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Treatment of Oilfield Produced Water Using Biosorptive Methods

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Abstract

This study investigated activated carbon from Banana peel and Luffa cylindrica biowaste for treating produced water. Banana peel and Luffa cylindrica waste were pretreated with alkaline of 5% NaOH concentrations to obtain Samples A and B respectively. Activated carbon was obtained by carbonizing another portion of adsorbents, using an impregnation ratio of 1:3 of H_3PO_4 for 24 hrs to obtain Samples C and D. The experimental runs were conducted using the batch adsorption method, where PW was treated using 0.15, 1, 1.5, and 2 g adsorbent dosage for 2, 4, and 6 hours. The results of the study showed that the concentration of TDS in PW after treatment achieved 66.2 %,76 %, 62.5 %, and 59.5 % reduction for sample A, sample B, sample C, and sample D, respectively. The TSS concentrations were found to reduce in decreasing order, reducing from 26.7 % to 21.2 % for samples A to D. Also, there were general reductions in the heavy metals' concentrations in PW. Sample A led to 81.40% decrease in Zn, 68.8 % in Cu, 72.7 % in Ni, and 52.2 % in Fe. Similarly, sample B led to 70.9 %, 86.1 %, about 100 %, 4.6 % decreases in Zn, Cu, Ni and Fe, respectively. Sample C led to 85.7%, 88.29 %, 54.7 %, 52.18 % reductions in Zn, Cu, Ni, and Fe, respectively, while Sample D followed a similar trend with 89.1% decrease in Zn, 85.4% in Cu, 70.7 % in Ni, and 35.2% in Fe. It could be concluded that activated carbon adsorbents have better adsorption capacity than the modified adsorbent for the treatment of produced water in the reduction of TDS, TSS, and heavy metals.

Keywords: ***Activated carbon; Banana peel; Luffa cylindrica waste; Heavy metals; Produced water***

1. Introduction

Petroleum crude is produced with large volumes of wastewater generally referred to as produced water. The amount of produced water could be due to formation water. Formation water is seawater or fresh water that has been trapped for millions of years with oil and natural gas in a geologic reservoir consisting of a porous sedimentary rock formation between layers of impermeable rock within the earth's crust (Olajire, 2020). During oil and gas exploration, when a hydrocarbon reservoir is penetrated by a well, the produced fluids may contain this formation water, in addition to the oil, natural gas, and/or gas liquids. Extraction of the oil and gas leads to a reduction in reservoir pressure, and as a result of this, additional water is injected into the reservoir water zone to enhance recovery and maintain reservoir pressure (Igunnu and Chen, 2014). In addition to the injected water, water breakthrough occurs from outside the reservoir area, and as oil and gas production continues, formation water penetrates the production well and is produced alongside the hydrocarbon. This water is known as produced water (or oilfield brine) and makes up for the largest waste stream generated during oil and gas recovery operations. Produced water often is generated during the production of oil and gas from onshore and offshore wells.

The Oil & Gas industry is marked by significant water usage and the generation of large volumes of wastewater. In the upstream sector, this primarily stems from extraction processes, resulting in what is known as produced water. In the downstream sector, complex wastewater is generated during refining operations. In both sectors, the wastewater produced is not only chemically complex but also poses a high pollution risk, requiring advanced and specialized treatment technologies for effective management. (Jiménez et al. 2018).

Globally, oilfield formation water contains 3000 to 9000 mg/L chloride ions, and continuous discharge of this water into freshwater environment has contributed to damage to aquatic life and agricultural resources. In

Nigeria, produced water is one of the major pollutants of the aquatic environment in areas where oil exploration and production activities are carried out and has attracted much attention in the past (Obire and Amusan, 2003). As a result, efficient treatment and reuse of treated produced water is one of the main technological challenges with regard to the management of the effluent.

