

# **EVALUATION OF BENZENE, TOLUENE AND P-XYLENE REMOVAL FROM PRODUCED WATER USING LOCALLY AVAILABLE ADSORBENTS AND GRANULAR ACTIVATED CARBON**

# Rahima Abubakar

# <sup>1</sup>Lecturer II, Dept. of Petroleum and Gas Engineering, Baze University, Abuja, Nigeria.

\*\*\* **Abstract** - The sorbent efficiency of two locally available adsorbents in the removal of benzene, toluene and p-xylene (BTX) from produced water were investigated; Moringa Oleifera Seeds and Banana Peel, and compared with the sorbent efficiency of Granular Activated Carbon. This was conducted by means of a batch adsorption experiment using a univariate approach and analysis was done using the HS-GCMS. The effect of parameters such as contact time (0-90mins), pH (4.0-10.0), adsorbate concentration (1-9mg/l), adsorbent dosage (0.2-0.8g) and temperature (15°C-30°C) were investigated. The maximum % removals of BTX compounds by the selected adsorbents are; BP- 29%, 46%, 53%, MOS-57%, 63%, 57% and GAC- 70%, 65%, 59% respectively and the maximum adsorption capacity obtained for BTX adsorption in this study are: BP-0.02mg/g, 0.038mg/g, 0.037mg/g, MOS-0.036mg/g, 0.039mg/g , 0.038mg/g and GAC -0.042mg/g, 0.041mg/g, 0.039mg/g respectively. Generally, BTX adsorption in this work was in the order of B<T<X. Moringa Oleifera has proven to be a promising adsorbent in the removal of BTX compounds as such the potential applicability of MOS in BTX removal could be further examined on a large scale.

#### ADSORPTION. PRODUCED WATER. Kev Words: SORPTION CAPACITY. MORINGA **OLEIFERA.** ADSORBENT

# 1. INTRODUCTION

The oil and gas industries are faced with challenges in the management and disposal of produced water. This is usually either re-injected into the well for secondary oil recovery or discharged into surface water and surrounding environment mostly only after the separation of oil from water without necessarily meeting the required standards of discharge limits for the various constituents of produced water. These practices are not without effects to humans, aquatic lives and the environment as a whole.

Produced water contributes to the biggest wastewater stream during oil exploration and production processes (Utvik 1999). Global produced water production is approximately 250million barrels/day as compared to the estimated 80million barrels/day of oil production. As such, water to oil ratio is 3:1 which indicates a water cut of 70%. An approximate 40% of this water is discharged offshore of which about 1.1 million cubic meters of produced water is disposed daily in the North Sea.

Produced water constituents are a mixture of chemicals originating from dissolved and dispersed oil components comprising organic acids, alkyl phenols, heavy metals and aromatic hydrocarbons to name a few (Boitsov, Mjos and Utvik 1999). However, aromatic Meier 2007, hydrocarbons constitute polycyclic aromatic hydrocarbons, Naphthalenes and monocyclic aromatic hydrocarbons (Benzene, Toluene, Ethylbenzene and Xylenes). Benzene, Toluene, Ethylbenzene and Xylenes (BTEX) constitutes about 97% of the aromatics while the PAHs and NPDs together make up just about 3%. Furthermore, Benzene has been established as a human carcinogen while toluene may also be carcinogenic, xylene is also harmful to marine animals. Together they all pose hazards to humans, aquatic lives and the environment hence necessitates the drive to investigate a cost effective way to treat these compounds from produced water which forms the basis of this work.

Produced water and its constituents are conventionally treated using various physical, chemical and biological methods. Amongst these methods are separation by hydroclones (Lohne 1994), Microfiltration (Campos et al. 2002), Ultrafiltration through polymer membranes (Cheryan and Rajagopalan 1998) and more recently, Electrodialysis (Dallbauman and Sirivedhin 2005). Biological treatment systems that were examined include; Activated sludge (Tellez, Nirmalakhandan and Gardea-Torresdey 2005), Airlift attached growth (Campos et al. 2002), Wetlands (Rambeau et al. 2004), Upflow Anaerobic Sludge Blanket (Rincon et al. 2003). However, existing technologies fail to eliminate small suspended oil particles and dissolved elements and most chemical methods of treatments have high initial and/or running costs and produce hazardous sludge.

Low cost adsorbents are now the emerging technologies for produced and waste water treatment as they are cost effective and environmentally friendly. Adsorbents from fruit waste (banana peel, orange peel, and tangerine peel), agricultural wastes (palm fruit fibre, plantain peels) and nut & shells to name a few are now being used for the removal of heavy metals, organic and inorganic constituents of produced water. U.A. El-Nafaty et al., (2013) worked on the bio sorption of the oil component of produced water using banana peels and have shown almost 100% removal efficiency. Treatment through sorption onto materials such as surfactant modified zeolite



(SMZ) have also been used to reduce BTEX levels in contaminated water but has its limitations in that the sorption capacity of SMZ for BTEX is low relative to other adsorbents (Lynn *et al.* 2003). Granular activated carbon and powdered activated carbon have also been used as sorbents in the treatment of produced water but have limitations in the fact that they are expensive and exhibit competitive sorption where the compound with higher affinity for the activated carbon exchanges with the lower affinity compound that has sorbed more rapidly.

The locally available adsorbents selected for this work are; moringa oleifera seeds and banana peel. These have been selected based on availability and cost effectiveness, as the work is aimed at finding a cost efficient adsorbent for removal of Benzene, Toluene and P-xylene (BTX) from produced water. Moreover, no previous work has appeared involving the removal of BTX compounds from produced water using moringa oleifera seeds and banana peel although work has been carried out using moringa oleifera seeds in Benzene, Toluene, Ethylbenzene and Cumene (BTEC) removal from aqueous solution and granular activated carbon for BTEX adsorption. This research will assess and compare with other adsorbents the potential application of moringa oleifera seeds and banana peel in the removal of BTX from produced water and if viable, recommend to oil and gas industries a cost effective and eco-friendly method of treating one of the most hazardous and dominant components of in produced water.

### **1.1 Research Objectives**

- To investigate and compare the sorbent efficiencies of moringa oleifera seeds, banana peel, and granular activated carbon in BTX removal from produced water.
- To assess the potential application of moringa oleifera seeds and banana peel as adsorbents in the removal of BTX from produced water.
- To investigate the effect of variable process configurations(pH,temperature,adsorbate concentration, dosage and aqueous matrix) on the rate of BTX adsorption.
- To compare the adsorption capacities of moringa oleifera seeds, banana peel, and granular activated carbon in BTX removal.

