

Article

Valorization of *Vigna trilobata* Rind Waste into Activated Carbon for Efficient Iron Removal from Aqueous Solutions

Vamsee Krishna Kodali ¹, Randhi Uma Devi ^{1,*}, K. Sri Lakshmi ², Damaraju Lakshmi Lavanya ³
and Bala chandu Koya ⁴

¹ School of Science, GITAM Deemed to be University, Hyderabad 502329, Telangana State, India; vkodali@gitam.in

² Geethanjali College of Engineering and Technology, Hyderabad 501301, Telangana State, India

³ Vignana Bharati Institute of Technology, Hyderabad 501301, Telangana State, India

⁴ B V Raju Institute of Technology, Narsapur 502313, Telangana State, India

* Correspondence: urandhi@gitam.edu

Abstract

Iron (Fe) contamination of water sources has become an increasing environmental concern, creating the need for effective, environmentally friendly, and cost-effective technologies for Fe(III) removal from aqueous systems. In the present work, the possibility of using the sulfuric acid-activated carbon made of *Vigna trilobata* rind waste for treating water contaminated with Fe ions was explored. The characteristics of the synthesized material were identified by physical, chemical, and spectroscopic methods, and its Fe ion sorption efficiency was studied experimentally in batch mode under various conditions. Equilibrium, kinetics, and thermodynamics of Fe ion removal by the prepared adsorbent were determined. The obtained adsorbent had a BET surface area of 20.55 m² g⁻¹ and showed high experimental adsorption capacity with the highest observed uptake of 19.81 mg g⁻¹. Based on the experimental results, the equilibrium data could be best described by the Langmuir equation ($R^2 = 0.978$). Kinetic analysis showed that the rate-limiting step in Fe ion sorption was intraparticle diffusion ($R^2 = 0.921$). Thermodynamic calculations indicated that the adsorption process occurred spontaneously ($\Delta G^\circ = -4.31$ to -6.53 kJ mol⁻¹) and endothermically ($\Delta H^\circ = +7.11$ kJ mol⁻¹). A comparative analysis showed that the sorption capacity of the studied adsorbent corresponded to that reported for the analogous materials produced from other biomasses.

Keywords: waste valorization; activated carbon; biosorption; *Vigna trilobata* rind

Academic Editors: Jandro L. Abot
and Jian Sun

Received: 9 June 2026

Revised: 1 July 2026

Accepted: 5 July 2026

Published: 9 July 2026

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1. Introduction

Contamination of water sources by dissolved metals such as iron, manganese, chromium, lead, and cadmium has been an issue of great concern over time due to the detrimental impacts these pollutants pose on water quality and ecosystems. Sources of water pollution by these dissolved metals include industrial discharge, mining, agriculture, and waste disposal [1]. Some of these include industrial activities, agriculture, mining, and ineffective waste disposal practices [2]. Being toxic, having high persistence rates, and tending to bioaccumulate in the food chain make these pollutants very dangerous for both ecological and public health reasons [3]. Hence, pollution from these metals can lead to devastating effects environmentally and health-wise. Therefore, there

is a need for novel solutions to these problems that will have long-term benefits to mitigate them [4]. Dissolved metal contamination in water can be managed through a number of approaches, such as chemical precipitation, ion exchange, and coagulation. In some ways, this process may face certain challenges, such as a lack of efficiency in removing these metals, being costly to operate, and the formation of other pollutants [5]. Using biomass, biosorption has proved to be a better alternative approach that can effectively remove dissolved metals in water. Besides being cheap to operate, it is harmless to the environment. This biological technique entails the use of biomass, which is inert or dead, in absorbing metal ions without changing metabolic, physical, and chemical conditions. The process of treating wastewater becomes easy and cost-effective [6].

Even though there are efforts towards adopting cheap and environmentally friendly adsorbents, agricultural by-products continue to be widely used as adsorbents with a prolonged lifespan. Biosorption has been found to possess great potential in terms of being an alternative way of removing metal ions from the environment using non-living biomass [7], which is in line with the concepts of green chemistry. Activated carbons (AC) from the *Vigna trilobata* rind as sustainable, cost-effective adsorbents to remove iron (Fe^{3+}) ions from aqueous solutions will be evaluated within the framework of this research, which includes extensive adsorption isotherm, kinetics, and thermodynamics investigation [8]. There has been increasing interest in the use of activated carbon as an adsorbent owing to its unique characteristics, including high surface area and tunable pore size [9]. Activated carbons may be produced economically and sustainably through the use of agricultural by-products, including the rind of the *Vigna trilobata* [10].

The selection of *Vigna trilobata* rind waste as the precursor was based on several reasons. First, this agricultural biomass has a relatively high amount in the environment and is of low commercial value. Second, biomass is composed of three major components, cellulose, hemicellulose, and lignin, known to promote porosity development during the carbonization and activation process. Third, *Vigna trilobata* rind waste usually serves no purpose except being disposed of after processing crops and hence represents an ideal choice to produce activated carbon as a valuable adsorbent [11]. Moreover, utilization of this biomass is aligned with green chemistry and circular economy concepts as it eliminates waste while developing a new product that would contribute to solving environmental problems. Lastly, not many researchers have studied the application of *Vigna trilobata*-derived activated carbon in dissolved metal removal from the environment.

Recent studies have demonstrated that biomass-derived activated carbons are promising adsorbents for Fe(III) removal owing to their abundant surface functional groups, heterogeneous pore structure, and tunable surface chemistry, all of which strongly influence adsorption performance [12]. Moreover, it is worth noting that the latest trends in adsorption research focus on the incorporation of advanced methods such as BET, Raman spectroscopy, FTIR, and SEM techniques in order to obtain more comprehensive data about the adsorption mechanism and adsorbent performance. Nevertheless, although there has been a rising trend to use agricultural waste in various fields, little attention has been paid to their application in Fe^{3+} ions removal using *Vigna trilobata* rind as a carbon precursor. This paper thus aims to fill this knowledge gap by investigating the adsorption properties of sulfuric acid-activated carbon derived from *Vigna trilobata* rind.