In order to find the most cost-effective and efficient method for the treatment of produced water for the removal of toxic heavy metals and contaminants, a lot of scientific research has been investigated. A large number of physicochemical methods earlier used are associated with many drawbacks; hence research on adsorption technology (Al-Kaabi et al. 2021*)*. Adsorption technology has been found to be fast, inexpensive, and universal compared to other technologies. The use of agricultural waste in the adsorption treatment of produced water has several advantages over conventional treatment methods, such as their low cost, regeneration ability, high adsorption efficiency, lower chemical or biological sludge, and the possibility of metals recovery (Burakov *et al.* 2018; Alalwan *et al.* 2018). These agricultural waste materials have shown promising adsorption removal efficiency for different pollutants in general and for heavy metals in particular from wastewater either in their natural form or after some physical or chemical modifications (De Gisi *et al*. 2016). The removal of heavy metals by adsorption using activated carbon is a prominent technology to treat domestic and industrial wastewater due to its easy operating requirements, high regeneration ability, and low cost (Jabbari, *et al.* 2016; Abbas and Alalwan 2019).

The aim of the reported study was to investigate the adsorption efficiency using formulated biosorptive materials for the removal of total dissolved solids, total suspended solids, and heavy metal ions from oilfield produced water.

2. Materials and Method

Produced water was obtained from an oilfield in the Niger Delta. Banana peels and *Luffa cylindrica* were collected from Ibadan, Oyo State, Nigeria. Reagents used were of high analytical grade.

2.1.Preparation of adsorbent

This study investigates the use of pretreated and activated carbon from Banana peel and *Luffa cylindrica* waste in order to extend the adsorbent shelf life and improve adsorption performance by improving the pore space and surface area of the adsorbent for the treatment of produced water and other contaminants.

2.2.Pre-treatment of adsorbents

Banana peels and *L. cylindrica* were thoroughly washed with distilled water to remove dirt. Then cut into pieces and air-dried for 7 days. Excess water was removed by oven-drying banana peel for 5 hours and *Luffa cylindrica* for 3 hours at 100°C. After which they were pulverized and were stored in airtight containers.

2.1.2 Modified bio-adsorbent

Alkali pre-treatment was achieved by immersing separately 20 g of the pulverized Banana peels and *L. cylindrica* in 50 mL of NaOH solution at room temperature for 24 hours. Then the resultant samples, now referred to as adsorbents, were thoroughly washed with distilled water, until pH 7 was attained in order to remove NaOH. The obtained adsorbent samples, modified *Luffa Cylindrica,* referred to as Sample A, and modified Banana peel as Sample B, were dried and stored in airtight containers.

Figure 1 (a) Alkali treatment of Luffa cylindrica. (b) Sample A (c) Alkali treatment of Banana peel (d.) Sample B

2.1.3 Bio-adsorbent activated carbon production

The activation of the pulverized adsorbents, *Luffa cylindrica* and Banana peel, was conducted based on the method explained by Akinsete *et al.* (2022). Briefly, the *Luffa cylindrica* and Banana peel were subjected to carbonization in a muffle furnace (Heraeus RO/ROF Tube Furnace) under a continuous nitrogen (N_2) atmosphere at 450°C for 1 hour and 0.5 hours. After carbonization, the resulting samples were allowed to cool to room temperature. Chemical activation of the carbonized biomass products was carried out through impregnation with phosphoric acid (H_3PO_4) at a mass ratio of 1:3, maintained for 24 hours. Upon completion of the impregnation period, the samples were filtered and repeatedly rinsed with distilled water to remove residual acid from the pore structure, ensuring the pH was neutralized. The acid-activated carbon (Sample C and Sample D) was then dried at 200°C for 2 hours and 5 hours.

