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Removal of Azo Dye (Acid Red 18) from Aqueous Solution using a Bioadsorbent from *Carica Papaya* Peels

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Abstract

This study evaluates the removal of Acid Red 18 (AR 18) dye from aqueous medium using an adsorbent developed from *Carica papaya* peel. The adsorbent was prepared by carbonization at 500^{o}C followed by activation with NaOH solution. Moisture content, ash content and volatile matter of the adsorbent were determined to be 2.87%, 11.0% and 89.0% respectively. FTIR characterization which was done before and after activation indicated some functional group modifications. The parameters affecting sorption such as initial sorbate concentration, adsorbent dosage, contact time, temperature and pH were evaluated and average percentage removal of AR 18 by the adsorbent was found to be 70%. Correlation of the experimental data with Langmuir and Freundlich adsorption isotherms revealed linear coefficient (R^2) values of 0.802 and 0.720 respectively; thus implying that the Langmuir isotherm best describes the adsorption process. Lagergren pseudo first order and pseudo second order models were employed to test the sorption kinetics and our findings indicated that the sorption of the dye followed pseudo second order kinetics.

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Keywords: Sorption, *Carica papaya*, Azo dyes, Acid red 18, Adsorption isotherms, Kinetics

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1. Introduction

One of the major causes of water pollution is the indiscriminate discharge of dyes into water bodies by textile dyers [1]. There are more than 10,000 dyes used in textile manufacturing alone, with about 70% being azo dyes which are synthetic in nature and complex in structure [2]. About 10-15% of these dyes are usually lost during

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different processes of textile manufacturing [3]. The release of such dyes into water bodies is mostly associated with their incomplete exhaustion onto textile fibres during dyeing processes. These highly coloured substances not only contaminate water sources but also destroy its aesthetic nature and interfere with the path of sunlight into the stream hence affects aquatic life by reducing photosynthetic action. Dye contaminated water may also contain some toxic or carcinogenic chemicals [4]. Examples of such dyes include alizarin, pure blue B, acid red 151, basic red 18, acid red 88, acid red 18 (azoic dyes), direct dyes, mordant dyes, vat dyes, reactive dyes and sulphur dyes. The ones with potential carcinogenic effects include dyes that contains heavy metals and aromatic azo dyes that release amines such as benzidine, 2-naphthylamine, 4-aminodiphenyl and 4-chloro-o-toluidine [5].

Azo dyes, characterized by a chromophoric azo (-N=N-) bond, account for 70% of the 9.9 million tons of industrial dye colorants used annually [6, 7]. These dyes have become an industrial choice worldwide due to low cost, ease of preparation, versatility, fastness, and intensity of colour [8]. Several industries like textile, leather, printing, fast food, and cosmetics flourish due to the prevalent use of azo dyes. Though, these industries contribute notably to a nation's economy, they have become an environmental challenge due to the injudicious discharge of untreated or partially treated effluents into the water, which is often directly used for irrigation [9].

The need to reduce the amount of residual dyes in textile effluents has become a major concern in recent years. The commonly used methods for the removal of dyestuff include sedimentation, ultra-centrifugation, coagulation, ozonation, reverse osmosis and adsorption [10]. Among all these methods, adsorption is one of the most important methods to remove the organic and inorganic substances from wastewater [11, 12]; it is preferably used due to its high efficiency, easy handling, availability of different bio-adsorbents and cost effectiveness.

Adsorption is the adhesion of molecules or ions on the surface of liquid or solid substances. Adsorbents used for adsorption processes can be natural such as clays, charcoal, zeolites, silica or Synthetic such as activated carbon, carbon nanotubes and chemicals. Modified bio-waste materials, ferrites, polymers/biopolymers, composites of various materials can also be used as adsorbents [13]. Among all these adsorbents, activated carbon prepared from bio-waste materials are widely used because they are low-cost adsorbents. These are mostly agricultural and industrial wastes converted into activated charcoal by means of physical and chemical modifications such as heating, pyrolysis and treatments with acids or bases [14].

The large quantity of C*arica papaya* peel (CPP) obtained from local consumption all over Nigeria and especially those from beverage industries have been of no use over the years. Studies have shown that these wastes consist of lignin and cellulose which are known to contain a variety of functional group such as hydroxyl and carbonyl that can help in ammonium ion binding [15]. These components could provide good adhesion surface area for the adsorption of azoic dyes from wastewater.

In this study, papaya peel activated carbon was prepared and evaluated for its efficiency to remove acid red 18 dye from aqueous medium. Sorption equilibrium studies were carried out through Langmuir and Freundlich isotherms while the kinetics were studied via the Lagergren pseudo first order and second order models. The effects of solution pH, adsorbate initial concentration and temperature one sorption capacity of the adsorbent were evaluated. This study could lead to the development of a cheap adsorbent for the treatment of dye-polluted waters.

2. Materials and Method

2.1. Collection and Preparation of Carica papaya Peels

Carica papaya peels were collected as wastes from fruit sellers within Wukari, Taraba State Nigeria. They were thoroughly washed with water and sun-dried for a day followed by oven-drying at 105 °C for 24 hours. The dried peels were then pulverized and stored in an air-tight container for further use.

2.2. Characterization of Carica Papaya Peel Powder (CPPP)

2.2.1. Determination of Moisture Content

One gram (1.0 g) of CPPP was placed in a weighed crucible and dried in an oven at 105 °C to a constant weight (W_2). The percentage weight loss gives the moisture content and it is calculated as follows:

$\% \text{ Moisture Content} = \frac{W1-W2}{W1} \times 100$

where W_1 and W_2 are the weights of sample before and after drying respectively

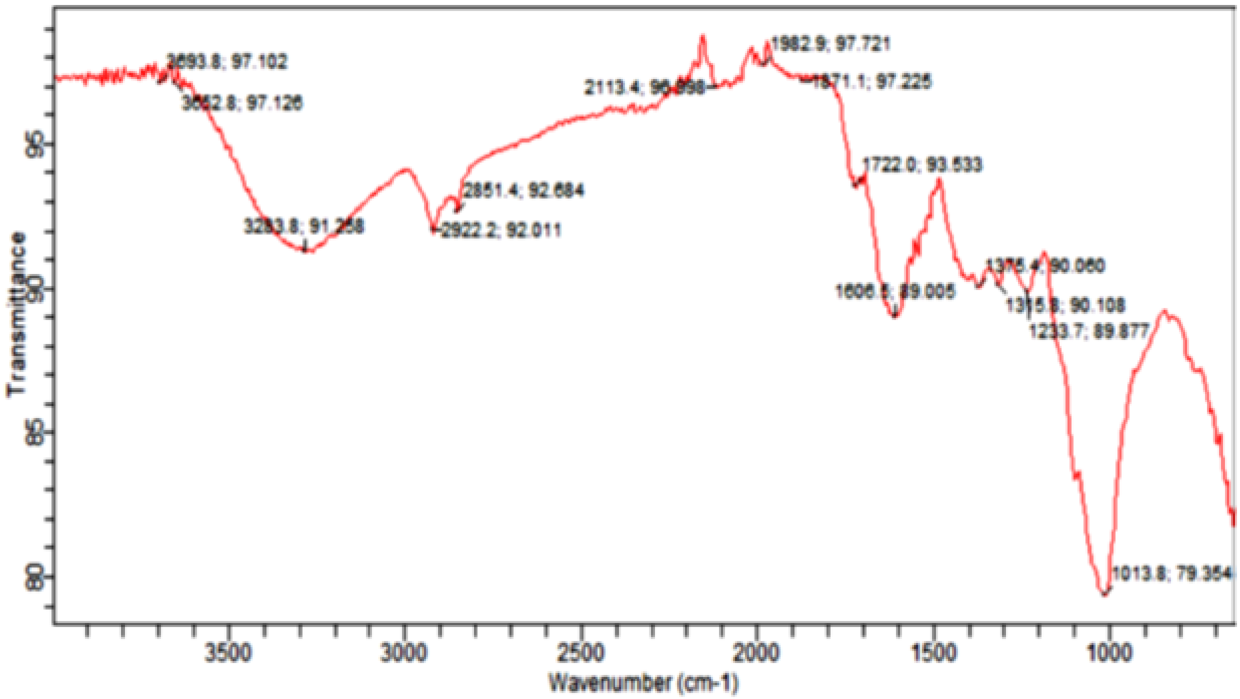

Figure 1. FTIR Spectra of *Carica Papaya* Peel Powder

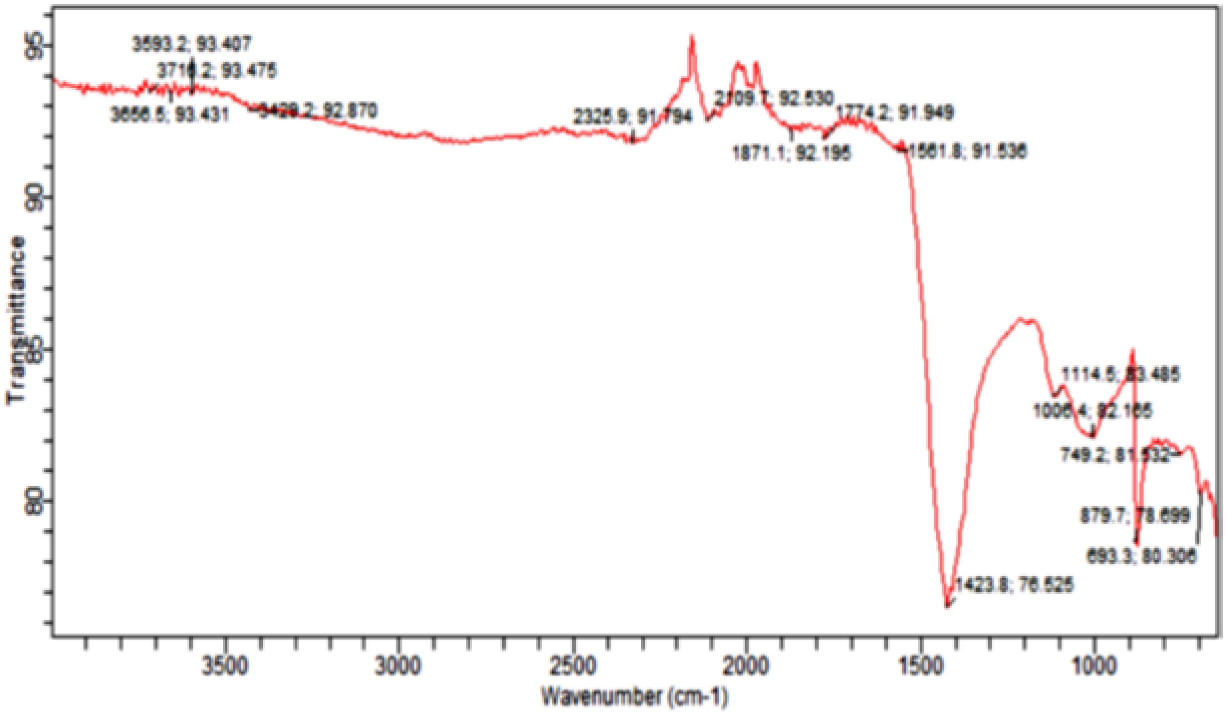

Figure 2. FTIR Spectra of Activated *Carica Papaya* Peel Powder (Adsorbent)

