Adsorption of Iron (II) ions from Simulated Industrial Wastewater utilizing Activated Carbon from Cogon Grass *(Imperata Cylindrica)*

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***Abstract*—In this study, activated carbon derived from cogon grass, an abundantly available and invasive weed, was employed as an adsorbent to Eliminate Iron (II) heavy metal from simulated wastewater. The research covered a series of sequential steps, commencing with the carbonization of cogon grass followed by chemical activation using phosphoric acid. Subsequently, aqueous solutions utilizing ferrous sulfate heptahydrate were prepared, leading to batch adsorption experiments. Analysis of the filtered samples was conducted using Flame Atomic Absorption Spectroscopy. Three pivotal factors—time, pH levels, and the adsorption dosage of activated carbon—were meticulously examined in the design of experiments. A Full Factorial Design was done and it did not only prove to be more economical but also provided a richer dataset. JMP® (SAS Institute) facilitated robust data management and analysis throughout the study. Impressively, the model obtained an R^2 value of 0.9968, signifying a strong fit and the model's representativeness concerning the dataset. Achieving a remarkable 99.33% iron removal, accompanied by a desirability value of 94.97%, highlighted the efficiency of the chosen parameters. Optimal conditions for this notable outcome included a pH of 2, an adsorption dosage of 5 grams, and a 30-minute time duration. The results show that Cogon Grass Activated Carbon (CGAC) is an effective adsorbent for the removal of iron heavy metal from wastewater.**

***Keywords*—Cogon Grass, DOE, adsorption, activated carbon, wastewater treatment**

I. Introduction

Water, the cornerstone of life, remains an indispensable resource critical for the survival of both human and wildlife populations across the globe. However, this essential source is facing an alarming decline in quality due to an array of human-induced factors. The surge in industrial growth, rapid urban expansion, and unchecked population growth have collectively propelled a significant deterioration in water quality in numerous regions [1].

In the dynamic landscape of swiftly urbanizing areas, the diminishing water quality raises heightened concerns, especially with the infiltration of inorganic contaminants [2] like Fe^{2+} or Iron (II) ions. The unregulated activities of industries, the relentless pace of urbanization, and the increasing population impose relentless pressure on water bodies, amplifying pollution levels. This influx of contaminants poses an imminent threat to the delicate balance of ecosystems reliant on these water sources, risking the health of aquatic life and the sustainability of the environment.

The treatment of wastewater emerges as a critical imperative in curbing environmental pollution and safeguarding human health. The discharge of Fe^{2+} ions into water bodies has the potential to induce severe ecological imbalances, posing significant risks to aquatic life and undermining both environmental sustainability and public well-being.

High concentrations of iron can alter water chemistry, affecting the solubility of nutrients and minerals crucial for aquatic plant and animal life. According to [3], consumption of water contaminated with high levels of iron can lead to adverse health effects, including gastrointestinal distress, and nausea. Prolonged exposure to iron-contaminated water may also increase the risk of developing iron overload disorders, such as hemochromatosis, which can result in liver damage, diabetes, and other serious health complications.

Conventional methods for removing Fe^{2+} often involve costly and chemical-intensive processes. However, the exploration of adsorption techniques, notably through the utilization of activated carbon sourced from natural origins, presents an environmentally friendly and potentially efficient alternative for remediating wastewater [4]. Within this framework, the current work explores the application of cogon grass-derived activated carbon for the adsorption of iron (II) ions in wastewater, to fill in research gaps and address associated problems. This research is novel because it focuses on sustainable alternatives to standard activated carbon—specifically, cogon grass is often viewed as a nuisance due to its rapid growth and invasive nature. By converting this biomass into activated carbon, researchers can repurpose an otherwise underutilized waste material. This study aims to investigate the potential of activated carbon generated from cogon grass as an efficient adsorbent for wastewater treatment.

This study aims to contribute valuable insights and support ongoing efforts in sewage adsorption treatment research by exploring the potential of cogon grass, an abundant material currently underutilized due to its lack of known value.

Aligned with the United Nations Sustainable Development Goal (UN SDG) 6 – Clean Water and Sanitation, the priority to address water quality concerns resonates profoundly. This goal underscores the key importance of ensuring access to clean water and sanitation for all, emphasizing sustainable water management practices and addressing issues stemming from industrial discharge-induced water pollution [5]. In this pursuit, the exploration of innovative and eco-friendly

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solutions like adsorption techniques holds promise in advancing the agenda for cleaner water resources and sustainable environmental practices.