2. Materials and Methods

2.1. Preparation of Activated Carbon

In this study, activated carbon has been synthesized from the rind of *Vigna trilobata*. For this purpose, rind has been subjected to solar drying for 120 h to ensure a reduction in extra moisture in it to achieve a consistent weight [11]. In the next step, dried rind has

been crushed using a combination and further sieved to prepare particle sizes of 250–500 μm . First, biomass has been dried at 120 $^{\circ}\text{C}$ for 48 h to get rid of extra moisture in it. Thereafter, dried biomass has been subject to chemical and thermal treatments to develop activated carbon at a temperature of 800 $^{\circ}\text{C}$. The resulting powder has been used as a biosorbent during the trial period. The careful preparation of the above biosorbent is necessary to provide consistent materials for subsequent activation and adsorption studies. Recently conducted studies have proved that KOH activation is very effective in producing highly developed micro- and mesopores, increasing the specific surface area, and enhancing the adsorption capacity of carbon materials [12]. Even though it is capable of providing extremely high surface areas, sulfuric acid activation has gained popularity because of its easy processing capability, lower cost, better surface modification, and ability to utilize sustainable biomass [13]. In the present study, attention will be paid to the use of sulfuric acid-activated carbon obtained from the rind of *Vigna trilobata* as an environmentally friendly and economical adsorbent to reduce iron ions from the aqueous solution.

For the activation of carbon materials, the solution of sulfuric acid was prepared by mixing concentrated H_2SO_4 and distilled water at a ratio of 1:1 (v/v). Activated charcoal had been subject to treatment with sulfuric acid solution, leading to charring, heat generation, and emission of odor. When the reaction process had been completed, the produced materials were allowed to stay in the air oven at 180–200 $^{\circ}\text{C}$ for 24 h. The product obtained after removing it from the oven was cooled down to room temperature and subsequently washed with deionized water, followed by soaking it in a 1% NaHCO_3 solution to neutralize sulfuric acid. Finally, it was rinsed again with distilled water till reaching a pH of 6, dried at 110 $^{\circ}\text{C}$, and activated at 800 $^{\circ}\text{C}$. Thus, sulfuric acid-activated carbon has been obtained and collected. It is very important to note that the above process is required to develop porosity and a higher surface area, both of which are necessary to facilitate adsorption [14].

2.2. Characterization of Activated Carbon

Properties of the produced sulphuric acid-activated carbon from *Vigna trilobata* rind, such as pH value, electrical conductivity, moisture content, ash content, and solubility, were determined following standard procedures [15]. In order to measure the pH value and electrical conductivity, 10 g of activated carbon was suspended in 100 mL of deionized (DI) water and stirred at 200 rpm for 1 h. Moisture content was determined after drying the sample at 110 $^{\circ}\text{C}$ to a constant mass. Ash content was obtained after combustion of the activated carbon at 800 $^{\circ}\text{C}$ in a muffle furnace. Physicochemical and structural properties, like morphology, elemental composition, surface functional groups, specific surface area, pore volume, and pore size distribution, were determined with the use of X-ray diffraction (XRD), scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM-EDX), Fourier transform infrared spectroscopy (FTIR), Raman spectroscopy, and Brunauer–Emmett–Teller (BET) surface area analysis [16].

2.3. Batch Adsorption Experiments

Removal efficiencies of Fe^{3+} ions from aqueous solutions by *Vigna trilobata* rind-activated carbon were investigated using batch adsorption tests. Stock solutions containing Fe(III) ions were prepared with the help of analytical grade ferric chloride (FeCl_3). Working solutions of the required concentration were prepared by appropriate dilutions of the stock solution. In each batch test, 50 mL of Fe ions was placed in a conical flask of 250 mL and was added with appropriate amounts of activated carbon samples. The mixture was shaken at 150 rpm in an orbital shaker for proper adsorption of Fe ions. After the desired contact time, the suspensions were separated by filtration for the

determination of residual Fe ion concentration in aqueous solution. Effects of pH value, contact time, adsorbent dose, initial Fe concentration, and temperature on the removal efficiency of Fe^{3+} ions were studied.

2.4. Adsorption Modeling and Thermodynamic Studies

The equilibrium adsorption studies were performed to elucidate the adsorption mechanism of Fe ions onto the surface of activated carbon. Batch equilibrium adsorption data were examined based on the Langmuir and the Freundlich models for the purpose of determining the adsorption mechanism, adsorption intensity, and adsorption capacity. Parameters of both models were estimated by means of linear regression analysis. Moreover, the appropriateness of each equation was validated based on the R^2 correlation coefficient. The isotherm study helped us determine the nature of the interaction between Fe ions and the surface of activated carbon. Kinetics studies were done to find the mechanism and rate of Fe ion adsorption by activated carbon [11]. Adsorption experiments were carried out to collect samples at predetermined time intervals, which were then examined to estimate the Fe concentration in solution. Residual Fe ions were further analyzed to determine the Fe ions adsorbed to activated carbon using pseudo-first order, pseudo-second order, and intraparticle diffusion kinetic models. Batch adsorption studies were carried out to examine the adsorption process under different temperatures. Equilibrium constant for thermodynamics (K_c) was calculated from the results of batch experiments, while the standard Gibbs energy (ΔG°) change was calculated using $\Delta G^\circ = -RT \ln K_c$. The values of standard enthalpy change (ΔH°) and standard entropy (ΔS°) change were calculated from the Van't Hoff plot ($\ln K_c$ versus $1/T$). Such thermodynamic parameters were useful for evaluating the feasibility and spontaneity of Fe ions adsorption on the activated carbon surface.

3. Results and Discussion

3.1. Structural Characterization of the Activated Carbon

The characterization findings offered a good basis for understanding the behavior of adsorption by *Vigna trilobata* rind activated carbon. From the XRD analysis, it was evident that there was a broad diffraction pattern obtained for *Vigna trilobata* rind-activated carbon, which had its diffraction peak centered at approximately 22° (2θ). Lack of any sharp and defined Bragg reflections indicated that there was an amorphous structure in it due to low graphitization, which is common for activated carbons produced from a lignocellulosic precursor.

The XRD pattern for *Vigna trilobata* rind activated carbon (Figure 1) shows a broad diffraction peak. Lack of any sharp and defined Bragg reflections confirmed that there was an amorphous structure in it due to low graphitization. This type of broadening is common for activated carbons made from a lignocellulosic precursor, whereby stacking disorder of the carbon layers and structural defects do not allow crystallinity [17]. The particle size distribution of the *Vigna trilobata* rind-based activated carbon sample was analyzed using the laser diffraction particle size analyzer, and the obtained data are depicted in Figure 2. The particle size distribution curve shows that the distribution is unimodal in nature, indicating a relatively uniform distribution of particle sizes in grinding and activation processes.

