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Investigating The Efficacy Of Silica-Pumice Nanocomposite In A Nanotechnology-Driven Adsorption Process For Phenol Removal From Wastewater, With Focus On Kinetics And Isotherms

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Abstract
The presence of priority and emerging aromatic based pollutants such as phenols in water sources is of growing concern due to their bio-accumulative mutagenic and carcinogenic effects. These compounds are not readily removed during conventional treatment and persist in plant feed streams, necessitating effective removal strategies. Adsorption has been widely accepted as a suitable remediation technology due to its simplicity, cost effectiveness, and regeneration potential. Silica based adsorbents have attracted significant attention because of their-environmental friendly properties and reusability. This study focused on the synthesis of silica-pumice Nanocomposite which was prepared by grafting silica extracted from rice husks onto pumice rock using the sol gel method, followed by sintering at 550°C. The efficacy of silica-pumice Nanocomposite material prepared was investigated by use of batch adsorption studies using real industrial wastewater within the Nairobi Industrial Area. The wastewater had phenol levels of 0.59- 8.83 mg/L. The experiments investigating the rate for phenol removal were done under optimal conditions for efficiency. The optimal contact time, adsorbent dose, pH and initial phenol concentration were 60 minutes, 1.0 g, 6 and 2.5mg/L, respectively. The results showed that phenol removal efficiency reached 100%. Adsorption equilibrium data were best described by the Freundlich isotherm model ($R^2 = 0.99899$) with a capacity constant Kf of 0.080 mg g $^{-1}$ and a slope 1/n of 2.91, indicating heterogeneous surface adsorption and cooperative multilayer formation. Kinetic analysis showed that the pseudo - second order model provided the best fit ($R^2 = 0.9749$) with a rate constant K of 0.015 and an equilibrium adsorption capacity (q_e) of 4.401 mg g $^{-1}$, confirming that chemisorption dominating the adsorption process through hydrogen bonding and π–π interactions. Phenol levels in wastewater decreased significantly using the adsorbent; sample (1) 0.59-0.00 (100%), sample (2) 8.83-1.62 (82%), sample (4) 8.64-1.47 (83%) and sample (6) 1.15-0.01 (99%). The silica-pumice adsorbent was confirmed to be an efficient and environmentally friendly adsorbent suitable for the removal of phenols from industrial wastewater.

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I. Introduction

Phenol is a toxic organic pollutant commonly found in industrial wastewater[1]. It causes unpleasant taste and odour in drinking water even at very low concentrations. Phenol and its derivatives are harmful to aquatic organisms, and prolonged exposure poses carcinogenic risks to humans[2]. When phenol concentrations exceed certain thresholds, they disrupt natural water self-purification processes and inhibit photosynthesis in aquatic plants[3]. The major sources of phenol pollution in water bodies are wastewaters from industries such as textile manufacturing, petroleum refineries, petrochemical plants, pharmaceutical production, pesticide, plastic manufacturing, and coking plants among others [4]. Chlorination of natural waters for disinfection has also been associated with byproducts such as chlorinated phenols, which are more toxic than phenol itself. Due to these risks, phenols are classified as priority pollutants by environmental agencies worldwide[5].

Several treatment methods have been used to remove phenolic compounds from aqueous solutions, including biological treatment, chemical oxidation, membrane filtration, solvent extraction, and photocatalytic oxidation[6]. However, many of these methods are cost intensive, time consuming, or ineffective when phenol concentrations are high. Adsorption on activated carbon is currently the most effective technique for treating phenolic wastewaters, particularly for high strength and low volume effluents[7]. The disadvantage of activated carbon is its high cost due to non-renewable and relatively expensive starting materials required such as coal. This has prompted growing research interest in producing alternative low cost adsorbents from locally available materials.

Agricultural by-products (agro-waste) and natural minerals have been used as suitable precursors for adsorbent production. They are cheaper, renewable, abundantly available, and have high carbon or silica content[8]. Rice husks for example contains over 90% silica and are widely available as post-harvest agricultural waste[9]. Pumice is a porous volcanic aluminosilicate mineral with high surface area and amorphous structure favorable for adsorption[10]. The sol gel method is considered a promising technology for synthesizing adsorbents because it allows precise control over particle size, porosity, and surface chemistry at relatively low temperatures[11].

This study focused on the synthesis of a silica -pumice nanocomposite adsorbent by grafting silica extracted from rice husks onto pumice rock using the sol gel method, followed by evaluation of its effectiveness for phenol removal from industrial wastewater.

