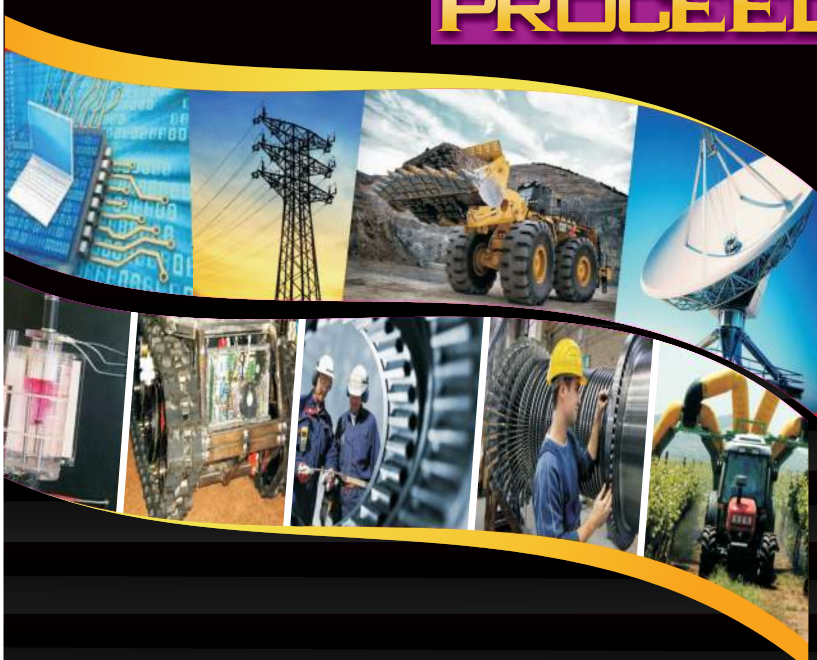
FEDERAL UNIVERSITY OF TECHNOLOGY, MINNA

SCHOOL OF ELECTRICAL ENGINEERING AND TECHNOLOGY &
SCHOOL OF INFRASTRUCTURE, PROCESS ENGINEERING AND TECHNOLOGY

3rd INTERNATIONAL ENGINEERING CONFERENCE IEC 2019

THEME THE ROLE OF ENGINEERING AND TECHNOLOGY IN SUSTAINABLE DEVELOPMENT

BOOK of PROCEEDINGS



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EDITED BY

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FORWARD

The School of Engineering and Engineering Technology, Federal University of Technology, Minna, organized the 1st and 2nd International Engineering Conference in 2015 and 2017 respectively. With the emergence of the new School of Electrical Engineering and Technology and the School of Infrastructure, Process Engineering and Technology, the two schools came together to organize this 3rd International Engineering Conference (IEC 2019) with the theme: "The Role of Engineering and Technology in Sustainable Development" considering the remarkable attendance and successes recorded at the previous conferences. The conference is aimed at offering opportunities for researchers, engineers, captains of industries, scientists, academics, security personnel and others who are interested in sustainable solutions to socio-economic challenges in developing countries; to participate and brainstorm on ideas and come out with a communiqué, that will give the way forward. In this regard, the following sub-themes were carefully selected to guide the authors' submissions to come up with this communiqué.

1. Engineering Entrepreneurship for Rapid Economic Growth.
2. Regulation, Standardization and Quality Assurance in Engineering Education and Practice for Sustainable Development.
3. Solutions to the Challenges in Emerging Renewable Energy Technologies for Sustainable Development.
4. Electrical Power System and Electronic as a Panacea for Rapid Sustainable Development
5. Promoting Green Engineering in Information and Communication Technology
6. Reducing Carbon Emission with Green and Sustainable Built Environment
7. Artificial Intelligence and Robotics as a Panacea for Rapid Sustainable Development in Biomedical Engineering
8. Petrochemicals, Petroleum Refining and Biochemical Technology for Sustainable Economic Development.
9. Advances and Emerging Applications in Embedded Computing.
10. Traditional and Additive Manufacturing for Sustainable Industrial Development.
11. Emerging and Smart Materials for Sustainable Development.
12. Big Data Analytics and Opportunity for Development.
13. Building Information Modeling (BIM) for Sustainable Development in Engineering Infrastructure and Highway Engineering.
14. Autonomous Systems for Agricultural and Bioresources Technology.

The conference editorial and Technical Board have members from the United Kingdom, Saudi Arabia, South Africa, Malaysia, Australia and Nigeria. The conference received submissions from 4 countries namely: Malaysia, South Africa, the Gambia and Nigeria. It is with great joy to mention that 123 papers were received in total, with 0.9 acceptable rate as a result of the high quality of articles received. Each of the paper was reviewed by two personalities who have in-depth knowledge of the subject discussed on the paper. At the end of the review process, the accepted papers were recommended for presentation and publication in the conference proceedings. The conference proceedings will be indexed in Scopus.

