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Green Synthesis of *Ananas Comosus* (Pineapple Peel)-Zinc Nanoparticles for Photocatalytic Degradation of Methylene Blue Dye

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ABSTRACT: Textile wastes are the main environmental issues in various dyeing industries. Several treatment methods are proposed to minimize the organic contaminants to reduce Pollutants from the effluents. In photodegradation many bio doped materials such as (PA-Zn Nps) have shown promising performance in destroying organic pollutants such as organic dyes with minimal side pollution. This study investigated the green synthesis of nanoparticles using pineapple peel extract and the photodegradation capabilities of bio doped zinc metal (Zn) nanoparticles for their efficiency in degrading organic dyes. By harnessing the unique properties of (PA-Zn Nps), this research aims to advance the field of photocatalysis, particularly in environmental applications such as wastewater treatment. The degradative capacity of the nanoparticles against methylene blue was assessed by a range of analytical methods, such as SEM, XRD and FTIR analysis. Optimisation studies are made to enhance the performance of photocatalyst in the removal of dye. Kinetics and Thermodynamics are also studied. According to our research, 'PA-Zn Nps' exhibits better pollutant degrading efficiency and is environmentally benign, making it a viable sustainable photocatalyst for water treatment applications.

KEYWORDS: Photocatalytic Degradation, PA-Zn Nps, Methylene Blue dye, SEM, XRD, FTIR, Optimisation Studies, Kinetics, Thermodynamics, Photocatalyst.

I. INTRODUCTION

The presence of organic dyes in effluent from textile, paper and apparel industries results in substantial environmental contamination. Several studies reported that dyes like Rhodamine B, Victoria blue, Indigo Red, Caramine, Eriochrome, Methylene Blue, Eriochrome Black-T, and Thymol blue are utilized annually in textile industries of which a major portion (~20%) is lost during synthesis and processing operations and ends up in wastewater [1]. These dye-polluted effluents comprise non-biodegradable, highly poisonous, and colored pigments that are harmful to living organisms. Conventional wastewater treatment techniques, such as adsorption, coagulation, and biological degradation, often fail to completely remove these pollutants or generate secondary waste [2]. Advanced oxidation processes, particularly photocatalytic degradation, have gained attention as an efficient and eco-friendly approach for degrading organic dyes into non-toxic byproducts [3]. The photocatalysts using zinc nanoparticles have emerged as a promising material due to their high chemical stability, non-toxicity, and excellent optical properties with a wide bandgap (~3.6 eV), making it effective under ultraviolet light for photodegradations [4]. However, the synthesis of Zn Nps often involves hazardous chemicals and energy-intensive processes, limiting their large-scale application in environmental remediation.[5]

To address this issue, green synthesis using peel extracts as reducing and stabilizing agents has gained popularity as a sustainable alternative to conventional chemical methods [6]. Plant mediated synthesis is eco-friendly, and cost-effective, and enables the formation of biocompatible nanoparticles with controlled size and morphology. Pineapple peel (Ananas comosus) extract is rich in bioactive compounds such as flavonoids, polyphenols, and organic acids, which can act as natural reducing agents for the synthesis of PA-Zn nanoparticles [7]. In this research, we studied the





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green synthesis of the most effective PA-Zn nanoparticles using pineapple fruit peels for the removal of organic dyes from textile industrial wastewater. This work focused on the economical synthesis and key parameters that influence the photocatalytic effective degradation process of dyes.

II. MATERIALS AND METHODS

Pineapple Peels were gathered from local areas.

- Chemicals Required: Zinc Sulphate (ZnSO₄), HCl, NaOH.
- Glassware: 1 lit beaker, Measuring jar, Pipette, conical flasks etc.,
- Equipments: pH meter, Mechanical stirrer, Spectrophotometer, Orbital Shaker and Glass Ware etc.

A. Estimation of Dyes:

1) Preparation of Congo Red Sample-

1g of Methylene blue dye was diluted in 1000 ml distilled water to prepare the methylene blue sample with a concentration of 1000 ppm. It is further diluted to prepare desired concentrations of the dye sample. [8]



Figure 1- Methylene Blue dye (powder)



Figure 2- Methylene Blue dye solution

B. Synthesis of (Ananas Comosus) Pineapple Peel-Zinc Nanoparticles (PA-Zn Nps):

1) Preparation of Pineapple Peel Extract-

Fresh pineapple peels had been collected. Now, the fresh peel extract was made individually using 200 grams of fresh pineapple peels gathered. Seize a 1000ml conical flask, fill it with 1000ml of deionized water, and add 200g of fresh peels. The mixture should be stirred at 80°C for 60 minutes using a magnetic stirrer, and then allowed to cool. After the combination reaches room temperature, it was extracted by straining the liquid with the aid of whatmann filter paper. The filtrate obtained is the prepared pineapple peel extract, which can be stored at 4°C and utilized within a week [9].