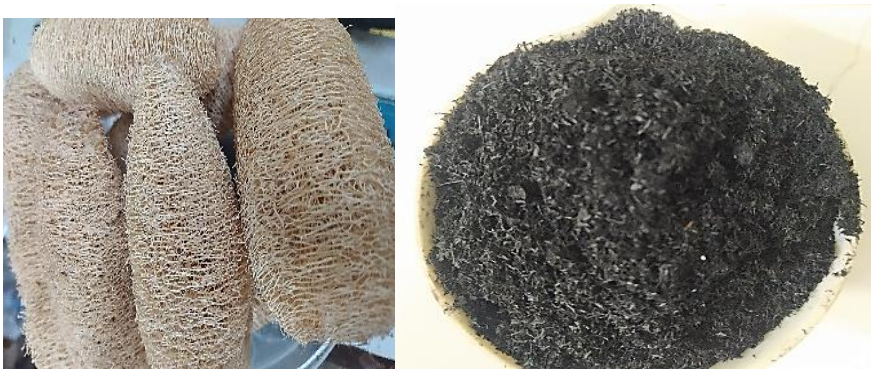

Figure 2 Processed Luffa cylindrica activated carbon

Figure 3 Processed banana peel activated carbon

2.1.4 Adsorbent characterization

The physicochemical properties of the activated carbon samples were assessed through a series of standard methods. Moisture content, ash content, and volatile matter were analysed using procedures described by Oladimeji *et al.* (2021).

The total and fixed carbon content were evaluated according to the method described by Isehunwa and Onovae (2011). Bulk density and surface area were determined following the approach outlined by Sugumaran *et al.* (2012).

2.2 Produced water treatment

To evaluate the effect of contact time on the treatment efficiency, 1 g of each adsorbent sample was added to 50 mL of distilled water in a bottle. The mixture was agitated using a mechanical shaker for varying contact times of 2, 4, and 6 hours. Then, after completion of the predefined contact time, the solutions were filtered. The filtrates were analysed for total dissolved solids (TDS), total suspended solids (TSS), and concentrations of heavy metals.

2.3 Determination of Total dissolved solids

The TDS was quantified by evaporating a measured volume of the water sample to dryness and weighing the remaining residue after cooling to room temperature. This process was repeated in triplicate, and the average value was recorded. The TDS was estimated using Equations (1) and (2) (Oladimeji *et al.* 2021).

$$TDS = [(Wt.\,of\ dish + residue - Wt.\,of\ dish) \times 1000) \quad (1)$$

$$TDS\left(\frac{mg}{L}\right) = \frac{TDS}{vol.\,of\ sample} \quad (2)$$

2.4 Determination of Total suspended solids

The TSS concentrations in produced water samples treated with 0.15 g of adsorbents were determined using the gravimetric method. The mass of suspended solids retained on a pre-weighed filter paper was used to calculate TSS according to Equations (3) and (4) (Oladimeji *et al.* 2021).

$$\text{TSS} = (\text{Wt. of filter paper} + \ \text{solids residue} - \ \text{Wt. of filter paper}) * 1000) \quad (3)$$

$$TSS = \frac{TSS}{Vol\ of\ sample} \quad (4)$$

2.5 Determination of Heavy Metals

The concentrations of heavy metals in both untreated and treated produced water samples were measured using an Atomic Absorption Spectrophotometer (AAS; PerkinElmer Analyst 200).

3. Results and discussion

3.1. Physicochemical properties of the adsorbents

The physicochemical properties of the adsorbents after chemical activation with phosphoric acid (H_3PO_4) are summarized in Table 1. The table shows total ash content of the activated carbons ranged from 2.8 to 3.23%, indicating that the majority of the raw biomass was composed of organic material, with only a small fraction remaining as inorganic residue after thermal treatment. Low ash content is desirable, as it typically reflects minimal mineral impurities and enhances the purity and quality of the carbon, which is crucial for efficient adsorption performance.

Moreover, the high carbon content observed in the activated samples signifies that the precursor biomass used in this study is suitable for conversion into bio-adsorbents. Carbon-rich materials tend to exhibit better structural

integrity, thermal stability, and greater surface reactivity, making them highly effective for capturing a wide range of contaminants in water treatment applications.

The measured bulk density of the chemically activated carbon samples ranged from 0.28 to 0.32 g/cm³. These values are consistent with those reported for phosphoric acid-impregnated carbonized plantain stem (0.32 g/cm³) (Ekpete *et al.* 2017), but slightly lower than the values (0.39–0.56 g/cm³) reported for phosphoric acid-activated carbon derived from spent coffee grounds (Ziezio *et al.,* 2020). Bulk density plays a vital role in practical adsorption systems, particularly for packed bed reactors or column designs, as it affects both adsorbent packing efficiency and mass transfer dynamics.

