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Effect of Contact Time on the Sorption of Metal Ions Associated with Produced Water on Pulverized Oyster Shell (OS) as Adsorbent

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Authors' contributions

This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.

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ABSTRACT

The aim of this research is to investigate Metal ion sorption potentials of pulverized Oyster Shell as adsorbent in produced water from selected flow stations in the Niger Delta region of Nigeria. Produced water samples were collected from six (6) different oil drilling installations (CC7T, CC8T, WELL 2 GSS, WELL 8 TEB, AZUZUAMA ST 1, AZUZUAMA ST 2) around the Niger Delta area of Nigeria. The waste oyster shell was collected in Akpan-Andem Market in Uyo, Akwa-Ibom State and processed using well established protocols. Atomic Absorption Spectrometer (AAS) was used to determine selected metals' Nickel (Ni), Cobalt (Co), Copper (Cu), Iron (Fe), Lead (Pb), Calcium (Ca) and Potassium (K) concentrations. The obtained values for Nickel ranged from 0.076mg/l at

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AZUZU-AMA ST1 flow station to 2.882 mg/l at WELL2-GSS flow station with average value of 0.745 mg/l. The obtained value for Cobalt ranged from <0.001 mg/l at CC-8T and CC-7T to 0.277 mg/l at AZUZUAMA ST1. Values obtained for Cu, Fe, Pb, Ca, and K ranged between: 0.195 mg/l at WELL2-GSS to 0.449 mg/l at AZUZU-AMA ST1; 2.785 mg/l to 89.279mg/l; <0.001 mg/l to 0.483 mg/l; 16.217 mg/l to 92.714 mg/l and 96.386 mg/l to 105.416 mg/l respectively. Generally, the amount of metal ions absorbed/adsorbed onto pulverized Oyster shell (OS) increased swiftly within 2 hours and slowly with further increase of contact time and then tended to be stable. For pulverized Oyster shell adsorbent, the adsorption/absorption capacity decreases with the increasing contact time. The adsorbent (OS) isn't good material for removal of Calcium and Cobalt in produced water but was very effective adsorbent for Ni, Fe, and Pb removal in produced water and less effective for Cu and K removal from produced water. The study underscores the need to further probe outcome of above research at different pH and other relevant variables as well as compares its efficiency with other detoxification technologies like Detoxification by chemical reaction (DCR) technology.

Keywords: Pulverized oyster shell; contact time; adsorption; produced water; DCR technology.

1. INTRODUCTION

Produced water is the largest waste stream in the oil and gas industry, containing relatively high concentration of highly volatile compounds (e.g., benzene, toluene, ethyl benzene, and xylenes: BTEX), and other compounds (e.g., phenols and organic acids), polycyclic aromatic hydrocarbons (PAHs), as well as metals and other pollutants, inorganic compounds causing hardness. All these have significant negative impacts on the economy and environment [1]. Globally, oilfield produced water (OPW) production is expected to increase due to increased demand for crude oil, coupled with increased global production activity. Current forecast shows a two-fold increase of produced water volume globally over the next 10 years from about 158,900 million barrels per day (mb/d) to 243,000 million barrels per day (mb/d). (Produced Water Society, 2020). This implies that very large volumes of produced water will be disposed in future, stressing the need to comply with treatment regulations; as environmental literature have also showed great concern with possible detrimental effect of continued produced water discharged in offshore and onshore areas [2]. The composition of produced water is inconsistent and, its physiochemical properties are dependent on factors such as the geographic location of the field, the type of hydrocarbon produced. Concerns about produced water discharges as a possible environmental danger has grown [2]. The wide variety of produced water treatment methods have been reported previously [3].

Water pollution is a worldwide problem and needs to be controlled [4]. The main sources of heavy metals in wastewater are natural and man-

made activities [5, 6]. The literature contains several notable reviews on the removal of heavy metals from polluted waters describing conventional methods for removing heavy metals from wastewater, including chemical oxidation, ion-exchange. precipitation. adsorption. membrane filtration, coagulation-flocculation, flotation, and electro-chemical methods [7,8]. Alternative approaches to modern technologies will often focus on using waste materials to remove heavy metals or readily available materials such as clay soils via ion-exchange [9]. Reviewed several methods available for removing heavy metals noting that among the chemical conventional methods available. precipitation a commonly used conventional method, is only effective in high concentration metal ion solutions.

