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Comparing the Effectiveness of Activated Carbons Synthesized from Palm Kernel Shell for the Removal of Rhodamine B Dye from an Aqueous Solution

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Abstract:

Activated carbons were prepared from Palm Kernel Shells (PKS) using Sulphuric acid ($PKS_{AC-H_2SO_4}$), Phosphoric acid ($PKS_{AC-H_3PO_4}$) and Zinc Chloride ($PKS_{AC-ZnCl_2}$). They were characterised using the Fourier Transformed Infra-Red and X-ray diffraction techniques and applied in the removal of 5 ppm and 10 ppm concentration Rhodamine B (Rh B) aqueous solutions. The effect of contact time, the mass of adsorbent and change in dye concentration were assessed. The maximum percentage dye removal for 0.5 g was 96.43% for both concentrations with $PKS_{AC-ZnCl_2}$. 99.54% was the highest for the 1.0g adsorbent of $PKS_{AC-H_2SO_4}$ obtained for both concentrations of the dye. The mass to concentration ratio 1.5g: 10 ppm indicated a 99.88% removal of Rhodamine B dye at the 180th minute by $PKS_{AC-ZnCl_2}$, signifying maximum percentage removal. The adsorption kinetics also showed that the process favoured the Pseudo second-order kinetics.

Keywords: Activated carbon, X-ray diffraction, FTIR analysis, Rhodamine B dye, kinetic models

1. Introduction

The African Oil Palm (*Elaeisguineensis*) is a celebrated tree crop that has been harvested in many parts of Africa for centuries [1]. Its yield varies considerably based on management practices, genetics and geography [2]. In 2010, Palm Oil and Palm Kernel Oil production represented 2% of Ghana's total agricultural production value [3]. This is demonstrated by an average annual increase in the production of fresh fruit bunches (FFB) from 1960 to 2010 [4]. The oil palm is the second most important tree crop in the Ghanaian economy after cocoa. [5]. There are problems associated with the waste generated in palm oil mills is that it is a major source of environmental pollution. In 2014, it was estimated that 93.38 million tonnes of empty fruit bunches (EFB), 21.03 million tonnes of oil palm fibre (OPF), and 4.46 million tonnes of oil palm shell (OPS) all together totaled about 118.9 million tonnes of solid Biomass were produced [6].

In promoting zero waste in the oil palm industry, according to Van Dam et al. [7], the palm kernel shells can be used as fuel for the mill, activated carbon, briquette, and particleboard. Activated Carbon (AC) or activated coal has attracted the attention of many researchers due to its excellent properties such as thermal stability, high performance, high adsorptive effect, large surface area and well-developed structure [8]. Commercially produced AC is prepared from solid carbonaceous materials such as a nutshell, wood, lignite and petroleum pitch. Several studies had been done to discover new, inexpensive and renewable raw materials [9]. Being an inexpensive agricultural waste material, it allowed researchers to convert this agricultural waste material into a useful product. PKS contains 51.6% of Carbon [10] which produces a high percentage of char and this increases the product yield. Therefore, it was believed that PKS will act as a good precursor in the production of AC [11]. The main source of discharge of dyes is textile industries where they are used to colouring products. Today there are over 1,000,000 dyes for commercial use and around 700 tons of dyestuffs are produced annually [12]. A dye is generally a substance that bears the affinity to the substrate to which it is being applied. It is often applied in an aqueous solution. They cause water pollution and also pose a serious threat to the environment [13,16]. The types of dyes are mainly basic dyes, acid dyes, direct dyes, reactive dyes, mordant dyes, azo dyes, disperse dyes and sulphur dye.

The Rhodamine B (Rh B) dye is one of fresh peach of synthetic dyes, and it is widely used as a colourant in the manufacturing of textiles and foodstuffs. It has been medically proven that drinking water contaminated with Rhodamine B dyes could lead to subcutaneous tissue born sarcoma, which is highly carcinogenic [14,15]. In this research work, PKS-AC was activated using three different chemical reagents namely: Sulphuric acid, Phosphoric acid and Zinc Chloride. They were used to adsorb Rhodamine B dye from an aqueous solution and their efficiencies were compared using their adsorption kinetics.

2. Materials and Methods

2.1. Sampling

Raw palm kernel shells were obtained from Twifo Oil Palm Plantation Limited in the Central Region of Ghana.

2.2. Synthesis of Adsorbent

2.2.1. Pre-Treatment of Raw Materials

Pre-treatment of raw Palm Kernel Shell (PKS) consists of two main treatments; physical and chemical.

2.2.2. Physical Treatment

The physical treatment process is mainly a size reduction process. The raw PKS were washed, soaked in tap water for two days, decanted and filtered to eliminate water-soluble impurities. It was then dried in an electrical oven (MMM MedcenterEcoCell) at 100 °C for two hours. The dried PKS was milled with a laboratory rotary mill (IKA M20 Universal Mill) to achieve a particle size of 0.75 µm. It was heat-treated in an electrical furnace (Nabertherm SN224350) at 500 °C for 2 hours to eliminate incorporated organic matter, impurities and obtain the pure char.

