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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. Therefore, the objective of this study is to investigate the effect of solution pH and temperature on adsorption of phenol onto rice husk activated carbon produced by chemical activation with phosphoric acid subsequent to carbonization at optimum temperature of 441.46°C. The adsorption experiment was conducted in batch mode over a pH range of 2-10 and temperature range of 30-50°C. Results obtained showed that when pH was increased from 2 to 4, adsorption capacity increased from 0.960 to 0.965mg/g, and thereafter, decreased progressively with additional increase in pH. Also, removal efficiency increased from 94.6 to 95.03% as pH was increased from 2 to 4, and thereafter, decreased progressively with additional increase in pH. Results also showed that when temperature was increased from 30 to 50°C adsorption capacity decreased from 0.958 to 0.948mg/g while removal efficiency decreased from 94.38 to 93.52%. Therefore, this study has established that the optimum solution pH for adsorption of phenol onto phosphoric acid modified rice husk activated carbon is 4. In addition, better performance of the adsorption process was noted to occur at lower solution temperatures.
Keywords: pH, temperature, solution, adsorption, experiment

1. INTRODUCTION

Adsorption is a process involving a solid phase and a liquid phase containing dissolved species to be detached where the dissolved species migrate from the liquid 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 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 activated carbon (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 in the solution, dissociation of functional groups on the active sites of the adsorbent and chemistry of the solute in solution [10-15]. It has been described that phenol adsorption onto activated carbon can occur via a complex interplay of electrostatic and dispersion interactions with three possible mechanisms: (i) π-π dispersion interaction between the π electrons delocalized on the graphene layers of activated carbon surfaces and the aromatic ring of phenol molecules, (ii) hydrogen bond formation, and (iii) electron donor-acceptor complex formation at the surface of activated carbon where the oxygen of the surface carbonyl group acts as the electron donor and the phenol aromatic ring as the acceptor [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. MATERIALS AND METHOD

— 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 μm), 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.

— **Production of Activated Carbon**
Rice husk was collected from National Cereal Research Institute (NCRI), Badeggi, Nigeria and washed with distilled water to remove dirt and surface impurity, then oven-dried at 100°C for 24h. For the carbonization process, rice husk was placed on a ceramic flat surface, charged into a furnace and heated to optimum temperature of 441.46°C at a heating rate of 20-25°C/min and residence time of 1h. The carbonized residue was collected and cooled at room temperature. For the chemical modification, the carbonized rice husk was activated with 1M H_3PO_4 for 3h at impregnation ratio of 2:1 (volume *mL* of acid/mass *g* of rice husk) and later oven-dried overnight at 200°C to ensure proper drying. The material was then removed from the oven, cooled for 2h and then washed with distilled water to bring the pH to 7.0 and again oven-dried overnight at 100°C [20,21].

— **Batch Adsorption Experiment**
The batch adsorption experiment was conducted in Multi-User Laboratory of Ahmadu Bello University Zaria, Nigeria, under the optimum operating condition developed from preliminary study that is initial concentration of 40.61mg/l and adsorbent dosage of 4g [22]. Thus, 4g of the activated carbon was added to 100ml of 40.61mg/l phenol concentration in 250ml conical flask and the mixture in the flask was placed on magnetic stirrer at 150 rpm for 90min at a known solution pH/temperature. Samples were then collected, centrifuged at 3000 rpm for 20min and the supernatant solution was collected from the centrifuge by decantation and filtered using a micro filter attached to a 5ml syringe. The micro filter was backwashed after each use with de-ionized water using a 50ml 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 HNO_3 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 analysis of phenol in each sample filtrate was carried out using UV spectrophotometer set at wavelength of 270nm 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.

— **Determination of Adsorption Capacity and Removal Efficiency**
Adsorption capacity at equilibrium was determined using the equation [26,27]:
$$q_e = \frac{(C_o - C_e)\, V}{m} \tag{1}$$
Adsorption removal efficiency was determined using the equation [28-30]:
$$RE = \frac{(C_o - C_e)}{C_o} \times 100 \tag{2}$$

— **Determination of Ionic Fraction**
The ionic fraction of phenolate was determined from the equation [31,32]:
$$\varphi_{ions} = \frac{1}{1 + 10^{(pKa - pH)}} \tag{3}$$
where: q_e = adsorption capacity at equilibrium (mg/g), C_o = initial concentration of solute (mg/L), C_e = equilibrium concentration of solute (mg/L), V = volume of solution (L), m = mass of activated carbon used (g), RE = removal efficiency (%), ϕ_{ions} = ionic fraction of phenolate, pKa = acid dissociation/ ionization constant