Biosorption is another method that exploits the benefits of low cost and environmentally friendly alternatives such as fungi and poplar trees etc., to provide sustainable methods for the removal of heavy metals in storm water. Chitin and its deacetylated form chitosan, is an example of a biosorption material for water purification, and can be extracted from the shells of shrimps, lobsters and crabs, or supporting mineral deposits. In biosorption research, both natural and modified absorbents are investigated [10]. Reviewed all the economical absorbents available in recent years based on the source of the absorbent. They noted that nano-sized, zerovalent particles and minerals, such as magnetite, laterite, and cement kiln dust, etc., show great efficiency in removing arsenic. Biosorption using calcium carbonate from seafood waste, such as oyster, and crab shells, is also gaining popularity.

Oyster shells are a type of mollusk shell in which over 90% of the shell's mass is calcium carbonate with organic matrices occupying less than 5% of the shell. Calcium carbonate has three crystal forms: calcite, aragonite, and vaterite. Calcite is the most stable form, followed by aragonite, then vaterite. Aragonite is the most common mineral in a mollusk shell, followed by calcite, which are crystal forms of calcium carbonate. The mechanism by which the oyster shell (CaCO₃ micro-particles) absorbs metal ions is through ion exchange in three steps: (i) absorption of metal ions on the porous surface area (involving dissolution of partial calcium carbonate because of higher solubility compared to most of the metal carbonates, releasing Ca2+ and CO_3^{2-} ; (ii) precipitation of metal ions on the surface; and (iii) the formation of heavy metal complex nucleation and crystals on the surface. To date, mollusk shells have been used in wastewater treatment for many purposes, such as purifying wastewater by trapping particulates (by forming a filter bed with shell powder), nutrient reduction; adjusting pH to provide an alkaline environment for specific reactions; or ion substitution for removing heavy metal ions. The mechanism of using mollusk shells for water treatment is mainly by using calcium carbonate for heavy metal sedimentation while releasing calcium into the water at the same time. The original hypothesis dates from earlv studies on the strong adsorption ability of metal on calcite, a calcareous geologic ions counterpart. Most research to date use shell powder with specific particle sizes. Most studies have proven that shell powder works well in solutions with hiah concentrations of contaminants.

Adsorption has proven to be economical and Adsorption of heavy metals on conventional adsorbents such as activated carbon is one of the methods that have been widely studied. However, activated carbon has a cost implication that is not sustainable [11]. Chitosan (CS) is a natural adsorptive polymer that has an affinity toward pollutants in wastewaters because it has amino (-NH₂) and hydroxyl (-OH) groups [12]. Despite its unique features, it suffers from low mechanical strength and poor stability, making the regeneration inefficient. Also, it is challenging to use CS in its powder or flake form because of its low porosity, low surface area, resistance to mass transfer, and high crystallinity [13]. Using natural minerals adsorbents such as zeolite, silica, and clay are considered good candidates for water purification with low operating costs [14]. However, the removal efficiency might decrease after a few cycles making their modification with its cost implication inevitable.

Shell powders, derived from sources like oyster shells, and egg shells, possess unique characteristics that make them effective adsorbents for heavy metal ions. These characteristics include high surface area, porous structures, and the presence of functional groups, such as hydroxyl and carboxyl groups, which play a crucial role in metal ion binding.

Zhang [15] My research team in 2021 had used Oyster and snail shells differently to remediate heavy metal and crude oil contaminated soils in the Niger Delta and the results are promising from the data obtained from Oyster shells [16,17].

The aim of this research is geared towards investigating the absorbent/adsorbent potentials of pulverized Oyster shells on metal ions in produced water obtained at different crude oil wells in the Niger Delta, at varying contact times. The outcome will be informative enough to policy makers and Environmentalists interested in conversion of abundant environmental waste material in the Niger Delta into huge and cost effective resource feedstock for detoxifying and decontaminating produced water with respect toxic metals that is released in abundance to the environment as a result of crude oil exploration.

