Chapter

Adsorption Technique an Alternative Treatment for Polycyclic Aromatic Hydrocarbon (PAHs) and Pharmaceutical Active Compounds (PhACs)

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Abstract

Water is essential to human consumption; however, its pollution is caused by populace activities from both organic and inorganic compounds sources that require serious attention, to provide clean water. Organic contaminants are known as persistent organic pollutants (POP). They are accumulated in the fat tissues of wildlife and human beings and are toxic to their organs. Degradations of POP are very difficult since they are persistent and also termed as semi-volatile, for example, polycyclic aromatic hydrocarbons (PAHs). Apart from POPs, others toxic organic contaminants with subtle ecological effects are the emerging organic contaminants (EOCs), like pharmaceutical actives contaminants (PhACs). They penetrate the aquatic environment and alter the natural quality. To obtain future discharge requirements, new technologies with granular activated carbon were developed using *Oxytenanthera abyssinica* and *Bambusa vulgaris* in remediating PhACs and PAHs. The activated carbon with KCl had removal efficiency of 73.3, 78.1, and 86.2%, which indicated the highest efficiency for PhACs removal, while adsorbent activated with H_3PO_4 gave 63.9, 66.7, and 82.2% for paracetamol, salbutamol, and chlorpheniramine, respectively. Removal efficiency of 42.5–81.2% and 8.9–65.5% ranges of PAHs were obtained for CBV and COA, respectively. The alternative adsorption treatment techniques are detailed in the chapter.

Keywords: wastewater, adsorption technique, organic pollutant, polycyclic aromatic hydrocarbon, pharmaceutical active compounds

1. Introduction

The earth's surface is covered by 70% of the water of which 97.5% are from seas and oceans which are salty for consumption, out of the remaining 2.5% water, 1.73% are in form of glaciers and ice-caps, left only with 0.77% available for freshwater supply. The amount of water available on the earth that can be renewable is only

0.0008% in the rivers and lakes for humans and agricultural use [1]. Fundamentally water is needed by all living creations of God, hence it must be provided in the rest state for their consumption, therefore clean water becomes a critical issue as the world population increases [2]. The populace activities are a factor that causes an increase in pollution from both organic and inorganic compounds sources that require serious attention to ensure clean water that the same growing world needs to consume.

Clean water becomes a critical issue as the world population increases. It has been estimated that by the year 2025, there would be an additional 2.5 billion people on the earth that will live in a region already lacking sufficient clean water [1]. Similarly, scholars have indicated that the recent problems in water treatment originate primarily from the increasing pollution of water by an organic compound that is difficult to decompose biologically because these substances resist the self-purification capabilities of the rivers as well as decomposition in conventional wastewater treatment plants [3, 4]. Further observations state that the conventional mechanical-biological purification is no longer sufficient and must be supplemented by an additional stage of processing [5].

Adsorption is the capacity of the adsorbate to form a bond with the adsorbent [6]. It is also defined as a physical and chemical process in which substances are accumulated at the interface between the faces which may be liquid-liquid, liquid-solid, or gas-liquid [7]. Adsorption differs from absorption in that it is the process by which the surface concentrates fluid molecules by chemical or physical force while absorption is the partial chemical bonds formed between adsorbed species or when the absorbate gets into the channels of the solids [8]. In other words, fluid molecules are taken up by a liquid or solid and distributed throughout the liquid or solid. Adsorbate is the substance that is removed from the wastewater or the amount of contaminant adhering to the surface of the adsorbent, while the adsorbent is the solid phase that accumulates the pollutant. This may be activated carbon or other biosorption materials [7]. For adsorption to take place, the adsorbate must have less free energy on the surface of the adsorbent in solution.

Organic contaminants are occasionally termed persistent organic pollutants (POP), their occurrences in the environment are frequent and possess the ability to move fast across the water and settle from where they are sources. Accumulated in the fat tissues of wildlife and human beings and are very toxic to their organics. Degradations of POP are very difficult since they are persistent and also termed semivolatile for example PAHs. Apart from POPs other toxic organic contaminants which can create subtle ecological effects are the Emerging Organic Contaminants (EOCs), the extent to which the environment can be adversely affected by EOCs is still under study. One of such is the (PhACs) which are products of synthetic chemicals, natural organic chemicals, or microorganisms not controlled. They possess the ability to penetrate the aquatic environment and alter the natural quality leading to adverse health issues in human and ecological disorders [9, 10].