3. RESULTS AND DISCUSSION
— **Effect of Solution pH**
It was observed in Figure 1 that when pH was increased from 2 to 4, adsorption capacity increased from 0.960 to 0.965mg/g 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 noted as 4. The pKa of phenol, a weak acid is 9.89 and as a result phenol is usually dissociated at pH>pKa. In a phenol solution, when pH<pKa 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 pH>pKa of phenol, the ionic form, which is a consequence of the aromatic ring been partially negatively charged by means of the hydroxyl group ionization (equation 4), 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].

$$C_6H_5OH + H_2O \leftrightarrow C_6H_5O^- + H_3O^+ \tag{4}$$

Table 1: Ionic fraction of phenolate at different pH

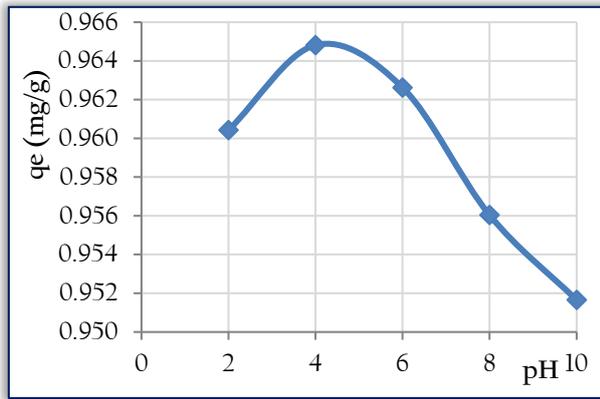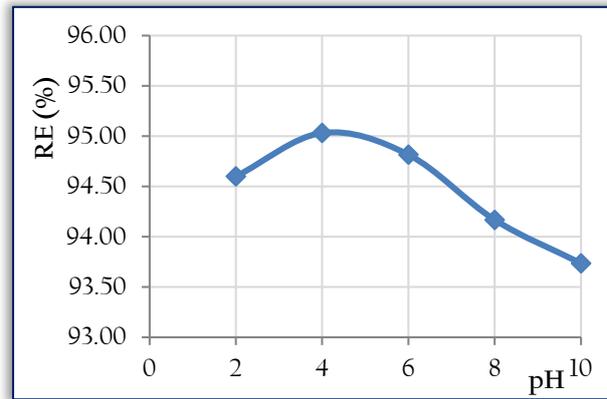| pKa of phenol, 9.89					
pH	2	4	6	8	10
ϕ_{ions}	1.288×10^{-8}	1.288×10^{-6}	1.288×10^{-4}	1.272×10^{-2}	5.63×10^{-1}

Figure 1: Effect of pH on adsorption capacity

Figure 2: Effect of pH on removal efficiency

— **Effect of Solution Temperature**

It was observed in Figure 3 that when temperature was increased from 30 to 50°C adsorption capacity decreased from 0.958 to 0.948mg/g. Figure 4 also shows that removal efficiency also 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].
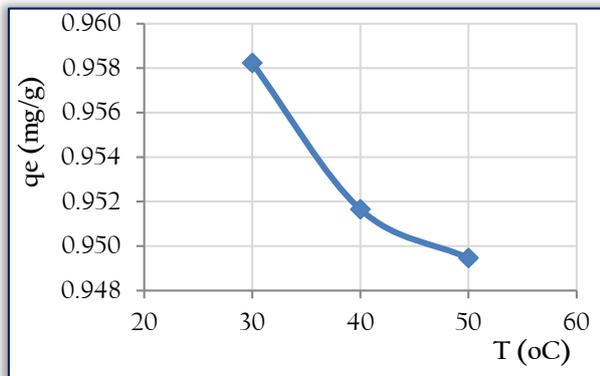

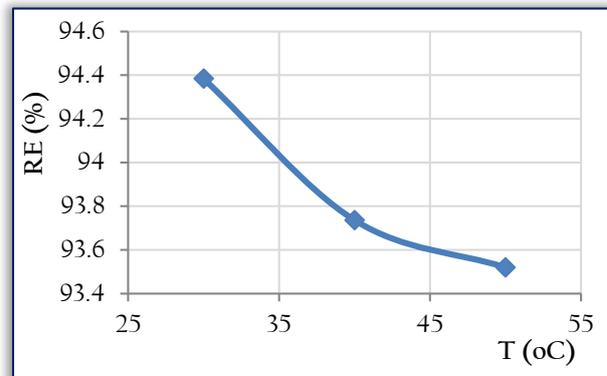Figure 3: Effect of temperature on adsorption capacity

Figure 4: Effect of temperature on removal efficiency

4. CONCLUSION

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. Removal efficiency was also observed to increase as pH was increased from 2 to 4 and thereafter, decreases

progressively as the pH was increased further. It also demonstrated that increase in temperature has a decreasing effect on adsorption capacity and removal efficiency. Though the effect of solution pH and temperature on the adsorption process were noted to be slight, explanation was given for the observed increase or decrease in performance. 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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