2.2.3. Chemical Treatment

The charred PKS was activated using three different chemicals namely; Zinc Chloride (ZnCl₂), Phosphoric acid (H₃PO₄) and Sulphuric acid (H₂SO₄). 20% v/v of each chemical compound was prepared in a volumetric flask. 30.0g of the prepared PKS was soaked in each of the prepared chemical solutions for 24hrs. They were then filtered using a Whatman filter paper and washed with distilled water to remove excess acid until a neutral pH is obtained.

2.3. Adsorbent Characterization

Analytical methods used for the characterization of PKS included X-Ray Diffractometry and Fourier Infrared analysis. The functional groups of both raw and synthesized activated carbons were determined by FTIR analysis and phase compositions and were used to measure PKS crystallinity and to reveal the compositional structure of the sample was obtained by X-ray diffraction.

2.3.1. Fourier Transform Infra-Red Analysis of the Synthesized Activated Carbon

The functional groups on the activated carbon were determined using the Mattson FTIR spectrometer which was equipped with a ZnSe crystal plate with a Mercury Cadmium Telluride A (MCTA) detector and KBr as a beam splitter. The samples were made thin enough for the infrared light to transmit through. An air background spectrum was collected at the start of the sample collection [18,19]. The measurements were done using 100 scans at 4 cm units of log (1/R) (absorbance), over the mid-region of 1200 - 400 cm⁻¹.

2.3.2. X-Ray Diffraction Analysis of the Synthesized Activated Carbon

The activated carbons were characterized using the PAN alytical Empyrean Powder X-ray Diffractometer of model 000000011136412 which was used to collect data using Bragg-Brentano's geometry and a slit configuration of a degree fixed divergence slit of 0. 0.4354°. It was equipped with a CuKα radiation source (λ=1.5406Å, K-Alpha1 [Å] =1.54060, KAlpha2[Å] =1.54443 and K-Beta [Å] =1.39225) and operated at 40 mA and 40kV). For phase identification, scans were taken from 2θ = 5.0150 to 69.9650 with a step size of 1.000 [17,18]. The diffraction patterns were identified by comparing them with reference data.

2.4. Preparation of Dye Solutions

The dye used in this work was Rhodamine B because it has strong adsorption onto solids and also for its high solubility in water. A stock solution of Rhodamine B was prepared by dissolving its 0.5 g in 1000 mL distilled water. The stock solution was diluted accordingly to obtain fresh solutions of desired concentrations.

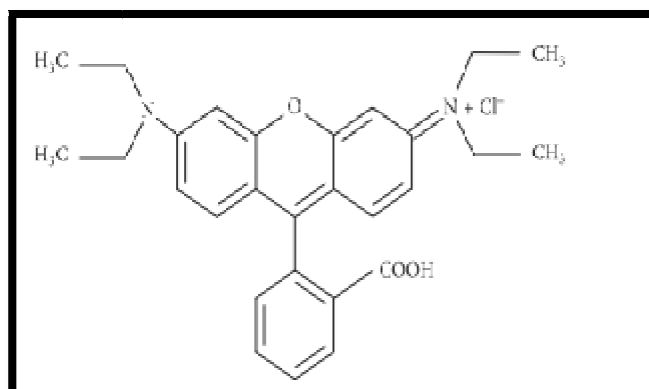


Figure 1: Molecular Structure of Rh-B Dye

2.5. Proximate Analysis of Char or Carbon

2.5.1. Moisture Content

A crucible with lid was taken and weighed. 2.0g of sample was taken in the crucible with lid and weighed. It was kept in a hot air oven at 155 °C for 2 hours. It was taken out and kept in the desiccator. Then the weight was taken out.

Where; M = moisture content

B = mass of crucible with lid plus sample

F = mass of crucible with lid plus dried sample.

G = mass of crucible with lid

2.5.2. Ash Content

A crucible was taken and weighed. 2.0 g of sample of char was taken in the crucible and weighed. The sample was kept in a muffle furnace for 3 hrs at a temperature of 650 °C. Then it was taken out and kept in a desiccator for half an hour to cool down. Then the weight is taken.

Where; A = ash content

G = mass of empty crucible

B = mass of crucible plus sample

F = mass of crucible plus ashed sample

2.5.3. Volatile Matter

A crucible with lid was taken and weighed, 2.0g of char sample was taken in the crucible with lid and weighed. It was kept in the muffle furnace for 7 minutes. Then it was taken out and kept in the desiccator for half an hour to cool down. The weight of the sample in a crucible with a lid was taken.

Where,

V = volatile matter

B = mass of crucible with lid plus sample

G = mass of empty crucible

F = mass of crucible with lid plus ash sample

M = moisture content

2.5.4. Carbon Content

Carbon content is calculated by

2.6. Determination of Percentage Removal /Reduction

The percentage removal of materials from the oil is calculated using the formulae.

