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Research Article

Comparative Study on Adsorption of Nickel Heavy Metal Using Different Acids (Sulfuric Acid, Hydrochloric Acid and Phosphoric Acid) Treated Activated Carbon of *Aegle Marmelos* Fruit Shells

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Abstract
Nickel, a prevalent metal in the ecosystem, is released into the environment through various anthropogenic activities, leading to adverse effects. This research explored comparison of various activating agents for nickel (II) removal from aqueous solutions. The adsorption characteristics were examined concerning contact duration, initial metal ion concentration and sorbent dosage. The findings revealed that among the acids tested, H_2SO_4 emerges as the most effective activator for enhancing porosity, making it a promising candidate for applications requiring high adsorption capacities. The higher removal efficiency of H_2SO_4-treated adsorbent underscores its potential as a cost-effective and efficient solution for treating wastewater containing Nickel (II) ions.

Keywords: Nickel (II) ions, *Aegle Marmelos*, adsorption, acid treatment etc.

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Introduction

Industries such as leather tanning, paint production, and battery manufacturing often produce liquid waste contaminated with toxic substances, including synthetic dyes and heavy metals. These pollutants can accumulate in living organisms through the food chain, resulting in significant health risks (Thanarasu et al., 2021). Studies have documented the harmful effects of dyes and heavy metals, which can lead to conditions such as kidney failure, diarrhea, lung cancer, liver damage, and respiratory issues like asthma. To mitigate these risks, it is crucial to treat industrial effluents containing such contaminants before their release into the environment (Loulidi et al., 2023).

Over the years, several conventional physical and chemical methods have been employed to remove heavy metals and dyes from industrial wastewater. These include filtration, coagulation and flocculation, chemical oxidation using agents like chlorine, ozone, and hydrogen peroxide, as well as advanced oxidation processes (El maguana et al., 2020). However, due to economic and technological limitations, only a few of these methods are widely adopted by industries. Among the available options, adsorption has gained prominence

for its efficiency, cost-effectiveness, simplicity, and versatility. Despite its advantages, the use of commercial activated carbon for adsorption can be expensive, as it is derived from costly raw materials (Nizam et al., 2021). As a solution, researchers have explored the development of low-cost adsorbents from agro-industrial waste, which are both abundant and affordable (Husien et al., 2022). Studies highlight the successful creation of activated carbons from materials such as orange peels, almond leaves, potato peels, water lilies, oil palm fruit waste, coffee husks, spent coffee grounds, and tea waste, offering a sustainable approach to wastewater management (Loulidi et al., 2019).

Various plant materials, including coconut husk, palm fiber, water fern, peat moss, duck weed, wheat bran, *Rhizopus nigricans*, modified corn starch, modified onion skins, saw dust, water lettuce, *Alterneuthera triandra*, *Citrus aurantium*, *Musa paradiasca*, and *Achras sapota*, have been the subject of numerous investigations by researchers (Bhati and Singh, 2012). We are aware of no study that compared the effects of various acid activation on the surface texture properties of AC derived from *Aegel marmelos* fruit shell for the adsorption of nickel II metal pollutants. The best models that provide a more accurate description of the adsorption process were investigated using adsorption kinetics and isotherms.

Materials and Methods

Preparation and characterization of activated carbon

To prepare activated carbon, locally available dried shells of *Aegle Marmelos* fruit were cleaned, dried, and ground to the desired mesh size. The resulting powder was treated with a 50% sulfuric acid, phosphoric acid and hydrochloric acid solution at an impregnation ratio of 1:1 (acid to raw material). Before impregnation, the raw material underwent pyrolysis, followed by carbonization for one hour at 700°C. The acid-treated material was then dehydrated in an oven at 105°C overnight. Subsequently, the sample was subjected to further pyrolysis in a furnace at 850°C for two hours to complete the activation process. After cooling to room temperature, the activated carbon was thoroughly washed with deionized water to remove residual chemicals. Finally, the product was dried again in a hot air oven at 105°C, yielding the prepared activated carbon.