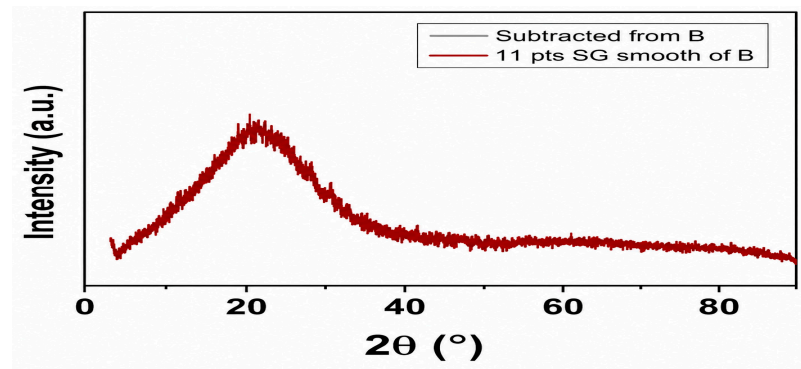


Figure 1. XRD patterns of *Vigna trilobata* rind activated carbon.

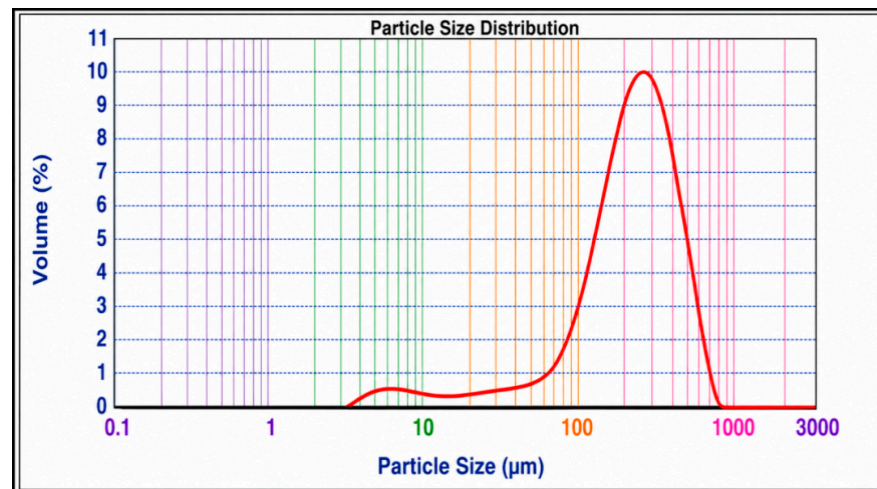


Figure 2. Particle size distribution for the *Vigna trilobata* rind-based activated carbon.

The mean particle size was determined to be 75.3 μm , which is evident from the extremely small value of the standard deviation, indicating that the distribution was uniform. Moderate particle size was desirable for use in adsorption techniques because it ensures sufficient surface area for adsorption without being too large in terms of particle size for easy handling and separation [18]. Diffuse background and absence of crystallized inorganic phases further confirm that there are minimal residues of mineral impurities in activated form. An amorphous structure of carbon matrix is desirable for adsorption since the disorderly arrangement of carbon layers, wide interlayer distance, and increased density of defects result in the development of microporous and mesoporous structures [19]. Flaked, cracked surface and macroporous canals were among some of the features that have been revealed through SEM images characterized by extremely uneven morphology. Dimensions of particles measured ranged from approximately 125–600 μm (Figure 2). The porous and fissured appearance of the particles suggested formation of structural defects during carbonization. These features altogether contribute to the adsorption abilities of the material through providing a sufficient amount of accessible points for metal ion adsorption [20]. This morphology allows rapid transport of the pollutants towards the particle surface, indicating the applicability of the material in environmental adsorption.

SEM analysis showed irregular shape and heterogeneous surface structure of the particles developed during the carbonization and activation processes (Figure 3). Structural features identified on the basis of fractured carbon surfaces indicate the formation of a carbonaceous matrix. Nevertheless, the development of pores was hardly noticeable

at the magnification used in the experiment. Thus, further investigation on the textural properties of the activated carbon was performed using BET and Raman spectroscopy [21]. SEM analysis revealed a heterogeneous and irregular surface morphology resulting from the carbonization and activation processes. Although surface irregularities were observed, the SEM images alone do not provide direct evidence of extensive pore development. Therefore, the observed morphology is interpreted as indicative of surface heterogeneity rather than confirmation of a highly porous structure. Combined with the oxygen-containing functional groups identified by FTIR, these surface characteristics may contribute to the adsorption behavior observed in this study.