Where:

R% = Percentage Removal / Reduction

Co = Initial Concentration (mg/L)

Ct = Final Concentration (mg/L)

2.7. Kinetic Studies

2.7.1. Determination of Pseudo-First and Second-Order Reaction

The Pseudo-First Order Reaction will be determined using the formulae;

Determination of Pseudo-Second Order Reaction (HO's Model)

Where: q_e = amount of the absorbent absorbed at equilibrium (mg/g)

q_t = amount of the absorbent absorbed at a time, t (mg/g)

K_2 = rate constant of second-order adsorption (g/mg/min)

t = time (min)

3. Results and Discussion

3.1. Proximate Analysis of Char

The analysis from Table 1 shows the various contents expressed in percentages where carbon content gave the highest yield. From this analysis, it can be concluded that palm kernel shell has a high carbon content. Hence, it can be used as a precursor to producing activated carbons. [20]

Proximate Analysis	Value (wt %)
Moisture content	3.0
Ash content	2.0
Volatile matter	44.61
Carbon content	50.0

Table 1: Proximate Analysis of Char or Carbon

3.2. Fourier Transform Infrared Analysis of the Synthesized Carbon

FTIR was used to analyse the surface functional groups on $\text{PKS}_{\text{AC-H2SO4}}$, $\text{PKS}_{\text{AC-H3PO4}}$, $\text{PKS}_{\text{AC-ZnCl2}}$ and raw PKS. These functional groups according to the literature are the volatile materials and impurities released at the applied elevated temperature [22, 26]. From Figure 2, it can be deduced that there was a strong and broadband of C=C stretching at 1580.89 cm^{-1} in the spectra of the activated PKS sample, proving the presence of an aromatic ring group [23].

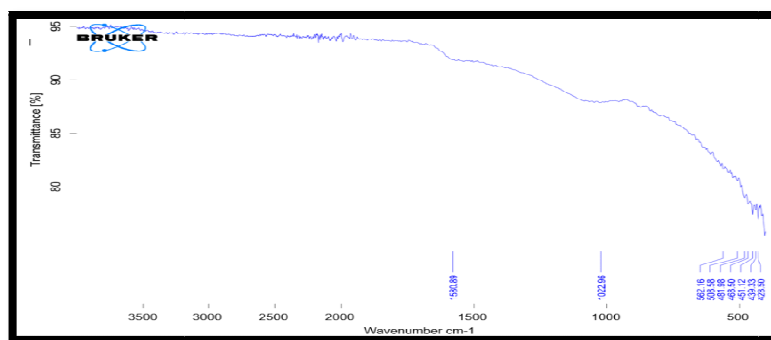
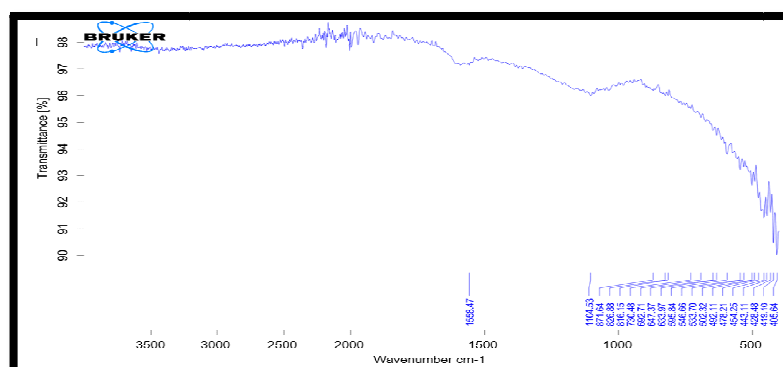


Figure 2: Fourier Transform-Infrared Spectra of $\text{PKS}_{\text{AC-H2SO4}}$

From Figure 3, it can be deduced that there was a strong and broadband of C=C stretching at 1558.47 cm^{-1} in the spectra of the activated PKS sample, proving the presence of an aromatic ring group [23].



From Figure 5, a strong band of wavelengths of 2334.39, 2033.76, 2010.48, 21156 cm^{-1} were observed in the FTIR spectrum for raw PKS with corresponding possible functional groups of C=C, C \equiv N Nitriles, C \equiv C stretching vibration for Alkyl groups and silicon compounds. However, this band is weaker and were mostly eliminated in the spectra during the carbonization or activation process, suggesting that decomposition of biomass constituents occurred. [23, 24].