The specific surface area of the activated carbon derived from BP (banana peel) was 920.0 m²/g, while that obtained from *Luffa cylindrica* was 830.0 m²/g. These high surface areas reflect extensive pore development during chemical activation, which is essential for efficient adsorption. A higher surface area provides more active sites for contaminant binding and is indicative of a highly porous structure. Compared to literature values, the surface areas in this study surpass those reported by Ramutshatsha-Makhwedzha *et al.* (2022) and are comparable to those obtained by Ziezio *et al.* (2020) using similar activation techniques.

In general, adsorbents with large surface areas and well-developed pore structures exhibit superior performance in removing pollutants from aqueous solutions due to the increased availability of adsorption sites (Ziezio *et al.,* 2020; Udeagbara *et al.,* 2021). These findings further support the potential application of phosphoric acid-activated agricultural waste materials as cost-effective and sustainable adsorbents for water purification.

Table 1: Characteristics of produced adsorbent^

Parameter	Banana peel activated carbon	*Luffa cylindrica* activated carbon
Moisture Content (%)	8.4 ± 0.1	7.966 ± 0.152
Volatile Matter (%)	0.8 ± 0.1	0.6 ± 0.1
Total Carbon (%)	86.533 ± 0.28867	90.966 ± 0.305
Total Fixed Carbon (%)	74.6 ± 0.2	79.166 ± 0.152
Total Ash (%)	2.7666±0.1527	3.233 ± 0.251
Bulk Density (Packed) (g/cm^3)	0.32083	0.2842
Surface area (m^2/g)	920	830

^: Akinsete *et al*. (2022).

3.2. Effect of bio-adsorbent dosage on total dissolved solids

The effect of the dosage of adsorbent and the amount of total dissolved solids present in the untreated and treated produced water sample is presented in Figure 5. The figure generally showed up to a 76 % reduction in TDS concentration of produced water following treatment with bio-adsorbents. Sample A initial TDS concentration at 0g dosage was 44 mg/L, which decreased to 25.2 mg/L at 0.15g and further reduced to 14.9 mg/L with an increase in adsorbent dosage to 2g. This steady decrease suggests effective surface functional groups and porosity introduced through modifications of the adsorbents, which enhances adsorption of dissolved ions.

Sample B showed the maximum TDS reduction in treated PW. TDS reduced from 44 to 22.5 mg/L at 0.15g, then to 10.6 mg/L at 2g, accounting for ≈ 49 to 76 % TDS reduction in treated PW. This phenomenon could be mostly due to enhanced microporous surface area and high content of functional groups such as carboxyl and hydroxyl in banana peels (sample B), which promotes ionic interaction, making it highly efficient for TDS removal.

Similarly, as a result of the microporous surface area that improves TDS efficiency, sample C led to a decrease in TDS amounts in treated PW from 44 to 26.3 mg/L at 0.15g and up to 16.5 mg/L at 2g. This finding is in agreement with Udeagbara *et al*. (2023) who reported a 20% TDS reduction using untreated luffa cylindrical. It could therefore be concluded that chemical modification of adsorbent improves adsorbent adsorptive capacity.