# 2. Materials and Method

# 2.1 Laboratory Equipments

- Gas chromatography Mass Spectrometer (Schimadzu QP2010).
- Glass vials with air tight screw caps
- Volumetric flasks
- Beakers
- Spatula

- 5ml Syringes
- Centrifuge
- Thermometer
- Temperature Controlled water bath shaker
- Pipette
- Funnels
- Weighing balance and pans
- Desiccator
- Mortar and Pestle
- pH meter
- Timer
- Oven and oven trays
- Sieve
- Ball mill

### 2.2 Reagents

- Stock solution of benzene, toluene and p-xylene (BTX)
- Methanol (HPLC grade)
- Nitric acid (0.1M)
- Hydrochloric acid (pH adjustment)
- Sodium Hydroxide (pH adjustment)
- Sodium Chloride

### 2.3 Adsorbent Preparation

## 2.3.1 Moringa Oleifera Seeds

Moringa Oleifera seeds were de-shelled and washed with de-ionized water three times. It was dried in the oven at 70°C for 8 hours. Dried moringa was pulverised with mortar and pestle and sieved to 1mm particle size (see figure 2.1a and b). This was then re-washed to eliminate fine particles and dried at 110°C for 4 hours. The dried powder is treated with 0.1M nitric acid and methanol for 4 hours to eliminate organic and inorganic matter from the surface. This was proceeded by thermal treatment at for 1 hour at 300°C to increase surface area. Sorbent was stored in an air tight plastic bag in a desiccator (Akhtar *et al.* 2007).

# 2.3.2 Banana Peel

Banana peels were cut into smaller pieces (about 5mm) and washed with de-ionized water three times to eliminate external impurities. Washed banana peels were dried in the oven at 105°C for 24hours. The dried banana peels were grounded using a ball mill and sieved to 1mm size (see figure 2.1c and d). This was then stored in an air plastic bag and kept in a desiccator (Hossain *et al.* 2012).





Figure2.1: A-moringa oleifera seeds, B- moringa oleifera powder, C- banana peel, D-banana peel powder.

# 2.3.3 Characterisation of Adsorbents

# 2.3.3.1 Properties of Locally Available Adsorbents of Choice

The ash content and organic matter percentages were determined according to standard methods of ASTM (ASTM, 2000) and the pH was determined by a water extract suspension procedure (Daifullah and Girgis, 2003). The results of the aforementioned tests and other properties of MOS and GAC are presented in table 3.1.

#### 2.2.3.2 Scanning Electron Microscopy Analysis

The SEM used in obtaining images of the present study is JEOL JSM -6060LV scanning electron microscope. SEM of MOS was obtained before and after adsorption (See figure 3.1a and b) as well as BP before and after adsorption (See figure 3.1c and d).

### 2.3.4 Granular Activated Carbon

The granular activated carbon used for this experiment was purchased from sigma Aldrich. Information on the properties and methods of preparation of the GAC used were provided by the manufacturer. The GAC was prepared by kiln burning peat bog until only carbon is left. The heating process in the kiln is what is known as the activation of the carbon. This process makes the carbon porous and eliminates all impurities (Sigma Aldrich, 2013). Other properties of the GAC used in the study have been stated in table 3.2

#### 2.4 Adsorbate Preparation

### 2.4.1 Produced Water Preparation

Saline produced water was simulated in the laboratory using salts of sodium; Nacl, Na<sub>2</sub>SO<sub>4</sub>, NaNO<sub>3</sub> and NaHCO<sub>3</sub> in concentrations of salts reported for the North Sea produced water (Johnsen et al. 2004). The aforementioned salts are the major inorganic constituents found in produced water from the North Sea produced water (Johnsen et al. 2004). The concentrations of anions reported by Johnsen et al., (2004) are; chloride-44.6mg/l, Sulphate-814mg/l, Nitrate -1mg/l and Bicarbonate -615mg/l. To prepare the saline produced water, salts of sodium were used; Nacl, Na<sub>2</sub>SO<sub>4</sub>, NaNO<sub>3</sub> and NaHCO<sub>3</sub>. The solution was prepared in a five litre bottle by adding 367.95g of Nacl, 6.025g of Na<sub>2</sub>SO<sub>4</sub>, 0.007g of NaNO<sub>3</sub> and 4.245g of NaHCO<sub>3</sub> to obtain 44.6mg/l, 814mg/l, 1mg/l and 615mg/l of Nacl, Na<sub>2</sub>SO<sub>4</sub>, NaNO<sub>3</sub> and NaHCO<sub>3</sub> respectively. The saline solution was used for diluting BTX stock solution and standards throughout the experiment.

# 2.4.2 BTX Stock Preparation

The chemicals tested in this work were benzene (Sigma Aldrich, purity: 99.8%), toluene (Sigma Aldrich, purity: 99.8%) and p-xylene (Fluka, purity:  $\geq$ 99.5%). BTX stock solution was prepared on the basis of the even concentration ratio, 1000ppm of each component (benzene, toluene and p-xylene) (Nourmoradi et al. 2013). 1000ppmv of BTX was prepared by pipetting 0.25ml each of benzene, toluene, and p-xylene into a 250ml volumetric flask and was made up with methanol (Sigma Aldrich, purity  $\geq$ 99.9%). This was transferred into an amber glass and stored in the fridge for daily preparation of standard solution and samples for adsorption experiments



# 2.4.3 Standard Preparation

Standard solutions of BTX in the following concentrations; Omg/l (blank), 1mg/l, 3mg/l, 5mg/l, and 7mg/l were prepared in produced water in order to calibrate the system. This was carried out in a 100ml volumetric flask by adding 0.1ml, 0.3ml, 0.5ml, and 0.7ml of the 1000mg/l BTX stock solution and made up to the mark with produced water for the preparation of 1mg/l, 3mg/l, 5mg/l and 7mg/l respectively. The solutions were each shaken to ensure homogeneity and were transferred into the GCMS glass vials containing 2g of NaCl. These were set up on tray 1 of the GCMS for analysis and a calibration graph was obtained. Standard solutions are prepared daily for calibration of the system. Similar method of preparation was carried out by Altare *et al.*, (2007).