Figure 3- Pineapple Peels



Figure 4- Stirring of the Mixture



Figure 5- Fresh Pineapple Peel Extract





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2) Preparation of Pineapple Peel-Zinc Nanoparticles (PA-Zn Nps)-

Pineapple Peel-Zinc nanoparticles (PA-Zn Nps) were made by combining freshly obtained pineapple peel extract and zinc sulphate solutions in the appropriate proportions. First, make a 0.02M ZnSO4 solution by dissolving 5.751 grams of zinc sulphate granules in 1000 mL of water. Now, this prepared 0.02M ZnSO4 is mixed with previously prepared fresh peel extract in a 5:2 ratio. The mixture should be stirred for 30 minutes using a mechanical stirrer at room temperature. After 30 minutes, the pH of the solution was measured and corrected by dropping some amount of 0.1 N NaOH solution upto the pH of the mixture reached to 8. The mixture was again stirred for 60 minutes. The stirred mixture is then centrifuged at a speed of 4000 rpm for 5 minutes [10]. Eventually, the obtained centrifuged pellets were collected into a petty dish and dried at a temperature of 65°C in a vacuum oven for about to a time period of 3 to 5 hours. Finally, the obtained dried pellets are crushed using a mortar and a pestle, therefore, the desired PA-Zn Nps are obtained and were stored carefully.

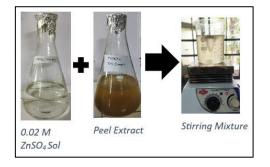




Figure 6- Stirring Mixture Preparation

Figure 7- Dried PA-Zn Nps

- C. Characterization of (Ananas Comosus) Pineapple Peel-Zinc Nanoparticles (PA-Zn Nps):
- 1) Scanning Electron Microscopy (SEM):

In Figure 8, there is a visible distribution, with some particles being finer and others slightly larger, contributing to a **granular or particulate morphology**. The overall size range varies in the range of 50- 250 nm which is consistent with a nanomaterial.

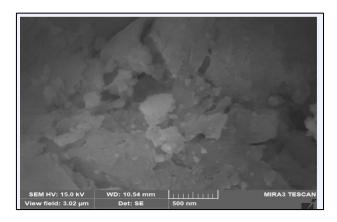


Figure 8- SEM image of PA-Zn Nps

2) X-Ray Diffraction (Analytics):

Figure 9 presents the XRD pattern, with its maximum intensity centered at peak positions of 22.920, 24.940, 25.310, 26.520, and 32.9928. The pattern is indicative of an **amorphous**, **non-crystalline and a disordered material** as well as also indicates that it's a nanocrystalline material.





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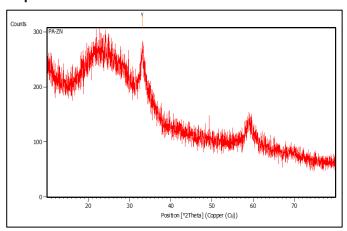


Figure 9- XRD Pattern of PA-Zn Nps

III. RESULTS AND DISCUSSIONS

The effects of contact duration, dose, solution pH, beginning concentration, and solution temperature were examined in order to maximise the percentage of Bisphenol A eliminated from the produced Bisphenol A (BPA) solution.

A) Characterization of PA-Zn Nps

1) Fourier Transform Infra-Red Spectroscopy (FTIR):

Figure 10 shows the FTIR spectrum of pure PA-Zn Nps. The peaks at various places correspond to distinct functional categories. The majority of functional groups consist of carboxylic, hydroxide, and C=C groups. The peaks at 2450cm-1 and 2500cm-1 for PA-Zn Nps before and after FTIR may be ascribed to the OH groups, CH2 groups, C=O stretching, NO2 aromatic nitro compound, C-O stretching, C-N stretching, and CH bending vibrations, respectively.