Batch equilibrium studies

Adsorption isotherms were carried out in a set of 250 mL flasks containing Nickel (II) chloride solutions (50 mL) of varying initial concentrations (50–250 mg/L). In order to achieve equilibrium in the solid-solution mixture, 0.1 g of activated carbon of equal mass was added to nickel solution and kept in an isothermal shaker at 30°C for one hour. Another set of Erlenmeyer flasks with the same metal concentration but no activated carbon was used as a blank and went through the same procedure. 0.1 mol sodium hydroxide or a few drops of diluted hydrochloric acid were added to bring the pH to 7. After that, the flasks were taken out of the shaker, and the metal concentration in the solution was checked. Prior to analysis, the samples were filtered to prevent carbon fines from interfering with the results. Each experiment was duplicated under identical conditions. The amount of adsorption at equilibrium, q_e (mg/g), was calculated by:

$$q_e = (C_0 - C_e)V/ W$$

where C_0 and C_e (mg/L) are the liquid-phase concentrations of nickel at initial and equilibrium, respectively. V is the volume of the solution (L), and W is the mass of dry adsorbent used (g).

Batch kinetic studies

The strategies of dynamic examinations were fundamentally indistinguishable from those of equilibrium tests. The metal concentrations were also measured, and the aqueous samples were taken at predetermined intervals. The amount of adsorption at time t, q_t (m/ g), was calculated by:

$$q_t = (C_0 - C_t)V /W$$

where C_0 and C_t (mg/L) are the liquid-phase concentrations of nickel at initial and any time t, respectively. V is the volume of metal solution.

Results and Discussion

Table 1 reveals that the adsorbent treated with H_3PO_4 exhibits the lowest BET surface area and total pore volume. This is attributed to the strong acidic nature of H_3PO_4, which hinders the optimal development of porosity. In contrast, the adsorbent treated with H_2SO_4 achieves the highest BET surface area, pore size, and total pore volume, measured at 2542.763 m²/g, 2.7562 nm, and 0.879 cc/g, respectively. These findings highlight the effectiveness of H_2SO_4 as a significant activator in enhancing specific surface area and pore volume. However, for H_3PO_4 and HCl-treated adsorbents, both the surface area and total pore volume were slightly lower compared to those treated with H_2SO_4.

The characterization results reveal distinct differences in the textural properties of adsorbents activated with different acids, highlighting the influence of the activating agent on pore structure and surface area. The adsorbent treated with H_3PO_4 exhibited the lowest BET surface area and total pore volume among the samples. This outcome can be attributed to the strong acidic nature of H_3PO_4, which promotes excessive dehydration and possible pore collapse during activation, as noted in previous studies (Foo and Hameed, 2012). Such effects hinder the optimal development of porosity, reducing the overall effectiveness of H_3PO_4 as a porosity-enhancing agent.

Conversely, the activation with H_2SO_4 demonstrated superior performance in terms of BET surface area, pore size, and total pore volume, reaching 2542.763 m²/g, 2.7562 nm, and 0.879 cc/g, respectively. These values significantly surpass those of the other acids used, indicating the high efficacy of H_2SO_4 in enhancing the specific surface area and pore volume. This can be attributed to the ability of H_2SO_4 to facilitate extensive carbonization and pore development through its strong dehydrating properties and effective removal of volatile

organic fractions during activation (Ioannidou and Zabaniotou, 2007). The observed increase in mesoporosity (indicated by the pore size) further supports its utility for applications requiring high surface area and accessibility, such as adsorption and catalysis. For adsorbents treated with HCl and H_3PO_4, the observed BET surface area and total pore volume were lower than those treated with H_2SO_4. HCl, although effective in some cases, often results in lower porosity due to limited pore expansion during activation. This aligns with findings in similar studies, where HCl-treated materials exhibited modest textural properties compared to those treated with H_2SO_4 (Alabdan et al., 2021).