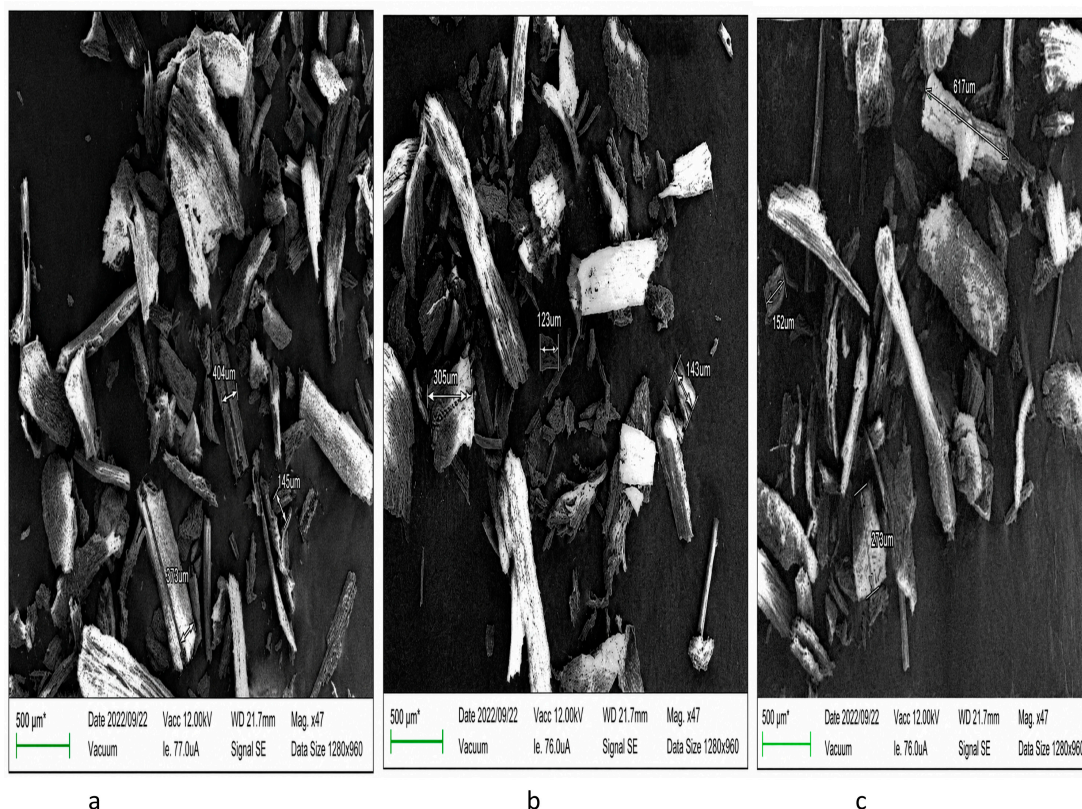


Figure 3. SEM micrographs of *Vigna trilobata* rind-derived activated carbon at different magnifications: (a) low, (b) medium, and (c) high magnification. * indicates the instrument-generated scale bar, corresponding to a length of 500 μm.

3.2. Surface Chemical and Textural Characterization

The FTIR spectrum of the activated carbon from the rind of *Vigna trilobata* (Figure 4) showed a broad absorption peak at 3200–3600 cm^{-1} , which is due to the stretching vibration of the O-H bond of hydroxyl groups and moisture molecules. The weak absorption peak at 2920–2950 cm^{-1} was due to aliphatic C-H stretching vibrations, which suggests the presence of some remaining organic structures as a result of the lignocellulose precursor. The absorption band at 1600–1650 cm^{-1} corresponds to stretching of the C=O and aromatic C=C bonds, suggesting the formation of carbon structures after activation of the precursor material. Other absorption peaks at 1000–1200 cm^{-1} correspond to C-O bond stretches of alcoholic, phenolic, and ether functional groups. The presence of oxygen-containing functional groups on the surface of the activated carbon implies the existence of chemically reactive sites on the surface that can interact with Fe ions by means of hydrogen bonding, electrostatic attraction, and complexing. Thus, the ability of the

adsorbent to absorb metal ions may be due to these chemical sites [22]. While FTIR analysis of the fresh activated carbon revealed the presence of oxygen-containing functional groups for effective adsorption, post-absorption spectroscopy has not been used to compare the adsorptive abilities of Fe-loaded activated carbon.

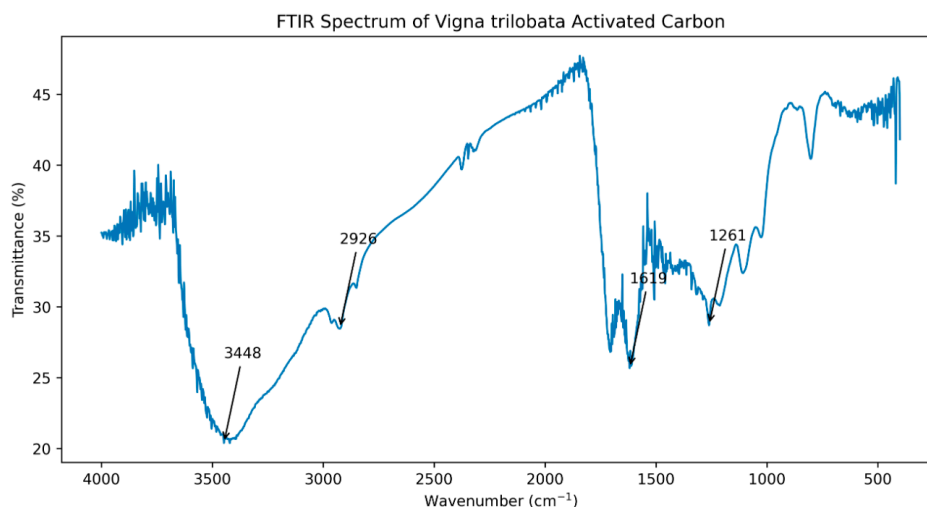


Figure 4. FTIR pattern of *Vigna trilobata* rind activated carbon.

This structural disorder, along with the presence of functional groups having oxygen atoms, identified using FTIR, helps achieve synergy between structural defects and heterogeneity, which are responsible for the enhanced absorption of pollutants. This type of structure formation is necessary for attaining high adsorption capacities in several carbonaceous adsorbents [23]. The results of the Malvern Mastersizer analysis showed that the activated carbon obtained from the rind of *Vigna trilobata* has a polydispersed particle size distribution, with a volume median diameter ($d(0.5)$) equal to 233.64 μm . $d(0.1) = 80 \mu\text{m}$ and $d(0.9) = 469 \mu\text{m}$ demonstrate that both fine and coarse particles were present in the sample. The span value of 1.664 confirms the existence of moderate broadness, common for activated carbons that were mechanically ground. The low specific surface area of $0.0656 \text{ m}^2/\text{g}$, as evaluated from the particle size distribution data, is due to the fact that laser diffraction estimates surface areas based only on geometrical parameters and does not consider internal microporous structures.

The analysis of the particle sizes demonstrated the heterogeneity of activated carbon particles [23]. Particle size variation could influence adsorption properties by changing the dispersion state of particles and affecting the external mass transfer rate, but further investigations should clarify these aspects. To assess the textural parameters of the synthesized activated carbon, BET surface area analysis was performed. It was found that the BET surface area of the material is $20.55 \text{ m}^2/\text{g}$ with a BET constant (C) of 46.99 and an extremely good correlation coefficient ($r = 0.99994$). The BET surface area of the synthesized activated carbon was measured as $20.55 \text{ m}^2 \text{ g}^{-1}$, which is relatively low compared with many commercial activated carbons. Therefore, the observed adsorption performance cannot be attributed solely to surface area. Instead, it is likely influenced by the combined contribution of surface heterogeneity observed by SEM and the oxygen-containing functional groups identified by FTIR. Analysis of the SEM images demonstrated the heterogeneity of the surface, while Raman and X-ray analysis proved that amorphous and partially graphitized carbon layers were present [24].