#### 2.5 BTX ANALYTICAL METHOD

The analytical method for BTX determination was selected based on the selectivity, sensitivity and reliability of that method. This aided in partitioning the analyte of interest from complex aqueous matrices. Among the methods that are being practiced for BTX analysis include GC-FID ; direct aqueous injection(DAI), liquid-liquid extraction (LLE), purge and trap (P & T), headspace gas chromatography mass spectrometry (HS-GCMS) and membrane based techniques (solid-phase microextraction- SPME) (Chary and Fernandez-Alba, 2012). Kubinec *et al.* (2005) studied the determination of BTX using DAI and reported a method LOD of 0.6-  $1.1\mu g/l$ .

The limitations reported include possible interferences arising from matrix effects and incompatibility of stationary films of the capillary columns with FID detectors. Purge and trap method has been used in a study to analyse BTEX and halomethanes in drinking water and was reported to have high sensitivity and recoveries but has its drawback in its complexity and interference of water vapour generated in the purge stage (Liu et al. 2009). Studies have been conducted on investigation of VOC in contaminated groundwater using LLE which yielded good reproducibility. Nevertheless, LLE is not preferred because of its requirement for large volume of solvent and alternative extraction units (Cabala and Bursová, 2012). BTEX and MBTE in Swiss lake water modelling studies have revealed that HS when used alone possesses good recovery values (LOD, 0.1µg/L) whereas HS-SPME has reproducibility values better than HS alone and has the lowest limit of detection. The results also indicate that the analysis time is shorter for HS-SPME than HS alone (Schmidt et al. 2004).

## **2.5.1 METHOD FOR BTX ANALYSIS**

The HS-GCMS analytical method was utilized for this work and the GCMS used for this work is a Schimadzu-QP2010 equipped with an AOC5000 auto sampler (See figure 2.2) which improves upon the accuracy of the equipment and allows for analysis of multiple numbers of samples.

Analyses of sample solutions were carried out using the method below following the Schimadzu procedure for analysing VOC (Lieshout, 2009).This method was however moderated after appropriate experimental optimization to suit the BTX analysis. See characteristics of the method below:

### **GC-MS** Conditions

Injector temperature: 300°C

Carrier gas: He

GC column: SPB<sup>TM</sup>-1 (30m×0.25mm×0.2 μ m)

Oven temperature:  $40^{\circ}$ C for 3mins at the rate of  $20^{\circ}$ C/min up to  $100^{\circ}$ C and hold for 5mins.

Ion source temperature: 200°C

GC–MS interface temperature: 300°C

### HEADSPACE

Sampler: AOC-5000

Sampling mode: Headspace

Vial volume: 20ml

Sample volume: 5ml

Injection volume: 1000µl

Salt addition: 2g of NaCl

Stirring: 1400rpm

Split ratio: 14.0

Solvent cut time: 1min

Run time: 11mins

The above method was applied in the system and saved for use throughout the experiment. 2g of salt (NaCl) was weighed out in 20ml glass vials after which sample for analysis were transferred into these glass vials and covered with a PTFE silicon septum screw cap and transferred onto tray-1 of the HS-GCMS. A batch file was created for the samples (for each set of analysis) specifying sample IDs, method file, data file name, and tray number after which the specified method was applied for analysis. IRJET

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Figure 2.2: Headspace Gas Chromatography Mass Spectrometry

# **2.5.2 VALIDATION OF ANALYTICAL METHOD**

The validation of this method was conducted by determining the following parameters for the specified method; Selectivity, precision, accuracy, linear correlation coefficient, limit of detection and limit of quantification.

Accuracy was evaluated as the recovery percentage in recovery tests while precision as RSD of replicate. The precision was determined by analysis of five replicates of produced water spiked with BTX at a concentration of 5mg/l.

The LOD and LOQ were determined using the following equations (1) and (2) respectively:

- 3s/S(1)
- 10s/S(2)

Where s is the standard deviation calculated from the calibration curve and S is the slope (Frena, Tonietto, and Madureira 2013).

The selectivity of the method was established by injecting a blank sample five times and was observed for elution of interfering compounds at the retention times of the individual analytes.

# 2.6 Batch Adsorption Studies

# 2.6.1 Experimental Design

In this study, the univariate experimental design was applied to optimise the removal of BTX onto the MOS/BP/GAC system was conducted using the univariate approach (Almeida *et al.* 2012). The factors considered in this work include; contact time (0-90mins), pH (4.0-10.0), temperature (15-30°C), adsorbate concentration (1-7mg/l). The adsorbent dosage and adsorbate volume were 0.4g and 10ml respectively which were kept constant throughout the experiment. These were selected on the

basis of preliminary tests conducted to estimate optimum value for variables. A temperature of 25°C was used throughout the experiments. The univariate approach has also been applied by Almeida *et al.*, (2012) for the optimisation of extraction procedures in BTEX removal.

### 2.6.2 Sorption Method

The initial concentration of BTX for all batch adsorption experiments was 3mg/l. This was prepared daily in a 500ml volumetric flask and used for all experiments. 0.4g of sorbent was measured into 20ml glass vials to which 10ml of 3mg/l initial concentration of BTX was added and immediately covered with a PTFE silicon septum screw cap (See figure 2.3). The vials were then fitted to a temperature controlled water bath shaker at 25°C and a speed Of 250 RPM for 0-90mins. At the end of each experiment, 5ml aliquot of the solution is taken from the vials using a syringe and transferred into a 20ml GCMS glass vial containing 2g of NaCl. The glass vials are then set up on tray 1 of the HS-GCMS for further analysis using the method outlined in section 2.6.1 above. In order to asses evaporation losses of BTX, a control sample (without sorbent) was placed on the same shaker without sorbent and the final concentration noted to determine the percentage of BTX adsorbed onto sorbents and the percentage loss to adsorption onto the glass bottle or volatilization during the experiment. All experiments were conducted in duplicates and only the mean values were reported. Similar sorption method was used by Almeida et al., (2012) and Nourmoradi, Khiadeni and Nikaeen, (2012). The percentage removal was calculated using equation (3) below and the adsorption capacity, ge, was determined using equation (4).