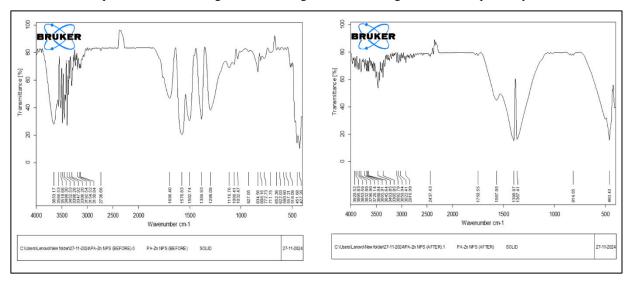


Figure 10- FTIR before and after analysis of PA-Zn Nps

B) Experimental Parameters:

Using PA-Zn Nps as an adsorbent, experimental data is gathered in batches to report the impact of various factors on Methylene Blue dye removal from aqueous solution (made in the lab). Experimental analysis provides the information on how various factors impact dye removal, followed by theoretical explanation of the results by graphical analysis.





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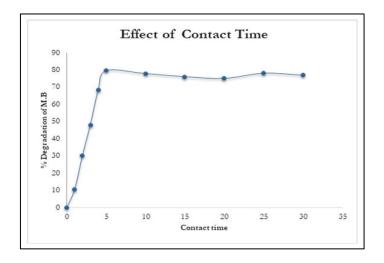
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The present reported data comprises a series of experimental runs. The parameters investigated include contact time (t), sorbent dose (W, g/L), solution pH, starting concentration (Ci, mg/L), and temperature (T,K).

1) Effect of Agitation Time (t)-

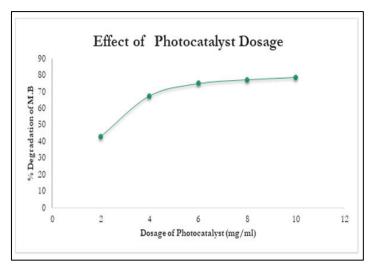
The plot shows variation of agitation time of dye removal at intervals of 0, 5, 10, 15, 20, 25, 30 and 35 minutes. The experimented data of the dye removal on used PA-Zn Nps revealed that a constant removal needed 5 minutes of contact time. As a result, the agitation time for this PA-Zn Nps adsorbent has been set at 5 minutes, as shown in Graph 1, which is the equilibrium period for future photocatalytic studies with varying parameters.



Graph 1- Effect of Agitation Time

2) Effect of Photocatalyst Dosage (W)-

The photocatalyst dose is raised from 0.162 to 0.32g 12 mg to assess the effect of dosage on dye removal. The agitation duration is set at 205 minutes. The proportion of dye removed rises with adsorbent dose and then fluctuates, as shown in Graph 2. At 0.2 g/LAt 10 mg/mL, the highest proportion of a 100ppm dye solution is removed. As a consequence, the ideal dose has been chosen at 0.2 g/L10 mg/mL for further tests with varied parameters.



Graph 2- Effect of Photocatalyst Dosage





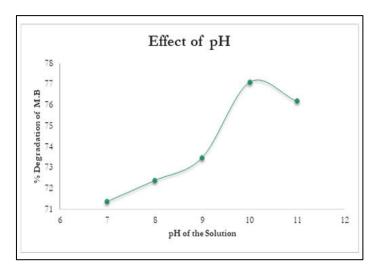
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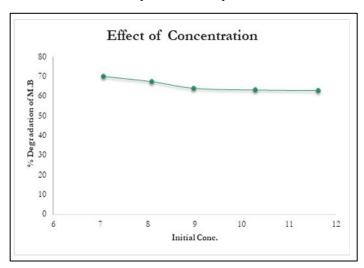
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3) Effect of Solution pH-

Graph 3 depicts the influence of beginning pH on dye removal with PA-Zn Nps in the solution. The proportion of dye removal increased from pH 6 to pH 12, but subsequently dropped as pH rose to 10. Thus, 10 was established as the ideal pH.



Graph 3- Effect of pH



Graph 4- Effect of pH

4) Effect of Initial Concentration of Aqueous Dye Solution-

Graph 4 shows the variation of elimination % with starting concentration. The graph revealed that when the original dye concentration grew, the proportion of dye removed dropped. As a result, the Optimal Initial Concentration of dye Solution is set at 10ppm.

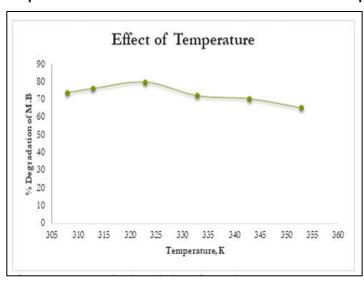




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Graph 5- Effect of Ph

3.2.5 Effect of Temperature (K)-

For constant temperatures as shown in Graph 5, the removal nature rose with temperature, for the initial concentration of 10ppm. Thus, this determines that the photocatalytic process involved in the experimentation is endothermic and therefore, the optimal temperature is determined as 50°C.