In order to study the structural properties of the sulphuric acid-activated carbon obtained from the peel of *Vigna trilobata*, the Raman spectroscopy technique was used (Figure 5). It was found that the spectrum contains two characteristic peaks located at

1370.3 cm^{-1} (D peak) and 1572.6 cm^{-1} (G peak). These peaks indicate the presence of carbon structures that have different levels of ordering: disordered carbon (D peak) and graphitic sp^2 carbon (G peak). In addition, it can be stated that the ratio between the intensity of these peaks, $I_D/I_G = 0.28$, indicates a small degree of structural disorder of the studied material and its graphitization. The simultaneous presence of D and G peaks means that carbonization of the biomass precursor took place, and a new carbon structure with amorphous and graphitic areas was formed. This structure provides active sites for ion adsorption and supports the suitability of the prepared activated carbon for wastewater treatment applications [25]. Thus, the performance of the prepared AC in the adsorption process is due to the combined effect of the surface functional groups identified by FTIR and the structural properties revealed by Raman, XRD, and SEM.

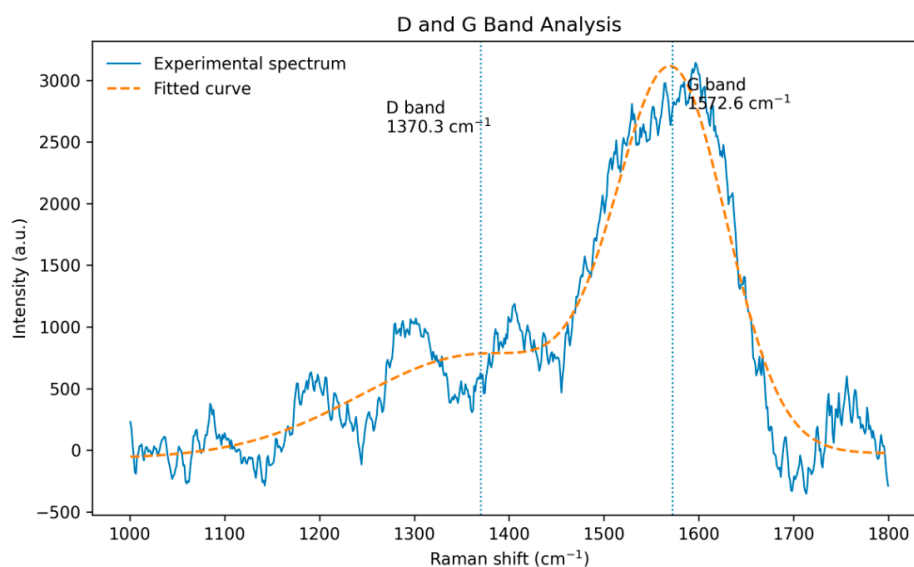


Figure 5. Raman spectroscopy of *Vigna trilobata* activated carbon.

3.3. Biosorption Isotherm Results

Adsorption capacity and kinetics of Fe ions adsorption were studied using the adsorption isotherm and kinetic experiments [26]. Adsorption experiments were carried out using Fe solutions prepared from FeCl_3 with different initial concentrations of iron (0.15–1 ppm), aiming to study the ability of the produced AC as an adsorbent for iron ion removal. Equilibrium adsorption experiments were performed by changing the initial concentrations of iron ions, allowing for the study of the adsorption capacity at the saturation level, while kinetics experiments aimed at determining the rate of the adsorption process. Batch biosorption experiments allowed evaluating the effect of several operational variables on Fe ions removal efficiency, such as pH, adsorbent dosage, contact time, and initial concentration of metal ions [27]. These investigations are crucial in optimizing the conditions of the adsorption process for predicting the performance of the adsorbent. Among the two models considered, namely, Langmuir and Freundlich, the former one described the adsorption data more adequately.

It was found that the adsorption of Fe ions on activated carbon produced from *Vigna trilobata* rind strongly depends on contact time, adsorbent dosage, solution pH, and temperature (Figure 6). As for contact time, Fe removal efficiency increases gradually, reaching the equilibrium state after 120–150 min of the interaction of Fe ions with the activated carbon, which can be explained by the occupation of adsorption sites and interaction of adsorbent and Fe ions. The increase in the adsorbent dosage in the range

0.125–1.0 g increases Fe removal efficiency since there will be more active adsorption sites and a higher surface area for Fe uptake. In addition, the adsorption capacity of AC increases with rising pH value, achieving the maximal values at pH = 8 because of decreased competition between H^+ and Fe and enhanced electrostatic attraction of ions. Finally, the adsorption efficiency is higher at rising temperatures since elevated temperatures favor the mobility and interaction of ions [28].