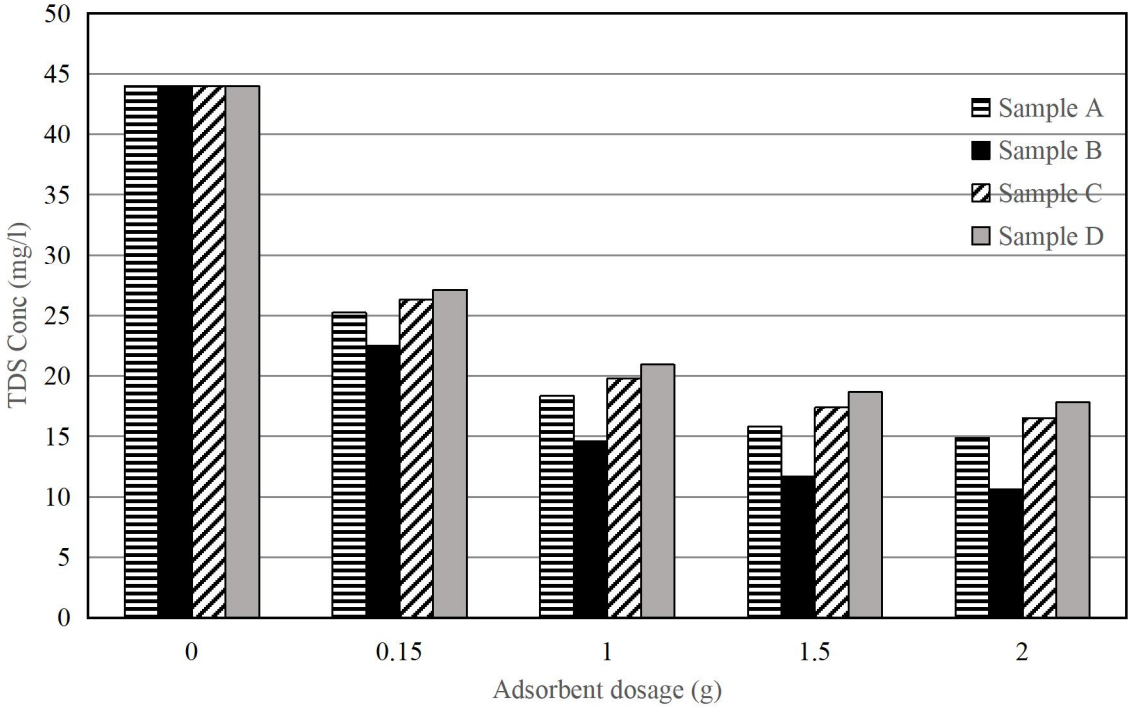

Figure 5 Effect of Adsorbent dosage on TDS

Sample A: Modified Luffa Cylindrica. *Sample B: Modified Banana peel*
Sample C: Luffa Cylindrica activated carbon. *Sample D: Banana peel activated carbon*

Sample D was found to follow a similar trend with samples A, B, and C, where up to 60 % TDS reduction in treated PW was observed. Although not as aggressive as Sample B. Sample D consistently outperforms sample A and C in overall TDS removal, likely due to greater porosity and enhanced chemical interaction.

This study has demonstrated that modified and carbonized adsorbent could be effective in the removal of total dissolved solids from produced water. Sample B shows the highest TDS removal efficiency (≈76 %), making it ideal for targeting dissolved contaminants. As the modified *Luffa cylindrica* (Sample A) activated carbon *Luffa cylindrica* (sample C), and activated carbon Banana (sample D) achieved 66.2 %, 62.5 % and ≈ 60 %, respectively. These results are within the range of previous reports reporting TDS removal from PW. For example, Ochi *et al*. (2022) achieved 60 %, 68 %, and 66% TDS removal when plantain peel activated carbon, chitosan and a blend of both were used to treat PW. Okologume & Olayiwola, (2019) reported 99.68% TDS removal efficiency from banana peel activated carbon.

3.3. Effect of bio-adsorbent dosage on total suspended solids

The data obtained for concentrations of total suspended solids (TSS) in PW after treatment of PW with samples A to D is shown in Figure 6. As shown in the figure, there was a reduction in TSS concentration, which is in agreement with previous reports (Ahmad *et al*. 2005; Okologume & Olayiwola, 2019; and Ochi *et al*. 2022) that bio-adsorbents are effective as adsorbents. Ahmad *et al*. (2005), Okologume & Olayiwola (2019), and Ochi *et al*. (2022) achieved 97%, 95.45%, and 80% TSS removal, respectively, following PW treatment. Modified *Luffa cylindrica* (sample A) had an initial TSS of 50 mg/L and decreased to 41.40 mg/L at 0.15g and to 36.7 mg/L with a further increase in adsorbent dosage of 2 g. Although removal is consistent, there was no

substantial decrease at higher dosages, suggesting site saturation or limited surface area for particulate adhesion. A similar trend was observed for samples B, C, and D. Sample C led to a TSS decrease from 50 mg/L to 42.92 mg/L at 0.15g and to 39.01 mg/L at 2g. The lower rate of decrease suggests that the activated form may favour ionic and molecular adsorption over larger suspended particulates.