The Petroleum and Pharmaceutical industries are seen among others as major contributors of organic contaminants because of continuous usage and pollutant from them are emerging and steady in the environment [10].

Produced water and crude oil spills are the major sources of pollutants generated by the petroleum industry. Produced water is the largest by-product of wastewater attributed to the petroleum industry and it is a mixture of salt, organic and inorganic compounds.

Among the organic constituent of crude oil is a group of hydrocarbons called PAHs [11]. These are large groups of organic contaminants, which are characterized

by the presence of at least two fused aromatic rings and are seen by the United States Environmental Agency (USEPA) as priority organic pollutants [12]. PAHs are highly lipophilic contaminants that are ubiquitously present in the environment [13] because of their low biodegradation and bioaccumulation in the adipose tissues of organisms and biomagnifications through the food chain, they are considered persistent organic pollutants POPs [14].

The pharmaceutical industries on the other hand have to do with the well-being of living organisms. Their products refer to a group of chemicals used for the diagnosis, treatment, or prevention of health conditions. Most of the chemicals or ingredients used in production can be active, inactive, additive, or preservative. When most of these ingredients are no longer used for the intended purpose and if the pharmaceutical product is designated for discarding, it is then classified as pharmaceutical waste. Active chemicals like paracetamol (acetaminophen), salbutamol, amoxicillin, ibuprofen, chloramphenicol, etc. can be referred to as pharmaceutically active compounds (PhACs), and preservatives such as parabens, e.g., ethyl, propyl, etc. are called excipient [15]. Pharmaceutical wastes are EOCs of concern and are mostly unregulated contaminants that need future regulation [16].

Like any EOCs, they do not need to persist in the environment to cause negative effects because they are continually being released into the environment mainly from manufacturing processes, disposal of unused products, and excreta [17]. At the 2005 Burger AEC programme on EDCs, it was reported that most EOCs can disrupt the endocrine system-a health condition called endocrine disruptors [18]. The WHO defines an endocrine-disrupting substance as an exogenous substance that alters the function of the endocrine system and consequently causes adverse health effects in an organism or its progeny or subpopulations [19].

Petroleum and pharmaceutical Industries have been seen as major generating sources of organic contaminants that create adverse effects on surface water which are the primary source of livelihood. For water to be available in its pure state, identifying and remediating processes of those contaminants is key, if clean water is a necessity [10].

Most research works had employed analytical techniques like gas and liquid chromatography, UV-spectrophotometers and gravimetric, etc. to identify organic contaminants, gas and liquid chromatography with mass spectrophotometer followed by a cleanup method such as solid-phase extraction (SPE) and solid-phase microextraction (SPME) [20] are seen as most effective techniques in determinations of organic pollutants in trace amount (or micro-pollutant).

Induced Gas Flotation (IGF) or the Induced Air Flotation cells are usually used as conventional treatment methods by petroleum industries to separate produced water from crude oil with Enviro-cell as the newest technology that uses the principle of gravity with differences in density between the oil and water [20].

The hydrocarbon content in either the produced water or water polluted with crude spills can be classified as free, dispersed, and dissolved oil [21, 22]. The conventional method is seen to be effective in the removal of dispersed oil and grease but cannot be used in the removal of the dissolved hydrocarbon which includes the PAHs. Different literature had reviewed that conventional wastewater treatment plants (WWTPs) are not the best in the removal of PAHs pollutants from wastewater, additional methods that had been researched and are still being researched is the adsorption mechanism. It had been suggested that carbon and membrane filtration with reverse osmosis are very effective in the removal of dissolved and emulsified oils [23, 24]. Many materials in their raw or waste form had been developed or modified

into adsorbent in adsorption of pollutants which could be agricultural materials, clay, zeolite, vibratory share enhanced process, etc.

The commonest adsorbent used by most industries for the removal and recovery of inorganic and organic substances from gaseous and liquid streams is activated carbon [25]. Because of its high internal surface area and porosity formed during the carbonization process, the adsorbent is said to have a high adsorption capacity. Similarly, the use of activating agents and heat during carbonization will influence the development of pore structure but its uses are limited to high cost hence the use of agricultural products or materials have been observed to be potential precursors in activated carbon production because of the abundant supply and low cost.