II. Materials And Methods

All solvents and chemicals were of analytical grade with 99% purity and obtained from laboratory reagent suppliers and manufacturers based in Kenya. Sodium citrate, starch, nitric acid, sulfuric acid, hydrochloric acid and dichloromethane were supplied by Merck, Germany. Distilled water was prepared at Murang'a University of Technology Analytical Laboratory.

Preparation of Ash from rice husk

The process started by weighing 200 g of rice husks. It was then washed with deionized water to remove dust and other impurities as illustrated in Figure 1 [12]. After washing, they were dried in an oven at 110°C [13]. Then the husks were soaked in 1 N hydrochloric acid at 75°C for 1 hour to get rid of undesirable minerals and pigments [14]. Afterwards, they were rinsed with distilled water until a neutral pH of 7 was achieved, then dried for 3 hours at 80°C in an oven [15]. After treating it with acid, the husks were then burned in a muffle furnace at 700°C for 2 hours to give rice husk ash (RHA)[16].

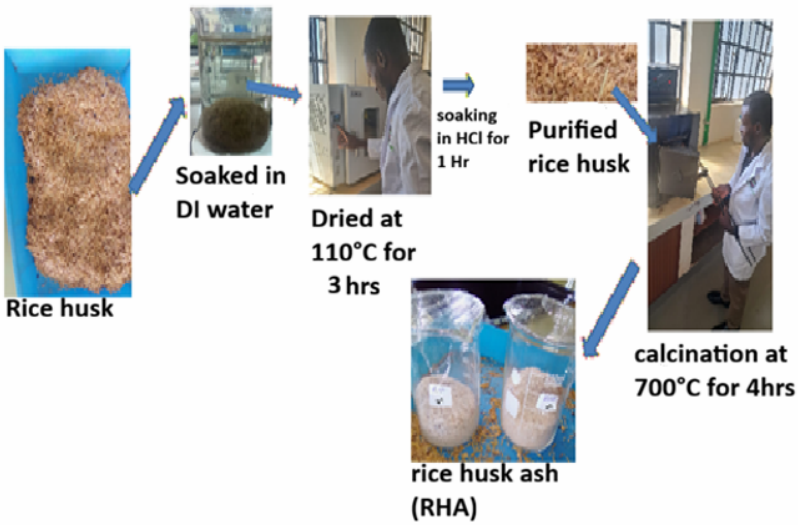

Figure 1: preparation of ash from rice husk

Synthesis of silica nanoparticles from RHA

An amount of 5 g of pretreated rice husk ash was weighed and placed in a flat bottomed flask with 100 ml 3 M NaOH for refluxing which took place for 2 hours. During the process, the temperature was maintained at 80°C as illustrated in Figure 2. At the end of the experiments, the sodium silicate and leachate solution were separated from insoluble residue using Whatman filter paper 41. Then the separated solution was neutralized to pH 7 with 3 M H_2SO_4 solution with continuous stirring, and allowed to settle for 48 hours to precipitate silicic acid. The precipitated produced was collected and washed thoroughly with distilled water to remove any water-soluble salt and dust. After washing, the final product was dried at 80°C for 24 hours for converting silicic acid to pure silica, activated at 550 °C for 3 hours and stored in an airtight sample holder for further use. The amount of silica extracted from pumice rock was calculated using Equation (1).

Equation (1).