Table 1: Results of Surface area, pore volume and pore size of activated carbon derived from *A. marmelos* with three different acid activators

Parameters	Method	H_3PO_4 activated	H_2SO_4 activated	HCl activated
Surface area	Single point BET	899.06 m^2/g	1376.23 m^2/g	1107.16 m^2/g
	Multipoint BET	710.15 m^2/g	1032.12 m^2/g	872.00 m^2/g
	Langmuir	1762.56 m^2/g	2542.76 m^2/g	2187.15 m^2/g
Pore volume	BJH cumulative micropore volume	0.426 cc/g	0.862 cc/g	0.567 cc/g
	DFT micropore volume	0.435 cc/g	0.563 cc/g	0.448 cc/g
	DR micropore volume	0.504 cc/g	0.879 cc/g	0.691 cc/g
Pore size	BJH desorption pore radius	0.8564 nm	2.7562 nm	1.673 mm
	DR micropore radius	1.6065 nm	1.9328 nm	1.679 mm
	DFT pore radius	0.54397 nm	1.4113 nm	0.864 mm

Adsorption Experiment

Batch experiments were carried out to investigate the effects of contact time, initial Nickel(II) concentrations, adsorbent dose on adsorption of the metal ions from its solutions.

Effect of adsorbent dosage

The removal of Nickel(II) by low cost adsorbent at different adsorbent doses (0.1-0.5 g in 100 mL) was studied. All process parameters like, contact time, agitation speed, and pH was fixed. Figure-1 shows that, the adsorption capacity of Nickel(II) increased rapidly with increase in the dose of adsorbent of *Aegel marmelos* fruit shell. The adsorption rate of adsorbent to remove Nickel(II) ion increase with increase in biomass dose from 0.1 to 0.5 g/L of solution and after then it becomes stationary. Highest adsorption was found in H_2SO_4 treated carbon 80.6% compare to phosphoric acid (63.6) and HCl treated (71.3%). The results (Figure 1) indicate a significant increase in the removal of Nickel(II) ions as the adsorbent dose increased from 0.1 to 0.5 g/L. This trend can be attributed to the increased availability of active adsorption sites and surface area with higher biomass loading, which enhances the adsorption process (Demirbas, 2008). However, beyond a dose of 0.5 g/L, the adsorption capacity plateaued, suggesting saturation of active sites or possible aggregation of adsorbent particles at higher concentrations, which reduces the effective surface area available for adsorption (Das and Mondal, 2011).

The superior performance of H_2SO_4-treated adsorbent can be linked to its higher BET surface area, pore volume, and acidic surface functional groups, which enhance the interaction between the adsorbent and the metal ions (Ioannidou and Zabaniotou, 2007). HCl-treated carbon showed moderate efficiency (71.3%), likely due to its lower pore development compared to H_2SO_4 activation (Baccar et al., 2013).

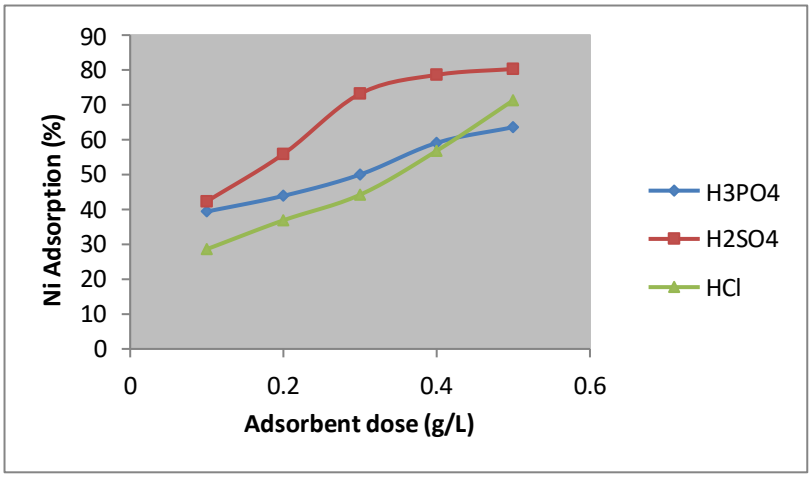


Figure 1: Effect of adsorbent doses on Ni (II) adsorption, Constant value: Metal Ni (II) -50mg/L contact time- 30 minute

Effects of Ni (II) metal ion concentration.