2. MATERIALS AND METHODS

2.1 Sample Collection

Produced water samples were collected from six (6) different oil drilling installations (CC7T, CC8T, WELL 2 GSS, WELL 8 TEB, AZUZUAMA ST 1, AZUZUAMA ST 2) around the Niger Delta area of Nigeria. The samples were collected and stored in a clean glass container, which was furthermore put into a cooler to regulate the temperature, as it was transported for analysis in an analytical laboratory. The waste oyster shell was collected in Akpan-Andem Market in Uyo, Akwa-Ibom State, Nigeria and washed in distilled water to remove residual sediments and musculature, and then dried in air.

2.2 Method of Analysis

Pre-analysis preparation and sample analysis procedure: The produced water was separated using a separating to remove all crude oil in the sample before analysis. Oyster shells were air dried and ground to powdery form using the crusher. The oyster shell powder was then sieved to a mesh size of 1.18 mm . The sieved oyster shell powder (OSP) was then stored in a plastic container for further use. A separating funnel was mounted on a retort stand and 200ml of the produced water was poured into the separating funnel. 20ml Of hexane (an organic solvent) was poured into the separating funnel and the funnel was corked and shook vigorously while intermittently opening the lower end of the funnel to allow removal of air bubbles. The produced water mixture was shook and hexane was returned to the retort stand allowing it to settle for two (2) minutes, after which water was removed from the separating funnel leaving behind the hexane. A column was created using layers of glass wool, sodium sulphate anhydrous and silica gel and the separated mixture was then passed through the column into a clean glass beaker.

2.3 Analysis of Heavy Metals in Produced Water

About 100 cm³ of a well mixed water samples was poured into a 150 cm³ beaker. 5cm³ of conc. HNO₃ was added. The solution was later evaporated to near dryness on hot plate, making sure that the sample does not boil by using low to medium heat. The beaker and contents was allowed to cool to room temperature before adding another 5 cm³ conc. HNO₃ into the beaker. The beaker immediately covered afterwards with a watch glass. The beaker was returned to the hot plate and set a gently reflux action of the solution by increasing the temperature of the hot plate (medium to high heat). We continued heating with addition of conc. HNO₃ as necessary until light-coloured residue is obtained (i.e digestion is completed). Add 1-2 cm³ conc. HNO₃ to the residue. Wash with distilled water. The solution was later filtered into 100 cm³ volumetric flask to remove silicate and other insoluble materials and made up to the mark with distilled water in the volumetric flask. Store the solution in 125 cm³ polypropylene bottle.

2.4 Analysis for Adsorption using Oyster Shell Powder (OSP)

A setup was put in place where the separating funnel was mounted on a retort stand and below it, glass column chamber was also mounted to the stand, while a clean glass beaker was placed below the column chamber. The column chamber was filled with the oyster shell powder and then the separating funnel was opened and the produced water was allowed to run through the chamber filled with oyster shell powder into a clean glass beaker below, where the filtrate was collected. This procedure was carried out for all the produced water sample, and was repeated, varying the time (hours) taken to pass through the oyster shell chamber. The concentrations of each of the metal was noted with AAS before and after injection in the column for the differences in concentrations of the respective metal in the produced water.