%Sorption= (Ci-Ce)/Ci \* 100 (3)

Where Ci= initial concentration of sorbate in mg/l

Ce= equilibrium concentration of sorbate in mg/l



Figure 2.3: BTX Sample Preparation

### 2.6.3 Comparative Studies

The capacity of locally available adsorbents (MOS AND BP) to adsorb BTX was compared with GAC. This comparison was conducted with GAC as GAC has been established to have high removal efficiency for organic pollutants from aqueous matrices. The study was conducted simultaneously for all three adsorbents (MOS, BP AND GAC) under variable experimental conditions as outlined in section 2.6.1. These varying operating parameters have been further discussed below. The results were used to compare the removal efficiencies of all three adsorbents.

#### 2.6.4 Procedure for Investigation of Effect of Contact Time

Samples were agitated in the bath shaker at time intervals of 30, 60 and 90 minutes following the procedure discussed in section 2.6.2 above having established from preliminary studies the equilibrium time for BP as 60minutes whereas MOS and GAC are both 90minutes.

### 2.6.5 Investigation of Effect of pH

The effect of pH was investigated on BTX removal by varying the BTX solution pH between 4.0 - 10.0 (4.0, 6.0, 7.0, 8.0 and 10.0) (Nourmoradi et al. 2012). The solution pH was found to be 7.9 and was adjusted by addition of 0.1M HCL and 0.1M NaOH appropriately after which the solution was continuously stirred to ensure complete homogenization of the mixture before taking readings off the pH meter. The procedure in section 2.6.2 was followed in carrying out the experiment after obtaining the desired solution pH. The pH of the solution was determined using Jenway 3505 pH meter which is illustrated in figure 2.4 below:



Figure 2.4: pH Meter Array

#### 2.6.6 Determination of Adsorbate Concentration Effect

This was investigated for BTX solutions at concentrations of 1mg/l, 3mg/l, 5mg/l and 7mg/l. The solutions were prepared by pipetting 0.1ml, 0.3ml, 0.5ml, and 0.7ml aliquots of BTX stock solution into 100ml volumetric

flasks and making up to the mark with produced water for 1mg/l, 3mg/l, 5mg/l and 7mg/l respectively. The experiment was then conducted following the procedure in section 2.6.2.

### 2.6.7 Determination of Effect of Adsorbent Dosage

This experiment was carried out using adsorbent masses of 0.2g, 0.4g, 0.6g, and 0.8g. The method discussed in 2.6.2 above was used to carry out the experiment while maintaining all other factors constant. The result obtained from effect of adsorbent dosage was expressed as the adsorption capacity of BP, GAC and MOS at different masses (see equation 4). This similar method was carried out by Akhtar *et al.*, (2007)

### 2.6.8 Determination of Temperature Effect

Adsorption experiments were carried out at temperatures of 15°C, 20°C, 25°C, and 30°C. This was carried out using the procedure discussed in 3.7.2 above with all other factors kept constant. The same temperature range has been applied in studies conducted by Aivalioti et al., (2012) and Nourmoradi, Khiadeni and Nikaeen, (2012).

### 2.7 Data Analysis

Data obtained from the variable factors discussed above (contact time, pH, dosages, concentration and temperature) during the adsorption experiments were computed as removal efficiencies and expressed as % removals whereas adsorption capacity was computed using equation (4) and expressed as sorption capacity qe (mg/g). These were all represented using tables, charts and graphs generated from excel appropriately. Standard errors have been calculated for each all factors using duplicate samples and error bars presented on all charts appropriately. Data was further analysed using regression analysis and the significance of variable configuration parameters were related to the F values obtained from results of regression analysis. F values≤0.05 indicate significant effect of a configuration parameter on the removal of BTX while F value>0.05 indicates that the configuration parameter has no significant effect on BTX removal.

## 3.0 RESULTS AND DISCUSSION

# 3.1 Characterisation of Adsorbents

# 3.1.1 Properties of Adsorbents

Table 3.1: Properties of the Locally Available Adsorbents Used in the Experiment

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## **3.0 RESULTS AND DISCUSSION**

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#### **3.1 Characterisation of Adsorbents**

#### 3.1.1 Properties of Adsorbents

Table 3.1: Properties of the Locally Available Adsorbents Used in the Experiment

Parameters	Moringa Oleifera	Banana
	Seeds	Peel
Ash Content (%)	3.3	11.3
Organic Matter	96.7	88.7
(%)		
рН	5.55	5.87
Particle Size	1mm	1mm
Surface Area	4.01 m <sup>2</sup> /g	1.856
	(Kumari <i>et al</i> .	m²/g
	2006)	(Kamsonli
		an <i>et al</i> .
		2011)

Table 3.2: Properties of Granular Activated Carbon Used in the Experiment (Sigma Aldrich, 2013).

Parameters	Granular Activated
	Carbon
Ash Content (%)	≤12
Moisture Content (%)	≤4
Surface Area (m <sup>2</sup> /g)	600-800
рН	9-11
Particle Size (mesh)	4-8

#### 3.1.2 Scanning Electron Microscope Analysis

The images of untreated adsorbents (before adsorption) and treated adsorbents (exhausted) were observed at a bar length equivalent of  $20\mu$ m, working voltage of 8KV and (X 800) magnification. The scanning electron micrographs of untreated MOS (figure 3.1a) depicts homogenous linear structures with large surface area whereas exhausted MOS (figure 3.1b) shows a significant change in structure with dense agglomerated structures which might be due to the presence of organic molecules partially covering its surface. The micrographs of untreated BP (figure 3.1c) reveals heterogeneous large clusters of irregularly shaped structures while exhausted BP (figure 3.1d) shows a partial change in structure with a rough surface.



Figure 3.1: A- SEM image of moringa oleifera before adsorption B- SEM image of moringa oleifera after adsorption, C- SEM image of banana peel before adsorption, D- SEM image of banana peel after adsorption

# **3.2 VALIDATION OF ANALYTICAL METHOD FOR BTX DETERMINATION IN PRODUCED WATER.**

The parameters examined in order to validate the method for determination of BTX in produced water are; Selectivity, precision, accuracy, linear correlation coefficient, limit of detection and limit of quantification.

The precision of the method was examined by analysing five replicates of produced water spiked with BTX at a concentration of 5mg/l. The %RSD obtained were 6.2%, 6.7% and 6.8% for benzene, toluene and p-xylene respectively. These are excellent values (<20%) and indicates the method has a very high precision. Results obtained from spike recovery tests showed >90% recovery indicating good recovery; 94.5%, 96.4% and 97.5% for benzene, toluene and p-xylene respectively. This further verifies the accuracy of the method. The selectivity of the method was assessed by analysing five replicates of a blank sample without adding any analyte and there were no interfering compounds eluting at the retention times of the analytes indicating that the method used is selective.