Nickel (II) adsorption is significantly influenced by the concentration of Nickel(II) in aqueous solutions. Figure-2 shows the effect of initial concentration on percentage removal of Ni (II) and adsorption capacity of the adsorbent. To see the effect of Ni (II) concentration, 0.5 g of the biomass was taken and mixed with 100 ml of Ni (II) solution of varying concentrations (50, 100, 150, 200, 250, and 300 mg/L) in different Erlenmeyer flasks. The mixture was continuously agitated in a temperature controlled water bath with agitation rate of 120 rpm. The adsorbed amount was determined from the difference in the initial and residual concentrations of Ni (II) in the liquid phase. The adsorption of Ni (II) ions by the adsorbent initially increased rapidly with increasing Ni (II) concentration and become constant when Ni (II) concentration reached 250 mg/L. It may be due to the interaction between the metal ions and the active sites of the adsorbent. At higher concentrations more Ni (II) ions are left un-adsorbed in solution due to the total saturation of binding sites of the adsorbent. The study found that among three different acid treated adsorbents, sulfuric acid treated carbon absorbed more nickel ion. The sequence of nickel adsorption is as follows HCl $<H_3PO^4<$ H_2SO_4.

However, as the concentration increased beyond 250 mg/L, the binding sites on the adsorbent surface became saturated, limiting further adsorption. This saturation effect is consistent with Langmuir's adsorption theory, which describes the formation of a monolayer of adsorbate on the adsorbent surface (Foo and Hameed, 2010).

At higher concentrations (>250 mg/L), the reduced percentage removal of Nickel(II) ions is likely due to an insufficient number of available binding sites relative to the excessive number of metal ions in the solution (Hellal et al., 2023). This highlights the need to optimize adsorbent dosage and initial metal ion concentration to achieve maximum removal efficiency (Ungureanu et al., 2024).