3. RESULTS AND DISCUSSION

3.1 Initial Concentration of Heavy Metals (mg/l) in Produced Water

Results obtained from initial concentration of metals in produced water from the selected sampling stations is along with effluent limitations for metals for Inland/Near shore oil and gas installations for oily waste water [18] is presented in Table 1. Metal composition in mg/L results from AAS analysis show that the produced water samples contain Nickel, Cobalt, Copper, Iron, Lead, Calcium and Potassium at variable concentrations. As shown, some of are present at metals these elevated concentrations in a particular produced water sample. The obtained values for Nickel ranged from 0.076mg/l at AZUZU-AMA ST1 flow station to 2.882 mg/l at WELL2-GSS flow station with average value of 0.745 mg/l but which was within the maximum limit recommended level set by EGASPIN (1.00 mg/L). The obtained value for Cobalt ranged from 0.001 mg/l at CC-7T to 0.277 mg/I at AZUZU-AMA ST1. Values obtained for Cu, Fe, Pb, Ca, and K ranged from: 0.195 mg/l at WELL2-GSS to 0.449 mg/l at AZUZU-AMA ST1; 89.279 mg/l to 2.785 mg/l; 0.001 mg/l to 0.483 mg/l; 16.217 mg/l to 92.714 mg/l and 96.386 mg/l to 105.416 mg/l respectively. Some of the well locations recorded metal concentrations above the maximum limit. Example WELL 2 location, the obtained metal values in produced water comparatively higher than the tabulated heavy metal limits (1.00mg/l) for Nickel and Fe (1.00mgL) in drinking water and effluent limitations for Inland/Near shore oil and gas installations for oily waste water by the EGASPIN [18] (Environmental Guideline and Standard for Petroleum Industries in Nigeria).

Parameters				Sample			
	CC7T	CC8T	WELL 2 GSS	WELL 8 TEB	AZUZUAMA ST 1	AZUZUAMA ST 2	EGASPIN (2018)
Nickel (mg/L)	0.116	0.708	2.882	0.254	0.076	0.438	1.00
Cobalt (mg/L)	<0.001	<0.001	0.026	0.049	0.277	<0.001	5.00
Copper (mg/L)	0.348	0.549	0.195	0.274	0.449	0.392	1.00
Iron (mg/L)	7.194	52.692	89.729	2.785	24.578	5.372	1.00
Lead (mg/L)	0.277	0.483	<0.001	<0.001	0.213	0.013	0.05
Calcium (mg/L)	71.751	66.176	16.217	92.714	61.654	82.616	75
Potassium (mg/L)	98.970	100.444	96.386	100.389	103.965	105.416	NA

Table 1. Initial Heavy Metal Analysis of Produced Water Samples

Guideline limits for produced water discharge (EGASPIN 2018) [18]

3.2 Effect of Contact Time on Adsorption of Metals Assayed

The effect of contact time on absorption/adsorption potentials of pulverized Oyster shell is shown from Tables 2. to 8. Adsorption experiments with various contact times (2hrs and 4hrs) were investigated after an initial metal concentration. Results obtained showed that the amount of metal ions adsorbed onto OS (Oyster shell) surface increased swiftly with the increase of contact time and then tended to be stable. The metal ion adsorption process was almost immediate and the adsorption rate was higher at the start of the experiment than later in the entire experiment. This was due to the sufficient number of available sites on the OS surface at the beginning of the adsorption process and the interactions of the notable functional groups on the OS adsorbent with the metal ions in solution. As the adsorption sites

were occupied, the adsorption rate of metal ions decreased until saturation. Generally Ni, Pb and Fe sorption by OS reached equilibrium before 4hrs (240 minutes), the maximum removal was obtained at the equilibration time of 60 minutes (1hr), and was then stabilized.

3.3 Comparison of Initial Metal Concentrations in Produced Water Samples at Different Time Intervals after Adsorption/Absorption

The data reveal the initial and concentrations of various metals Nickel, Cobalt, Copper, Iron, Lead, Calcium, and Potassium in produced water samples from different locations after Adsorption/Absorption. The concentrations after Adsorptions/Absorptions were measured at two distinct times: after 2 hours and after 4 hours.

Table 2. Comparison of Initial Nickel Concentrations (mg/L) in Produced Water Samples at Different Time Intervals after Adsorption/Absorption

Sample	Initial	After (2hrs)	After (4hrs)	
CC7T	0.116	0.076	<0.001	
CC8T	0.708	0.321	<0.001	
WELL 2 GSS	2.882	1.241	0.312	
WELL 8 TEB	0.254	0.038	<0.001	
AZUZUAMA ST 1	0.076	<0.001	<0.001	
AZUZUAMA ST 2	0.438	0.029	<0.001	

Table 3. Comparison of Initial Cobalt Concentrations (mg/L) in Produced Water Samples at Different Time Intervals after Adsorption/Absorption