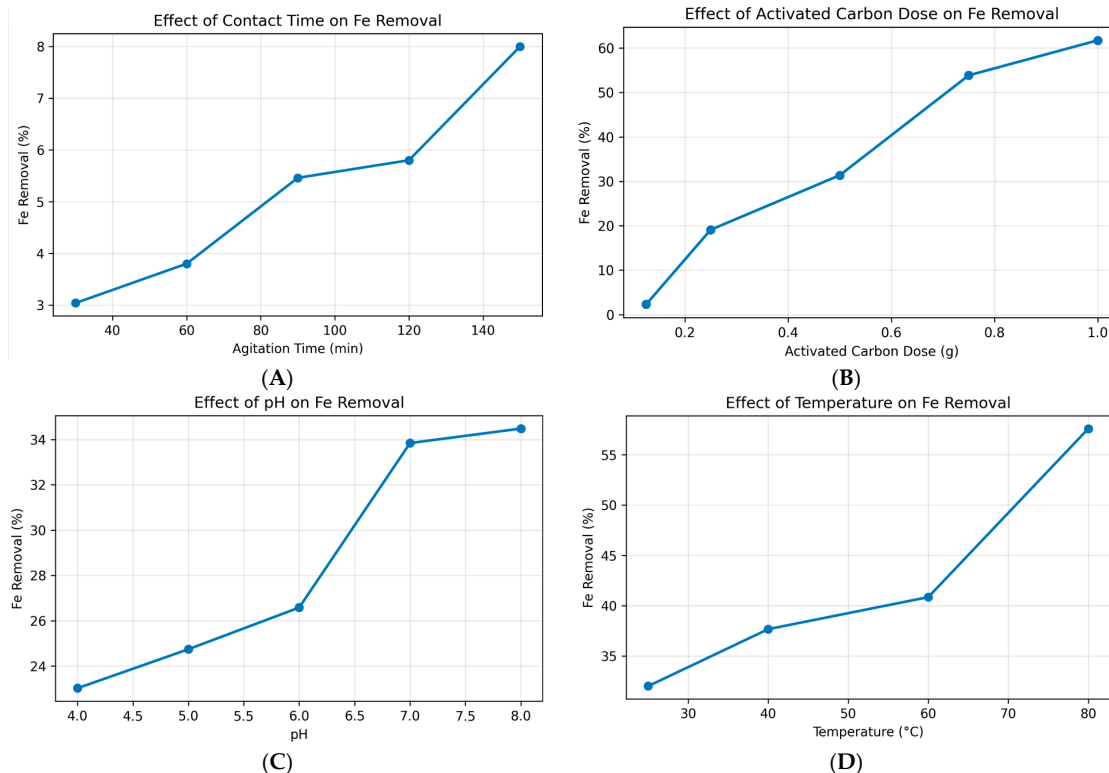


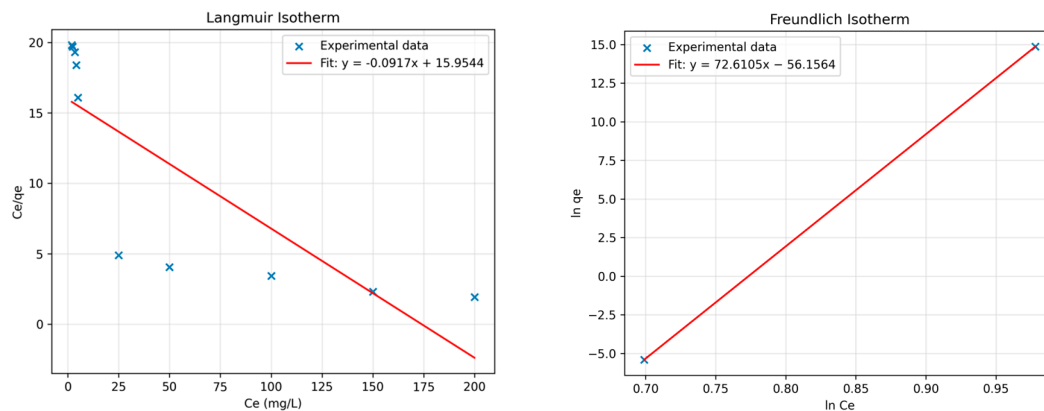
Figure 6. Influence of adsorption parameters on Fe removal efficiency: (A) contact time, (B) adsorbent dosage, (C) pH, and (D) temperature.

The equilibrium adsorption data fit the Langmuir model ($R^2 = 0.978$) better than the Freundlich model ($R^2 = 0.699$). This shows that the Langmuir model describes the adsorption behavior well under the studied conditions (Figure 7). However, a good fit for the Langmuir model does not prove that adsorption occurs only as a monolayer or on a completely uniform surface. Activated carbon has a heterogeneous surface and different pore sizes, as shown by the SEM, FTIR, Raman, and BET results [29]. Therefore, the adsorption process may involve several mechanisms, including surface heterogeneity, pore filling, and interactions between Fe ions and oxygen-containing functional groups on the activated carbon surface. The negative Langmuir and Freundlich constants obtained from the regression analysis indicate that these adsorption models are not fully suitable for describing the present experimental dataset under the investigated concentration range. Such non-physical parameter values generally arise when the experimental data do not adequately satisfy the assumptions of the corresponding isotherm models or when the concentration range is insufficient for reliable parameter estimation. Therefore, these constants were not interpreted in terms of adsorption affinity, and the model fitting results should be considered with caution (Table 1).

Table 1. The isotherm data of the Langmuir and the Freundlich models.

Model	R ²	q (mg/g)	b or n	Kf	Interpretation
Langmuir	0.97807	14.86165	−1.67619	−	Best-fitting equilibrium adsorption model.
Freundlich	0.698935	−	−5.40641	22.97219	Poor fit; lower predictive capability compared to Langmuir model.

Among the evaluated adsorption isotherms, the Langmuir model exhibited the highest coefficient of determination ($R^2 = 0.978$), whereas the Freundlich model showed a comparatively lower correlation coefficient ($R^2 = 0.699$). However, the negative Langmuir and Freundlich constants obtained from the regression analysis indicate that the experimental data do not fully satisfy the assumptions of these models under the investigated concentration range. Therefore, although the Langmuir model provided a comparatively better statistical fit, the estimated model parameters should be interpreted with caution, and the results primarily indicate the limitations of applying these classical isotherm models to the present dataset rather than the adsorption affinity of the adsorbent (Figure 7). The adsorption isotherm experiments were conducted at pH 5 and 25 °C using a fixed adsorbent dosage and solution volume. Understanding the adsorption kinetics is paramount, with pseudo-first-order and pseudo-second-order models frequently employed to differentiate between physisorption and chemisorption rate-limiting steps, respectively [30].

**Figure 7.** Langmuir and the Freundlich isotherm models.

The negative Langmuir and Freundlich constants obtained in this study indicate limitations in applying these models to the present adsorption data. Such non-physical parameter values suggest that the experimental concentration range and regression fitting were insufficient to satisfy the assumptions of these classical isotherm models. Therefore, although the Langmuir model exhibited a comparatively higher coefficient of determination ($R^2 = 0.978$), the corresponding parameter values should be interpreted with caution [31]. This affinity is also proven by substantial amounts of the adsorbed material reported previously for different activated carbons (for example, 10.020 mg g^{−1} for banana peel-activated carbon and 10 mg/g for white lupine HSUK) [32]. In order to increase the reliability of results, it is proposed to conduct further experiments using a broad concentration range for the determination of the whole adsorption isotherm curve before saturation. Thus, a wider range of experimental data is recommended to fit different adsorption models and to reveal all the processes taking place at the interface between solid and liquid phases [33]. The adsorption isotherms were obtained under conditions of relatively low Fe concentration (0.15–1.0 ppm), which was used to simulate pollution in an environmental sample. Even though the Langmuir model had the high-

est coefficient of determination ($R^2 = 0.978$), this concentration range can fail to show some saturation features of the adsorbent. Therefore, it is necessary to take into account this issue while analyzing the adsorption isotherm results and to conduct further investigations on a broad range of Fe concentrations in the future.