The limit of detection was estimated was obtained using equations (1) and (2). The values obtained for BTX determination in produced water varied from 0.002ng/l to

0.008ng/l indicating that the projected method has adequate detection capacity to be used for BTX analysis.

A calibration graph was constructed using data obtained from BTX standard solutions prepared at five concentration levels; 0mg/l (blank), 1mg/l, 3mg/l, 5mg/l, and 7mg/l. The linear correlation coefficient (R<sup>2</sup>) obtained is 0.991, 0.9959 and 0.995 for benzene, toluene and pxylene respectively.

### **3.3 BTX ADSORPTION**

#### **3.3.1 EFFECT OF CONTACT TIME**

The removal percentages of benzene, toluene and pxylene at equilibrium time are shown in figure 3.2 for MOS, BP and GAC. It can be seen that % removal increased gradually to equilibrium (60mins for BP and 90mins for MOS and GAC). The % removal of benzene at equilibrium is are 30%, 53% and 55% for BP, MOS and GAC respectively whereas toluene had a % removal of 45%, 56%, 58% for BP, MOS and GAC respectively and p-xylene had a % removal of 49%, 58% and 63% for BP, MOS and GAC respectively. The adsorption of BTX onto BP has shown significantly lower % removals when compared to the % removals of MOS and GAC. The % removal of BTX for all three adsorbents as depicted in the figure 4.2 below was in an increasing order of benzene<toluene<p-xylene. This order corresponds to BTX ascending order of water hydrophobicity (based on BTX octanol-water coefficient log values: 2.13, 2.73, and 3.12 respectively), molecular weight (106, 92, and 78 respectively) and in a descending order of water solubility (1780, 500, and 150 respectively). In general, it appears the hydrophilic and most soluble (benzene) has a lesser affinity for BP, MOS and GAC adsorbents. This similar order of adsorption is in agreement with the adsorption order in previous studies reported by Nourmoradi et al., (2013), Akhtar et al., (2006), and Aivalioti et al. (2012). However, a contrary of adsorption (increasing order order of pxylene>toluene>benzene) was reported by Costa et al., (2012). This was attributed to the fact that relative adsorptions of different compounds can vary based on the substrate used. They added that the chemical properties of the sorbate (water solubility, molecular weight and hydrophobicity) cannot alone explain adsorption, the chemical groups present in the adsorbent also plays a role in adsorption (Costa et al. 2012). Results obtained from the regression analysis of benzene adsorption onto BP, MOS and MOS have shown F value of 0.212, 0.05 and 0.121 respectively indicating that contact time has a significant effect in the removal of MOS as the F value is 0.05 whereas for BP and GAC, F>0.05 indicating that contact time has no significant effect in the removal of benzene onto BP and GAC. Although, there was a gradual increase in % removal with contact time according to the results obtained from the experiment. The F value obtained for toluene adsorption onto BP, MOS and GAC are 0.282,0.140 and 0.144 respectively showing that there is no significant %

removal in relation to contact time for toluene adsorption onto BP, MOS an GAC. P-xylene adsorption onto BP, MOS and GAC has an F value of 0.310, 0.09 and 0.112 respectively showing that there is no significant % removal of toluene with respect to contact time onto BP, MOS and GAC.



Figure 3.2a: Effect of contact time on benzene adsorption using banana peel, and granular activated carbon.
Conditions: 0.4g of the adsorbents; 3ppm of BTX solutions; 10ml volume; agitation at 200 rpm; temperature of 25°C



Figure 3.2b: Effect of contact time on toluene adsorption using banana peel, moringa oleifera seeds and granular activated carbon. Conditions: 0.4g of the adsorbents; 3ppm of BTX solutions; 10ml volume; agitation at 200 rpm; temperature of 25°C



Figure 3.2c: Effect of contact time on p-xylene adsorption using banana peel, moringa oleifera seeds and granular activated carbon. Conditions: 0.4g of the adsorbents; 3ppm of BTX solutions; 10ml volume; agitation at 200 rpm; temperature of 25°C.

#### 3.3.2 EFFECT OF pH

The effect of pH of solution in the removal of BTX (benzene, toluene and p-xylene) was investigated at pH of 4.0, 6.0, 7.0, 8.0, and 10.0. It is evident as seen in figure 3.3 (See data in appendix A2.2) that variation of solution pH does not have a significant effect on BTX adsorption for all three adsorbents and the increase from pH 4.0 to pH 10.0 has not considerably affected BTX adsorption on all three adsorbents. Nevertheless, adsorption of BTX onto MOS and GAC at pH6 has slightly higher percentage removal whereas pH 7.0 and pH 8.0 show higher removal percentages for banana peel. The insignificant influence of pH on BTX adsorption has also been reported by other studies and has been explained to be as a result of high stability of the adsorbents over wide-ranging solution pH (Nourmoradi et al. 2013). This finding also agrees with the BTEX (benzene, toluene, ethylbenzene and xylenes) adsorption on carbon nanotubes documented in literature (Su, Lu and Hu 2010). Conversely, a study carried out by Almeida et al., (2012) has shown that an increased %removal of BTEX onto moringa oleifera seed cake was observed when the solution pH was increased from 5.0 to 7.0. This pH dependence of BTEX adsorption was further explained to be related to the functional groups of moringa oleifera seeds (mainly amino and acid groups) whose ability to interact with the adsorbate is pH dependent (Almeida et al. 2012).

Nonetheless, a contrary result was reported in literature indicating an inverse relationship of BTEC (benzene, toluene, ethylbenzene and cumene) uptake with increasing pH. An increased uptake of BTEC molecules was observed at lower pH whereas at a higher pH, a reduced uptake of BTEC molecules was observed. This was related to the presence of hydrogen ions at lower pH which might have aided the increase in removal whereas the presence of hydrogen ions at a higher pH was suggested to have suppressed BTEC uptake (Akhtar *et al.* 2005). Regression analysis of data obtained for benzene adsorption onto BP, MOS and GAC show F values of 0.172, 0.114 and 0.718 respectively (See appendix A3.4) which are all >0.05 indicating that pH has no significant effect in the removal of benzene onto BP, MOS and GAC. The F-values obtained for toluene adsorption onto BP, MOS and GAC are 0.923, 0.275 and 0.09 respectively showing no significant effect of pH on toluene adsorption. Results of p-xylene regression analysis shows F-values of 0.345, 0.531 and 0.201 for BP, MOS and GAC respectively (See appendix A3.6) depicting that pH has an insignificant effect in pxylene adsorption onto BP, MOS and GAC.