Sample	Initial	After (2hrs)	After (4hrs)
CC7T	<0.001	0.412	0.110
CC8T	<0.001	0.013	0.241
WELL 2 GSS	0.026	0.319	0.259
WELL 8 TEB	0.049	<0.001	0.273
AZUZUAMA ST 1	0.277	0.315	1.131
AZUZUAMA ST 2	<0.001	<0.001	0.173

Table 4. Comparison of Initial Copper Concentrations (mg/L) in Produced Water Samples at Different Time Intervals after Adsorption/Absorption

Sample	Initial	After (2hrs)	After (4hrs)
CC7T	0.348	0.316	0.301
CC8T	0.549	0.495	0.543
WELL 2 GSS	0.195	0.110	0.130
WELL 8 TEB	0.274	0.139	0.249
AZUZUAMA ST 1	0.449	0.218	0.392
AZUZUAMA ST 2	0.392	0.310	0.267

Sample	Initial	After (2hrs)	After (4hrs)	
CC7T	7.194	<0.001	<0.001	
CC8T	52.692	32.51	<0.001	
WELL 2 GSS	89.729	65.11	0.092	
WELL 8 TEB	2.785	<0.001	<0.001	
AZUZUAMA ST 1	24.578	9.271	<0.001	
AZUZUAMA ST 2	5.372	<0.001	<0.001	

Table 5. Comparison of Initial Iron Concentrations (mg/L) in Produced Water Samples at Different Time Intervals after Adsorption/Absorption

Table 6. Comparison of Initial Lead Concentrations (mg/L) in Produced Water Samples at Different Time Intervals after Adsorption/Absorption

Sample	Initial	After (2hrs)	After (4hrs)
CC7T	0.277	<0.001	<0.001
CC8T	0.483	<0.001	<0.001
WELL 2 GSS	<0.001	<0.001	<0.001
WELL 8 TEB	<0.001	<0.001	<0.001
AZUZUAMA ST 1	0.213	<0.001	<0.001
AZUZUAMA ST 2	0.013	<0.001	<0.001

Table 7. Comparison of Initial Calcium Concentrations (mg/L) in Produced Water Samples at Different Time Intervals after Adsorption/Absorption

Sample	Initial	After (2hrs)	After (4hrs)
CC7T	71.751	73.427	71.894
CC8T	66.176	68.524	70.200
WELL 2 GSS	16.217	17.114	20.154
WELL 8 TEB	92.714	96.381	96.817
AZUZUAMA ST 1	61.654	64.001	69.135
AZUZUAMA ST 2	82.616	85.173	88.101

Table 8. Comparison of Initial Potassium Concentrations (mg/L) in Produced Water Samples at Different Time Intervals after Adsorption/Absorption

Sample	Initial	After (2hrs)	After (4hrs)
CC7T	98.970	103.420	94.528
CC8T	100.444	116.130	99.872
WELL 2 GSS	96.386	100.404	92.386
WELL 8 TEB	100.389	105.217	93.971
AZUZUAMA ST 1	103.965	109.724	100.01
AZUZUAMA ST 2	105.416	112.426	100.92

For Nickel, the initial concentrations ranged from 0.076 mg/L in AZUZUAMA ST 1 to 2.882 mg/L in WELL 2 GSS as shown in Table 2. The concentrations of Nickel ions in the produced water after 2 hours showed significant reductions, dropping below 0.001 mg/L in the produced water samples from AZUZUAMA ST 1. The same trend continued after 4 hours, the sorption efficiency of the adsorbent increased for Nickel ions in all the samples and the concentration of Nickel ions in the produced water were below 0.001mg/kg for all samples

from all sites excerpt in WELL2 GSS were its concentration is 0.312 mg/L.

Cobalt concentrations were mostly below the detection limit initially as shown in Table 3 except for WELL 2 GSS, WELL 8 TEB and AZUZUAMA ST 1. After 2 hours, adsorption led to noticeable reductions in WELL 8 TEB only and remained undetected in AZUZUAMA 2 and decreased in other samples. These negatives in concentrations after adsorption suggest possible leaching from the adsorbent and these trends

continued at 4 hours across all sites indicating that the adsorbent isn't good material for removal of Cobalt in produced water.