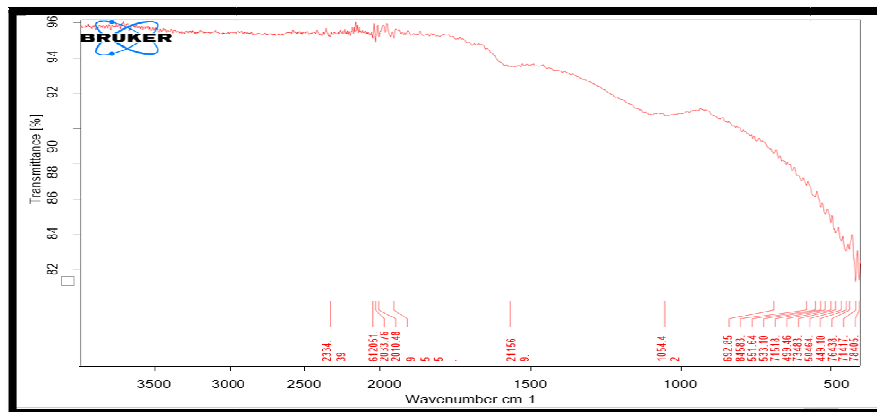


Figure 5: Fourier Transform-Infrared Spectra of Raw PKS

3.3. X-Ray Powder Diffraction (XRD)

Figure 6, shows the broad peak shape of the diffractogram which suggests that the PKS powder was in the amorphous state while very small sharp peaks suggest a small number of microcrystalline materials could be present. PKS is largely amorphous as its crystallinity is only 14.38%, which is indicative of its high lignin and hemicellulose contents [25, 27].

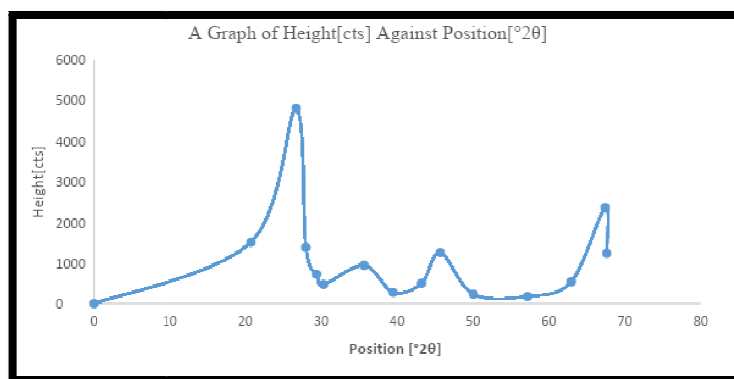


Figure 6: X-ray Diffraction Spectrum of PKS_{AC-H2S04}

Figure 7 also shows the broad peak shape of the diffractogram which suggests that the PKS powder was in an amorphous state while very small sharp peaks suggest a small number of microcrystalline materials could be present. PKS is largely amorphous as its crystallinity is only 14.38%, which is indicative of its high lignin and hemicellulose contents [25, 27].

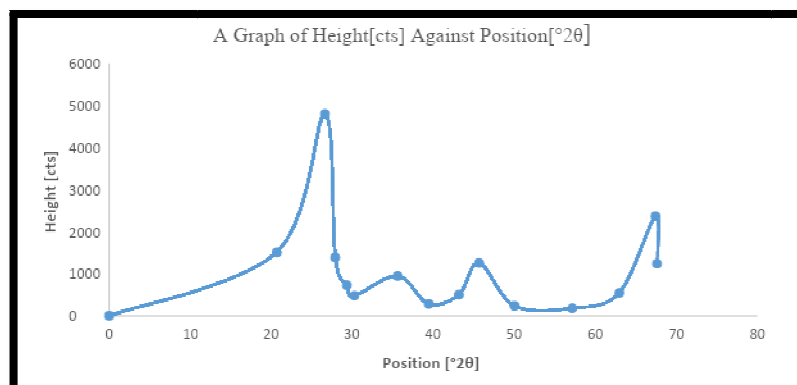


Figure 7: X-ray Diffraction Spectrum of PKS_{AC-H3P04}

Figure 8, the results of XRD show crystal peaks corresponding to d-4.28, d-4.29 and d-4.25. A peak at d-4.26 and 2θ equal to 20.6 is likely silicon oxide, which is a component of PKS. Oleic acid, identified at d-4.19, is likely residue from palm oil and palm kernel oil [25, 27].

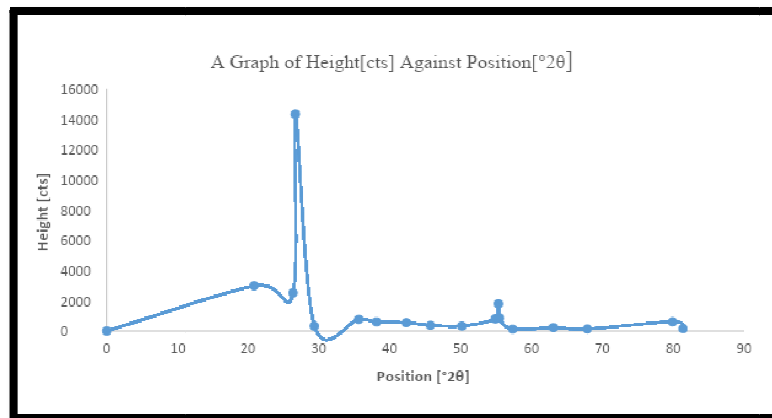


Figure 8: X-ray Diffraction Spectrum of $PKS_{AC-ZnCl_2}$

Figure 9 shows the broad peak shape of the diffractogram which suggests that the PKS powder was in an amorphous state while very small sharp peaks suggest a small number of microcrystalline materials could be present. PKS is largely amorphous as its crystallinity is only 14.38%, which is indicative of its high lignin and hemicellulose contents [26, 27]. From this, it can be concluded that silicon oxide was present in all samples. Since adsorption proceeds with weaker bonds, it can be concluded that these kinds of AC will find application in waste management such as the elimination of Chemical Oxygen Demand (COD) and adsorption of organic pollutants [29, 30].