II. Literature Review

A. Activated Carbon

Activated carbon (AC) has emerged as a versatile adsorbent extensively utilized for the removal of contaminants from water and gas. Traditionally sourced from wood, coal, coconut shells, and other materials, AC boasts high porosity and sorption capacity. Its effectiveness in eliminating harmful impurities has made it indispensable in water treatment [6]. The production of AC from agricultural and waste materials has proven to be a cost-effective, high-capacity sorbent, and green alternative to previously used nonrenewable sources [7]. Its reputation as a potent adsorbent comes from its wide surface area, numerous active adsorption sites on the surface, and exceptional adsorption capacity [8].

The characteristics of activated carbon are contingent upon the raw material and the activation process employed, which can be physical or chemical [9]. Physical activation involves carbonization or pyrolysis of the raw material, resulting in variations of activated carbon forms: powdered, granulated, or pelletized. Each form exhibits distinct adsorption properties, determined by the source and activation method [10]. To reiterate, despite its efficacy in removing contaminants, the high production cost of AC restricts its widespread use. This limitation has spurred research into more economical production methods.

B. Cogon Grass (Imperata Cylindrica)

Cogon Grass (CG) is a persistent grass [11] species found in various regions globally. Its prevalent presence makes it easily accessible and a potentially sustainable resource for use in wastewater treatment processes. CG possesses attributes conducive to the development of activated carbon with desirable adsorption properties. There is a growing focus on agricultural waste as a natural sorbent due to its rich lignocellulosic content, environmentally friendly attributes, non-toxic and degradable nature, as well as its widespread availability and cost-effectiveness [12]. Its fibrous nature and structural composition provide the potential for creating activated carbon with suitable pore structures, enhancing its adsorption capability.

C. Adsorption Process

Adsorption, recognized as a reliable and environmentally benign technique, stands out as a promising approach for metal removal from wastewater. AC, owing to its exceptional adsorption capacity and surface characteristics, has emerged as a versatile adsorbent [13]. Chemical activation is a process used to enhance the porosity and surface area of carbonaceous materials and transform them into activated carbon with superior adsorption properties. Unlike physical activation, which primarily involves the use of high temperatures in an inert atmosphere, chemical activation utilizes chemical agents to create porosity. The process typically involves impregnating the carbonaceous precursor material with a chemical activating agent. Commonly used chemical agents include phosphoric acid, potassium hydroxide, or zinc chloride. These agents penetrate the precursor material, reacting with the carbon atoms, and promoting the development of pores and active sites.

This study aims to investigate and optimize the adsorption efficiency of iron (II) from simulated industrial wastewater using Cogon Grass Activated Carbon (CGAC). Moreover, it will underscore the utilization of locally available biomass resources for producing value-added materials, aligning with the principles of circular economy and environmental sustainability.

D. JMP Software

JMP® (SAS Institute) serves as a robust statistical analysis and discovery platform, empowering scientists, engineers, and business analysts to delve into data exploration and derive meaningful insights. Within this environment, statistically designed experiments stand out as a potent method to initiate the discovery process [14]. JMP's strength lies in its ability to facilitate well-designed experiments, resulting in data rich in information.

A substantially constructive study should not only address methodological flaws but also embrace a more rigorous experimental design. This entails using valid measures of core variables and implementing a systematic adjustment of important factors to assess their independent and combined impacts on the phenomenon under study [15]. To investigate their impacts on the adsorption process, specific parameters are specifically chosen and varied within predetermined limits, such as adsorption dosage, pH levels, and contact time. Furthermore, following good experimental design guidelines improves the repeatability and generalizability of study results, providing a strong basis for additional research and real-world applications in pollution removal and wastewater treatment.

Notably, the prediction profiler feature stands out as a powerful tool, enabling researchers to forecast future outcomes by manipulating study parameters [16]. This interactive tool showcases the effects and correlations of each factor, providing a visual representation that simplifies comprehension of process dynamics and relationships.

The Full Factorial Method serves as a comprehensive experimental design strategy employed in research studies including those investigating adsorption processes like the removal of Fe (II) from solutions. This method systematically evaluates all possible combinations of factors and their levels, allowing researchers to assess the individual and combined effects of various parameters [17]—such as adsorbent dosage, contact time, and pH—on the % Fe Removal. Various studies, including research by [18], have emphasized the effectiveness of Design of Experiments (DOE) compared to traditional One-Factor-At-A-Time (OFAT) experiments. DOE methodologies have demonstrated their effectiveness in enhancing cost-efficiency and elevating accuracy and precision through statistical approaches. This method provides a comprehensive understanding of how changes in each factor independently and collectively impact the response variable.