Figure 3.3a: Effect of pH on BTX adsorption using banana peel. Conditions: 0.4g of the adsorbents; 3ppm of BTX solutions; 10ml volume; agitation at 200 rpm; temperature at 25°C.



Figure 3.3b: Effect of pH on BTX adsorption using moringa oleifera seeds. Conditions: 0.4g of the adsorbents; 3ppm of BTX solutions; 10ml volume; agitation at 200 rpm; temperature at 25°C.

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Figure 3.3c: Effect of pH on BTX adsorption using granular activated carbon. Conditions: 0.4g of the adsorbent; 3ppm of BTX solutions; 10ml volume; agitation at 200 rpm; temperature at 25°C.

### **3.3.3 EFFECT OF ADSORBATE CONCENTRATION**

The effect of BTX concentration over a range of 1mg/l -9mg/l was examined and the % removals shown in figure 3.4 were obtained for BP, MOS and GAC. The %removal of benzene and toluene by BP as seen in figure 3.4a increased significantly with an increased concentration whereas there was only a slight increase in the % removal of pxylene with increase in concentration. The % removals of BTX by BP at 9mg/l were 31%, 47% and 56% respectively. The adsorption of BTX onto MOS and GAC has shown a gradual increase with increased concentration with the highest % removals at 9mg/l; benzene- 56% for MOS and 68% for GAC, toluene- 61% for MOS and 64% for GAC and p-xylene- 61% for MOS and 62% for GAC (See figure 3.4b and 3.4c). This may be related to the increased driving force of BTX compounds as well as van der Waal's forces to active adsorption sites of the adsorbents, this occurs at higher concentrations. Similar findings have been reported by Akhtar et al., (2007), Nourmoradi, Khiadani, and Nikaeen, (2012) and Nourmoradi et al., (2013).

The results of regression analysis of benzene adsorption onto BP, MOS and GAC show F values of 0.136, 0.137 and 0.106 respectively (See appendix A3.7). The F values obtained for toluene adsorption are 0.225, 0.699, and 0.064 for BP, MOS and GAC respectively (See appendix A3.8) whereas p-xylene has F values of 0.06, 0.179, and 0.027 for BP, MOS and GAC respectively (See appendix A3.9). The results indicate that concentration has no significant effect on the removal of BTX onto BP, MOS and GAC except for the removal of p-xylene onto GAC which has F value of <0.05.



Figure 3.4a: Effect of adsorbate concentration on BTX adsorption using BP. Conditions: 0.4g of the adsorbent; 1-9ppm of BTX solutions concentration; 10ml volume; agitation at 200 rpm; temperature at 25°C.



Figure 3.4b: Effect of adsorbate concentration on BTX adsorption using MOS. Conditions: 0.4g of the adsorbent; 1-9ppm of BTX solutions concentration; 10ml volume; agitation at 200 rpm; temperature at 25°C.



Figure 3.4c: Effect of adsorbate concentration on BTX adsorption using GAC. Conditions: 0.4g of the adsorbent; 1-9ppm of BTX solutions concentration; 10ml volume; agitation at 200 rpm; temperature at 25°C.



### **3.3.4 EFFECT OF TEMPERATURE**

The influence of temperature on BTX adsorption was assessed for temperatures between 15°C-30°C. The figures below (fig 3.5,3.6 and 3.7) show the effect of varying temperatures (x-axis) on BP, MOS and GAC and the corresponding % removals (y-axis) whereas the legend (on the right of the graph) indicates the time intervals (i.e. 5mins-blue, 20mins-red, 40mins- green, 60mins- purple for BP and 10mins- blue, 40mins-red, 60mins -green, 90mins- purple for both MOS and GAC) at which samples were taken during the experiment. The % removal of BTX onto BP at different temperatures increased in the order of 30°C< 25°C<20°C<15°C while MOS and GAC had the same order of % removal of BTX at different temperatures; 30°C<25°C<15°C<20°C. This indicates that the optimum temperature for BTX adsorption onto MOS and GAC in this study was 20°C whereas 15°C was the optimum temperature for BTX adsorption onto BP suggesting that removal of BTX is higher at lower temperatures. Akhtar et al., (2007) stated that enhanced adsorption for all BTEC components is noted at low temperatures and suggested that the sorption process is of exothermic nature. The %removal of BTX onto BP at optimum temperature (15°C) was 29%, 46% and 53% respectively while the %removal of BTX onto MOS and GAC at optimum temperature (20°C) were 57%, 62%, 60% and 70%, 65% and 59% respectively.

Therefore, the adsorption of BTX by all three adsorbents is in the increasing order of BP<MOS<GAC suggesting GAC to have the highest removal of the BTX molecules though having close values of % removals with MOS. BP has again recorded the lowest removal percentage of BTX molecules. The results of regression analysis shows that significant removal of benzene onto BP was only obtained at 20°C with F value of 0.03 while toluene and p-xylene adsorption onto BP show a significant removal at 25°C with F value of 0.02 and 15°C with F value of 0.04 respectively. Results obtained for the regression analysis of BTX adsorption onto MOS shows temperature has no significant effect in the removal of BTX onto MOS whereas the results for regression analysis of BTX adsorption onto GAC indicate that at 25°C, there was a significant removal of benzene onto GAC as the F value obtained is 0.02. Toluene adsorption onto GAC has shown F value of 0.05 at 30°C indicating significant removal of toluene at this temperature while regression analysis of results of pxylene adsorption onto GAC has shown no significant removal at all temperatures. Generally, according to results obtained from the experimental data, the optimum temperature for BTX removal onto the adsorbents was observed to be 20°C.



Figure 3.5a: Effect of temperature on Benzene adsorption onto BP. Conditions: 0.4g of the adsorbent; adsorbate 3ppm; 10ml volume; agitation at 200 rpm; temperatures (15, 20, 25, and 30) °C; time interval (5, 20, 40, 60) minutes.



Figure 3.5b: Effect of temperature on toluene adsorption onto BP. Conditions: 0.4g of the adsorbent; adsorbate 3ppm; 10ml volume; agitation at 200 rpm; temperatures (15, 20, 25, 30)°C; time interval (5, 20, 40, 60) minutes.