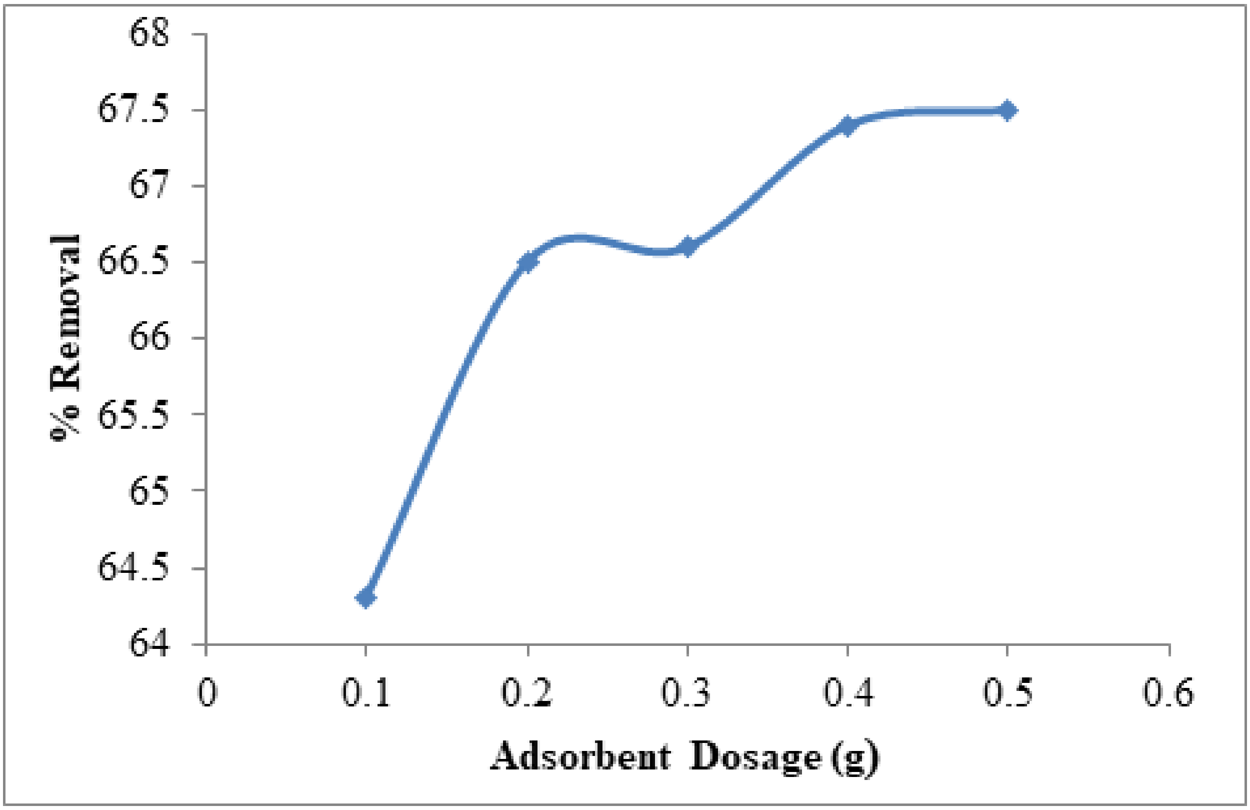

Figure 3. Percentage removal of AR 18 Dye from aqueous solution at varying adsorbent Dosage

2.2.2. Determination of Ash Content and Volatile Matter

Two grams (2.0 g) of CPPP were placed in a crucible and charred on a stove flame until smoking stops. After cooling, it was transferred into the muffle furnace and ashed at 550 ^{0}C for 24 h. The ash was placed in a desiccator to cool and then weighed. Ash content and volatile matter were calculated as follows:

$$\%\text{Ash Content} = \frac{\text{Final weight}}{\text{Initial weight}} \times 100$$

$$\%\text{Volatile matter} = 100 - \%\text{Ash Content}$$

2.3. Preparation of Adsorbent

Carica papaya peel powder (CPPP) was activated by soaking in 2 % NaOH solution for 48h and then filtered. The resulting activated *Carica papaya* peel powder (ACPPP) was washed with distilled water to neutrality and dried in the oven. It was then carbonized at 500 ^{0}C in a Muffle furnace for 5 h, cooled and stored in an air-tight container for further experiments.

2.4. FTIR Analysis

FTIR analysis was carried out on the untreated and the NaOH-treated CPPP. A blank KBr disc was prepared as the background control then, 1 mg of sample was mixed together with 200 mg KBr to produce the sample disc for analysis. FTIR spectra were recorded using Perkin Elmer Spectrophotometer in the range from 400 to 4000 cm^{-1}.

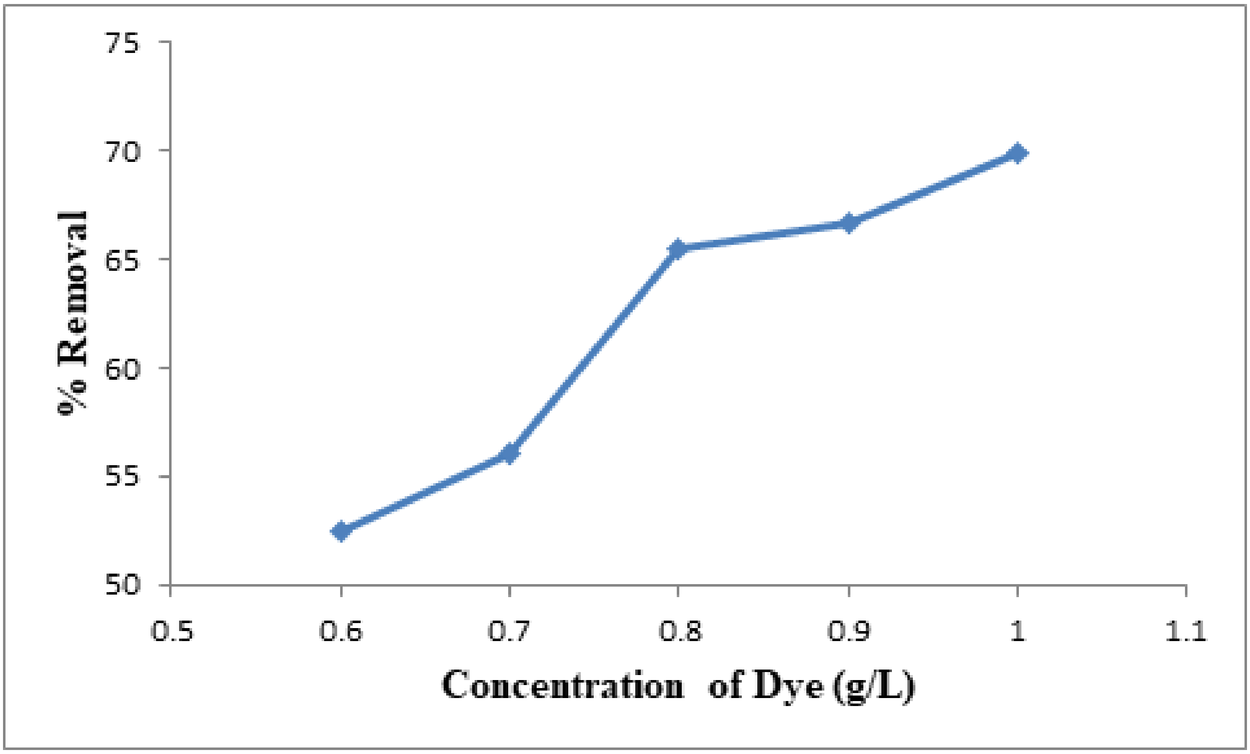

Figure 4. Percentage removal of AR 18 dye from aqueous solution at varying initial dye concentration

2.5. *Sorption Studies*

The stock solution of Acid Red 18 was prepared according to the method described by Bharathi and Ramesh [16]. One gram (1.0 g) of the azoic dye was dissolved in 1000 ml of distilled water to make a 1g/L solution. This prepared solution serves as the ideal dye solution for the study. Batch adsorption tests were carried out using 100 ml screw-cap conical flasks into which were placed known amounts of adsorbent and 50 ml of test solution for each experiment.

2.5.1. *Effect of Adsorbent Dosage on Sorption*

Fifty (50) mL of Acid red dye solution in beakers; A, B, C, D and E were mixed with 0.1, 0.2, 0.3, 0.4 and 0.5 g of the adsorbent respectively and stirred continuously at room temperature for 30 minutes. The mixtures were filtered and the residual dye concentrations (C_e) were determined at a wavelength 506 nm using a UV-Vis Spectrophotometer. The percentage dye removal was estimated as follows:

$$\%\text{Removal} = \frac{Initial\ conc.\ \ (Co) - Residual\ Conc.\ (Ce)}{Initial\ sorbate\ conc.} \times 100$$

2.5.2. *Effect of Initial Dye Concentration*

Five different concentrations (0.6, 0.7, 0.8, 0.9 and 1g/L) of AR 18 dye solution were prepared and 50 mL of each concentration was stirred continuously with 0.1 g of ACPPP for 30 min. The residual concentration of AR 18 at equilibrium was determined as previously described.

