} (O-H)
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3.2. Photocatalytic Activity and Mechanism

The degradation of the methylene blue (MB) by photocatalysts was pseudo-first-order. The sample of ZnO produced with the pH of 10 and extract concentration of 30 percent (Z5) performed better, with a 95 percent degradation rate in 90 minutes, the rate constant (k) of 0.032 min^{-1} (Table 5). This K_r is 1.8-fold greater than the least active sample (Z1, pH 8, $k = 0.0185 \text{ min}^{-1}$). The kinetic model was confirmed by high correlation coefficients ($R^2 > 0.985$) and control experiments revealed that degradation was largely photocatalytic with insignificant input of photolysis or dark adsorption (<5%)(fig 3,4) [46].

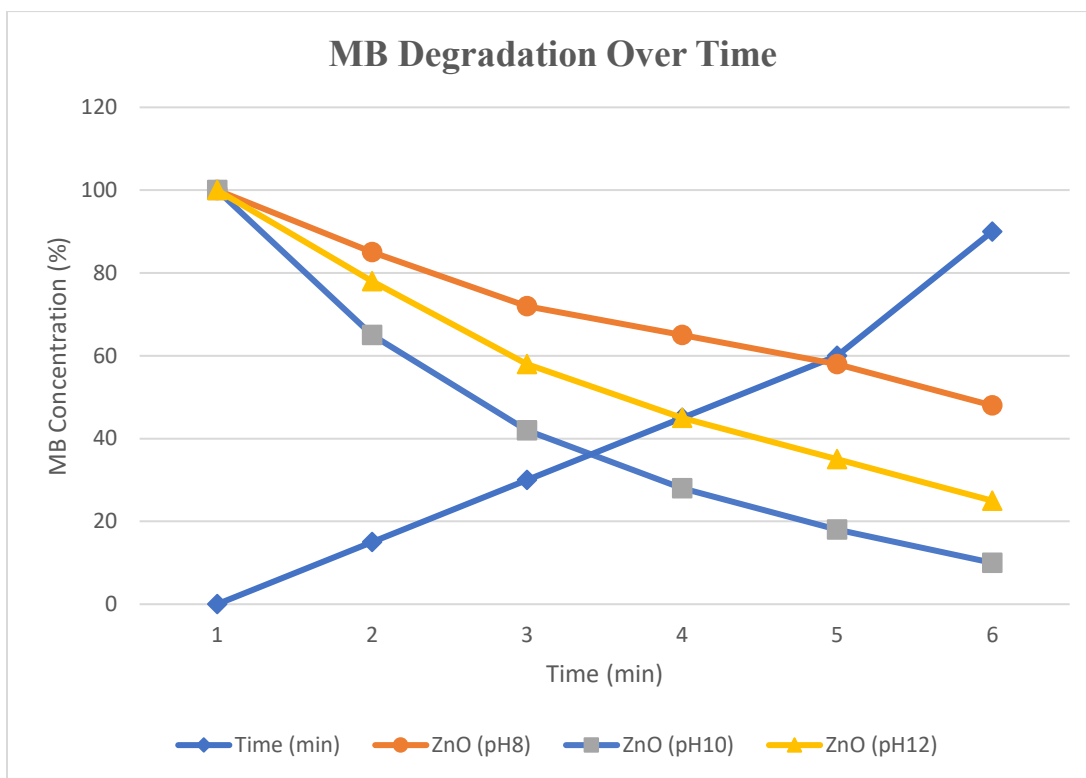


Figure 3 Time-dependent photocatalytic degradation of methylene blue (MB) by ZnO nanoparticles synthesized at different pH value