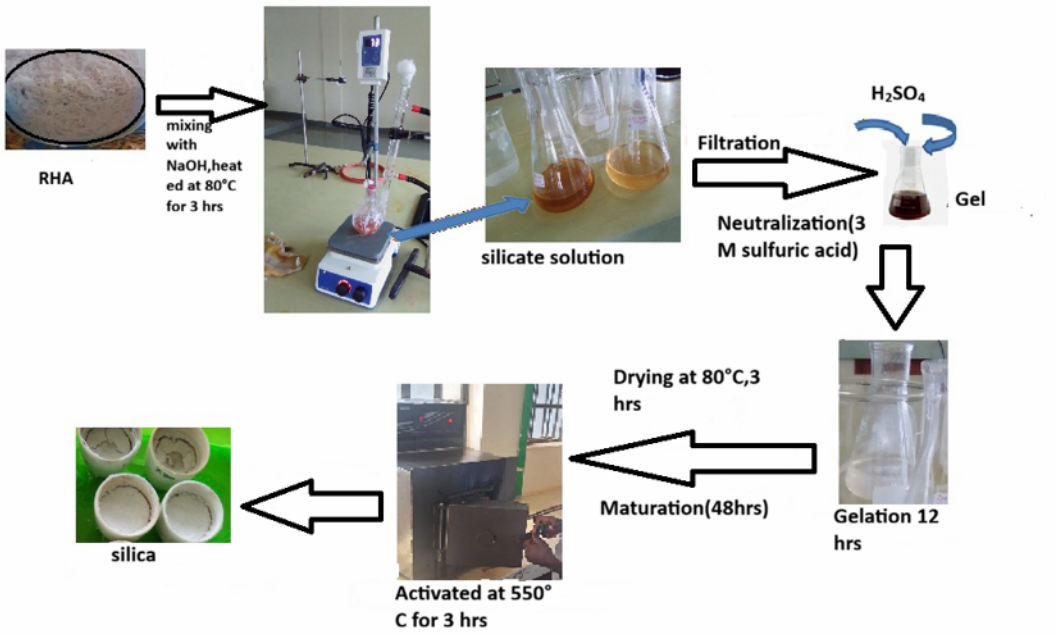

Figure 2: Rice husk ash processed into silica particles

Preparation of Pumice rock for grafting

The pumice rock was rinsed with deionized water several times to get rid of impurities. It was then dried in an oven at 100°C and then crushed using a mortar and pestle to obtain fine and uniform particles. The crushed powder was then passed through a 200 µm sieve to eliminate coarse particles and then activated in a muffle furnace at 550 °C for 3 hours [23].

Sol-gel preparation, grafting and sintering

The silica sol was prepared by dispersing silica nanoparticles in water-ethanol at a ratio of 1:2 while stirring [24] as illustrated in Figure 3. 0.1M HCL was added to adjust the PH to 5 for stability, and then sonicated for 30 min to form a stable silica sol. Pumice powder and sodium citrate were added into silica sol to form slurry. The mixture was then homogenized by magnetic stirring for 1 hour to form a stable suspension. The slurry was cast into crucibles and then left for 24 hours for gelation to occur through solvent evaporation. The gel was dried at 100°C in an oven to remove residual solvent [25]. The dried adsorbent was sintered at the temperature of 700°C in a muffle furnace for 5 hours to enhance mechanical strength and stability of pore structures.