Figure 3.5c: Effect of temperature on P-Xykene adsorption onto BP. Conditions: 0.4g of the adsorbent; adsorbate 3ppm; 10ml volume; agitation at 200 rpm; temperatures (15, 20, 25, and 30) °C; time interval (5, 20, 40, and 60) minutes.



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Figure 3.6a: Effect of temperature on Benzene adsorption onto MOS. Conditions: 0.4g of the adsorbent; adsorbate 3ppm; 10ml volume; agitation at 200 rpm; temperatures (15, 20, 25, and 30) °C; time interval (10, 40, 60, and 90) minutes.



Figure 3.6b: Effect of temperature on Toluene adsorption onto MOS. Conditions: 0.4g of the adsorbent; adsorbate 3ppm; 10ml volume; agitation at 200 rpm; temperatures (15, 20, 25, and 30) °C; time interval (10, 40, 60, and 90) minutes.



Figure 3.6c: Effect of temperature on P-Xylene adsorption onto MOS. Conditions: 0.4g of the adsorbent; adsorbate 3ppm; 10ml volume; agitation at 200 rpm; temperatures (15, 20, 25, and 30) °C; time interval (10, 40, 60, and 90) minutes.



Figure 3.7a: Effect of temperature on Benzene adsorption onto GAC. Conditions: 0.4g of the adsorbent; adsorbate 3ppm; 10ml volume; agitation at 200 rpm; temperatures (15, 20, 25, 30)°C; time interval (10, 40, 60, 90) minutes



Figure 3.7b: Effect of temperature on Toluene adsorption onto GAC. Conditions: 0.4g of the adsorbent; adsorbate 3ppm; 10ml volume; agitation at 200 rpm; temperatures (15, 20, 25, 30) °C; time interval (10, 40, 60, 90) minutes.



Figure 3.7c: effect of temperature on P-Xylene adsorption onto GAC. Conditions: 0.4g of the adsorbent; adsorbate 3ppm; 10ml volume; agitation at 200 rpm; temperatures (15, 20, 25, 30) °C; time interval (10, 40, 60, 90) minutes.



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# **3.3.5 EFFECT OF ADSORBENT DOSAGE**

The effect of adsorbent dosage was investigated within a range of 0.2g-0.8g. This was reported as adsorption capacity of BP, MOS and GAC as seen in figures 3.18a, b and c respectively (See data in appendix A2.5). The adsorption capacity of BP for BTX increased progressively as the dosage was varied from 0.2g to 0.8g whereas for MOS and GAC, the increase in adsorption capacity was only distinct when the dosage was increased from 0.2g to 0.4g but has remained constant when dosage was increased from 0.4 to 0.8g. This suggests that 0.4g is sufficient sorbent dosage (MOS and GAC) to adsorb BTX molecules. The adsorption capacities of BP, MOS and GAC obtained in this present study were; benzene: 0.020, 0.036, 0.042 (mg/g), toluene: 0.038, 0.039, 0.041 (mg/g) and p-xylene: 0.037, 0.038, 0.039 (mg/g) respectively. This result conforms to studies conducted on adsorption of organic pollutants using MOS (Akhtar et al. 2007). The order of adsorption in this result for three adsorbents is in the order B<T<X suggesting that order of adsorption is related to solubility, molecular weight and hydrophobicity of the sorbate as earlier envisaged (Nourmoradi et al. 2013). The sorption capacities of the selected adsorbents used in this study have been compared with sorption capacities of other sorbent in different studies (See table 3.3). As shown on table 3.3, the adsorption capacity of MOS obtained in this study is higher than those obtained for BTX adsorption onto peat and diatomite in other studies. The regression analysis of benzene adsorption onto BP, MOS and GAC have shown F values of 0.04, 0.06 and 0.22 respectively indicating that dosage has a significant effect only for benzene adsorption onto BP. The F values obtained for toluene adsorption onto BP, MOS and GAC are 0.02, 0.26 and 0.22 respectively depicting that dosage has a significant effect on the adsorption of toluene onto BP and otherwise for MOS and GAC. P-xylene adsorption onto BP, MOS and GAC has shown F values of 0.07, 0.17 and 0.10 respectively indicating that dosage has no significant effect on p-xylene removal onto BP, MOS and GAC.



Figure 3.8a: Adsorption capacity of BP, MOS and GAC on Benzene removal. Conditions: (0.2-0.8)g of the adsorbent; adsorbate 3ppm; 10ml volume; agitation at 200 rpm;temperatures 25°C.



Figure 3.8b: Adsorption capacity of BP, MOS and GAC on Toluene removal. Conditions: (0.2-0.8)g of the adsorbent; adsorbate 3ppm; 10ml volume; agitation at 200 rpm; temperatures 25°C.



Figure 3.8c: Adsorption capacity of BP, MOS and GAC on Pxylene removal. Conditions: (0.2-0.8)g of the adsorbent; adsorbate 3ppm; 10ml volume; agitation at 200 rpm; temperatures 25°C

Adsorbent	Benzene	Toluene	Р-	Reference
	(mg/g)	(mg/g)	xylene	
			(mg/g)	
Peat	0.049	0.062	0.0142	Costa et al. 2012
Modified	3.98	5.15	6.98	Nourmoradi
Clay				et al. 2013
(TTAB-				
MT)				
Diatomite	0.031	0.037	0.042	Aivalioti et al.
				2012
Banana	0.020	0.038	0.037	This study
peel				



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Moringa Oleifera seeds	0.036	0.039	0.038	This study
Granular activated carbon	0.042	0.041	0.039	This study

# **3.3.6 MATRIX EFFECT OF AQUEOUS SOLUTION**

In order to investigate the effect of the aqueous matrix (saline produced water) on BTX uptake onto the selected adsorbents, an adsorption experiment was carried out simultaneously in produced water and de-ionised water under the same condition as all other experiments. The results are shown in figure 3.21a, b, and c for BP, MOS and GAC respectively. The % removals of BTX in produced water across the three adsorbents were  $\leq 55$ ,  $\leq 57$  and ≤59% respectively whereas a higher % removal was obtained in de-ionised water for all three adsorbents;  $\leq$ 70% - B,  $\leq$ 73% - T and  $\leq$ 74% - X. This indicates that the highly saline produced water hinders the adsorption of BTX onto the selected adsorbent. The low polarity of BTX might have hindered their extraction from a matrix of high ionic strength and thus a lower % removal onto the adsorbents. Aivalioti et al., (2012) reported similar results in literature on the adsorption of BTEX, MTBE and TAME onto diatomite. However, a contrary result was reported in literature stating that the removal of BTEX from saline produced water was not hindered and suggested that the high salinity of the produced water aided removal and increased the availability of active sites on the adsorbents (Costa et al. 2012)



Figure 3.9a: Effect of aqueous matrix solution on BTX adsorption onto BP. Conditions: 0.4g of the adsorbent; adsorbate 3ppm; 10ml volume; agitation at 200 rpm; temperatures 25°C.