Most of the research conducted lately made use of agricultural product such as adsorbent in the removal of heavy metals from water and wastewater, such agricultural products are coconut shell and rice husk [26, 27], palm kernel shell, and oil palm fruit fiber [28], bamboo [3, 29], maize cob [6]. Other works had been done on the identification and remediation of organic contaminants in petroleum [30–31] and pharmaceutical wastes [32–33] but few works have been done on the use of adsorbents in remediating organic pollutants (PAHs and EOCs).

Since water is the prime necessity of life and very essential for the survival of all living organisms it is imperative to improve the quality of available water. The presence of pharmaceutical residues (PhACs) and PAHs as newly recognized contaminants in aquatic systems is one of the current environmental issues [34]. It should be noted that organic contaminants usually occur in multi-component in aquatic environments. Thus, it is expected that there will be interspecies interaction among these pollutants which will cause chemical reactions that can generate other metabolites compared to when the single contaminant is present [35].

Adsorption method of bioremediation had proven to be the chosen treatment option for PAHs and PhACs and other micro-pollutant in aqueous or any environmental media because it is easier to understand and has obvious advantages of convenience, easy operation, efficiency, effective, and very simple to design as compared to another kind of treatment. Apart from the identified attributes, it does not add harmful degradation metabolites or undesirable by-products [36].

Adsorption is better than any other wastewater treatment method due to its insensitivity to toxic substances and is economically based on types of materials employed as adsorbents [37]. But, the complete use of adsorption processes in purifying water is impeded by the insufficiencies of the commercial adsorbents like activated carbon and synthetic polymer resins, synthetic Nanomaterial. Hence, there is a need to develop a low-cost adsorbent for environmental research. So, the adsorbent of agricultural products is becoming the popular alternative for commercial and synthetic adsorbents due to the hydrophobic-oleophilic potential that is needed for bioremediation processes [38]. With this new trend in mind, this chapter will seek to explain courses of organic pollutants with a special interest in industrial wastewater and adsorption techniques as an alternative treatment.

2. Assessment of production activities that leads to the generation of wastewater in the selected industries

To determine wastewater characteristics, generation disposal processes and assessment of production activities that lead to wastewater generation within the

pharmaceutical and petroleum industries were conducted through interviews, observation, and experimental analysis. From the information gathered, most pharmaceutical industries in a country located in West Africa produced more syrups than any other form of drugs because it is cheaper to produce. Production of syrups takes more than 50% of production water and an average of 48,000 L of water per day is being discharged as effluent during the production of syrups. Syrups can be analgesic, antacids, or pain relievers. Effluent discharge may contain codeine phosphate, paracetamol, chlorpheniramine maleate, ephedrine HCl, parabens, etc. which are regarded as active pharmaceutical compounds. PAHs and PCBs can be found within the effluent as a result of chemical metabolites. Most of the pharmaceutical industries around the States visited in that country discharge their effluents through the drains into the surface water.

The petroleum industries visited were located within the Delta area of the country. The industries have two sources of liquid waste which are produced water and water pollutant with crude spills. Averagely about 30 million barrels of produced water are said to be discharged per day into the environment. Most of the oil and gas industries in the Delta region utilize the common treatment technique like the Induced Gas Flotation (IGF) or the Induced Air Flotation called WEMCO with the Enviro-cell as the latest technology of the group. The orthodox separation technique used the standard of gravity with variances in density between oil and water.

The oil and grease in the produced water can be classified as free, dispersed, and dissolved oil [21, 22]. The conventional method is seen to be effective in the removal of dispersed oil and grease and cannot be used in the removal of the dissolved oil and grease which are the PAHS (**Figure 1**).