For Copper, initial concentrations ranged from 0.195 mg/L in WELL 2 GSS to 0.549 mg/L in CC8T as shown in Table 4. After Adsorption at 2 hours resulted in moderate reductions across all samples. By 4 hours, the adsorption effectiveness generally increased slightly.

Iron concentrations were initially very high in some samples, especially WELL 2 GSS and CC8T as shown in Table 5. After 2 hours, there were significant reductions in most samples, with some showing complete removal, such as CC7T and WELL 8 TEB. After adsorption at 4 hours it continued to show further reductions, particularly in WELL 2 GSS.

Lead was initially low or below the detection limit in most samples as shown in Table 6, except for a few like CC8T. After 2 hours, adsorption effectively reduced the lead concentrations in the produced water to below the detection limit in all samples, a trend that remained consistent at 4 hours.

Calcium concentrations were, ranging from 16.217 mg/L in WELL 2 GSS to 92.714 mg/L in WELL 8 TEB as shown in Table 7. Interestingly, after adsorption its concentrations at 2 hours showed slight increases, suggesting possible desorption or measurement variability. This trend continued at 4 hours, with slight increases in concentration. These increases in concentrations after adsorption suggest possible leaching from the adsorbent and these trends continued at 4 hours across all sites indicating that the adsorbent isn't good material for removal of Calcium in produced water.

Potassium had initial concentrations, around 100 mg/L. After 2 hours as shown in Table 8, there was an increase in concentration across all samples, which could indicate either leaching from the adsorption medium or experimental variability. At 4 hours, the concentrations slightly decreased across all samples compared to its corresponding value after 2 hours but remained slightly lower than the initial concentrations.

4. CONCLUSION

Oyster shell was evaluated for absorption/adsorption of metal ions from

produced water. This study showed that produced water samples from the study areas contained traces of metals. The outcome of this study has established that untreated produced water contains varying quantities of chemicals and metals, which are toxic to human and other life-forms. This was an evidence of a build-up of toxic metals in the produced waste water in the study areas. The essence of the study is to make contribution to efforts at sensitizing the various stakeholders concerning this challenge and the prescribed solutions using ecofriendly and cost effective remediation methods. Of the metals studied, the mean concentration of Ni and Fe were relatively higher than [18] regulatory limits in samples analyzed. Generally, there is an indication that the water quality has been extremely compromised. The adsorption process is a function of the pH, contact time and other variables. These results can be used as a good indicator for the design of produced water or runoff treatment systems for the elimination of soluble and particle-bound metals. For pulverized Oyster shell adsorbent, the adsorption/absorption capacity decreases with the increasing contact time. These negatives in some sorptions suggest possibly that the adsorbent (OS) isn't good material for removal of Calcium and Cobalt in produced water but was effective adsorbent for Ni, Pb and Fe, but was less effective as adsorbent for Cu and K with time.

Currently, there are set limits for concentrations of metals in produced water and other effluents before they are discharged into the environment. These limits, set by various regulatory agencies and others interested in environmental protection, are expected to be adhered to by the industrial outfits concerned. Nonetheless, there are documented reports that some of these sometimes do not outfits ensure strict compliance to these limits. This claim is however subject to scientific monitoring and verifications. Ensuring compliance to these limits can only be achieved if the mandatory sampling, analysis and monitoring of effluents, such as produced water, is carried out regularly to determine the prevalent physical and chemical parameters before and after treatment. The results of this study show that the OS is an effective and low-cost adsorbent for the removal of Ni, Pb and Fe ions in aqueous solution. Use of low cost adsorbents such as OS can be considered as a viable option for the treatment of produced water pollution from metals. The present study demonstrates that it is possible to carry out an efficient and economic treatment of produced water by using OS as adsorbent to remove Lead, Iron and Nickel.

DISCLAIMER (ARTIFICIAL INTELLIGENCE)

Author(s) hereby declare that NO generative AI technologies such as Large Language Models (ChatGPT, COPILOT, etc.) and text-to-image generators have been used during the writing or editing of this manuscript.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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