III. Materials and Methods

A. Chemically Modified Activated Carbon Preparation

The cogon grass used for the study was obtained from

Cabuyao, Laguna, Philippines. The collected CG was washed with deionized water to remove dirt and unnecessary substances clinging to it. It was naturally dried for 6 days in the presence of sunlight. Then it is oven-dried at 110°C for 2-3 hours until constant weight is achieved. The oven-dried cogon grass was carbonized in a sealed cylindrical container at 400°C for 30 minutes in the absence of oxygen. It was crushed using mortar and pestle and passed through a 63 µm mesh sieve. The cogon grass-derived pure carbon underwent chemical activation to enhance both its surface area and pore structure. This process involved impregnating the cogon grass powder with a 40% H_3PO_4 solution in a 1:5 weight ratio. Subsequently, the mixture was heated by means of oven drying at 500°C for 2 hours to ensure thorough incorporation of the chemicals within the particles.

Sieving was then done once again through a 63 µm mesh, cooling to room temperature, and was finally stored in an air-tight container for subsequent analyses and applications, designated hereafter as CGAC (Cogon Grass Activated Carbon).

B. Simulated Industrial Wastewater Preparation

A stock iron solution was prepared by dissolving iron sulfate heptahydrate ($FeSO_4{\cdot}7H_2O$) with distilled water in a volumetric flask to create a solution with a concentration of 200 mg/L. The solution is then transferred into a proper container.

Table 1. 2 Levels of the factors considered for the Full Factorial DOE

Level	pH	Adsorbent Dosage (g)	Contact Time (min)
Low	2	1	15
High	9	5	30

C. Batch Adsorption Experiments

Table 1 shows the Low and High levels of each of the predetermined parameters that is the basis of generation of Table 2. 100 mL of Iron (II) solutions were added in an Erlenmeyer flask and the pH was adjusted to predetermined values according to the DOE matrix in Table 2 using NaOH or HCl solution. CGAC was then added to the flasks at different amounts along with adjusted adsorbent dosage and pH according to the same table, all of which are at room temperature. The contents of the flask were mixed using a magnetic stirrer. All the samples were filtered using filter paper, and the filtrate was analyzed for Fe (II) concentration with a Flame Atomic Absorption Spectroscopy (FAAS). FAAS is an analytical technique employed to determine the concentration of metals within a sample. The percent removal of iron (II) ions is calculated with the equation

$$\%Fe\ removal = \frac{(c_i - c_f)}{c_i} \times 100 \quad (1)$$

where C_i and C_f are the initial and final phosphate concentrations of the solution in mg/L.

The JMP software was employed to input and analyze the impact of pH level, adsorption dosage, contact time, and the resulting percentage removal of Fe (II). This analysis aimed to identify the optimal combination of parameters that would yield the highest %Fe removal, utilizing the software's prediction profiler tool.

IV. Result and Discussion

Adsorbent dosage determines the availability of adsorption sites, directly influencing the capacity of the activated carbon to remove contaminants. Findings indicated a direct correlation: higher adsorbent dosages yielded superior results. Generally, this relationship is attributed to the increased availability of adsorption sites for iron, theoretically suggesting that higher dosages would lead to enhanced performance. However, there exists a threshold for optimal dosage. Excessive adsorbent quantities can saturate the available adsorption sites on CGAC, diminishing its effective utilization and consequently reducing its adsorption capacity noticeably.

Contact time reflects the duration for which the adsorbent interacts with the solution, affecting the extent of adsorption. pH levels, on the other hand, modulate the surface charge of the adsorbent and the speciation of the target contaminants, thereby influencing their adsorption behavior. The experiment revealed effective iron removal at a solution pH of 2, attributed theoretically to the heightened presence of protonated active sites, specifically amino, carboxyl, and phosphate groups, which might have amplified iron adsorption. However, as the solution's pH increased to 9, a decline in iron removal was observed.

Table 2. DOE Matrix and %Fe removal result

Leg	Pattern			Adsorption Dosage (mg)	pH	Time (min)	%Fe Removal
1	-	-	-	1	2	15	97.64%
2	-	-	+	1	2	30	97.92%
3	-	+	-	1	9	15	88.95%
4	+	-	-	5	2	15	98.96%
5	0	0	0	3	5.5	22.5	95.37%
6	+	+	-	5	9	15	93.28%
7	+	-	+	5	2	30	99.43%
8	-	+	+	1	9	30	89.81%
9	+	+	+	5	9	30	93.13%

This decline can be attributed to the reduction in hydrogen ion concentration, diminishing the positive charge intensity on the CGAC surface. The low pH value could potentially have adverse effects due to its acidic nature in practical applications.