2.1 Production of local Adsorbent using local Technology

The production of local adsorbent from the natural agricultural material using local Technology is a crucial alternative to commercial absorbent since adsorbent is used in carrying out adsorption which is better than any other wastewater treatment methods due to its insensitivity to toxic substances and economically

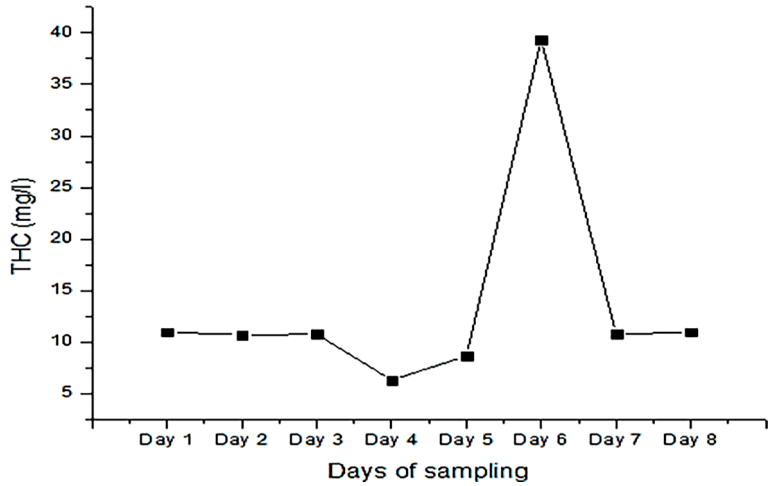


Figure 1.
Concentrations of oil and grease in produced water at different days of sampling.

based on types of materials employed as adsorbents [37]. But, the complete use of adsorption processes in purifying water is impeded by the insufficiencies of the commercial adsorbents like activated carbon and synthetic polymer resins, synthetic Nanomaterial. Hence, there is a need to develop a low-cost adsorbent for environmental research. So the adsorbent of agricultural products is becoming the popular alternative for commercial and synthetic adsorbents due to the hydrophobic-oleophilic potential that is needed for bioremediation processes [38]. The Activated Charcoal which is also known as activated carbon is a kind of carbon treated under heat to be extremely porous as processes very large surface area making room for adsorption or chemical reactions [39]. The activated carbons that are employed were formed from two different species of fresh bamboo culms that were cut at the height of 20 cm from the soil level and were chopped into 20 cm each as the peripheral materials were detached. The chopped bamboo culms were left to dry at the normal ambient temperature of 26-28°C and later cut down to 5 cm. The bamboo species were weighted and the aluminum foil was used to tightly cover in preparation for carbonization, the wrapping with the foil was done to complete deoxygenated processes. It was carbonized at 350°C for 2 hrs in an electric muffle furnace. Carbons were cooled and oven-dried at 105°C for 360 min. The carbonated samples were granulated and sieved to 1.18 m size and stored. Activation was done with Phosphoric acid (H_3PO_4) and Potassium chloride (KCl) as dehydrating agents. 26.25w/w of activator was used in the activation of the carbonated samples. Characterization was done chemically using Point of zero charge (pH pzc) and The Scanning Electron Microscopy (SEM) was used to view the surface structure of the samples at a magnification of 100, 300, 500, 2000, and 5000 times the original size to view the pore space development and reveal other information such as texture (external morphology) and structural orientation see **Figure 2** [10]. The point of zero charge (pH pzc) was used for further determination of pore and adsorption capacity. The point of zero charge is the point where the pH of the net total particle charge is zero. It is important in describing variable charge surface and it indicates the approximation equilibrium time at which the carbon is required to adsorb. Points of zero charge of the adsorbents were determined by measuring 20 ml of 0.01 m Nacl solutions into 9 separate beakers. The pH of each solution was adjusted to between 2 and 10 by adding 0.1 M of HCl or NaOH solution to each of the flasks. The flasks were thereafter placed in a water bath shaker at 25°C. The suspensions were agitated for 30 min and allowed to equilibrate for 48 hrs to ensure equilibrium point (pH pzc) after which the final pH(s) were measured see **Figure 3**. The differences between the initial and final were calculated as:

$$\Delta pH = pH_i - pH_f \tag{1}$$

Where ΔpH = Change in pH, pH_i = Initial pH, and pH_f = Final pH.

The values of changes in pH were then plotted against the initial pH values.