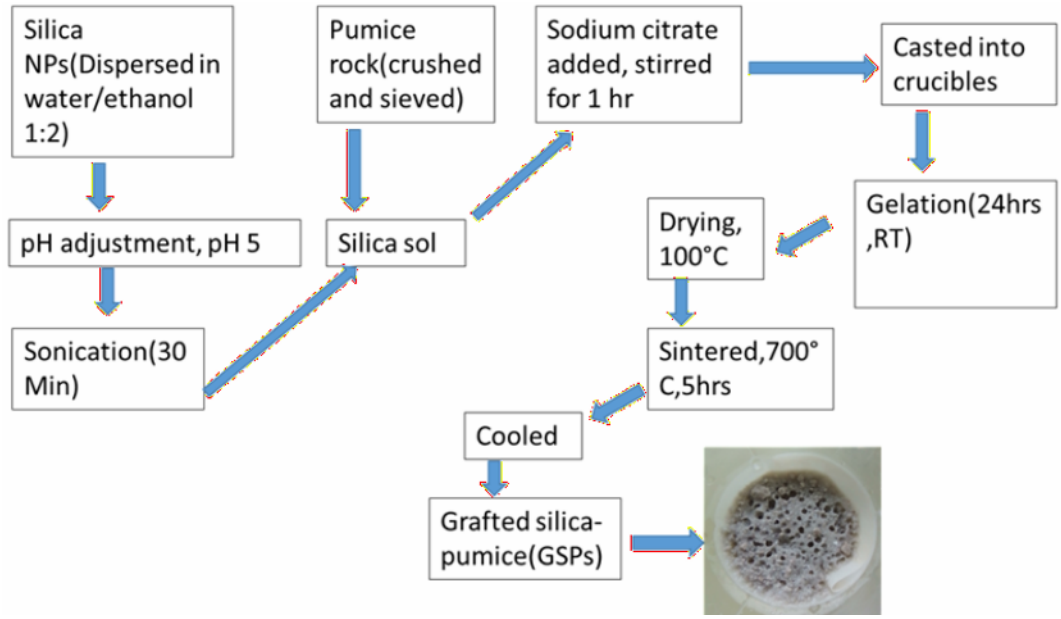

Figure 3: Sol-gel preparation, grafting and sintering

Adsorption studies

Adsorption studies were particularly done by batch technique to identify the key parameters influencing the sorption process and to obtain the optimal conditions for maximum phenol removal. The effects of various parameters on the rate of adsorption were assessed by varying initial concentration of phenols (0.5, 1, 2.5, 5, 10, and 15 mg/L), pH of solution (2, 5, 7, 9, and 10), contact time (10, 30, 45, 90, and 120 Min) and adsorbent dose (0.2, 0.8, 1.5, 2.0, and 2.5 g/L). The experiments were carried out in triplicates at room temperature using 250ml beakers containing 50ml of phenol concentration. Continuous swirling was enabled during the experiment at a constant mixing speed for better mass transfer with high interfacial area of contact. The residual phenol concentration in each sample after sorption was determined by use of UV-Vis spectrophotometer after filtration with whatman filter No 41. For pH adjustment, HCl and NaOH solutions were used. The analysis of these parameters provided a clear understanding of the interaction mechanisms between the grafted silica-pumice particles and phenol and allows assessment of the suitability of this material for large scale water treatment applications. The amount of phenol adsorbed onto per gram of adsorbent (qe) and the percentage removal efficiency (R %) were calculated using Equations (2) and (3), respectively.

Equation 2

Where C_i and C_e are initial and equilibrium concentrations (mg/L), V is volume (L), and m is adsorbent mass (g). The percentage removal was obtained as:

% removal = Equation 3

III. Results And Discussion

Effect of adsorbent dose

As illustrated in Figure 4, Phenol removal efficiency increased with increase in adsorbent dose, since contact surface of adsorbent particles increased.

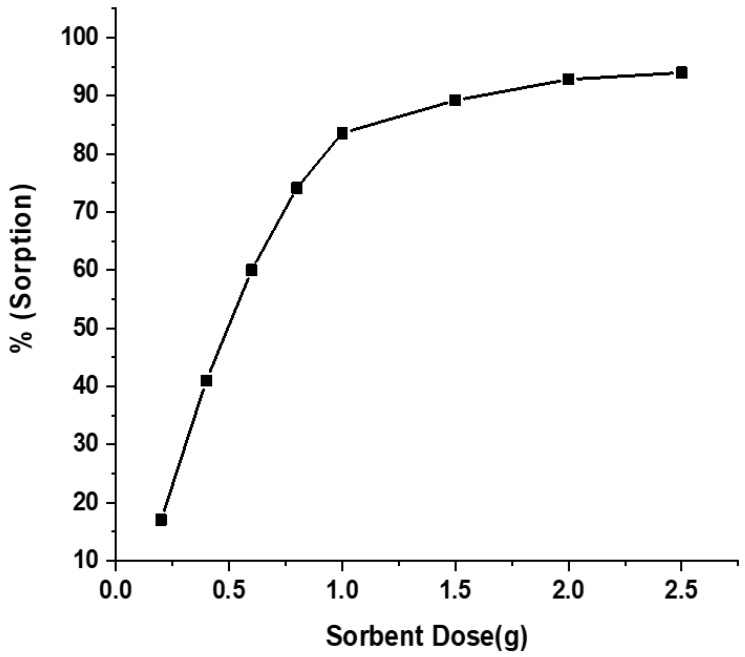

Figure 4: Effect of Adsorbent dosage on the Adsorption Capacity of Silica-Pumice nanocomposites.

Increase in the adsorbent dosage enhanced the availability of more active sites for the adsorption, thus making easier penetration of aromatic ions to the adsorption sites. The adsorption capacities for phenols increased from 17.5 ± 0.53% to 85.5 ± 0.69% with doses ranging from 0.2 -2.5 g. The dosage of 1.0 g, corresponding to 85.5 ± 0.82% removal, was optimal. Minimal removal efficiency at 17.5 ± 0.53% is attributed

to the decreased number of active sites on the adsorbent surface relative to the number of phenol molecules present in solution. An increase of 22% of removal efficiency from 0.2 to 0.4 g is due to proportional increase in total surface area with increase in silanol (Si - OH) and grafted functional groups capable of binding phenol through hydrogen bonding and hydrophobic interactions.

Further increases in dosage produced continued improvements in removal efficiency. The near linear increase in removal efficiency across this dosage range confirms that the adsorbent surface remained under-saturated with respect to phenol concentration, and each increment of adsorbent mass contributed additional binding sites that were readily occupied by phenol molecules as depicted in Figure 4 above.

The slight increase of 0.05% when the dosage increased from 2.0 g to 2.5 g indicates that the adsorption was approaching equilibrium. This trend is enhanced by the availability of active sites and larger total surface area provided by increased adsorbent dose. The grafting of highly porous silica from rice husks onto porous pumice provides more micro-pores and functional groups for the formation of chemical or hydrogen bonds with phenol molecules. Each increment of adsorbent dose increase binding sites, allowing more phenol molecules to be removed from solution.

