

EFFECT OF SOLUTION pH AND TEMPERATURE ON PHENOL ADSORPTION ONTO CARBONIZED RICE HUSK CHEMICALLY ACTIVATED WITH PHOSPHORIC ACID

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Abstract: Operating conditions such as pH and temperature are important factors that influence the performance of activated carbon and solute uptake in adsorption process. Thus, this research work was aimed at studying the effect of solution pH and temperature on phenol adsorption onto rice husk activated carbon. Results obtained showed that when pH was increased from 2 to 4, adsorption capacity and removal efficiency increased, and thereafter, decreased progressively with additional increase in pH. Results also showed that when temperature was increased from 30 to 50 °C adsorption capacity and removal efficiency decreased. Therefore, this study has established that the optimum solution pH for adsorption of phenol onto phosphoric acid modified rice husk activated carbon is 4, and better performance of the adsorption process was occurred at lower solution temperatures.

Keywords: phenol, adsorption capacity, rice husk, concentration, equilibrium

1. INTRODUCTION

Adsorption is a process involving a solid phase and a liquid/gas phase containing dissolved species to be detached where the dissolved species migrate from the liquid/gas phase and form an attachment with the solid phase by different mechanisms as a result of stronger affinity. Adsorption using agricultural wastes as activated carbon for removal of contaminants from wastewater is receiving great attention due to economic and environmental concerns. Phenol is an organic compound that is continuously being generated in industries such as petroleum refinery as wastewater contaminant thereby necessitating treatment before eventual disposal into receiving bodies while incidentally, rice husk is a potential source of activated carbon that is locally available in Nigeria but underutilized and thereby constituting solid waste menace in the environment [1-7].

Reports on adsorption of solute onto activated carbon in literature have demonstrated that performance is largely dependent on the characteristics of the adsorbent (surface area, pore size distribution and surface functional groups), properties of the solute (molecular size, solubility, pKa and electron distribution) and operating conditions such as pH and temperature [8, 9]. Solution pH is an important factor in adsorption process because it affects the properties of solute and adsorbent such as type and ionic state of functional groups, degree of ionization of the solute contained in solution, dissociation of functional groups existing on the active sites of the activated carbon and chemistry of the solute contained in solution [10-15].

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The three possible mechanisms of complex interplay of electrostatic and dispersion interactions through which phenol adsorption onto activated carbon can occur are: (i) complex formation of electron donor-acceptor at the surface of adsorbent where the electron donor is the oxygen of surface carbonyl group and the electron receptor is the phenol aromatic ring, (ii) formation of hydrogen bond, and (iii) π - π dispersion interaction between aromatic ring of the molecules of phenol and π electrons delocalized on the graphene layers of adsorbent surfaces [16-19]. However, mechanisms 1 and 3 cannot occur at the same time as the presence of one rule out the possibility of the other. Solution temperature also has considerable effect on the adsorption process. Therefore, the objective of this study was to investigate the effect of solution pH and temperature on adsorption of phenol onto carbonized rice husk chemically activated with phosphoric acid.

2. EXPERIMENTAL SETUP

2.1. Materials

The materials used during the experimentation include furnace, ceramic flat surface, oven drier, de-ionized water, measuring cylinder, pH meter, digital weighing balance, conical flask, magnetic stirrer (Jenway 1000 Hotplate and Stirrer), micro filter (0.2 μ m), syringe, centrifuge (Horizon Clinispin 842VES), UV spectrophotometer (Jenway 6404UV), quartz cuvettes. The agricultural material used in this study was rice husk and the reagents used were phosphoric acid, nitric acid, sodium hydroxide and phenol, all of which were of analytical grade.

2.2. Production of adsorbent/activated carbon

The source material for the production of activated carbon was rice husk which was obtained from National Cereal Research Institute (NCRI), Badeggi, Nigeria. After collection, it was cleaned with distilled water for removal of any associated dirt and impurity. Thereafter, the cleaned rice husk was oven-dried at 100 °C for 24 h. The carbonization process was carried out by putting the oven-dried rice husk on a ceramic plate, then placing it inside a furnace where it was heated at a rate of 20-25 °C/min to an optimum temperature of 441.46 °C and residence time of 1h, after which the carbonized residue was collected and cooled at room temperature. Furthermore, the chemical modification process was carried out by activating the carbonized rice husk with 1M H₃PO₄ for 3h at impregnation ratio of 2:1 representing volume (mL) of acid to mass (g) of rice husk, after which it was oven-dried at 200 °C overnight in order to guarantee proper drying. The material was then removed from the oven, cooled at room temperature for 2h and then cleaned with distilled water to adjust the pH to 7.0 and again oven-dried overnight at 100°C [20, 21].