2.5.3. *Effect of Temperature on Sorption*

ACPPP (0.1g) was added to 50ml of the AR 18 dye solution at an initial concentration of 1g/L. Sorption studies were carried out at different temperatures (30, 40, 50, 60 and 70 ° C). In each case, the mixture was stirred for 30 minutes using heat magnetic stirrer and percentage dye removal was determined as previously described

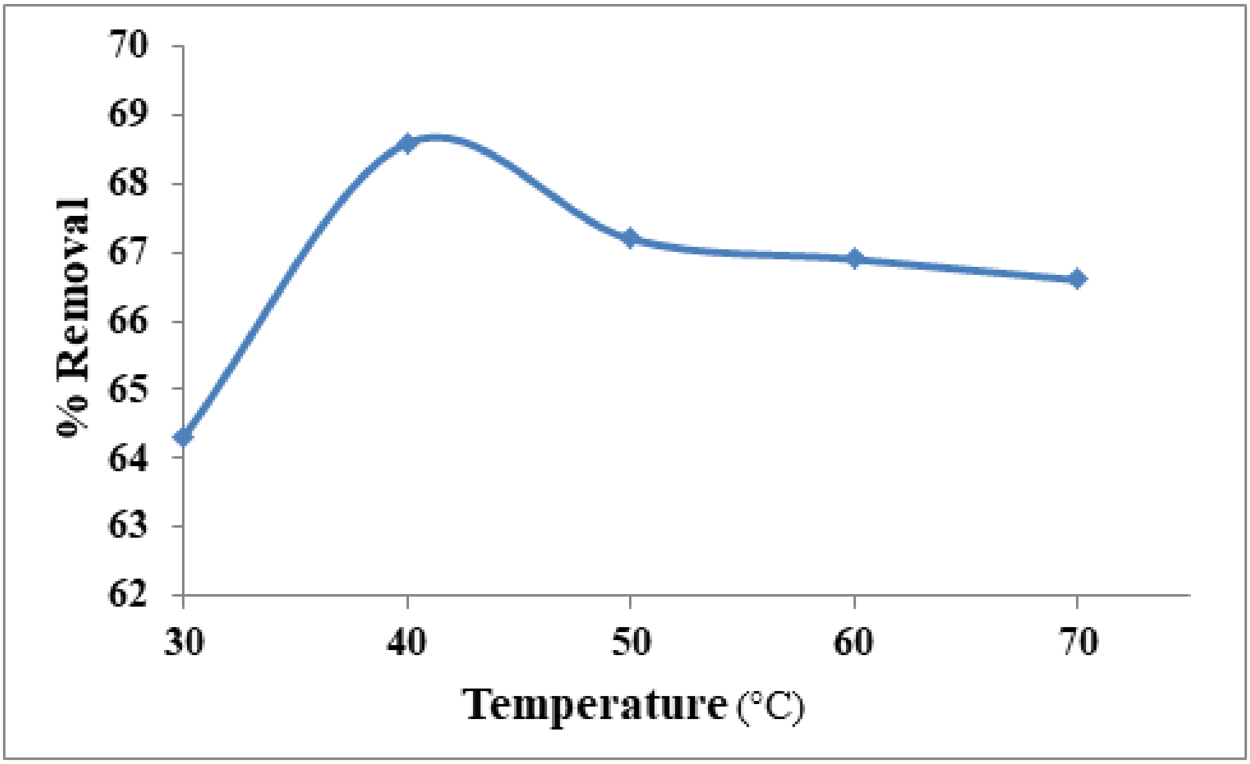

Figure 5. Percentage removal of AR 18 Dye from aqueous solution at varying temperatures

2.5.4. *Effect of Contact Time on Sorption*

Batch experiments were done using 0.1g of adsorbent in 50ml of dye solution (1g/L) at normal room temperature. The mixtures were stirred continuously for different time interval (5, 20, 40, 60 and 90 min) and then percentage dye removal was determined

2.5.5. *Effect of pH on Sorption*

The effect of pH was determined by batch experiments using adsorbent dosage of 0.1g in 50 ml of dye solution at room temperature. 0.1 M HCl and 0.1 M NaOH were used to adjust the pH values of the solutions followed by the addition of the adsorbent. The mixtures were allowed to run on a magnetic stirrer for 30 min followed by the determination of percentage dye removal.

2.6. *Sorption Isotherms*

Langmuir and Freundlich Models were used characterize the adsorption process. The Langmuir treatment is based on the assumption that a maximum adsorption corresponds to a saturated monolayer of solute molecules on the adsorbent surface. The linear form of the equation is expressed as;

$$\frac{Ce}{Qe} = \frac{1}{kqm} + \frac{1}{qm}Ce$$

where qm is the monolayer capacity (mg/g) and k is the adsorption equilibrium constant which expresses the extent of affinity between the sorbate and the adsorbent, Ce is the residual concentration of adsorbate, qe is the amount of adsorbate (Acid red 18) sorbed per gramme of adsorbent (mg/g). The monolayer coverage is obtained from the straight line plot of $\frac{Ce}{Qe}$ versus Ce.