From **Figure 3** above the pH pzc for carbon activated with salt is greater than pH value of the activated with acid, i.e., pH pzc < pHi COA KCl and pH pzc for CBV H3PO4 is lower than that pHi implying that pH > pH pzc This implies that the surfaces of these carbons are positively charged and this arises from the basic site that combines with protons from the medium. These results also confirmed [40, 41]

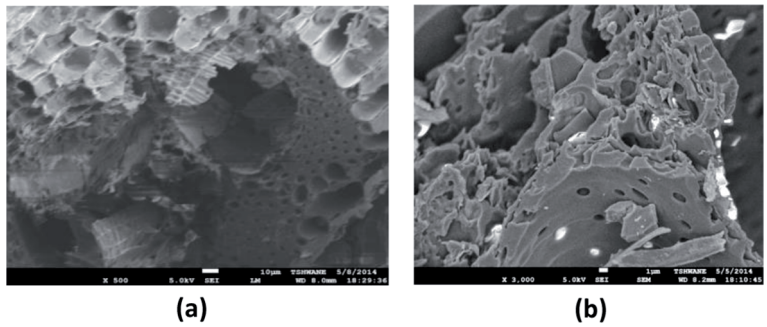


Figure 2.
SEM image of (a) CBV 350°C H_3PO_4 and (b) COA 350°C KCl at magnification of 2000.

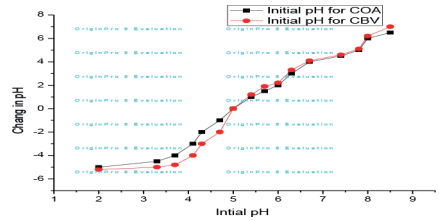


Figure 3.
Potentiometric titration curves (pH pzc) for CBV H_3PO_4 and COA KCl.

studies that a positive surface charged adsorbent would strongly attract to acidic compound in any polluted water, while a negative surface charge would strongly attract pollutant in a natural media.

2.2 Adsorption of PAH and PhACs from industrial wastewater

The behavior of Adsorption for PhACs in pharmaceutical effluents and polycyclic aromatic hydrocarbon (PAHs) in petroleum wastewater onto activated carbon produced from bamboo was studied in a batch process. Experiments were done at room temperature and the adsorption efficiency of activated carbon made from bamboo was determined using contact time. Half a liter of Pharmaceutical effluent was poured into conical flasks with a capacity of 600 ml. Selected bamboo activated carbon of two grammes each was weighed into conical flasks to make an adsorbent/solute solution. Solutions were mixed at a stirring speed of 160 rpm to ensure propped contact of the adsorbent and solute in the solution. 6 hrs contact time was used to observe each solution before reaching a dynamic equilibrium. Thereafter, solutions were filtered with filter paper 0.45 μm size. 300 ml filtrate was poured into sampling bottles with a tie cap sealed with aluminum foils and kept at a temperature of 4°C for further analysis of extraction, clean-up, and Vis-UV. For accuracy, all experimental analysis was repeated. Similarly, 200 ml of petroleum wastewater simulated, was poured into different conical flasks of 250 ml capacity. 1 g of each selected bamboo activated carbon was weighed into the conical flasks to form an adsorbent/solute solution. Solutions were mixed at a stirring speed of 160 rpm to ensure propped contact of the adsorbent and solute in solution while observing each solution in equilibrium for 5 hrs contact-time

to attain dynamic equilibrium. After 5 hrs of clean up and solutions were filtered with filter paper 0.45 μm and the filtrate of 150 ml was poured into amber bottles with a Teflon cap and kept at a temperature of 4°C for further analysis of and extraction were done before analying with GC-MS. The 5 hrs contact time was informed based on the experiment performed at the terminal station of the oil and gas industry. To obtain accuracy, all experimental analysis was duplicated [10]. The amount of PhACs (qe) and PAHs (qe) adsorbed by bamboo activated carbons can be expressed mathematically as:

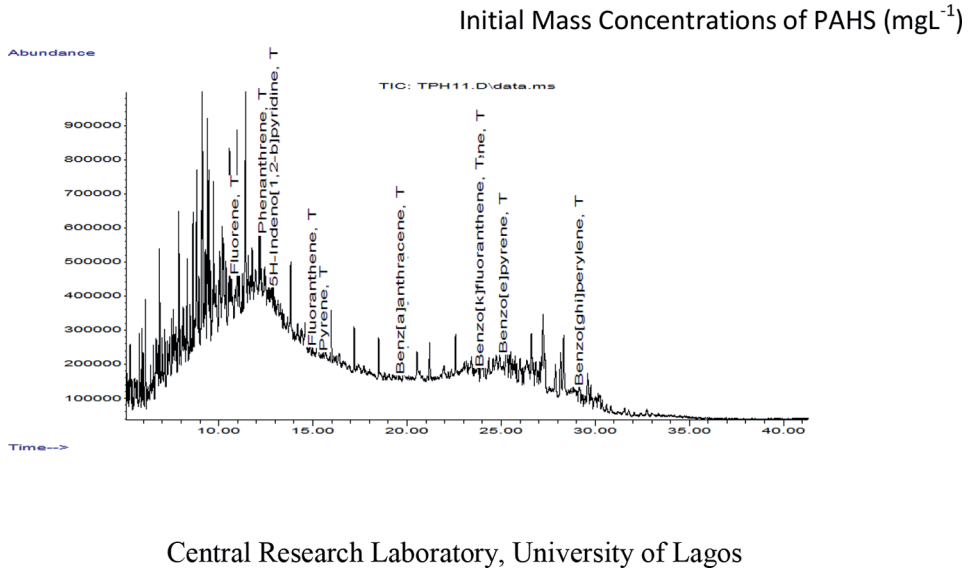
$$qe = ((Co - Ce)/M) \times v \tag{2}$$

The percentage removal is evaluated using.

$$\%\,Removal = ((Co - Ce)/M) \times 100 \tag{3}$$

Where V is the volume of PAHs and PhACs in solution (L), Co is initial concentrations of PAHs and PhACs ($mg\,L^{-1}$), Ce is equilibrium concentrations of PAHs and PhACs ($mg\,L^{-1}$), M is the mass of the adsorbent (g).

The Spectra in **Figure 4** shows, 16 priority PAHs in Simulated Petroleum wastewater. The efficiency of adsorbent based on contact time in the removal of PAHs from simulated petroleum wastewater by COA KCl and CBV H_3PO_4 are stated in **Figures 5** and **6**. The various contact times used were 30 min, 2 hrs, and 12 hrs, and there were otnotany changes differences in the adsorption rate with time. It was deduced that the percentage removal efficiency of PAHs with time by COA KCl was not consistent. At 30 min of adsorption rate; the percentage removal efficiency was 49.8% of total PAHs, while 39.1% of total PAHs were adsorbed at 2 hrs and about 72.3% of total PAHs were adsorbed at 12 hrs contact time.



Central Research Laboratory, University of Lagos

Figure 4.
Spectra of 16 priority PAHs in Simulated Petroleum wastewater.

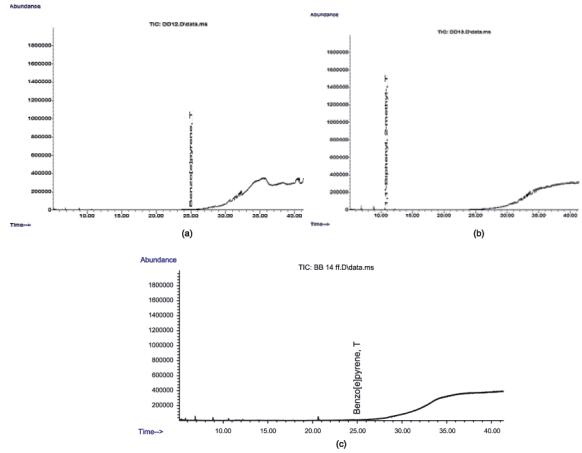


Figure 5.
Effect of contact time on adsorption rate of PAHs by COA KCl at (a) 30 mins, (b)2 hrs and (c) 12 hrs.