2.3. Adsorption experiment

The batch mode adsorption experiment was conducted in Multi-User Laboratory of Ahmadu Bello University, Zaria, Nigeria, under the optimum operating condition developed from our preliminary study, that is, initial concentration of 40.61 mg/L and adsorbent dosage of 4 g [22]. Thus, 4g of the activated carbon was added to 100 ml of 40.61 mg/L phenol concentration in a 250 mL conical flask and was placed on a magnetic stirrer at 150 rpm for 90 min at a known solution pH/temperature. Thereafter, samples were collected and centrifuged at 3000 rpm for 20 min, after which the supernatant solution was taken from the centrifuge by means of decantation and then filtered with a micro filter attached to a 5 mL syringe. After each use, the micro filter was backwashed with de-ionized water using a 50 mL syringe. The procedure outlined above was carried out at solution pH of 2, 4, 6, 8 and 10 and the pH was adjusted by adding a few drops of HNO₃ or NaOH. It was also carried out at solution temperatures of 30, 40 and 50 °C on a hot plate magnetic stirrer [6, 22-25]. The concentration of phenol in each sample filtrate was determined using UV spectrophotometer set at wavelength of 270 nm absorbance. Prior to analysis, a technical calibration curve was prepared and it was linear over the concentration range used. Therefore, un-adsorbed phenol concentration in the filtrate was determined by interpolation using the calibration curve.

2.4. Determination of Adsorption Capacity and Removal Efficiency

Equation (1) and (2) were used to determine the adsorption capacity at equilibrium [26, 27] and removal efficiency [28-30] respectively:

$$q_e = \frac{(C_o - C_e)V}{m} \quad (1)$$

$$RE = \frac{(C_o - C_e)}{C_o} \times 100 \quad (2)$$

2.5. Determination of Ionic Fraction

Equation (3) was used to determine the ionic fraction of phenolate [31, 32]:

$$\phi_{ions} = \frac{1}{1+10^{(pKa-pH)}} \quad (3)$$

where q_e is equilibrium adsorption capacity (mg/g); C_o = initial concentration of the solute (mg/L); C_e = concentration of solute at equilibrium (mg/L); V = volume of the solution (L); m = mass of adsorbent used (g); RE = removal efficiency (%); ϕ_{ions} = ionic fraction of phenolate; pK_a = acid dissociation/ionization constant.

3. RESULTS AND DISCUSSION

3.1. Effect of solution pH

The effect of solution pH on adsorption capacity and removal efficiency of phenol adsorption onto rice husk activated carbon are presented in Figures 1 and 2.

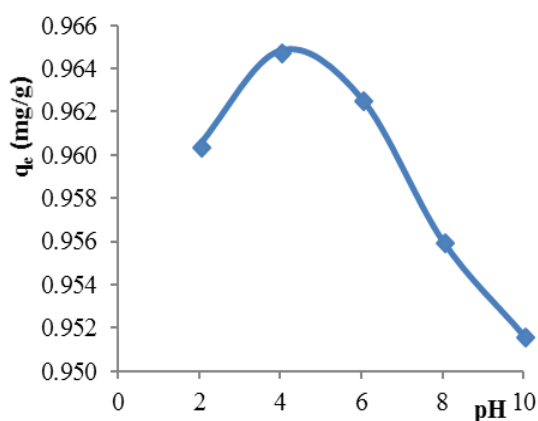


Fig. 1. Effect of pH on adsorption capacity.

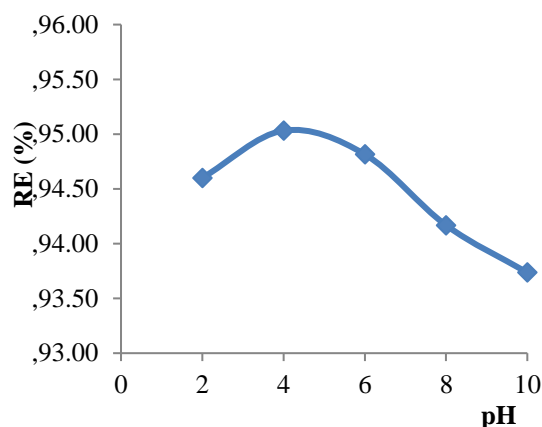


Fig. 2. Effect of pH on removal efficiency.

It was observed in Figure 1 that when pH was increased from 2 to 4, adsorption capacity increased from 0.960 to 0.965 $\text{mg}\cdot\text{g}^{-1}$ and thereafter, as the pH was increased further, adsorption capacity was observed to decrease progressively. Figure 2 also shows that removal efficiency increased from 94.6 to 95.03 % as pH was increased from 2 to 4 and thereafter, as the pH was increased further, removal efficiency was also observed to decrease progressively. Therefore, the optimum pH for phenol adsorption was taken as 4. The pK_a of phenol, a weak acid is 9.89 and as a result phenol is usually dissociated at $\text{pH} > pK_a$. In a phenol solution, when $\text{pH} < pK_a$ of phenol, the molecular form of phenol are the predominating species which are either neutral or positively charged. Therefore, maximum adsorption performance is observed at low pH because phenol is not dissociated and the driving force for adsorption is dispersion which is based on π - π interaction between the π electrons delocalized on the activated carbon graphene layers and the aromatic ring of the phenol molecules [17, 33-37].