The pH of the solution significantly influenced its electrochemical properties, directly impacting the coupling of hydrogen. This coupling not only affected the liquid's surface tension but also dictated the form of iron ions present in the water. Hence, the solution's pH emerged as a critical factor influencing Fe (II) adsorption.

In the initial phase of the experiment, a remarkable increase in iron adsorption was observed. This surge was primarily due to the abundant availability of adsorption sites in the early stages. The uptake of iron ions followed a two-stage pattern: an initial rapid phase succeeded by a slower one. When the quantity of available sites exceeds the number of metal species to be adsorbed, the adsorption process accelerates.

The parameters—adsorption dosage, pH, and contact time—stand as influential factors in determining adsorption efficiency. To assess the impact of each parameter, this stage of the experiment maintained constant temperature (room temperature) while varying adsorption dosage (1, 3, and 5 mg/L), pH levels (2, 5.5, and 9), and contact time (15, 22.5, and 30 min). This systematic variation aimed to discern the specific effects of these factors on the adsorption process.

The experimental results are presented in Table 2. The JMP software's prediction profiler feature proves invaluable in optimizing outcomes and identifying precise values for individual parameters. This functionality proves instrumental in overcoming financial and resource constraints for current and future researchers. The lowest percent removal of iron was obtained at Leg 3 with 88.95% removal.

Employing in Leg 7 an adsorbent dosage of 5 g, pH of 2, and a contact time of 30 min yields a remarkable 99.43% removal of iron from the solution. Validation runs were omitted due to the alignment of the optimal condition, as indicated by the prediction profiler's outcome, with the settings of Leg 7, demonstrated in Fig. 1. These settings projected a removal percentage of 99.33% alongside a desirability rating of 0.9497. Comparing the two values in terms of percent difference (%diff), it only results in a value of 0.1007 %. The %diff provides a quantitative measure to evaluate the disparity or similarity between two numerical values. That specific value of %diff implies an exceedingly small variation or discrepancy between these values. It suggests that the difference between the two quantities is notably minimal relative to their average, emphasizing their proximity or near equivalence. This minute difference signifies a high level of similarity or consistency between the compared values.

Table 3. Statistical Treatment of Effect Summary

Parameter	Log Worth	p-value
Adsorbent Dosage	1.796	0.01599
pH	2.732	0.00185
Contact Time	0.440	0.06032

Log worth, which is in Table 3, is essentially a transformed representation of the significance of effects in a factorial design. It's a logarithmic transformation of the p-value associated with each effect in an experimental design. This transformation helps in visualizing the significance levels more clearly, especially when dealing with multiple effects or several factors simultaneously.

This transformation compresses the range of p-values into a more manageable scale. Larger log worth values indicate more significant effects, while smaller values represent less significant effects.

In Table 2, the results demonstrate the significant influence of pH and dosage on iron (II) removal in the solution, warranting the rejection of the null hypothesis. Meaning, that there exists a significant relationship between at least one of the parameters and the % Fe Removal from the solution.

The p-value represents the threshold for rejecting the null hypothesis, indicating statistical significance. According to [19], a p-value below 0.05 indicates the statistical significance of both the main and interaction effects of variables. When a factor demonstrates a p-value equal to or less than 0.05, it signifies significance, thereby prompting the rejection of the null hypothesis. This threshold indicates that there's less than a 5% probability that the observed results are purely due to random chance.