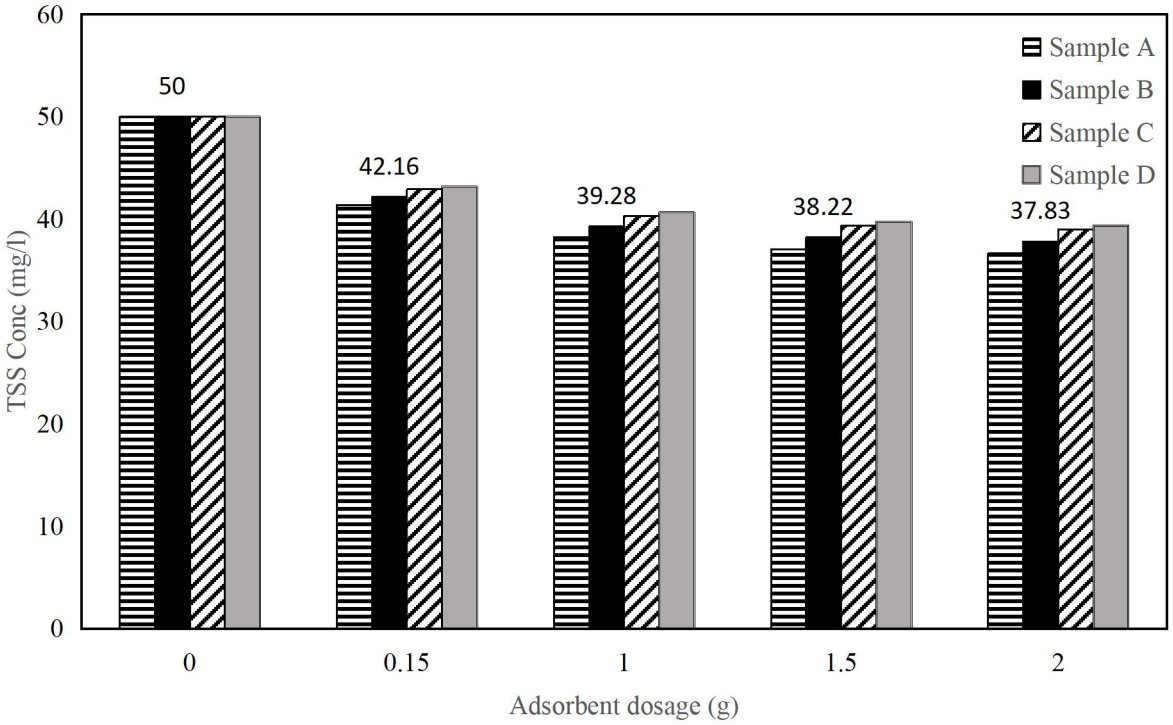

Figure 6 Effect of Adsorbent dosage on TSS

Sample A: Modified Luffa Cylindrica. *Sample B: Modified Banana peel*
Sample C: Luffa Cylindrica activated carbon. *Sample D: Banana peel activated carbon*

With the use of Sample B, TSS concentration reduced from 50 mg/L to 42.16 mg/L at 0.15g and to 37.83 mg/L at 2g while Sample D led to TSS concentration reduction from 50 mg/L to 43.17 mg/L at 0.15g and to 39.40 mg/L at 2 g. Moderate performance here indicates good particulate trapping and suspended solid reduction.