But when $\text{pH} > pK_a$ of phenol, the ionic form, which is a consequence of the aromatic ring been partially negatively charged by means of the hydroxyl group ionization, is the predominating specie. This is confirmed in Table 1 which revealed that the ionic fraction of the phenolate ions, ϕ_{ions} increases as the pH was increased. Therefore, the major mechanism is electrostatic interaction which may be attractive or repulsive. Thus, the observed progressive decrease in adsorption performance could be a consequence of electrostatic repulsion between phenolate ions and negatively charged activated carbon surfaces; or phenolate ions and already adsorbed ones [8, 17, 31, 38]. In addition, these phenolate ions are more soluble in aqueous solution and the stronger adsorbate-water molecule must be overcome before adsorption takes place [33]. Another possible reason for the observed reduction in adsorption performance is the resultant large quantity of hydroxyl ions formed, which via physical adsorption, lead to additional negative charges on the surface of the activated carbon thus, establishing competitive adsorption between hydroxyl and phenolate ions [39-42]. Similar trend was also reported in the literature [32, 38, 43-45].

Table 1. Ionic fraction of phenolate at different pH pKa of phenol, 9.89

pH	2	4	6	8	10
ϕ_{ions}	1.288×10^{-8}	1.288×10^{-6}	1.288×10^{-4}	1.272×10^{-2}	5.63×10^{-1}

3.2. Effect of solution temperature

The effect of solution temperature on adsorption capacity and removal efficiency of phenol adsorption onto rice husk activated carbon is as presented in Figures 3 and 4.

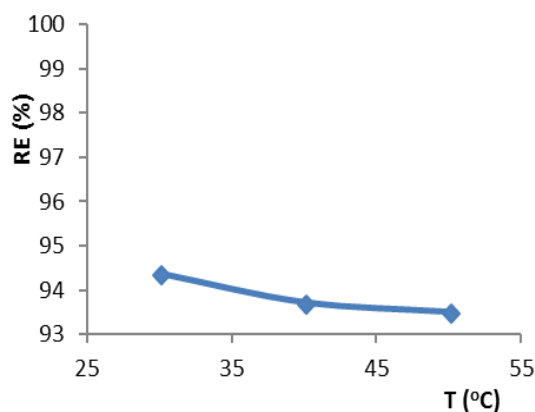
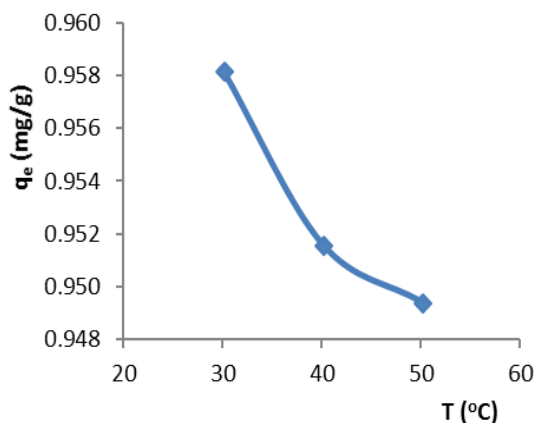


Fig. 3. Effect of temperature on adsorption capacity.

Fig. 4. Effect of temperature on removal efficiency.

It was observed in Figure 3 that when temperature was increased from 30 to 50 °C adsorption capacity decreased from 0.958 to 0.948 mg.g⁻¹. Figure 4 also shows that removal efficiency decreased from 94.38 to 93.52 % as the temperature was increased from 30 to 50 °C and according to [46, 47], this indicates less chemical interaction between the phenol molecules and the surface of the active carbons. It also implies that low temperatures favour higher adsorption capacity and removal efficiency most probably because adsorption process is exothermic. This observation is in agreement with the trend reported by Ozturk and Kavak [48]. The observed decrease in adsorption performance as the solution temperature increases could be attributed to the weakening of the physical bonding between phenol molecules and active sites of the activated carbon or the increased solubility of phenol molecules which enabled stronger interaction force between phenol molecules and solvent than phenol molecules and active sites of activated carbon [49].

4. CONCLUSIONS

This study has demonstrated that increase in solution pH from 2 to 4 has increasing effect on adsorption capacity and thereafter, further increase in pH results in progressive decrease in adsorption capacity. Also, removal efficiency was observed to increase as pH was increased from 2 to 4 and thereafter, decreases progressively as the pH was increased further. The study also demonstrated that increase in temperature has a decreasing effect on adsorption capacity and removal efficiency. Therefore, the optimum solution pH for adsorption of phenol onto rice husk activated carbon modified with phosphoric acid has been established as 4. In addition, better performance of the adsorption process was noted to occur at lower solution temperatures

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