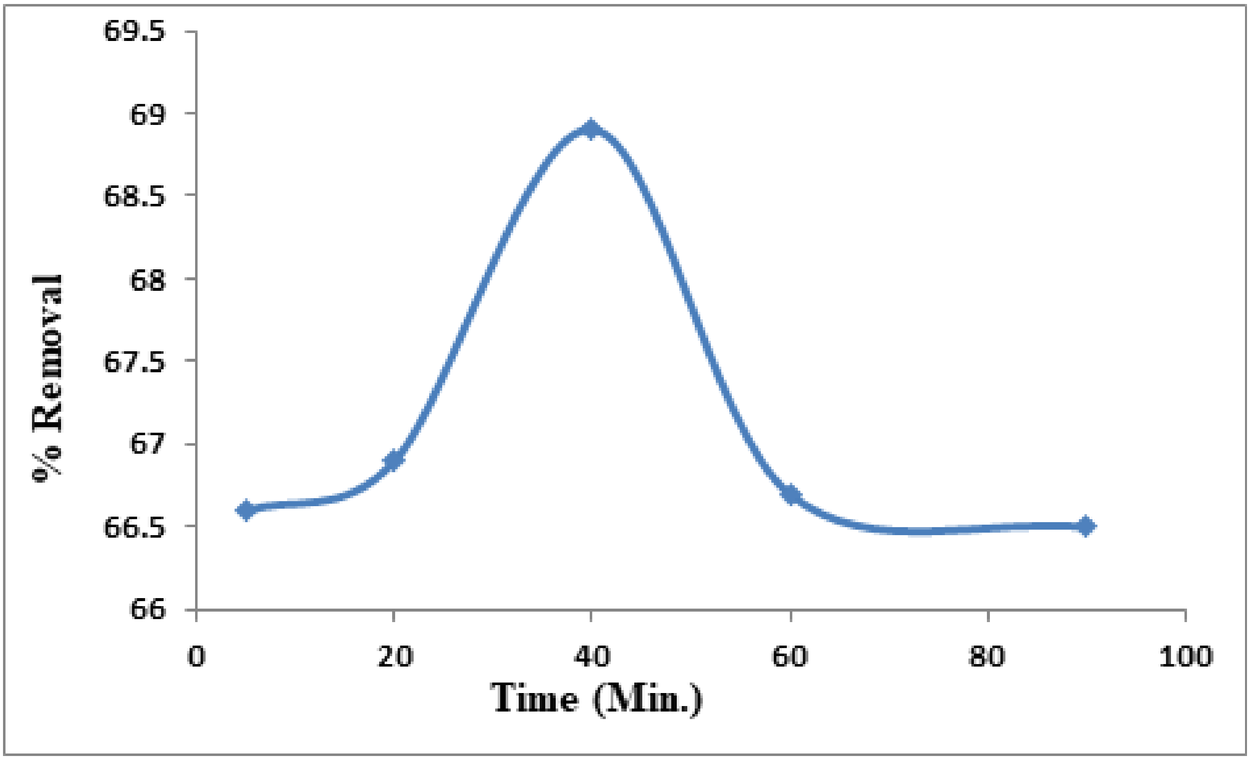

Figure 6. Percentage removal of AR 18 Dye from aqueous solution at varying contact time

The Freundlich isotherm is an empirical expression which shows the heterogeneity of the surface and multilayer adsorption to the binding sites located on the surface of the sorbent. The equation is given as follows:

$$lnqe = \ln(Kf) + \frac{1}{n}Ce$$

where Kf is the measure of sorption capacity and n is the adsorption intensity are Freundlich coefficients, obtainable from the slope ln(qe) against ln(Ce).

2.7. *Sorption Kinetics*

The pseudo first-order and second order kinetic models were applied to describe the kinetics of dye sorption by ACPPP. This first order equation is given as

$$\ln(\,q_e - q_t) = q_e - k_1 t$$

The pseudo second-order kinetic model is represented by the equation

$$\frac{t}{Qt} = \frac{1}{k_2 q_e^2} + (\frac{1}{qe})t$$

where q_t and q_e are the amounts of solute sorbed per mass of sorbent (mgg^{-1}) at any time (t) and at equilibrium respectively and k_1 and k_2 are the rate constants for first order and second order sorption respectively.

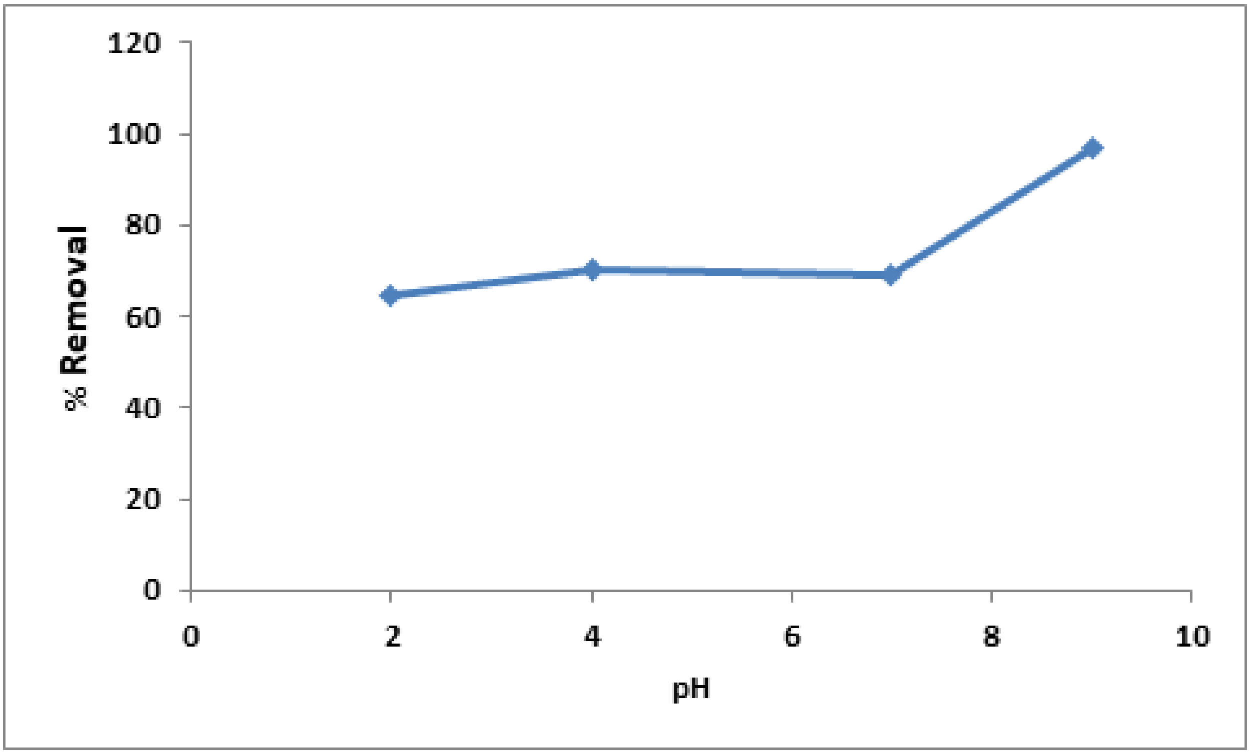

Figure 7. Percentage removal of AR 18 from aqueous solution at varying pH 3.3 Sorption Isotherms

Table 1. Moisture content, Ash content and Volatile matter of *Carica Papaya* peel

PARAMETER	(%)
Moisture Content	2.87
Ash Content	11.0
Volatile Matter	89.0

3. Results and Discussions

3.1. Characterization of Adsorbent material

The moisture content of papaya peel was determined to be 2.87% (Table 1). High moisture content in an adsorbent material could be an indication of low pore volume, low storage stability and low sorption capacity [17]. The low moisture content obtained in this study therefore suggests a good sorption potential. The ash content of *Carica papaya* peel powder determined in this study was 11.0%. Ash content reflects the total mineral content of a material and minerals are known to occupy space and create low pore space within the surface of an adsorbent and consequently lead to low sorption capacity. Volatile matter was estimated to be 89.0 %; this indicates the presence of high organic matter which is expected in plant materials.