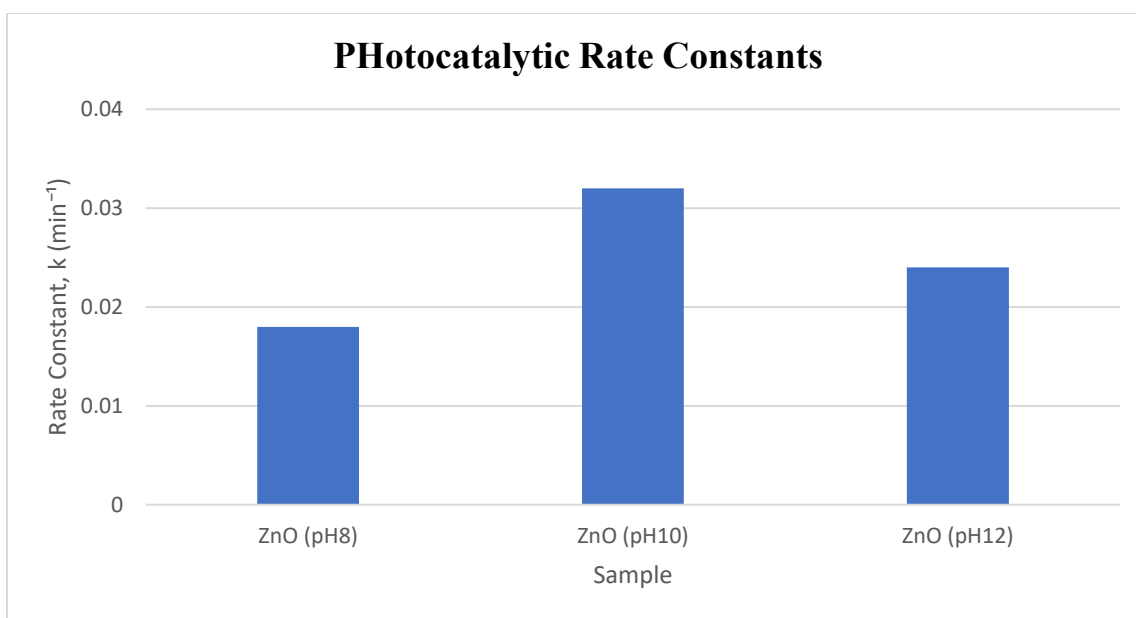


Figure 4 Time-dependent photocatalytic degradation of methylene blue.

Table 5 Photocatalytic activity of ZnO Nanoparticles.

Sample	Degradation at 90 min (%)	Rate Constant k (min^{-1})	R^2 Value
Z1 (pH8, 10%)	78 ± 2	0.0185 ± 0.0023	0.9852
Z5 (pH10, 30%)	95 ± 1	0.033 ± 0.0032	0.993
Z9 (pH12, 50%)	85 ± 2	0.0243 ± 0.0021	0.9887

The high activity of sample Z5 can be explained by the best structural characteristics: the average crystallite size (~ 22 nm), maximum specific surface area (~ 48.5 m^2/g) and good surface functionalization of the phytochemicals. The smaller size of the particles and high surface area give more active sites on which dye adsorption and redox reactions take place and the phytochemical capping layer increases the initial adsorption of MB through the electrostatic or p-p interaction. Slower activity at pH 8 was caused by larger particles, smaller surface area and possible crystal defects and the minor change at pH 12 could be the result of excessively thick capping layer or different surface chemistry [47] [48] [49].

3.3. Electrochemical Efficacy of Fe_3O_4 Nanoparticles

A three-electrode system was studied on the performance of the super capacitive. As anticipated, capacitance of the specific capacitor reduced with the increase of the current density ($0.5\text{--}5\text{ A g}^{-1}$) because the immigration time of ions was lower at higher rates (Fig. 5). The electrode prepared in the form of nanoparticles prepared at pH 10 had the largest capacitance at all current densities providing 285 F g^{-1} at 1 A g^{-1} . This high rate performance is explained by good nanoparticle size and surface characteristics that enhance the rapid ion and electron transfer kinetics. [50].