It can be seen that the intercept of this model is positive, which means that the rates of two types of diffusion occur—external film diffusion and internal pore diffusion. Thus, the process involves the transport of Fe ions through the liquid film, which surrounds the adsorbent particle, and then their penetration into the adsorbent pores. The fast establishment of equilibrium during 30 min shows the high uptake capacity of this material (Figure 8). The relatively rapid adsorption observed during the initial stage may reflect the availability of surface adsorption sites under the investigated experimental conditions. Moreover, the fast adsorption stage of Fe ions also confirms the high availability of adsorption sites on the material surface and, thus, high activity and surface area of the adsorbent [34].

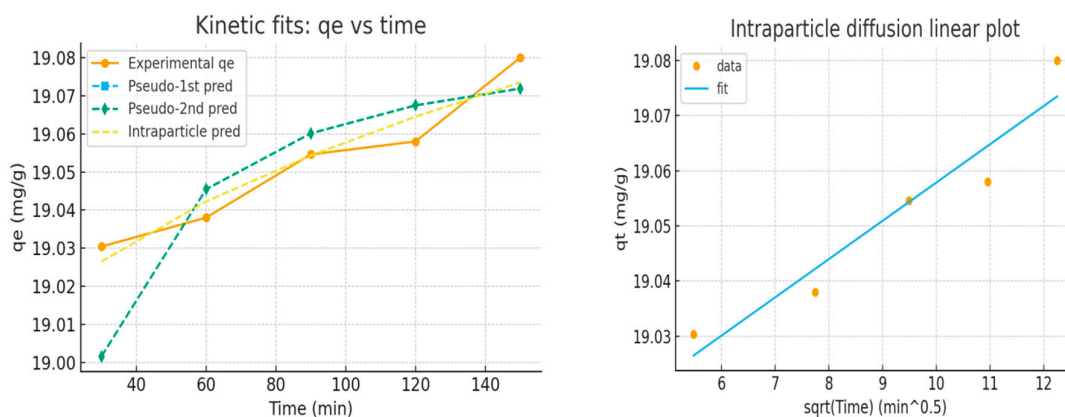


Figure 8. Kinetic fits and Intraparticle diffusion linear plot.

3.4. Biosorption Kinetic Results

Kinetic models such as pseudo-first order, pseudo-second order, and intra-particle diffusion were employed to establish the adsorption rate and mechanism of adsorption of Fe ions (Figure 9). Kinetic models aid in understanding the type of adsorption (whether physical or chemical) and also determine which stage dominates the adsorption process. In addition, the increase in Fe ion adsorption capacity with increasing concentration of the metal ion suggests that the adsorption is a mass transfer-driven process [35]. This behavior is attributed to the increased chances of metal ions colliding with the available active sites at high concentrations. The pseudo-first order model did not yield a satisfactory linear correlation to the experimental data, probably due to the quick establishment of adsorption equilibrium and the limited range of adsorption capacity during experimentation [36]. Accordingly, the kinetic constants obtained using this model were disregarded. Conversely, the intra-particle diffusion model yielded the best correlation coefficient value ($R^2 = 0.921$), implying that intra-particle diffusion contributed greatly to the overall adsorption process [37].

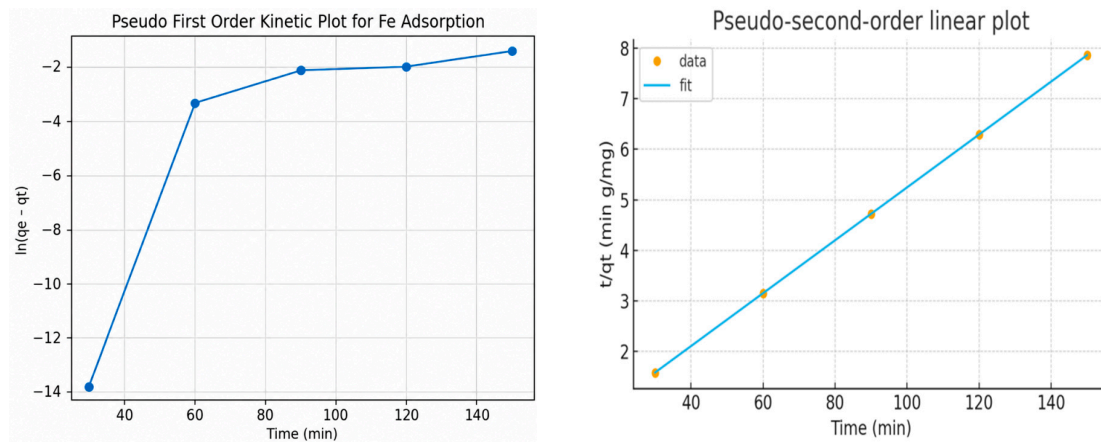


Figure 9. Pseudo first-order and second-order linear plots.

Since the intraparticle diffusion model has the largest R^2 (0.921) and lowest RMSE, diffusion within the pores must be considered an important stage that dictates the speed of the reaction. The fact that the intercept is not zero ($C \neq 0$) in the case of intraparticle diffusion suggests that both film diffusion and pore diffusion contribute to the rate. Thus, in the process, both film diffusion and pore diffusion are significant factors (Table 2). At 30 min, the reaction came close to equilibrium. The pseudofirst-order failed due to fast attaining equilibrium, while the pseudosecond-order did well but gave a low R^2 .

Table 2. Data of pseudo-first-order, second-order, and intra-particle diffusion.

Model	R^2	Parameters	Interpretation
Pseudofirst-order	Not applicable	-	Poor linearity; unsuitable for kinetic interpretation.
Pseudosecond-order	0.279	$\approx 19.09 \text{ mg/g}$, $\approx 0.377 \text{ g} \cdot \text{mg}^{-1} \cdot \text{min}^{-1}$	Low R^2 indicates poor fit, though estimate was reasonable.
Intra-particle diffusion	0.921	$\approx 0.00695 \text{ mg} \cdot \text{g}^{-1} \cdot \text{min}^{-0.5}$, $C = 18.988 \text{ mg/g}$	Highest R^2 among tested models; indicative of diffusion-controlled adsorption. Intercept $C \neq 0$ indicates contribution from film diffusion.