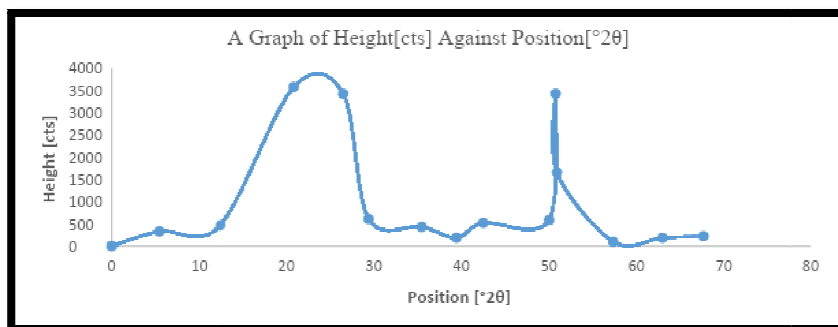


Figure 9: X-Ray Diffraction Spectrum of Raw PKS

3.4. Adsorption Studies

Results for percentage reduction for the three activated adsorbent dosages (0.5, 1.0, 1.5g) on 5ppm concentration of Rhodamine B dye solution are shown in the figures below. Considering Figure 10, the activity of the activated carbons was rapid and most effective in the first 30mins with 60-80% dye reduction. The highest percentage reduction was achieved by $PKS_{AC-ZnCl_2}$ which was 96.44% in the 180th minute.

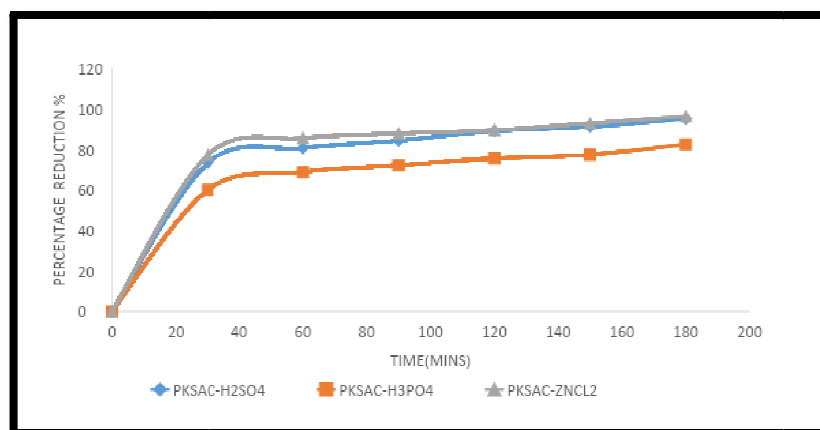


Figure 10: A Graph Showing the Percentage Reduction with Time for PKS_{AC} (0.5g) Application on 5 Ppm Aqueous Solution

From Figure 11, it can be seen that the activated carbons were rapid and most effective in the first 30mins with 70-90% dye reduction. The reduction is however appreciable with a gentle curve up to about two hours when the reduction level is about 99% of activity. The highest percentage reduction of the dye was achieved by $\text{PKS}_{\text{AC-H}_2\text{SO}_4}$ which was 99.54% in the 180th minute.

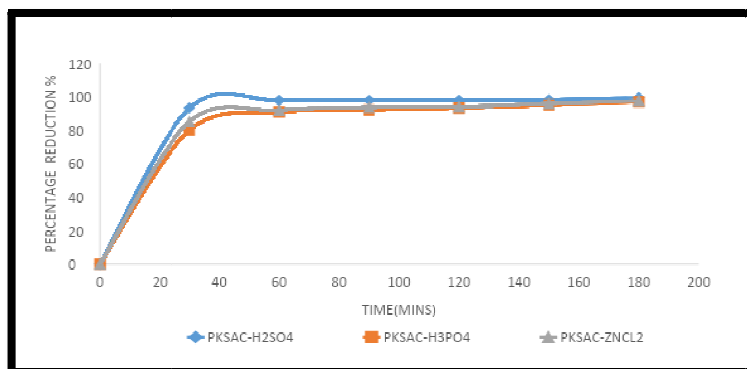


Figure 11: A Graph Showing the Percentage Reduction with Time for PKS_{AC} (1.0g) Application on 5 Ppm Aqueous Solution

From Figure 12, the activity of the activated carbons was rapid and effective in the first 30mins with 90-92% dye reduction. The highest percentage reduction was attained by the $\text{PKS}_{\text{AC-H}_2\text{SO}_4}$ was 99.84% in the 180th minute.