C) Photocatalytic Kinetics:

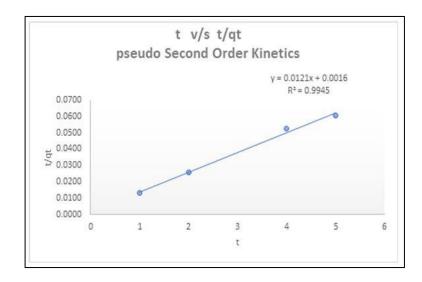
The pseudo 1st order and 2nd order kinetics are studied from the graph below and it is observed that reaction fits pseudo second order more promptly. As shown in the Graph 6.

q = Adsorption capacity (milligram of methylene blue dye adsorbed per gram of EuTiMg Nc) at some time t in minutes. qe = Equilibrium Adsorption capacity in (mg/g).

$$q = [(C_0 - C_f)/W] *100$$
 (1)

where C₀ and C_f are the initial and final concentrations in ppm respectively.

W is the optimum dosage in milligrams of EuTiMg NC per ml of Methylene blue solution.



Graph 6- Second Order Kinetics





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Calculating the value of rate constant from the line equation of the plot time V/s t/qt,

The equation obtained was y = 0.0121x + 0.0016

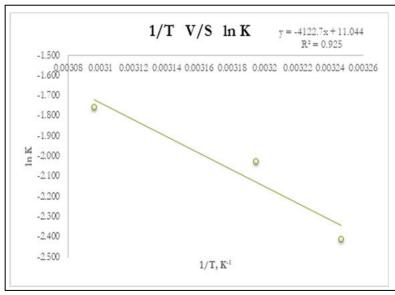
From the equation, positive value of slope gives the rate constant of the reaction process. Therefore, the rate constant for the degradation reaction of MB dye = 0.091 g/mg.min.

Equilibrium adsorption capacity, experimentally qe (exp) = 82.510 mg/g.

Equilibrium adsorption capacity, theoretically qe = 82.644 mg/g.

D) Thermodynamic studies:

The plot of ln k Vs 1/T was shown in the Graph 7 below.



Graph 7- Thermodynamic Studies

From the graph shown above the Graph 7, comparing the line equation with the Arrhenius law equation, the slope gives the value of activation energy and when the line equation was compared with the Vant Hoff's equation, the values of ΔH , ΔS and ΔG were determined.

Arrhenius law equation is
$$\ln K = [(-Ea/R) * (1/T)] + \ln A$$
 (2)

A line equation obtained from the plot drawn between ln K Vs 1/T for finding the activation energy. The slope of this line equation gives the value of activation energy.

The line equation obtained from the plot was y = -4122.7x + 11.044 Therefore, Slope = (-Ea/R), Ea = -(-4122.7*8.314) =34,276.12 J/mol. From VantHoff equation is ln K = - (Δ H/RT) +(Δ S/R) The linear equation obtained was y = -4122.7x + 11.044 Δ H = 34.276 KJ/mol, Δ S = 91.819 J/mol K. Δ G = Δ H - T Δ S = -RTlnK = 34.276 - (323*91.819) = -29.623 KJ/mol.

IV. CONCLUSION

- Maximum Dye removal occurred after 5 minutes of agitation.
- Increasing the photocatalyst dosage resulted in higher percentage removal, with the Optimum Dosage of 10 mg/mL.
- The pH vs % elimination of dye plot revealed considerable adsorption at a pH of 10.

(3)





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- Increasing the initial dye concentration resulted in lowering dye concentration, with 10 ppm being the optimal value.
- The percentage removal of dye rose as the temperature increased, and the optimal temperature dye removal on to the Pineapple peel zinc nanoparticles is 50° C.

The kinetics of BPA adsorption on PA-Zn Nps can be better explained using **pseudo second-order kinetics**. The thermodynamic parameters such as free energy, enthalpy, and entropy changes are computed. The positive ΔH^0 value of 34.276 KJ/mol provides that adsorption is **endothermic**. As temperature (T) increases, so does the equilibrium constant (K_c). The entropy shift of 91.819 J/mol-K indicates **reduced randomness** at the solid-solution interface, whereas no substantial variations may arise in the adsorbent's internal structure. The reported ΔG^0 is -29.623 KJ/mol.

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