Generally speaking, the activated carbon is characterized by suitable adsorption performance due to its amorphous nature, high porosity, and oxygen-containing groups. Such properties provide numerous active sites. This way, activated carbon can be used effectively for advanced water purification applications, particularly for removing dissolved metal contaminants from aqueous solutions. The impregnation of the activated carbon with iron results in changes in the surface properties of this compound, which increase the removal of target contaminants due to the creation of meso/micropore structure that improves mass transfer and sorption performance [38]. In such a case, the estimated kinetic parameters will be vulnerable to experimental error and deviations in the determination of equilibrium concentrations. Hence, despite the fact that the intraparticle diffusion model showed the highest coefficient of correlation ($R^2 = 0.921$), the obtained results have to be considered as an indication of the dominating adsorption mechanisms rather than evidence of the specific sorption process.

The thermodynamic parameters such as ΔG° , ΔH° , and ΔS° of Fe ions adsorption on the prepared *Vigna trilobata* rind-derived activated carbon have been obtained. These parameters are shown in Table 3. Since the negative values of Gibbs free energy (ΔG°)

(from -4.31 to -6.53 kJ mol $^{-1}$) are observed at all investigated temperatures, one can assume that the adsorption process is spontaneous and therefore, thermodynamically possible [39]. Moreover, the decreasing values of ΔG° show that the adsorption process is getting more favorable with the increasing temperatures. Additionally, as shown in Table 3, the obtained positive enthalpy change ($+7.11$ kJ mol $^{-1}$) confirms that the adsorption process has an endothermic nature. The obtained result shows that the heat improves the interaction between Fe ions and the active sites of the developed activated carbon. Based on the obtained low value of enthalpy, one can conclude that the adsorption takes place through physical adsorption mechanisms. The positive entropy change (37.96 J mol $^{-1}$ K $^{-1}$) indicates an increase in randomness at the interface between adsorbate and adsorbent. It can be explained by the detachment of water molecules from the hydrated Fe ions and the adsorbent surface during adsorption. A satisfactory correlation between the inverse temperature and $\ln K_c$ (the Van't Hoff plot) provides a further confirmation of the spontaneity and endothermicity of the Fe ion adsorption on the developed activated carbon ($R^2 = 0.840$). Thus, the negative ΔG° values (from -4.31 to -6.53 kJ mol $^{-1}$) observed during the increasing temperature confirm that the process is favorable and spontaneous [40].

Table 3. Thermodynamic parameters for Fe adsorption.

Temperature (K)	Kc	ΔG° (kJ mol $^{-1}$)
298.15	5.684	-4.31
313.15	6.218	-4.76
333.15	6.564	-5.21
353.15	9.234	-6.53

To determine the effectiveness of the developed adsorbent, a comparative study of the adsorption properties of Fe ions with various adsorbents reported in the literature sources was performed. According to data provided in Table 4, the adsorbent developed in the current work demonstrated a competitive adsorption capacity (19.81 mg g $^{-1}$). It should be assumed that the obtained result is related to synergistic effects of surface functional groups, surface heterogeneity, and adsorption sites formed during the sulfuric acid activation of the biomass raw material. The usage of cheap biomass as a source material adds an additional advantage to the developed adsorbent. It allows achieving high ecological and economic effects due to inexpensive adsorbent preparation and its utilization [41]. Despite the positive adsorption characteristics of the prepared activated carbon, the reusability or regeneration of the activated carbon was not studied in the current research. The future research should aim at studying the efficiency of regeneration of the activated carbon adsorbent using suitable desorption agents like acids or salts. This would help in determining the practical application value of the activated carbon prepared from *Vigna trilobata*.

Table 4. Comparison of Fe adsorption performance from biomass-based activated carbons (AC).

Adsorbent Material	Activation/Modification Method	Metal Ion	Max. Adsorption Capacity (mg g $^{-1}$)	Reference
Banana peel AC magnetic composite	Chemical activation and magnetic modification	Fe(III)	10	Yanti et al. [28]
White lupine HSUK biosorbent	Biomass biosorbent	Fe	10	Richards et al. [11]
Rice husk AC	H ₂ SO ₄ activation	Fe(II)	12.4	Osasona and Kanuhor [9]
Mesoporous AC from waste fibers	Chemical activation	Fe(II)	17.3	Azizi and Azizi [29]
AC from local	Biomass-derived activat-	Fe/Mn	15.2	Akl [7,25]

agro-residues	ed carbon			
Chemically activated biomass carbon	Chemical activation	Fe(III)	18.5	Elewa et al. [6]
<i>Vigna trilobata</i> rind AC	H ₂ SO ₄ activation	Fe ³⁺	19.81	Present work

4. Conclusions

Sulfuric acid activation of *Vigna trilobata* rind yielded activated carbon with the ability to adsorb Fe³⁺ ions from aqueous solutions. The presence of a predominantly amorphous structure with a heterogeneous surface and the presence of oxygenated groups were determined by characterization methods. According to BET, the specific surface area was found to be 20.55 m² g⁻¹, showing the formation of adsorption sites due to the activation process. The highest experimental observed adsorption capacity of the activated carbon was 19.81 mg g⁻¹. In the range of concentrations tested, the Langmuir equation described adsorption equilibria better than other isotherm models with a correlation coefficient R² = 0.978. It was shown from kinetics experiments that the intra-particle diffusion mechanism plays the key role in Fe adsorption (R² = 0.921), whereas film diffusion contributes to the process. Thermodynamic characteristics obtained in our work prove that Fe adsorption is endothermic ($\Delta H^\circ = +7.11$ kJ mol⁻¹) and spontaneous ($\Delta G^\circ = -4.31$ to -6.53 kJ mol⁻¹) and is accompanied by an increase in adsorption enthalpy with temperature elevation. The results showed that the activated carbon produced using *Vigna trilobata* rind could be considered as an efficient, inexpensive, and environmentally friendly adsorbent for Fe ion removal from aqueous solutions.

Author Contributions: Conceptualization, R.U.D.; methodology, R.U.D. and V.K.K.; Analysis: V.K.K. and K.S.L.; writing—original draft preparation, V.K.K., B.c.K. and D.L.L.; writing—review and editing, R.U.D. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Data Availability Statement: The original contributions presented in this study are included in the article. Further inquiries can be directed to the corresponding author.

Acknowledgments: The authors are thankful to the GITAM Deemed to be University for the research facilities to carry out this research work.

Conflicts of Interest: The authors declare no conflicts of interest.

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