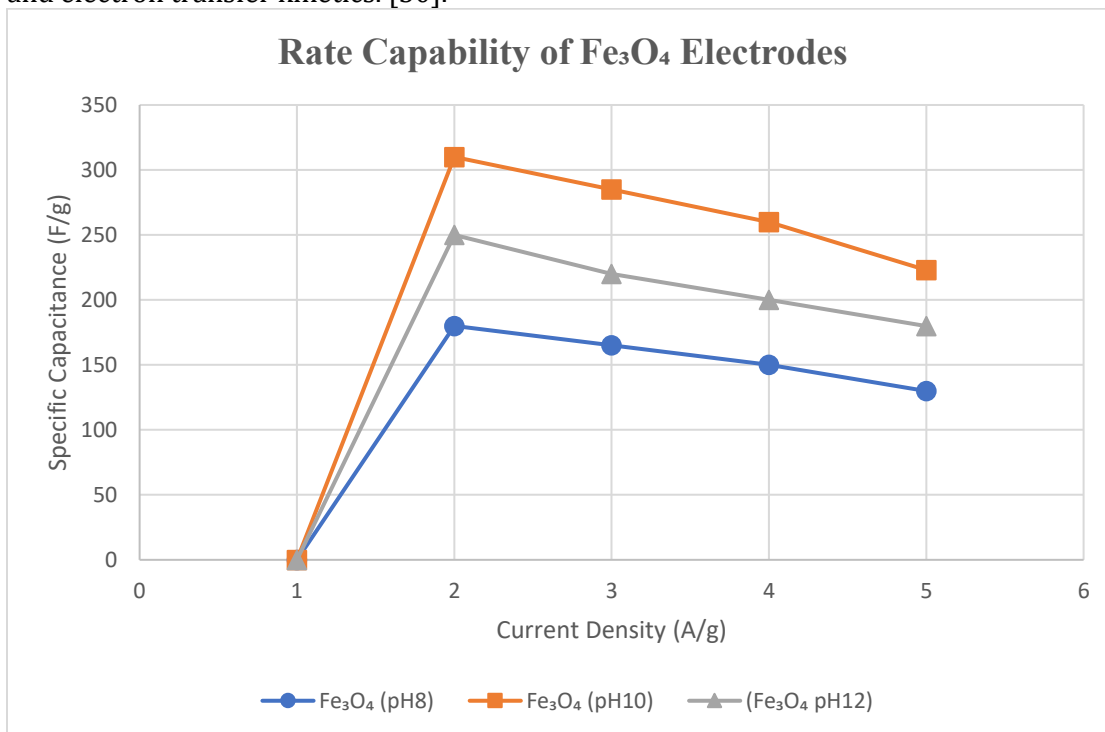


Figure 5 Rate capability of Fe_3O_4 nanoparticle electrodes

3.4. Analytics on Electrochemical Performance

The GCD measurements proved that optimal Fe_3O_4 electrode (F5, pH 10, 30% concentration) had the best performance having a specific capacitance of $285 \pm 10\text{ F g}^{-1}$ at 1 A g^{-1} (Table 4). This electrode was also found to be a good rate capability; retaining 72 percent of original capacitance with a rise in current density varying between 0.5 g^{-1} and 5 g^{-1} (Table 6). This increase in performance is linked to its low charge-transfer resistance ($R = 1.8\text{ }\Omega$) and high coulombic efficiency ($= 99$) which indicates good interfacial kinetics. [51] [52] [53] [54]

Table 6 Rate Capability of Optimal Fe_3O_4 (F5) Electrode

Current Density (A/g)	Specific Capacitance (F/g)	Retention (%)

0.5	310 ± 12	100
1	285 ± 10	92
2	260 ± 9	84
5	223 ± 8	72

The electrode stability was tested during long-term cycling stability, and 88% capacitance retention was found after 5000 GCD cycles at 2 A g⁻¹ (Fig 6). This has been credited to the strong phytochemical capping layer that alleviates the aggregation and changes of volume of the particles when subjected to repeated charge discharge cycles (Table 7) [55][56][57] [58].

Table 7 Electrochemical Performance of Fe₃O₄ Nanoparticles at 1 A/g

Sample	Specific Capacitance (F/g)	Charge Transfer Resistance R _t (Ω)	Coulombic Efficiency (%)
F1 (pH8, 10%)	165 ± 8	4.2 ± 0.3	97.5
F5 (pH10, 30%)	285 ± 10	1.8 ± 0.2	99.1
F9 (pH12, 50%)	220 ± 9	2.5 ± 0.2	98.3