On behalf of the conference organizing committee, we would like to seize this opportunity to thank you all for participating in the conference. To our dedicated reviewers, we sincerely appreciate you for finding time to do a thorough review. Thank you all and we hope to see you in the 4th International Engineering Conference (IEC 2021).

Engr. Dr. S. M. Dauda
Chairman, Conference Organizing Committee

ACKNOWLEGEMENT

The Chairman and members of the Conference Organizing Committee (COC) of the 3rd International Engineering Conference (IEC 2019) wish to express our gratitude to the Vice Chancellor and the management of the Federal University of Technology, Minna, the Deans and all staff of the School of Electrical Engineering and Technology (SEET) and the School of Infrastructure, Process Engineering and Technology (SIPET) for the support towards the successful hosting of this conference. We also thank the entire staff of the university who contributed in one way or the other. We are sincerely grateful to you all.

OPTIMISATION STUDY ON THE REMOVAL Pb(II), Cd(II) and Ni(II) FROM PHARMACEUTICAL WASTEWATER USING CARBONIZED AFRICAsN GIANT SNAIL SHELL (Archachatina marginata) AS AN ADSORBENT.

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ABSTRACT

Rapid expansion of the pharmaceutical industry resulting to increased wastewater disposal containing heavy metals calls for concern. Therefore, carbonised Archachatina marginata was used in order to understand how better the Pb(ii), Cd(ii) and Ni(ii) ion in pharmaceutical wastewater can be efficiently adsorbed. A Response Surface Method (RSM) Central Composite Design (CCD) was used to study the adsorption efficiencies of these heavy metals using DESIGN EXPERT Version 7.0.0 software. This software was used for the model fitting and also to evaluate the statistical significances of models. Batch adsorption studies was then carried out at optimum conditions. Raw sample was analysed using the X-ray Fluorescence (XRF) Spectrometry to contain 54.565 % CaO, 1.35 % SiO_2 and 0.67 % Al_2O_3 among others. It was also subjected to Thermo-Gravimetric Analysis (TGA) to establish its thermal response before the production of activated carbon. Brunauer Emmet Teller (BET) analysis carried out on carbonised samples revealed an increasing surface area and pore volume with increase in temperature causing irregular pore sizing. Pharmaceutical wastewater was analysed using the Flame atomisation adsorption spectrometry (AAS) to contain 0.09 mg/l Pb(ii) 0.0439 mg/l Cd(ii) and 0.1034 mg/l Ni(ii). Percentage removal of Pb(ii) and Ni(ii) increased with increase in adsorbent dosage while that of Cd(ii) decreased. Removal of all three increased with increase in temperature and time as well. Removal efficiencies of 95.44, 90.06 and 90.89 % were recorded for Pb(ii), Ni(ii) and Cd(ii) respectively. Determination coefficient (R^2) for the adsorption models of Cd, Ni and Pb are 0.9513, 0.9694 and 0.9598.

Keywords: Absorbent dosage; Response surface methodology; Removal efficiency; Snail shell; Temperature; Time.

1 INTRODUCTION

Massive urbanization has resulted in the release of wastewater from various industrial processes into the environment which in turn pollutes the ecosystem and eventually harms living beings with their toxic nature (Hossain, et al., 2012). Process industries such as electroplating industries, hospitals, pharmaceuticals, powerplant, refineries, leather tanning, mining, dyes and pigments, steel fabrication, canning industries and inorganic chemical production plants are at the helm of affairs in the release of waste water into the environment (Radaideh, et al., 2017). The term 'heavy metal' denotes group of metals and metalloids with a density greater than 5 g/cm^3 with atomic weights between 63.5 and 200.6 and a specific gravity greater than 5.0 (Chen, et al., 2018; Singh & Gupta, 2016). Chromium (Cr), Cadmium (Cd), Copper (Cu), Mercury (Hg), Nickel (Ni), Iron (Fe), Arsenic (As), Lead (Pb), Zinc (Zn) and Gold (Au) are toxic are examples of this group which has attracted attention of several researchers. Methods engaged to reduce or out rightly remove these metals are not limited to precipitation, solvent extraction, ion-exchange, reverse osmosis, oxidation/reduction, sedimentation, filtration, electrochemical techniques and cation surfactant (Czikkely et al., 2018). In this study however, the use of low-cost adsorbent in the adsorption of Lead (Pb), Cadmium (Cd) and Nickel (Ni) from pharmaceutical water is considered. The recommended limit of Cadmium (Cd) in waste water is only 0.005 mg/L, Nickel (Ni) is 0.02 mg/L and Lead (Pb) is 0.006 mg/L (Singh & Gupta, 2016). The surge demand for pharmaceutical product stems from ground breaking research that has been made in the field of medicine. Hence, volume of wastewater from this ever-increasing industry calls for concern to bring its component within environmentally acceptable limits at minimum cost. Pharmaceutical process is water consuming therefore, the recycle of wastewater especially in the Sahara region of Africa cannot be over emphasized. African giant snail shell is abundant in the coastal region of Nigeria and can serve as a bio-sorbent subject to further studies. Therefore, this study intends to focus on the use of carbonized African giant land snail shells (Archachatina marginata) as a biomass-derived adsorbent for the removal of Lead (Pb), Cadmium (Cd) and Nickel (Ni) from pharmaceutical wastewater.