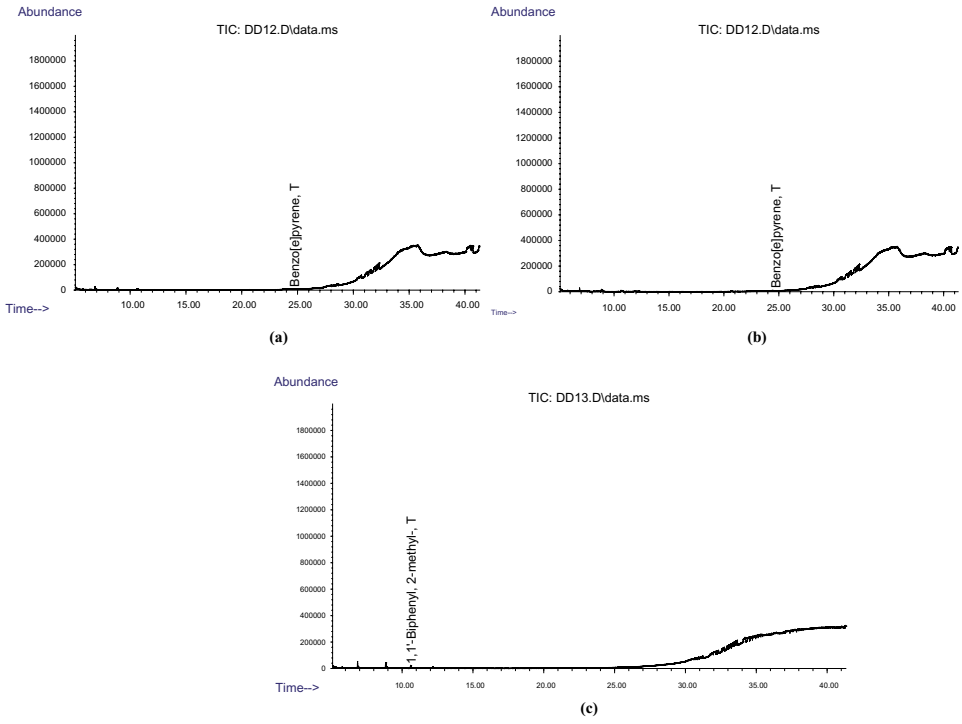


Figure 6.
Effect of contact time on adsorption rate of PAHs by CBVH3PO4 at (a) 30 mins, (b) 2 hrs and (c) 12 hrs.

Also, the adsorption rate of PAHs by CBV H_3PO_4 was seen not to follow the adsorption pattern wherein the adsorption rate increased with time. About 85.1% was adsorbed at 30 min; with an increase in contact time to 2 hrs its shows a reduction in adsorption rate to 25.1% but a further increase in time to 12 hrs increases adsorption efficiency to 87.7% which is the maximal contact time for CBV H_3PO_4 in adsorbing PAHs. It was deduced that adsorption patterns do not follow the norms of adsorption, hence adsorption of PAHs is in

two stages. In the first stage, the PAHs were adsorbed easily onto the accessible hydrophobic site within the adsorbent or granular activated carbon matrix for the first 30 min. These may have resulted from the chemical interaction between the PAHs and the adsorbent. The reduction in adsorption rate implies that in the second stage, the adsorption rate may be restricted by the slow movement of PAHs to less available sites associated with the micro-spores within the adsorbents matrix which could take hours [10].

Figure 7a-c revealed the adsorption trend of PhACs, it was deduced that a slight reduction of adsorption rate was observed before an increase after which equilibrium was observed for COA KCl adsorbent. Similarly, the adsorption trend of CBV H_3PO_4 shows a sharp reduction to a level, and thereafter an increase was seen before an equilibrium point was reached. These observations are similar to [42–45] findings. It can be explained that absorption of PhACs with COA KCl and CBV H_3PO_4 occurred in two different stages. The first stage occurred during the first 30-360mins