Figures 1 and 2 show the FTIR spectra of the sorbent material before and after activation with NaOH. The spectra show bond stretching that indicate the presence of phenols (O-H), carboxyl group (COOH), carbonyls (C=O), amine (N-H), esters (COOR), ethers (COR), alkane (C-H) and alkyne. The disappearance and reduction in the intensity of some peaks were observed after activation implying some form of modification of the adsorbent material.

3.2. Sorption Studies

3.2.1. Effect of Adsorbent Dosage

Figure 3 shows the percentage dye removal as a function of adsorbent dosage. It can be observed that the percentage dye removal increased with increasing adsorbent dosage from 0.1 to 0.4 g. Above this dosage, no further increase

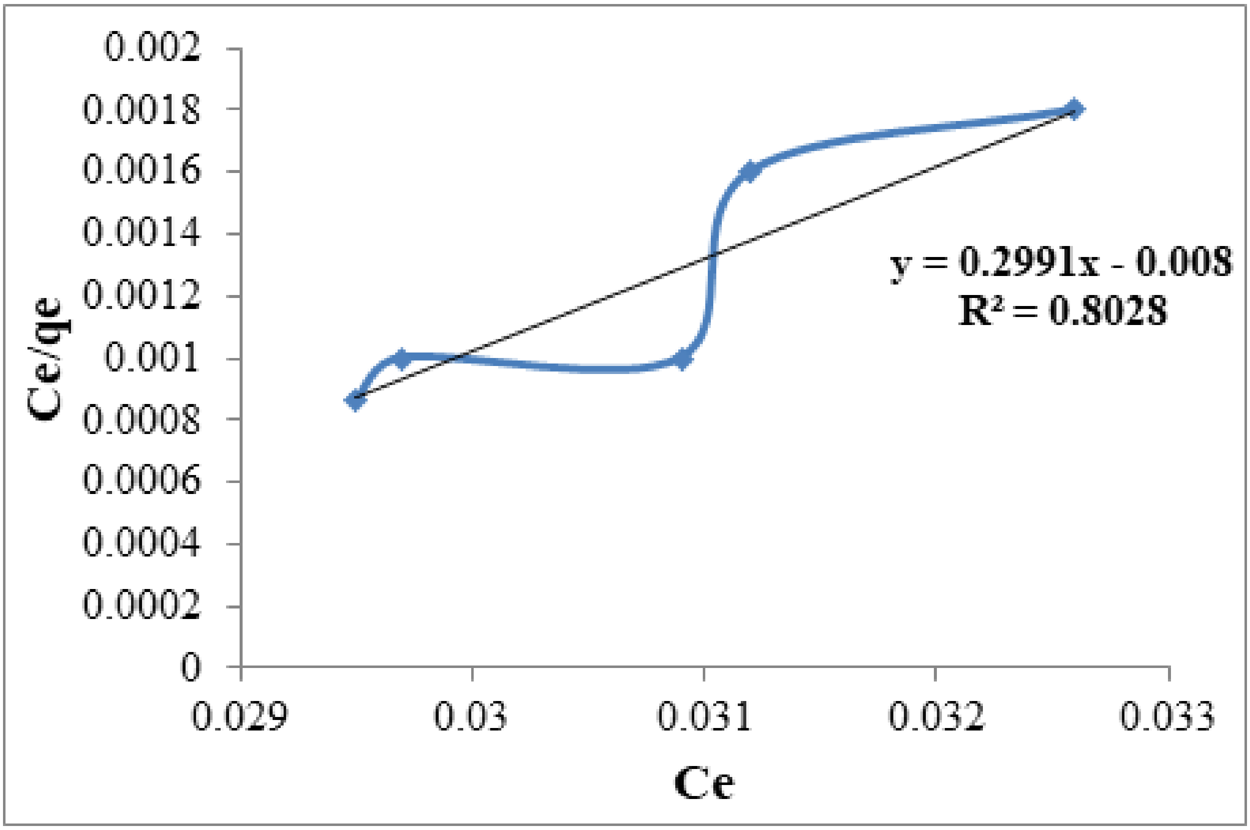

Figure 8. Langmuir Isotherm Plot for AR 18 Removal by ACPPP

in dye removal was evident. This can be seen from the curve (Figure 3) which shows a steady trend in the sorption process between 0.4 – 0.5 g. The obtained values here indicate that the optimal removal for the dye concentration of 1g/L was reached with 0.4 g of adsorbent. The reason for the initial increase in dye removal with increase in dosage can be attributed to the fact that the number of active sites is increased with the high dosage of adsorbent while adsorbate molecules in the solution remain constant. However, the presence of excess adsorbent than necessary would expectedly have no additional dye removal efficiency hence leading to the steady sorption observed at a much higher dosage. Also, the aggregation of adsorbent particles due to the high adsorbent dosage may cause a significant drop in surface area and an increase in diffusional path length [18] thus leading to a limiting adsorption process.

3.2.2. Effect of Initial Dye Concentration

Figure 4 shows the dependence of dye removal on initial adsorbate concentration. As can be seen, percentage dye removal increases with increase in dye concentration within the 0.6 – 1.0g/L concentration considered in this study. This observation is consistent with reports from related literature which showed that the amount of adsorbate removed onto adsorbent material increased with the increment in initial adsorbate concentration [19, 20]. The reason for this is likely because the initial concentration provides an important driving force to overcome mass resistances between the aqueous and solid phases. Also, the observed trend could be due to the availability of greater number of active sites with respect to the available dye molecules at these concentrations. However, with higher dye concentration, aggregation of particles would likely reduce the availability of binding sites for adsorption and hence limit the dye removal efficiency.