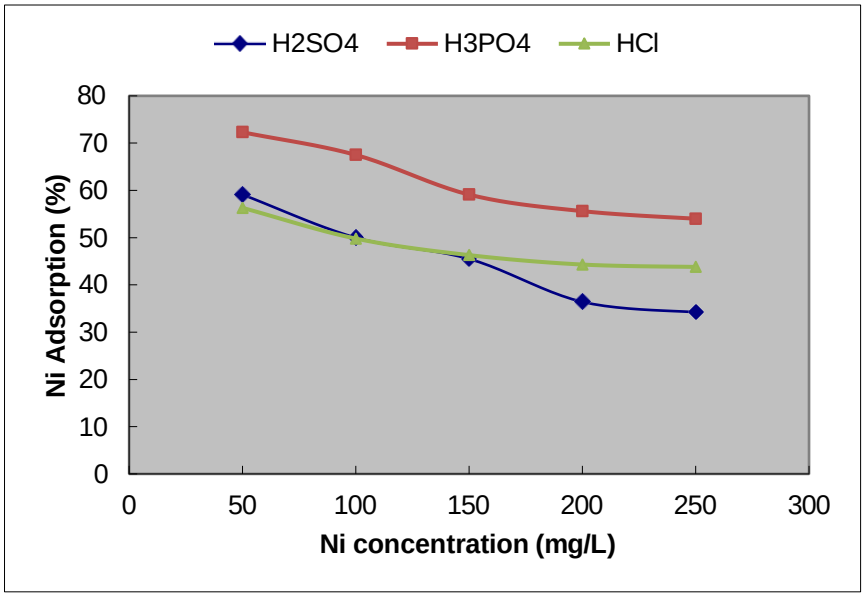


Figure 2: Effect of Ni (II) concentration variation on adsorption

Constant value: adsorbent dose- 0.5 g/L; contact time- 30 minute

Effect of contact time on Ni (II) adsorption

The effect of contact time on the adsorption of Ni (II) from its solution is shown in Figure-3. It is observed that the adsorption increases with the contact time to a particular level then in becomes slow because of non-availability of active sites on the adsorbent. The maximum adsorption 81% of Ni (II) was obtained at 70 min by sulphuric acid treated adsorbent, that was maximum compare to H_3PO_4 (76.1%) and HCl (72.3%).

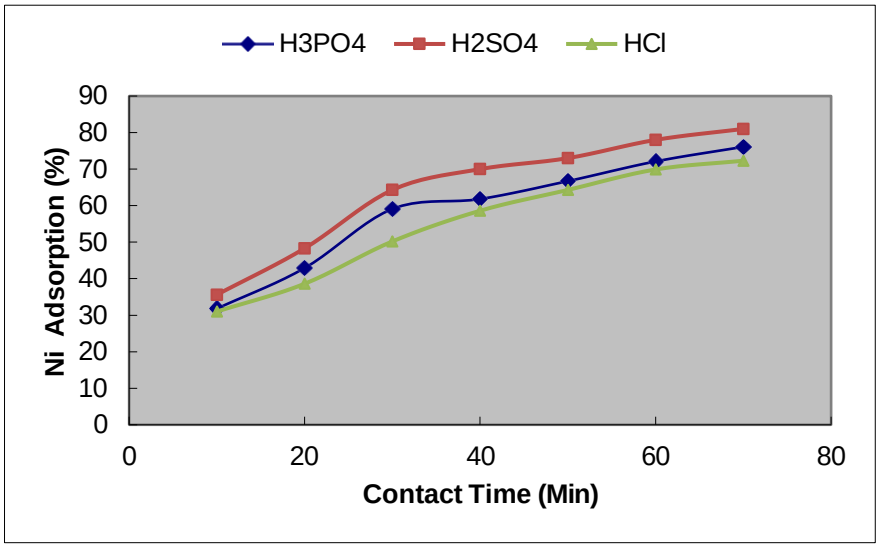


Figure 3: Effect of contact time on Ni (II) adsorption Constant value: Metal Ni (II) - 50 mg/L; Adsorbent dose- 0.5g/L

Adsorption isotherm

The adsorption behavior of Nickel (II) ions onto the acid- activated carbons was analyzed using two widely used isotherm models: Langmuir and Freundlich. The experimental data exhibited a non-linear relationship with equilibrium concentrations, suggesting complex interactions between the adsorbent and adsorbate. The isotherm parameters and correlation coefficients (R^2 values), as summarized in Table 2, were utilized to evaluate the model's goodness of fit.

The results indicate that the Freundlich isotherm model provided the best correlation with the experimental data for all acid-activated carbons, with R^2 values ranging from 0.976 to 0.998 (average 0.987). This suggests that the adsorption of Nickel (II) ions follows a heterogeneous surface adsorption process, where binding sites of varying affinities are involved. Freundlich's model, which assumes multilayer adsorption, is more suitable for such systems and has been widely observed in studies involving activated carbons (Qingping Yia et al., 2020; Baccar et al., 2013). The strong fit of this model underscores the complex nature of adsorption, influenced by both chemical interactions and surface heterogeneity.

The maximum adsorption capacity (qm) values derived from the Freundlich model further highlight the effectiveness of different acid-activated carbons. The adsorption capacity was found to follow the sequence **H_3PO_4 < HCl < H_2SO_4**, consistent with earlier observations of the textural and chemical properties of the adsorbents. Sulfuric acid-treated carbon exhibited the highest adsorption capacity, likely due to its enhanced porosity, surface area, and the presence of acidic functional groups that facilitate metal ion binding (Ioannidou and Zabaniotou, 2007; Demirbas, 2008). Conversely, the lower qm values for H_3PO_4 and HCl-treated carbons reflect their comparatively limited pore development and fewer active binding sites.