Figure 3.9b: Effect of aqueous matrix solution on BTX adsorption onto MOS. Conditions: 0.4g of the adsorbent; adsorbate 3ppm; 10ml volume; agitation at 200 rpm; temperatures 25°C.



Figure 3.9c: Effect of aqueous matrix solution on BTX adsorption onto GAC. Conditions: 0.4g of the adsorbent; adsorbate 3ppm; 10ml volume; agitation at 200 rpm; temperatures 25°C.

# 3.3.7 Comparative Analysis

A study to investigate the sorption efficiency of two locally available adsorbents; banana peel and moringa oleifera seeds in the removal of benzene, toluene and pxylene from produced water was carried out. This was carried out simultaneously with granular activated carbon in order to compare results obtained from the selected locally available adsorbents with a standard adsorbent (GAC). The parameters varied in order to obtain optimum removal efficiency include contact time, pH, dosage, adsorbate concentration and temperature. The results obtained from variations of these parameters were employed in the comparative analysis of the sorption efficiency of all three adsorbents. The % removals obtained for BP, MOS and GAC in BTX removal when contact time experiments were carried out are; benzene-30%,53%,55%; toluene-45%, 56%, 58%; p-xylene-



49%,58%,63% respectively. The adsorption of BTX onto the three adsorbents was in an increasing order of; BP<MOS<GAC indicating that GAC and MOS had a higher % removal of BTX over BP. In an experiment to investigate the effect of pH in BTX removal, the % removals of BTX obtained at optimum pH for BP, MOS and GAC are; benzene-24%,56%, 65%; toluene-46%,59%,61%; pxylene-55.5%,56%,57% . BTX removal in pH experiments was in the order of BP<MOS<GAC with GAC having the highest % removal of BTX compounds and closely preceded by MOS. The effect of dosage experiments carried out for BTX adsorption onto BP, MOS and GAC yielded adsorption capacities of; benzene-0.020mg/g, 0.036mg/g,0.042mg/g; toluene- 0.038mg/g, 0.039mg/g, 0.041mg/g; p-xylene- 0.037mg/g, 0.038mg/g, 0.039mg/g.

The BTX removal order for adsorption capacity is in the increasing order of BP<MOS<GAC and in the effect of adsorbate concentration experiment, the following %removals were obtained from BP, MOS and GAC when the initial concentration of BTX was increased to 9PPM; benzene- 31%, 56%, 68%; toluene-47%,61%,64%; pxylene- 56%, 61%, 62% respectively. The order of BTX removal onto the adsorbents is BP<MOS<GAC indicating that GAC has the highest % removal although in the same range with MOS % removal. The temperature effect experiment was carried out to investigate the adsorption efficiency of BP, MOS and GAC at varying temperatures. At optimum temperature, the following % removals of BTX onto BP, MOS and GAC were obtained; benzene-29%, 57%, 70%; toluene-46%, 63%, 65%; p-xylene-53%, 57%, 59% respectively. The sorption order was again in the order of BP<MOS<GAC indication that GAC has highest percentage removal. These results reveal that for all experiments carried out, GAC was observed to have the highest removal efficiency for BTX although the % removals obtained for MOS were only slightly lower than the % removals of GAC. BP was observed to have the lowest removal of BTX as compared to MOS and GAC.

#### **4.0 CONCLUSION**

This study was undertaken in order to evaluate the sorption efficiency of MOS and BP in the removal of BTX from produced water and to compare these efficiencies with a standard adsorbent; GAC. Following the results obtained from the analysis of the three adsorbents (BP, MOS and GAC), BP seemed to possess the lowest surface area (1.856  $m^2/g$ ) and GAC had the highest surface area  $(600-800m^2/g)$  while MOS had a surface area of 4.01 m<sup>2</sup>/g. The low surface area of BP might have hindered significant removal of BTX as seen in the results. It was observed that the treatment of MOS with nitric acid increased the pore area of MOS but the acidity on the MOS surface might have reduced the uptake of the hydrophobic BTX onto MOS as adsorption of the BTX molecules is not only a function of porosity of the adsorbent but also a function of the surface nature of the adsorbent.

BTX adsorption onto BP was observed to be higher between 30-60 minutes whereas uptake of BTX by MOS and GAC were highest between 60-90 minutes suggesting that significant adsorption of BTX onto BP, MOS and GAC were achieved at 60mins(BP) and 90mins (MOS and GAC). Variations of pH had no significant effect on the uptake of BTX indicating the stability of the adsorbents over a wide range of pH. However, a slightly higher percentage removal was observed at pH6 for MOS and GAC and at pH8 for BP. This suggests that pH6 might be the optimum pH for the uptake of BTX compounds onto MOS and GAC while it is pH8 for BP. Results from variations of adsorbent concentration indicate that BTX adsorption/ removal is proportional to its concentration in an aqueous medium as the highest uptake of BTX molecules was observed at the highest concentration used (9PPM). This is a result of increased mobility of BTX molecules towards active binding sites of the adsorbents in an adsorption process. This suggests that with an increased concentration of BTX, a higher %removal might be achieved using MOS and GAC. Increased sorbent dosage (above 0.4g) has shown no effect on BTX uptake indicating that 0.4g of MOS and GAC is a sufficient mass for the adsorption of 3PPM of BTX concentrated solutions. The sorption of BTX compounds was observed to be more favourable at lower temperatures, the optimum temperature for BTX removal established in this study was 20 °C.Generally, GAC has the highest % removal of BTX compounds although was only <10% higher than the %removals obtained for MOS.

Conclusively, based on all the results obtained from this study, it can be deduced that MOS is a promising potential adsorbent in the effective adsorption of BTX compounds under favourable conditions as suggested above while BP has not shown significance removal of BTX compounds.

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