3.2.3. Effect of Temperature

The adsorption of AR 18 by ACPPP at varying temperatures is presented in Figure 5. Temperature is a crucial parameter in adsorption reactions. The percentage removal of dye increased with an increase in temperature from 30

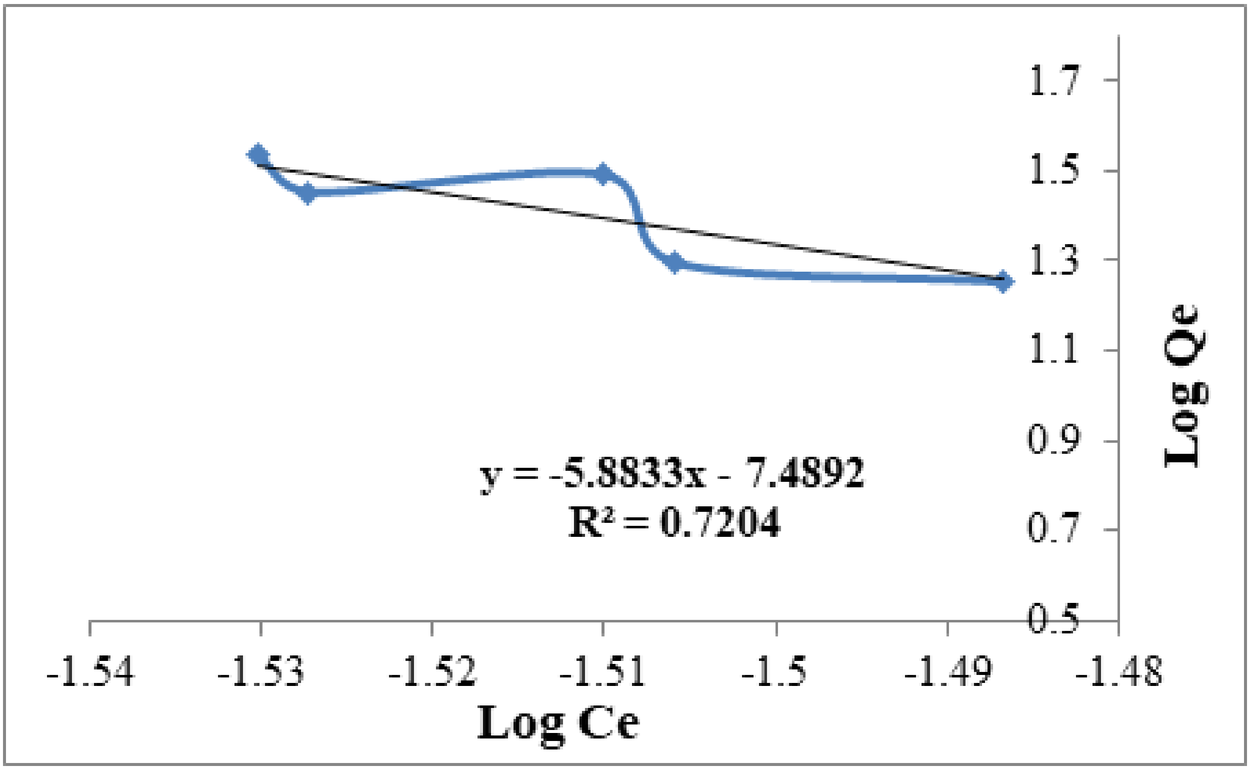

Figure 9. Freundlich Isotherm Plot for AR 18 Removal by ACPPP

to 40 °C. Above this temperature there was a decline in the sorption efficiency. The reason for the increase in sorption at the initial elevated temperature could be attributed to the enlargement of the pore size of adsorbent particles. This trend could also be due to an increase in the number of the adsorption sites because of breaking of some internal bonds near the edge of active surface sites of adsorbent [21]. The decline in sorption which occurred from 50°C indicates desorption of adsorbate molecules. This can be explained on the basis of the adsorption theory which states that adsorption decreases with increase in temperature and molecules adsorbed earlier on a surface tend to desorb from the surface at elevated temperatures. The reason for this is that with increasing temperature, the attractive forces between biomass surface and adsorbate molecules are weakened hence leading to desorption.

3.2.4. Effect of Contact Time

The effect of contact time on dye removal (Figure 6) indicates an initial rapid sorption with optimal removal reached within 40 minutes. Thereafter, a rapid decline in removal efficiency was observed. As seen from the graph, the % dye removal value at longer contact time (60 - 90°C) clearly indicates desorption. The reason for the initial rapid sorption process can be attributed to the availability of many sorption sites at the beginning of the process. However, as these sites are progressively filled the sorption process becomes more difficult and tends to be unfavourable. The desorption of the dye molecules at longer contact time suggests weak attraction between the adsorbent and adsorbate molecules.

3.2.5. Effect of pH

The % dye removal by ACPPP versus changes in pH is represented in Figure 7. Solution pH has been recognized as one of the important parameters that affects the removal of a solute from the solution and is been termed a master variable [22]. At acidic pH of 2.0 and 4.0, the percentage removal was observed to be 64.7% and 70.0% respectively. Neutral pH of 7.0 gave 68.9% dye removal while at pH 9 (basic), the percentage removal was highest high (96.9%). It has been reported that sorption capacity is higher at low pH for anionic dyes and high at basic pH for cationic dyes