Table 2: Isotherm parameters for the adsorption of Nickel (II) ions on different activated carbon samples

Adsorbent			**Langmuir Isotherm**		**Freundlich Isotherm**		
	q_m	b	Correlation Coefficient (R^2)	Separation Factor (R_L)	K_F	n	Correlation Coefficient (R^2)
H_3PO_4	114	0.0166	0.984	0.35	6.80	1.99	0.976
HCl	83.3	0.005	0.907	0.71	1.02	1.37	0.998
H_2SO_4	71.4	0.0137	0.960	0.51	2.57	1.66	0.991

Kinetic results

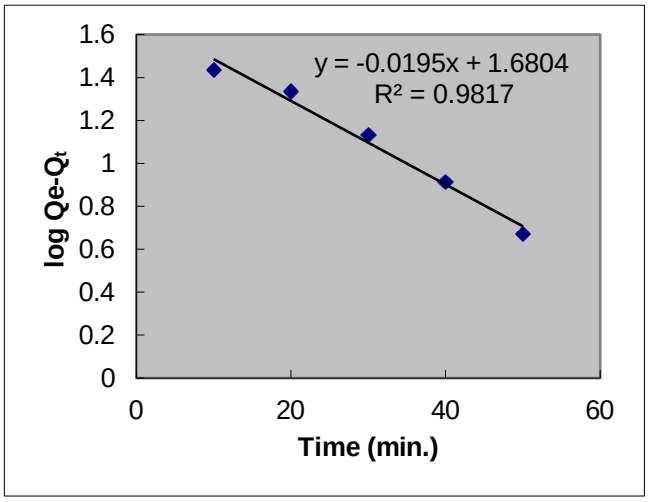
The kinetics of Nickel (II) adsorption onto the acid-activated carbons was analyzed using the pseudo first-order and pseudo second-order models to evaluate the adsorption mechanism and rate-controlling steps. The integration of these models and the application of boundary conditions yielded equations used to fit the experimental data. The kinetic parameters, including rate constants (k_1 and k_2) and equilibrium adsorption capacities (q_e), are summarized in Table 3.The results indicate that the pseudo first-order model did not adequately fit the experimental data, as evidenced by R^2 values less than 0.95 across all adsorbents. Moreover, the calculated equilibrium adsorption capacities obtained from this model showed significant deviations from the experimental values. These discrepancies suggest that the adsorption of Nickel (II) ions onto activated carbon is not primarily governed by physical adsorption processes alone, as described by the pseudo first-order model (Ho and McKay, 1999).Conversely, the pseudo second-order model provided a much better fit, with R^2 values exceeding 0.95 for all adsorbents. Additionally, the q_e (calculated values) derived from this model closely matched to the q_e (experimental values)

indicating its robustness in describing the adsorption process (Bhagat and Khandeshwar, 2024). The superior fit of the pseudo second-order model suggests that the adsorption of Nickel (II) ions onto the activated carbons is primarily controlled by chemisorption, involving valence forces or electron sharing between the adsorbent's surface and the metal ions (Elovich and Larinov, 1962).