3.4.Effect of contact time on heavy metal removal

The concentrations of heavy metals obtained after treatment of PW with various samples at varying contact times are presented in Table 2. Zinc (Zn), Nickel (Ni), Iron (Fe), and Copper (Cu) were the metals studied. The data presented in Table 2 shows a substantial reduction in the concentrations of metal ions in treated PW when compared to initial amounts in untreated PW. Thus, the increase in adsorption can be attributed to the availability of free and highly unsaturated adsorbent sites at the beginning of the process (Ullah *et al*. 2023). The foregoing results also revealed that when PW was treated with activated *Luffa cylindrica*, the rate of metal ion reduction rose rapidly. Sample B excelled in Ni and Cu removal but was weak in Fe handling. Sample A was also effective for most metals, and Sample D underperformed for Zn and Fe but had strong Ni/Cu performance. Although the produced water used in this study contained higher concentrations of heavy metals than reported in recent studies from the Niger Delta area of Nigeria (Okologume & Olayiwola 2019; Udeagbara *et al*. 2021; Popoola *et al*. 2022; Gajendiran *et al*. 2025). These results showed that bio-adsorbent have great efficiency of reducing the concentration of heavy metal in produced water.

Furthermore, Table 3 shows that with an increase in contact time, the increase in the reduction in metal ion concentrations. For example, sample A led to an increase in Zn reduction from 27.1 % at 2 hr to 81.4 %. Also,

sample B achieved a 37.2 to 70 % reduction in Zn with an increase in contact time from 2 to 6hr. The % removal of heavy metals from treated PW at 6 hr is presented in Table 4. It was found that the decrease was more pronounced with the activated carbon bio-adsorbent, except for Nickel treated with a modified absorbent sample that achieved a 100% removal.

Table 2: Concentration of heavy metals in produced water after treatment

Adsorbent	**Time (hours)**	**Metal concentration (mg/l)**			
		Zn	Cu	Ni	Fe
Untreated PW	-	0.258	0.144	0.300	3.600
Sample A (Modified *Luffa cylindrica*)	2	0.188	0.061	0.108	3.289
	4	0.165	0.058	0.083	2.76
	6	0.048	0.045	0.082	1.72
Sample B (Modified Banana peel)	2	0.221	0.031	0.125	3.431
	4	0.162	0.021	0.00	3.443
	6	0.075	0.020	0.00	3.434
Sample C (*Luffa cylindrica a*ctivated carbon)	2	0.201	0.093	0.289	3.442
	4	0.199	0.090	0.238	3.369
	6	0.154	0.049	0.211	3.052
Sample D (Banana Peel activated carbon)	2	0.257	0.140	0.266	3.340
	4	0.171	0.113	0.252	3.336
	6	0.168	0.056	0.152	3.330

Table 3 Percentage Performance of adsorbent on heavy metal removal after 6 hours

Adsorbent	**Zn Removal (%)**	**Cu Removal (%)**	**Ni Removal (%)**	**Fe Removal (%)**
Sample A	81.4	68.8	72.7	52.2
Sample B	70.9	86.1	100	4.6
Sample C	85.7	88.2	54.7	52.1
Sample D	8.9	85.4	70.7	2.17

This study has shown that the adsorbent was very effective in the adsorption of heavy metals in produced water. Also, the activated carbon adsorbent was more effective than the modified adsorbent, and with an increase in the contact time there would be high removal efficiency of heavy metals. Future studies could investigate blends of adsorbents or chemical modifications to enhance adsorption capacity.

Conclusion

This study demonstrated that bio-adsorbents, owing to their enhanced surface area and porosity, are effective in removing both heavy metals and other contaminants from produced water. Adsorption experiments indicated that contact time plays a critical role in the efficiency of metal ion uptake. Furthermore, Luffa cylindrica—

whether chemically modified or carbonized—exhibited a greater affinity for heavy metal adsorption compared to banana peel. Among the materials tested, Sample B was particularly effective in the removal of dissolved pollutants, whereas Sample A showed superior performance in eliminating suspended solids. Activated carbon-based adsorbents (Samples C and D) delivered more consistent performance across both total dissolved solids (TDS) and total suspended solids (TSS), making them suitable for comprehensive water treatment applications.

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