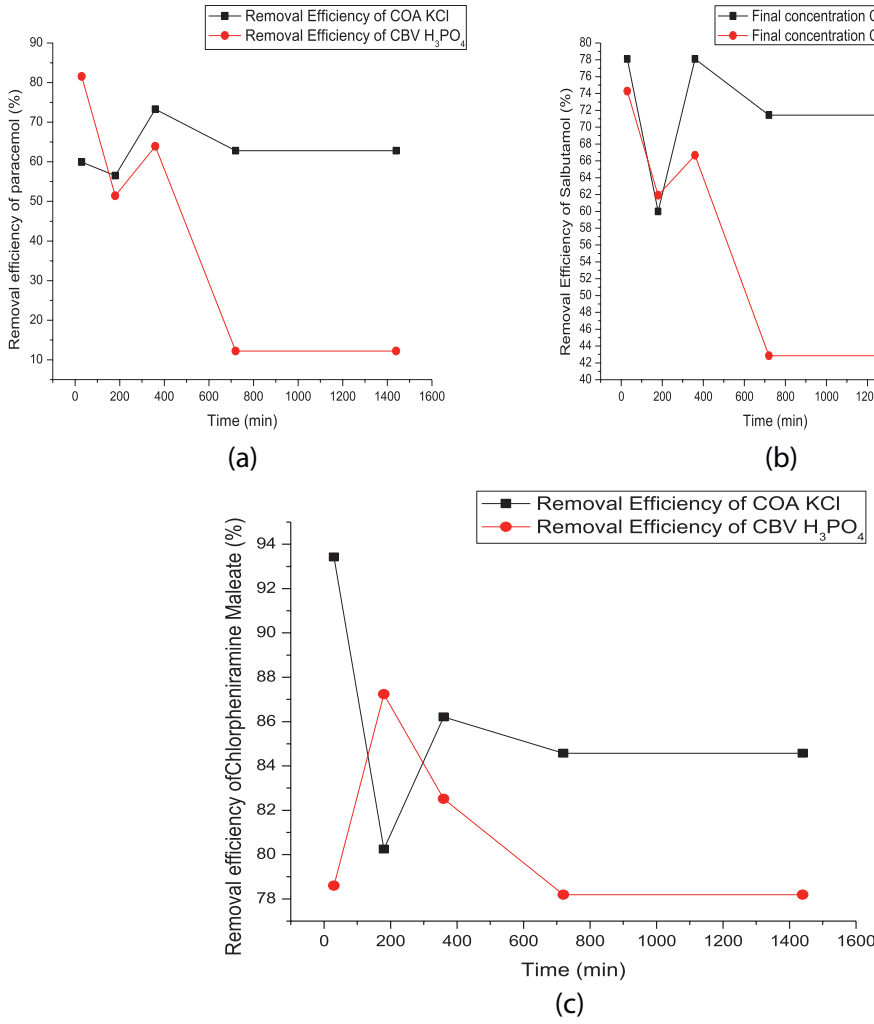


Figure 7.
(a-c). Effect of contact time on adsorption rate of PhACs by COA KCl and CBVH_3PO_4 at 30, 180, 360, 720, and 1440 mins.

contact time, with a high number of active binding sites on the adsorbent's surfaces. The adsorption rate is rapid in this stage and the points to adsorption are being controlled by diffusion processes of paracetamol, salbutamol, and chlorpheniramine molecules from the bulk phase to the adsorbent surface. The second stage of adsorption is an attachment-controlled process due to a decrease in the number of the active sites available for Paracetamol, salbutamol, and chlorpheniramine. Adsorptions graphs showing the rate and removal efficiency of PhACs by COA KCl and CBV H_3PO_4 at varying contact times of 30, 180, 360, 720, and 1440 min are shown below.

3. Conclusion

The ever-growing human population cannot do without water; hence clean water becomes a critical issue. Therefore, the various conventional ways of treating water have been established by existing researchers, most of which are said to be inadequate in the removal of organic contaminants. This study showed that adsorbents made from Oxytenanthera Abyssinia and Bambusa vulgaris can efficiently adsorb selected PhACs and PAHs in industrial Contaminated Water. *Oxytenanthera abyssinica* (COA 350°C KCl) had a Removal efficiency of (73.3%, 78.1%, and 86.2%) for PhACs while *B. vulgaris* (CBV 350°C H_3PO_4) had (63.9%, 66.7%, and 82.2%) in remediating Pharmaceutical actives contaminants such as paracetamol, salbutamol, and chlorpheniramine, respectively. For polycyclic aromatic hydrocarbons (PAHs) Removal efficiency of COA and CBV ranged from 42.5–81.2% and 8.9–65.5% respectively. The adsorption mechanism of trace organics followed the same pattern though with little differences. For all organic pollutants, adsorption rate is in two stages viz.: optimization and reduction followed by equilibrium. In comparison, COA showed the highest removal efficiency for PhACs and PAHs. The characterization of the adsorbent developed from agricultural materials was also revealed by Scanning Electron Microscopy (SEM) and point of zero charge (pH pzc)

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Conflict of interest

The author declares no conflict of interest

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