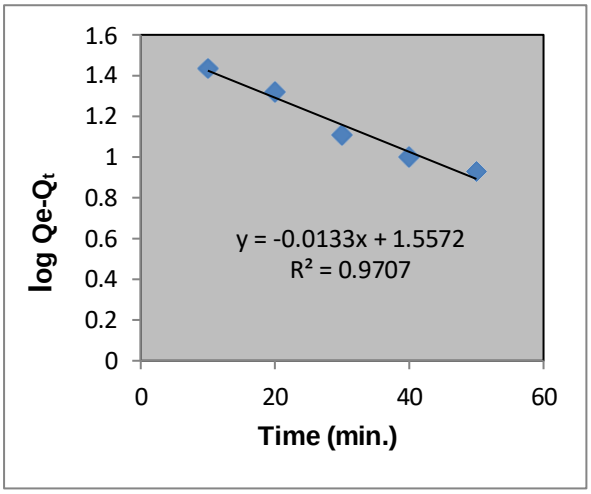
The rate constant k_2, determined from the slope of the linear plot of t/q_t versus t, provides insights into the adsorption rate. Among the adsorbents, sulfuric acid-treated carbon exhibited the highest k_2 value, reflecting its superior adsorption kinetics, which can be attributed to enhanced porosity and the presence of functional groups that facilitate rapid ion exchange. Phosphoric acid-treated and HCl-treated carbons showed lower k_2 values, consistent with their relatively lower adsorption capacities.

Table 2: Kinetic parameters for adsorption of Nickel (II) ions on activated carbon samples.

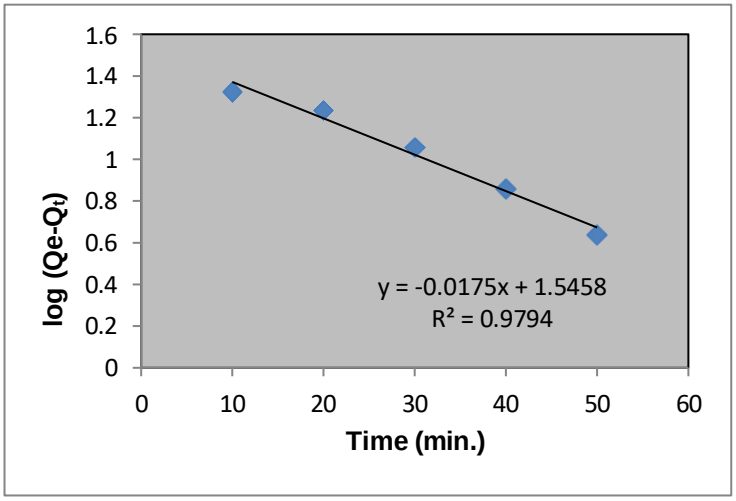
		First order kinetic model			Second order kinetic model		
	Qe (Experimental)	**k_1**	**Qe (Calculated)**	**R^2**	**k_2**	**Qe (Calculated)**	**R^2**
HCl	36.5	0.035	33.88	0.979	0.0009	47.6	0.962
H_2SO_4	45	0.03	36.05	0.892	0.0009	52.6	0.986
H_3PO_4	43	0.045	47.91	0.981	0.0004	64.1	0.954



Pseudo first order kinetic for H_3PO_4



Pseudo first order kinetic for H_2SO_4



Pseudo first order kinetic for HCl

Figure 4: Experimental kinetic data and linear fitted curve with kinetic models for pseudo-first-order