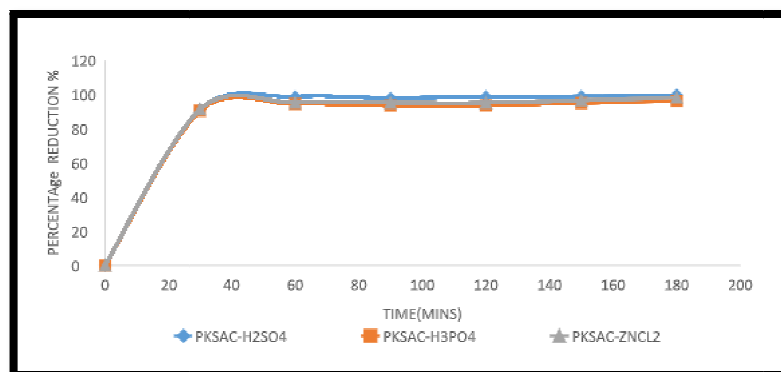


Figure 12: A Graph Showing the Percentage Reduction with Time for PKS_{AC} (1.5g) Application on 5ppm Aqueous Solution

From Figure 13, it can be deduced that the activity of the activated carbons was effective in the first 30mins with 48-61% dye reduction. The highest percentage reduction was attained by the $\text{PKS}_{\text{AC-ZnCl}_2}$ was 92% in the 180th minute.

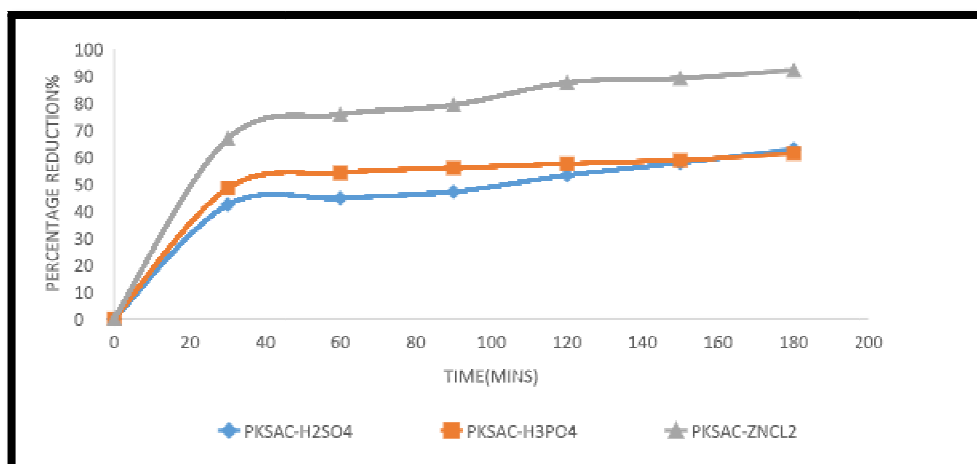


Figure 13: A Graph Showing the Percentage Reduction with Time for PKS_{AC} (0.5g) Application on 10 ppm Aqueous Solution

Figure 14, shows that the activity of the activated carbons was rapid and effective in the first 30mins with 29-85% dye reduction. The highest percentage reduction was attained by the $\text{PKS}_{\text{AC-H}_2\text{SO}_4}$ was 96.13% in the 180th minute.

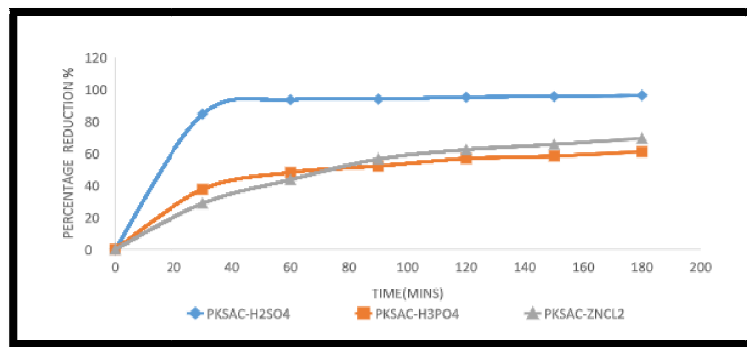


Figure 14: A Graph Showing the Percentage Reduction with Time for PKS-AC (1.0g) Application on 10ppm Aqueous Solution

Considering Figure 15, it can be deduced that the activity of the activated carbons was effective in the first 30mins with 67-87% dye reduction. The highest percentage reduction was attained by the PKS_{AC-ZnCl2} was 99.88% in the 180th minute.