Effect of pH

As it is shown Figure 5, the optimum removal efficiency of 80.32 ± 0.99% was observed at pH of 6 and by increasing pH, a radical decrease in adsorption was observed. This trend shows that maximum phenol uptake occurs under near neutral to weakly alkaline conditions, rather than at strongly acidic pH as commonly reported for other adsorbents. This observation is illustrated in Figure 5.

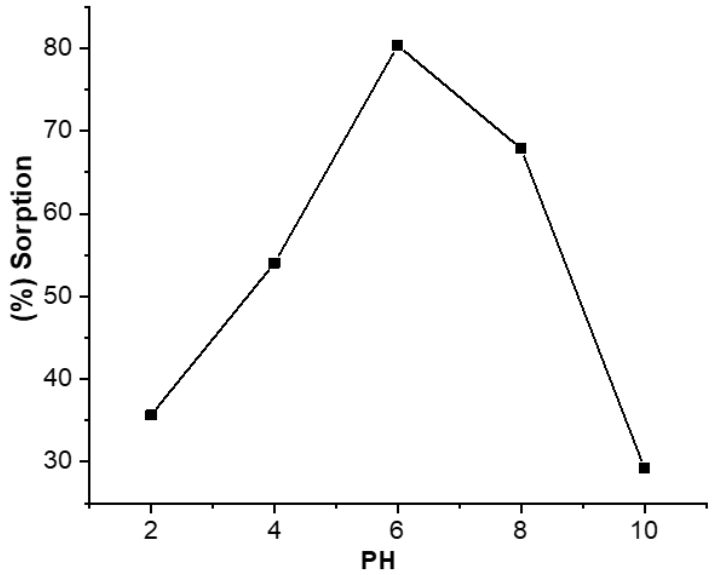

Figure 5: Effect of pH on Adsorption capacity of Silica-Pumice nanocomposites.

At optimum pH with maximum removal efficiency is attributed to the strong electrostatic force of attraction between the oppositely charged phenol molecules and the adsorbent and also hydrogen bonding interactions. The silanol groups (Si–OH) present on the rice husk derived silica and the aluminosilicate framework of pumice provide abundant binding sites. At this pH, which is close to the point of zero charge (pHpzc) of typical silica based materials ranging pH 6.5 -7.5, the adsorbent surface has either a neutral or weakly negative charge, while phenol with a pKa of about 10 remains undissociated molecular form. These factors contributes to minimization of electrostatic repulsion and promotes hydrogen bonding between the hydroxyl group of phenol and the silanol or siloxane groups on the nanocomposite(adsorbent) surface. At these pH (8, and 10) for alkaline conditions, the electrostatic repulsion force develops leading to reduction in sorption capacity. The silica-pumice surface becomes strongly negatively charged as silanol groups' deprotonate to form SiO^- species as the phenol begins to dissociate into phenolate anions ($C_6H_5O^-$). The resulting electrostatic repulsion between the negatively charged adsorbent surface and the negatively charged phenolate species reduces the affinity between the adsorbate and the adsorbent. However, at high pH, hydroxyl ions (OH) may compete with phenol for binding sites, further suppressing adsorption.

Effect of contact time

As indicated in Figure 6, adsorption rate increased rapidly to about 60 minutes for any initial concentration. The adsorption process involved two stages: an initial rapid phase followed by a slower phase (90 to 120min). This clearly indicated that the rapid adsorption process occurs when the number of available sites are much larger than the number of phenols to be adsorbed. At 10 minutes, the removal efficiency was 50.3 ± 1.72%, indicating that almost half of the available phenol molecules were rapidly adsorbed onto the available vacant active sites on the nanocomposite (adsorbent) surface. Longer contact times allow the system to approach equilibrium, ensuring maximum adsorption capacity. This observation is as illustrated in Figure 6.

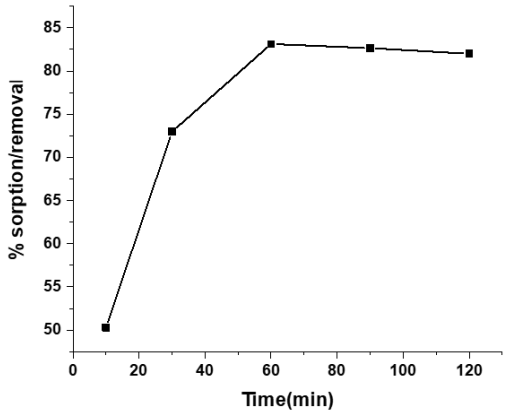

Figure 6: Effect of contact Time on the adsorption capacity of Silica-Pumice nanocomposites.