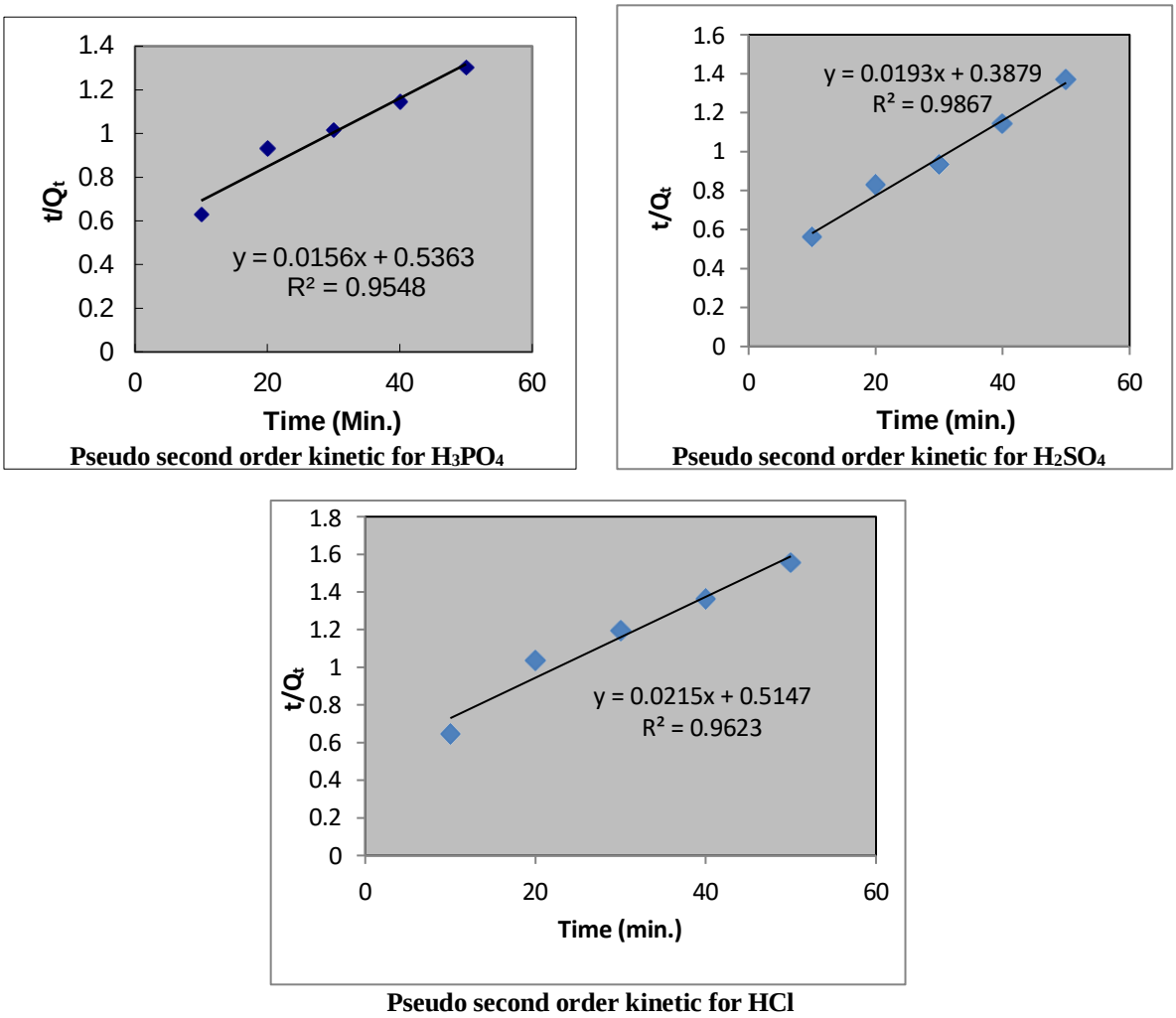


Figure 5: Experimental kinetic data and linear fitted curve with kinetic models for pseudo-second-order

Conclusion:

The findings underscore the pivotal role of the activating agent in tailoring the adsorbent's properties. Among the acids tested, H_2SO_4 emerges as the most effective activator for enhancing porosity, making it a promising candidate for applications requiring high adsorption capacities. The higher removal efficiency of H_2SO_4-treated adsorbent underscores its potential as a cost-effective and efficient solution for treating wastewater containing Nickel (II) ions. The strong agreement between experimental and Freundlich model data suggests that surface heterogeneity plays a significant role in adsorption. These findings reinforce the importance of chemisorption in the removal of Nickel (II) ions by acid-activated carbons and highlight the pseudo second-order model as a reliable tool for understanding the adsorption kinetics.

Future work should focus on optimizing the activation process parameters and exploring the regeneration and reusability of these adsorbents to improve their practical application in industrial-scale wastewater treatment. Future research should explore additional kineticmodels, such as intra-particle diffusion and Elovich models, to further elucidate the mechanism and identify potential rate-limiting steps.

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