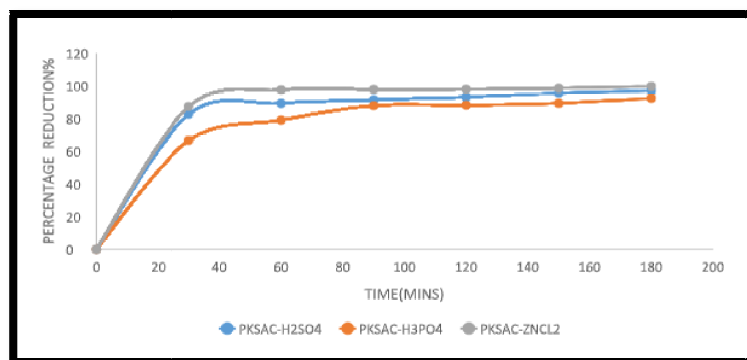


Figure 15: A Graph Showing the Percentage Reduction with Time for PKS-AC (1.5g) Application on a 10ppm Aqueous Solution

3.5. Kinetics Studies

The kinetics of the adsorption process was investigated using the pseudo-first and second-order kinetic models. The graphical representations of the first and second pseudo kinetic models are presented below.

3.5.1. Pseudo-first order Kinetics

The graphs shown below illustrate the Pseudo First-order kinetics for the reduction of dye concentration.

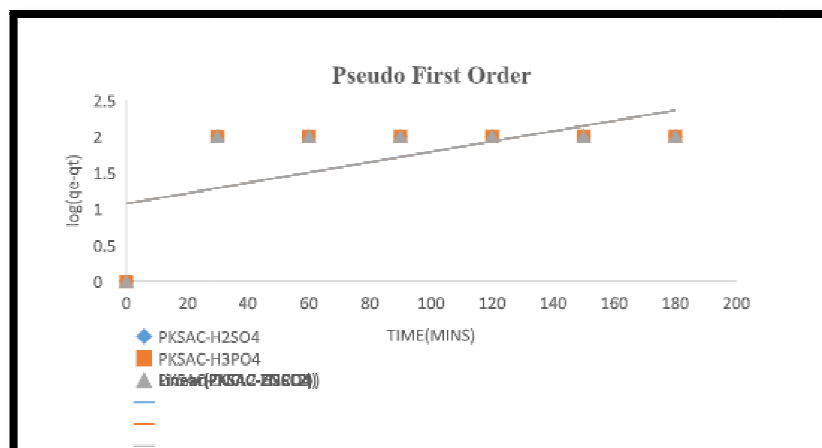


Figure 16: Pseudo First Order Kinetics Graph for 5ppm Dye Concentration

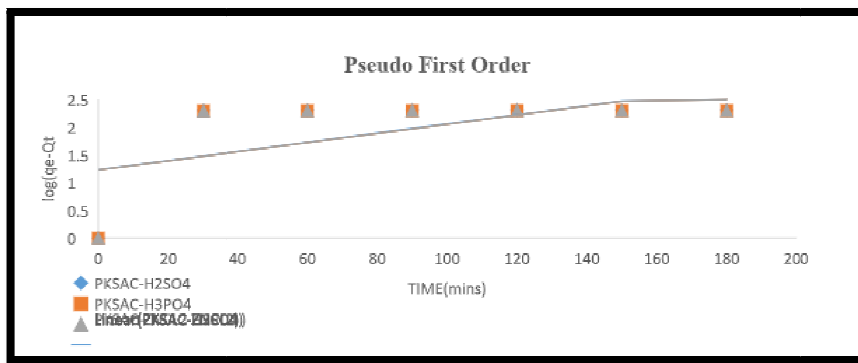


Figure 17: Pseudo First Order Kinetics Data for 10 Ppm Dye Concentration

It can be seen from Table 2 and Table 3 that the reduction of dye concentration in the dye solution process does not favour the first-order process because their R^2 values were found to be 0.8079 for all the three activated carbons respectively for 5ppm concentration of the dye solution. The kinetic constants (K_1) for 5ppm is 0.0154 for all the carbons with 101.035 as there.

The summary of the 10ppm dye concentration shows that the R^2 values obtained are 0.8085 for all the carbons respectively and kinetic constants (K_1) obtained for 10ppm is 0.0177 for all carbons with 205.805 s their respectively.

Sample			
PKSAC-H ₂ SO ₄	0.8079	0.0154	101.035
PKSAC-H ₃ PO ₄	0.8079	0.0154	101.035
PKSAC-ZnCl ₂	0.8079	0.0154	101.035

Table 2: Pseudo First Order Kinetic Data for 5ppm

Sample			
PKSAC-H ₂ SO ₄	0.8085	0.0177	205.805
PKSAC-H ₃ PO ₄	0.8085	0.0177	205.805
PKSAC-ZnCl ₂	0.8085	0.0177	205.805

Table 3: Pseudo First Order Kinetic Data for 10ppm

3.5.2. Pseudo-Second Order Kinetics

From the graphs shown below, it can be seen that after three (3) hours of treatment of the solution have improved reduction of the dye, and it could be better after that time of treatment.