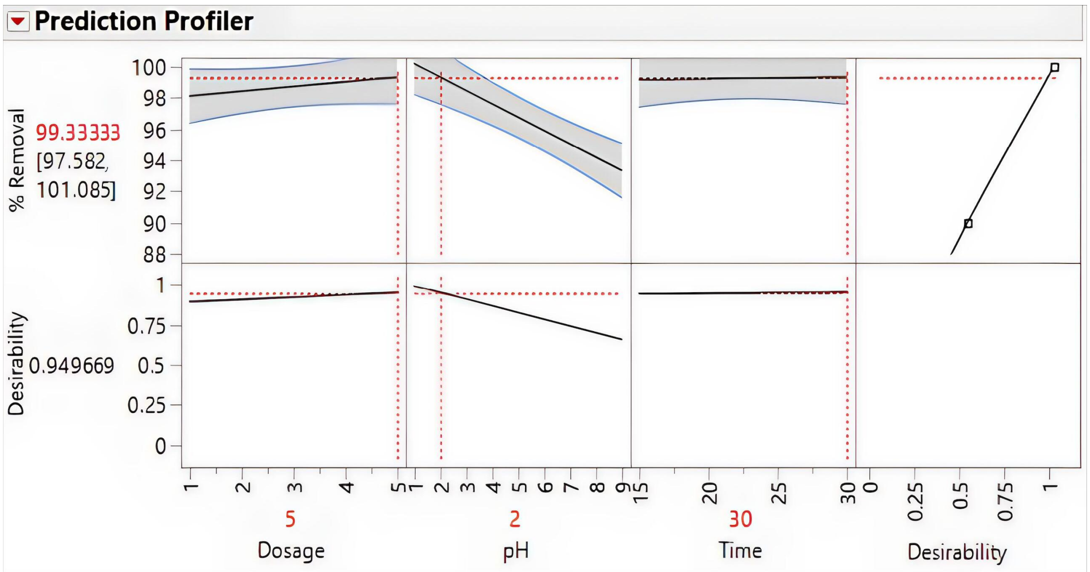

Fig. 1. JMP® prediction profiler outcome.

In the context of the study or analysis, it signifies that the variables under consideration—both their main effects and potential interactions—are likely not due to random fluctuations but are indeed influential or impactful factors. Consequently, it leads to the rejection of the null hypothesis in favor of the alternate hypothesis, highlighting the meaningfulness of the observed effects.

The Pearson product-moment correlation coefficient, as depicted in Table 4, often referred to as Pearson's correlation coefficient, measures the strength and direction of the linear relationship between two continuous variables [20]. According to [21], the value of this coefficient ranges between -1 and +1. A correlation coefficient close to +1, such as the value for pH, indicates a strong positive linear relationship, meaning that as one variable increases, the other tends to increase as well. A correlation coefficient around 0 suggests a weak or no linear relationship between the variables, such as that for adsorbent dosage and contact time.

However, it's important to note that correlation does not imply causation [22]. Even if two variables have a strong correlation, it doesn't necessarily mean that changes in one variable cause changes in the other; it simply implies a relationship in their changes.

Table 4. Pearson Correlation Test results between the %Fe removal versus each of the parameters: adsorbent dosage, pH, and contact time

Parameter	Pearson Product Moment Correlation
Adsorbent Dosage	0.0672
pH	0.9254
Contact Time	-0.0467

Additionally, the Pearson correlation coefficient measures only linear relationships. It might not capture nonlinear associations between variables, and it doesn't account for other types of relationships or dependencies that might exist.

While these results provide valuable insights, a deeper investigation into the interactive effects could significantly enhance the overall study. This is particularly crucial as the findings contrast with those from some analogous adsorption studies involving similar contaminants.

The inability to provide cost information and offer details on the production costs related to using cogon grass as a raw material for the creation of activated carbon is one of the study's limitations. This restriction results from a lack of production facilities and limited resources, which made thorough cost analysis impossible. Nevertheless, given the importance of cost factors in determining the viability and scalability of these processes, future studies should try to overcome this constraint by looking into efficient manufacturing techniques and offering comprehensive cost analysis.

V. Conclusion

In this study, activated carbon was produced from cogon grass, an invasive weed, through carbonization and chemical activation with phosphoric acid. Batch adsorption experiments were conducted using simulated industrial wastewater spiked with ferrous sulfate heptahydrate. The effects of contact time, pH, and adsorbent dosage on iron (II) removal were investigated using 2^k+1 Full Factorial DOE. JMP software was employed to evaluate the effectiveness of CGAC as an adsorbent using pertinent statistical analyses. Notably, employing 5 g of CGAC at pH 2 and a contact time of 30 min resulted in a remarkable 99.43% removal of iron (II) from the solution. However, variations in adsorbent dosage influenced removal efficiency, with higher dosages yielding superior results due to increased active sites for adsorption comparing Leg 2 and Leg 7 where the pH and contact time are constant.

While this study demonstrates the potential of CGAC as an effective adsorbent, financial constraints during the COVID-19 pandemic hindered the comprehensive characterization of the material. Future research should focus on characterizing CGAC to optimize its efficiency and explore its applicability in diverse adsorption applications. Comprehensive analyses, including surface area analysis, pore size distribution, and determination of functional groups, elucidate the material's porosity and surface chemistry, directly influencing its adsorption capacity. Surface morphology examination provides insights into structural aspects impacting adsorption behavior. Additionally, Fourier Transform Infrared Spectroscopy (FTIR) can identify functional groups on the CGAC surface, correlating them with adsorption capabilities.

Conflict of Interest

We declare that there is no conflict of interest about the research conducted or the publication of its findings.

Author Contributions

DB. Centeno, KMM. Pedrosa, and J. Tumamao played primary roles throughout the research process. Their contributions encompassed material procurement, laboratory work, and statistical analysis, prominently utilizing JMP. TRL. Panaligan and JR. Aquino provided guidance and direction to the group throughout the entirety of the project.

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