In chemisorption, longer exposure times enable pollutants to bind to adsorbent surfaces or penetrate the pores, leading to higher removal efficiencies. Though, prolonged contact times can sometimes lead to secondary reactions, such as desorption or degradation of adsorbed molecules, which must be managed for optimal system performance. The rice husk derived silica provides a high surface area with numerous silanol groups, while the porous pumice structure offers macro-pores that enhances rapid diffusion of phenol molecules to binding sites. The progressive increase in removal efficiency with time is characteristic of typical adsorption systems, where phenol molecules gradually diffuse from the bulk solution to the adsorbent surface, penetrate the porous structure, and bind to active sites via hydrogen bonding and hydrophobic interactions. After 1 h, there was no significant change in equilibrium concentration when the adsorption phase reached to equilibrium. Therefore, the best contact time is at 60 min for all experiments.

Effect of initial phenol concentrations

Phenol adsorption is a chemical process where phenol molecules interacts with Si-OH molecules through hydrogen bonding and mass transfer at the boundary between a solid and a liquid. The study focused on evaluating the effect of the initial phenol concentration as other parameters remained constant. As shown in Figure 7, the removal efficiency decreases from 92% to 55% when the phenol concentration increased from 0.5 to 2.5 mg/L.

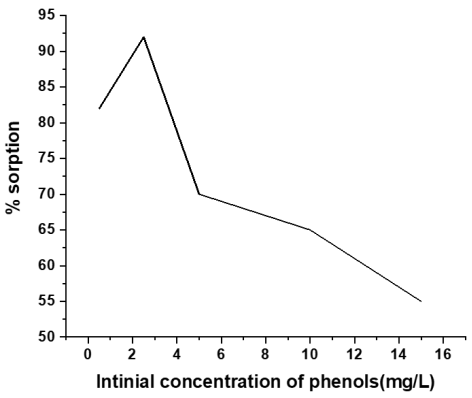

Figure 7: Effect of Initial concentration on the adsorption capacity of Silica-Pumice nanocomposites.

Similarly, the removal percentage reduced from 65 to 55 as the initial phenol concentration increases from 0.5 to 2.5 mg/L. Decrease in removal efficiency with increase of initial phenol concentration is attributed to the few number of active adsorption sites on the adsorbent. At lower concentrations of 0.5 to 2.5 mg/L, the number of available active sites exceeds the number of phenol molecules in solution, resulting in high removal efficiencies above 80% where almost all phenol molecules gets attached on the adsorbent surface. At 10 mg/L, the abundant silanol groups (Si–OH) and grafted functional sites on the rice husk derived silica and porous pumice framework are fully accessible, allowing maximum binding through hydrogen bonding and hydrophobic interactions. However, at higher concentrations, phenol molecules outnumber the available active sites, and therefore the removal efficiency of phenols which depends on the initial concentrations, decreases. Additionally, at higher initial concentrations, excess phenol molecules act as the barrier to mass transfer between the liquid phases block the pores of the adsorbent, hence limiting further adsorption by steric hindrance and pore plugging. Nonetheless, the pumice rock impregnated onto silica served as a crucial material to overcome this hindrance in the mass transfer. As a result, barrier to mass transfer decreased, improving the efficiency of phenol removal as the initial concentration decreased

Adsorption isotherms

The adsorption of phenol onto grafted silica pumice particles was better described by the Freundlich model ($R^2 = 0.99899$) than the Langmuir model ($R^2 = 0.98200$), indicating a heterogeneous sorbent surface with active sites of varying energy levels as indicated in Figure 8 and 9.

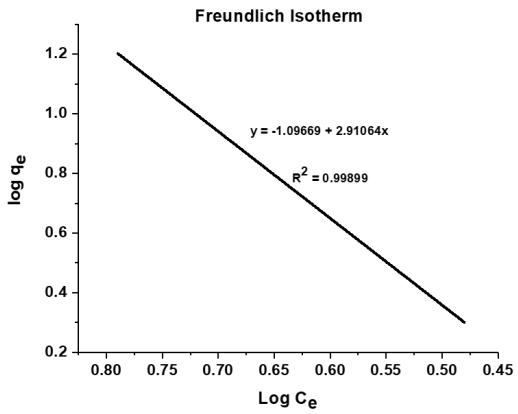

Figure 8: Freundlich Isotherm Plot for Phenol removal using Silica-Pumice nanocomposites.