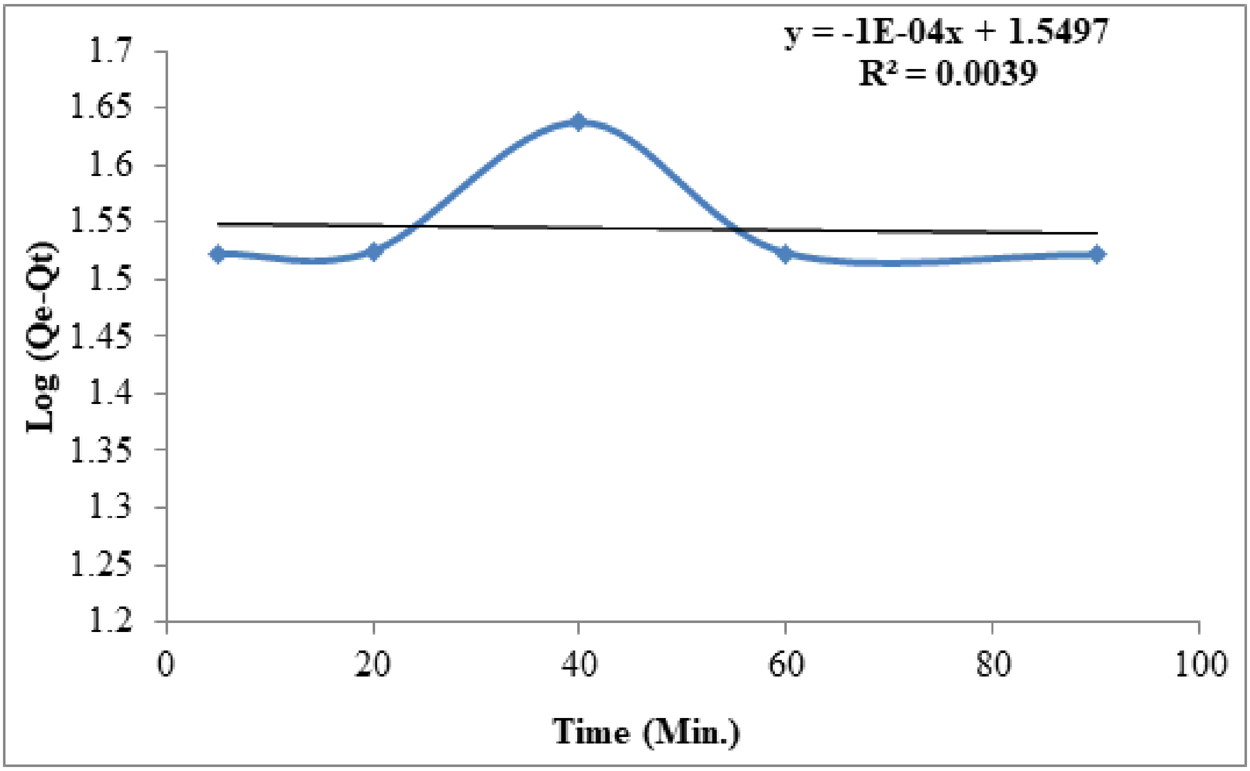

Figure 10. Pseudo First Order Kinetics Plot for AR 18 sorption by ACPPP

[23]. Our findings in this study agree with this trend. Being a cationic dye, the lower sorption observed at acidic pH could be attributed to the presence of excess H^+ ions competing with the cation groups on the dye for adsorption sites. Also, as the surface density decreases with an increase in the solution pH, the electrostatic repulsion between the positively charged dye and the surface of the adsorbent is lowered, this subsequently results in increased adsorption efficiency [24].

Isotherm studies are fundamental requirements for comprehending the mechanism of adsorption. It is the basis to understanding the practical application of an adsorbent and helps to ascertain the extent of affinity of an adsorbent to a particular adsorbate. Langmuir and Freundlich Isotherms models were adopted for this investigation. The appropriate isotherm type is determined based on the correlation coefficient (R^2) of the linear equation of each isothermic curve; linear equation with R^2 value close to or equal to 1 (one) indicates a better fit for the sorption process. Langmuir and Freundlich isotherm curves for AR 18 sorption by ACPPP are presented in Figures 8 and 9. The R^2 value for the Langmuir curve was 0.8028 while that of the Freundlich curve was 0.7204. These findings suggest that the adsorption process has a greater tendency to follow the Langmuir isotherm model thus it can be inferred that the surface of the sorbent is homogenous and at maximum adsorption only monolayer was formed on the free surface of the adsorbents [25].

3.3. Sorption Kinetics

Lagergren pseudo first order and second order models were used to evaluate the kinetics of adsorption of acid red 18 by the investigated biosorbent and our findings are presented in Figures 10 and 11. The correlation coefficients (R^2) for the pseudo first and second order kinetics were determined to be 0.0039 and 0.998 respectively. The values here strongly indicate that the pseudo second order kinetic best describes the sorption process. The Pseudo second order model supports chemisorption; the removal of adsorbate from bulk liquid is said to be due to physiochemical interaction between adsorbent and adsorbate [26]. The adsorption rate is also related to the availability of active sites on the adsorbent [27].

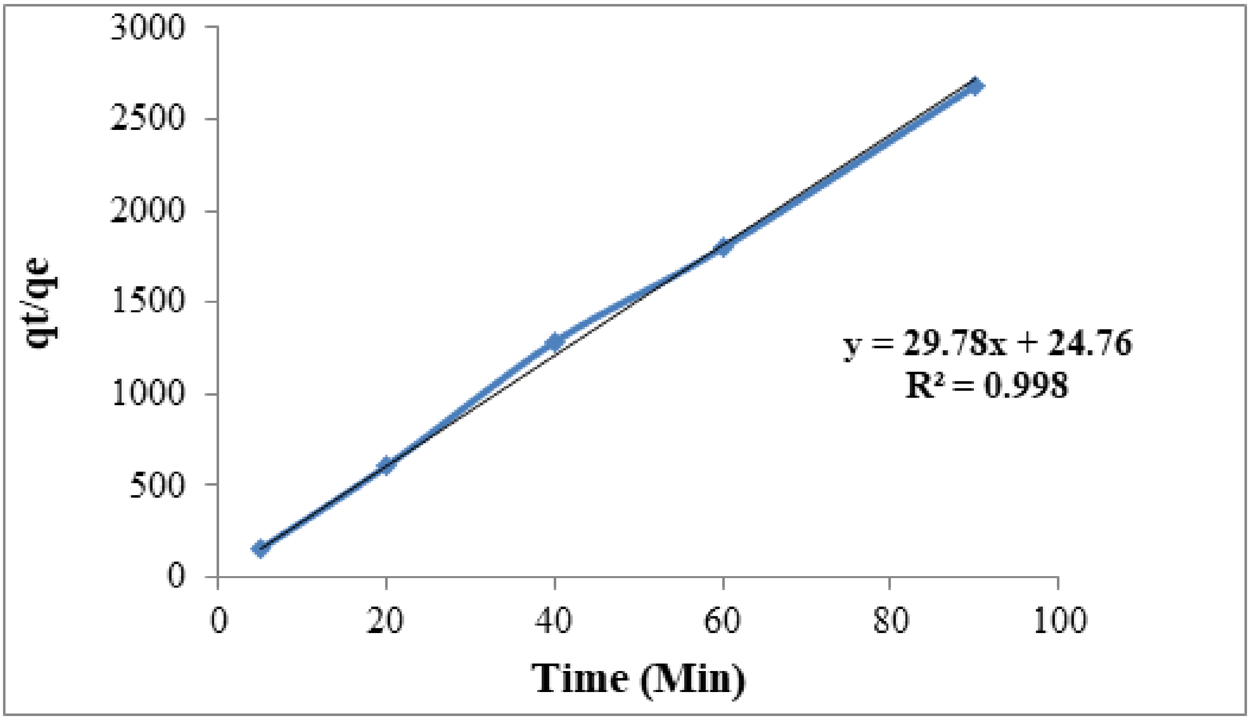

Figure 11. Pseudo Second Order Kinetic Plot for AR 18 Sorption by ACPPP

4. Conclusion

Sorption studies for the removal of AR 18 dye from aqueous solutions have been carried out using a biosorbent developed from *Carica papaya* peel. The investigated adsorbent shows great potential for uptake of the dye from water-based solutions. Isotherm and kinetic analysis revealed that Langmuir and pseudo-second-order models best describes the sorption process. Our findings further showed that the sorption capacity of the adsorbent is dependent on factors such as pH, temperature, initial dye concentration, adsorbent dose and contact time. This study uncovers *Carica papaya* peel as a good sorption material for potential use in the treatment of dye waste water. Being an agricultural waste, the peels constitute a nuisance to the environment hence converting them into a sorbent would help "clean up" the environment in addition to the immense benefit it could offer in water treatment processes. The use of such plant-generated waste materials could be better options compared to the conventional treatment processes because they are low cost and environmentally friendly. It is recommended that further studies look into the application of the investigated adsorbent in the removal of other dyes. This would help to fully uncover its potential in the holistic treatment of dyeing wastewaters.

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