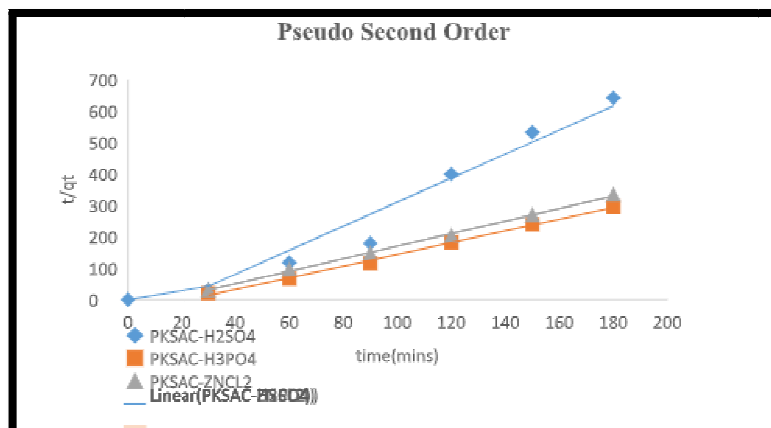


Figure 18: Pseudo Second Order Kinetic Data for 5ppm

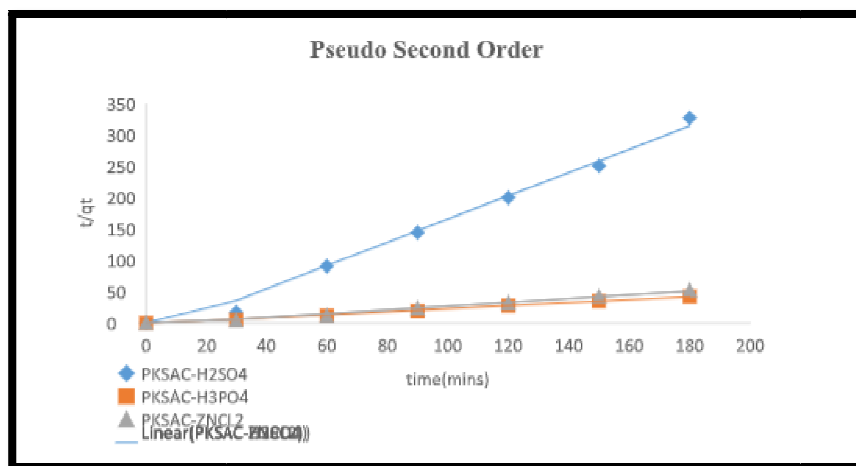


Figure 19: Pseudo Second Order Kinetic Data for 10ppm

The adsorption kinetics favours the second-order [31] as shown in Table 4 and Table 5 with R^2 values of 0.9683, 0.9882 and 0.9957 for 5ppm and R^2 values for 10ppm dye concentration are 0.9923, 0.9970 and 0.9917 for all the three carbons respectively. The kinetic constants (k_2) for 5ppm were 3.2681, 1.5369 and 1.7689 with 101.035 as their $1/k_2$ respectively. For 10ppm dye concentration, their kinetic constants (k_2) were 1.7017, 0.2280 and 0.2739 with 205.805 as their $1/k_2$.

Sample	R^2	k_2	$1/k_2$
PKSAC-H ₂ SO ₄	0.9683	3.2681	101.035
PKSAC-H ₃ PO ₄	0.9882	1.5369	101.035
PKSAC-ZnCl ₂	0.9957	1.7689	101.035

Table 4: A Table of Pseudo-Second Order Kinetic Data for 5 Ppm Dye Concentration

Sample	R^2	k_2	$1/k_2$
PKSAC-H ₂ SO ₄	0.9923	1.7017	205.805
PKSAC-H ₃ PO ₄	0.9970	0.2280	205.805
PKSAC-ZnCl ₂	0.9917	0.2739	205.805

Table 5: A Table of Pseudo Second Order Kinetic Data for 10 Ppm Dye Concentration

4. Conclusion

Activated carbons prepared from Palm Kernel Shell was used to eliminate the majority of the Rhodamine B dye from the solution through the adsorption process. Kinetics of adsorption was best described by Pseudo Second Order Kinetic Model. From the data obtained from the variations of the adsorbent doses at a different contact time and different dye concentrations, it can be deduced that an adsorbent dose of 1.5g of PKS_{AC-ZnCl₂} at the 180th minute had the highest percentage reduction thus 99.88%, this indicates that this adsorbent and dose was very effective for this particular concentration thus 10ppm. The findings showed that the removal mechanism of the pollutants by PKS-AC is dependent on the adsorption process proceeding through the surface functional groups available on PSK-AC which might contribute to the treatment of Rh B dye solution. Conclusively, this research work has demonstrated that palm kernel shell, an agricultural waste, non-toxic and bio-degradable material could be used as an effective adsorbent for the removal of Rhodamine B dye from wastewater.

5. Authors' Contributions

This work was carried out in collaboration among all authors. Author ES designed the study, performed the statistical analysis and wrote the manuscript. Authors MAA and AKA managed the analysis of the study. All authors read through the final manuscript.

6. Data Availability

All the data concerning this research work are with the authors.

7. Conflicts of Interest

There are no conflicts of interest regarding this research work.

8. Acknowledgements

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