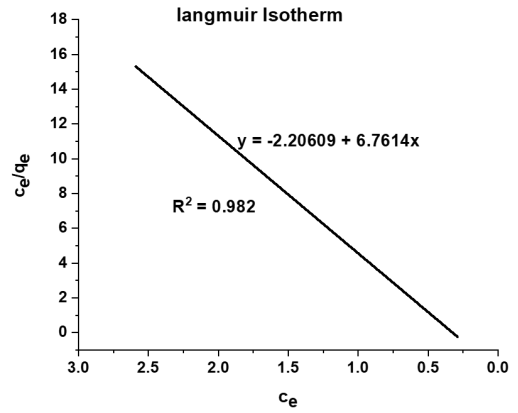

Figure 9: Langmuir Isotherm Plot for Phenol Removal using Silica-Pumice nanocomposites.

The Freundlich parameters gave an adsorption capacity constant (K_f) 0.080 mg g ^{1} and a slope 1/n=2.91, suggesting cooperative adsorption where bound phenol molecules facilitate additional binding, leading to multilayer formation. The Langmuir model, despite its relatively high R^2, assumes homogeneous surfaces and non-interacting sites, which does not fully capture the composite's complex dynamics. These

results in Table 1 confirm that the grafted silica pumice particles function as a multi-site adsorbent capable of sustained phenol uptake through diverse chemical and physical interactions.

Table 1: Calculated Freundlich and Langmuir Isotherm Parameters.

Model type	Intercept	Slope(1/n)			
Freundlich isotherm	-1.09669	2.910064	0.99899	0.08	
Langmuir isotherm	-2.20609	6.7614	0.98200	_	-3.06487

Adsorption Kinetics

Adsorption Kinetics play a crucial role in describing the effectiveness of adsorption. It essentially defines the rate at which solute is adsorbed and the contact time of the adsorbate on the solid-liquid interface. Adsorption rate relies on the number of particles to be adsorbed on the adsorbent surface and the number of particles colliding in unit area per second.

Sorption kinetics of silica-pumice adsorbent.

This study determined the kinetics of phenol removal by silica-pumice adsorbent applying both pseudo-first and pseudo-second order kinetics. The kinetic parameters for the adsorption process were studied for contact times ranging from 10 to 120 min by monitoring the removal percentage of the phenols as presented in Table 2 and 3.

Table 2: Pseudo-first-Order kinetics Raw Data at optimum conditions.

Time(minutes)	qt(mg/g)	log(qe-qt)
10	2.25	0.28
30	2.951	0.15
60	3.02	0
90	3.87	-0.56
120	4.011	-0.6

Table 3: Pseudo-Second-Order kinetics Raw Data at optimum conditions.

Time(minutes)	qt(mg/g)	t/qt
10	2.25	4.44
30	2.951	10.17
60	3.02	19.87
90	3.87	23.26
120	4.011	29.92

Figure 10 and 11 represents a first order kinetics and a pseudo-second-order kinetics results. The pseudo-second order (PSO) model gives an R^2 value of 0.9749, while the pseudo-first-order (PFO) model gives an R^2 value of 0.9267. The PSO model provides a better fit to the experimental data. In adsorption science, an R^2 value greater than 0.95 for the PSO model strongly suggests that the rate-limiting step of the mechanism is chemisorption.

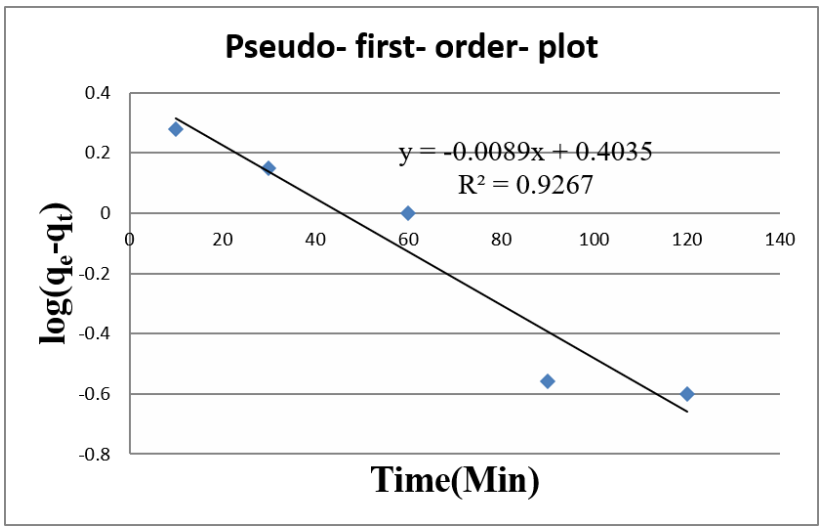

Figure 10: Pseudo - First - Order plot for phenol removal using GSPs.