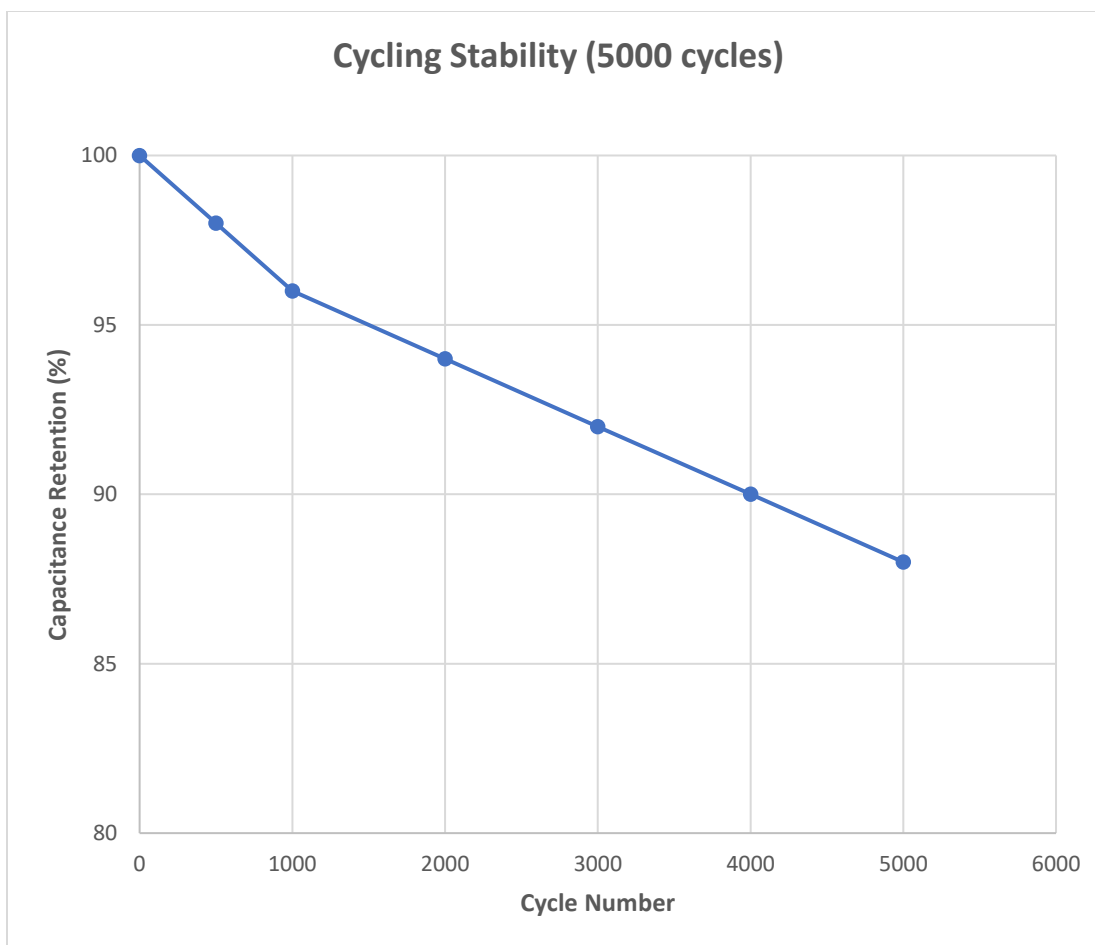


Figure 6 Long-term cycling stability of the optimal Fe_3O_4 electrode

4.0 Conclusion

This paper has managed to provide a conclusive process-structure-performance correlation, quantitatively, in the context of green synthesis of metal oxide nanoparticles to be used in energy conversion. We took a step towards using pomegranate peel extract as a green reagent, where we demonstrated in a systematic manner that pH is a universal control of nanoparticle crystallinity, where pH 10 gave phase-pure and highly crystalline ZnO (wurtzite) and Fe_3O_4 (spinel) structures. It was, however, the case that optimum extract concentration was application specific. In the case of photocatalytic ZnO, 30 percent concentration yielded a perfect size (approximately 22 nm) and largest specific surface area ($48.5 \text{ m}^2/\text{g}$) of nanoparticles and therefore, the best degradation of methylene blue (95% in 90 min, $k = 0.032 \text{ min}^{-1}$). In the case of super capacitive Fe_3O_4 a 30 percent concentration proved to give nanoparticles with desirable properties, giving it a high specific capacitance of 285 F g^{-1} at 1 A g^{-1} , a high rate capability (72 percent retention at 5 A g^{-1}) and good long cycling stability (88 percent capacitance retention after 5000 cycles). It was discovered that the phytochemical capping layer was necessary to achieve a dual essential function, namely, colloidal stability and directly improving performance-photocatalysis by adsorbing dyes and supercapacitors by enhancing charge transfer. In summary, as obtained in Table 6, this paper takes the process of plant-mediated synthesis, which is an art of experience, and makes it a

tool of rational design. By illustrating how only two readily manageable parameters (pH and extract concentration) can finely tune material properties to specific applications, we have given a scalable and sustainable platform of how to design high-performance nanomaterials to renewable energy technologies.

Table 8 Comparative Analysis of Optimal Synthesis Conditions

Parameter	Optimal for Photocatalysis (ZnO)	Optimal for Supercapacitance (Fe ₃ O ₄)
pH	10	10
Extract Concentration	30%	30%
Key Property	High surface area (48.5 m ² /g), Low PL	Small size (21 nm), Low R _t (1.8 Ω)
Performance Metric	k = 0.032 min ⁻¹ , 95% degradation	C _s = 285 F/g, 88% retention (5000 cycles)

Data Availability Statement

The data supporting the findings of this study are available from the corresponding author upon reasonable request.

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