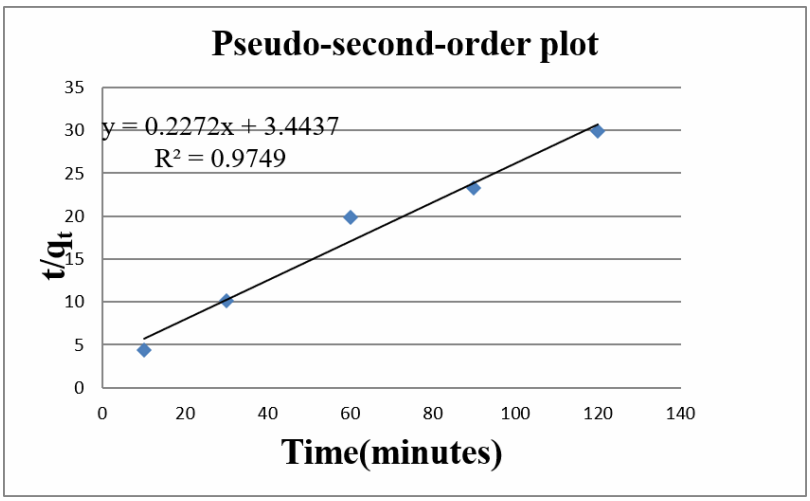

Figure 11: Pseudo- Second - Order plot for phenol removal using GSPs.

The dominance of the PSO model implies that chemical adsorption controls the phenol removal process through hydrogen bonding or π–π interactions, with the silica-grafted sites on the pumice. This behavior is highly desirable for industrial wastewater treatment because chemical bonds are much stronger than physical interactions, ensuring that the adsorbed phenol does not easily desorb back into the aqueous phase. The outcome values for the kinetic parameters q_e, K_1, and K_2 adsorption performance are presented in Table 4.

Table 4: Constants of Kinetic Models.

Pseudo-first-order					Pseudo-second-order				
slope	K1	interce pt	R^2	q_e	slope	K2	interce pt	R^2	q_e
-0.0089	0.0089	0.4035	0.9267	1.497	0.2272	0.015	3.4437	0.9749	4.401

The PSO rate constant (K) has a value of 0.015, which represents the velocity of the adsorption process. A rate constant in this range is typical for modified mineral adsorbents used in industrial wastewater treatment and indicates a steady and controlled uptake of phenol. The calculated equilibrium adsorption capacity (q_e) differs between the two models, with the PFO model showing a q_e value of 1.497 mg g ^{1} and the PSO model with a q_e value of 4.401 mg g ^{1}. Since the PSO model shows the better statistical fit, the q_e value of 4.401 mg g ^{1} is considered the more reliable model for determination of the actual phenol-holding capacity of the GSP adsorbent at equilibrium.

IV. Conclusion And Recommendations

Conclusion

The Industrial effluent had Phenol concentrations (0.59 -8.83) mg/L above the recommended limits by WHO and EPA. This is a clear indication that some industries did not comply with the regulations that require phenol concentrations below 1.o mg/L as per the WHO/EPA regulations. This work reported a low-cost method for extracting silica from RHA, with 87.4% pure amorphous silica obtained.

The study confirmed that that the silica-pumice nanocomposite was an effective adsorbent for the removal of phenol from aqueous solutions. It was observed that the process of adsorption is strongly affected by the experimental parameters such pH, contact time, adsorbent dose and initial concentration of phenol. The optimal conditions were confirmed as pH 6, initial concentration of phenols 2.5 mg/L, contact time 60 minutes, and adsorbent dose 1.0 g.

The experimental data showed a strong match with a Freundlich model, using the regression coefficient of $R^2 = 0.9989$. The adsorption favorability, obtained as n = 2.910064. The kinetics data showed that the adsorption process is controlled by a pseudo-second-order mechanism. The characteristics of silica-pumice adsorbent facilitate the treatment of wastewater by abating phenols.

Recommendations

The condition of water bodies around industrial areas should be tested to check phenol levels and other contaminants before allowed to be consumed by the residents in that vicinity. People should also be sensitized on the adverse effects coming into contact with water having high levels of phenols which might be through food as result of irrigation using water polluted with phenols.

The wastewater phenol levels are above acceptable range thus its recommended treatment using silica-pumice adsorbent before discharge from the industries as mitigation measure for rampant pollution.

Rice husk yielded high amounts of silica and is recommended as low cost source for production of adsorbents which can be modified for abatement various pollutants in water and wastewater

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Conflict of interest

The authors declare no conflict of interests that could have influenced the work reported in this paper.

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