2 METHODOLOGY

2.1SAMPLE PREPARATION

The Snail Shells (SnS) were washed with detergent, they were then dried in an oven and crushed to smaller particle sizes (Sunday & Magu, 2017) after which it was sieve with 1mm meshed sieve coupled to a mechanical shaker. The pulverized snail shells of 1mm particle size are placed in an airtight sample bottle and stored for further use. The chemical properties and thermal response of snail shell sample was investigated with the aid of X-ray fluorescence (XRF) and Thermo-Gravimetric Analysis (TGA) respectively. Grab sampling method was used in collecting the wastewater used in this study. Wastewater was collected in 5 L jerry can previously washed with distilled water which was then, rinsed with the wastewater. Sufficient quantities of the wastewater were collected and transported and stored under refrigerated condition. This was done to inhibit the ageing effect, biodegradation and changes in the pharmaceutical wastewater physiochemical properties (Bolade & Sangodoyin, 2018).

2.1.1 X-RAY FLUORESCENCE (XRF) SPECTROMETRY

X-rays fluorescent (XRF) analysis was conducted using PANanalytical XRF spectrometer (MiniPal 4). X-RF analysis was carried out by placing 2 g of 100 µm size of the sample on a clean stainless-steel lid which was placed in the cubicle of the spectrometer to determine its elemental composition. When the sample was irradiated by X-rays, the system software measures the individual component wavelengths of the fluorescent emission produced by atoms in the sample (Sani, et al., 2017).

2.1.2 THERMO-GRAVIMETRIC ANALYSIS (TGA)

This analysis was carried out based on the method reported by Kolawole et al., (2017) where thermal transition and decomposition of the sample was done via TGA analysis using Perkin Elmer TGA 4000 thermogravimetric Analyzer at 10 °C/min constant heating rate in nitrogen atmosphere following the ASTM D6370 standard procedure.

2.1.3 FLAME ATOMISATION ADSORPTION SPECTROMETRY (AAS)

Thermo scientific ICE 3000 AA02134104v1.30 was used to detect heavy metals present in pharmaceutical wastewater samples. Spectrometer was set to absorbance mode and a bandpass of 2 nm. It was connected to an ethylene flame flow of 0.9 L/min. calibration was then carried out at a scaling factor of 1.0 for each metal to be analysed.

2.2 ADSORBENT PREPARATION AND CHARACTERIZATION

Snail shell (SnS) sample was weighed in batches of 5 g and each was placed in a crucible and carbonised at temperatures 600, 700, 800 and 900 °C in a Gallen Kamp muffle furnace for two hours respectively after which it is exposed to free air for four hours in other to increase its surface area (Adiotomre, 2015; Odoemelam, & Eddy, 2009). Each batch of highly active calcium oxide catalyst were labeled S-600, S-700, S-800, and S-900 respectively and placed in an air tight container. Afterwards, pore volume, pore size and surface area were determined using the Brunauer Emmet Teller (BET) method (Zhang et al 2014; Adiotomre, 2015).

2.2.1 BRUNAUER EMMET TELLER (BET) ANALYSIS

Specific surface area and pore volume analysis of the adsorbent was carried out using BET surface area Nitrogen adsorption procedure. The prepared adsorbent was out gassed under vacuum condition at 300 °C for 4 hours. Out gassed carbon sample was tested for surface area (m^2/g) and pore volume (m^3/g) at 77 K using a 15-point BET NovaWin Quantachrome, 2013 version 11.03.

2.2.2 ADSORBENT RE-USABILITY

Determination of the adsorbent reusability gives an insight into the adsorbent's chemical, thermal, mechanical and physical stability of the adsorbent during consecutive rounds of adsorption – desorption. The desorption was carried out by reacting the spent adsorbent with 1M HCl. This was carried out five consecutive times (Khan & Lo, 2016).

2.3 OPTIMIZATION STUDIES

The experimental design was setup up in other to determine the optimum conditions and removal efficiency of Cd, Ni and Pb metals from the pharmaceutical wastewater. A statistical software (DESIGN EXPERT Version 7.0.0, Stat Ease, Inc., USA) was used for the model fitting and in the evaluation of the statistical significance. The Response Surface Method (RSM) Central Composite Design (CCD) was used to study the adsorption efficiencies of these metals.

Table 1. Process Parameters under Investigation for design purposes

Variables	Low Value	High Value
Adsorbent dosage (mg)	15	50
Temperature (°C)	40	60
Contact time	10	30

Table 3. Process parameters under statistical investigation

Variables	Low Value	High Value	- alpha	+ alpha
Adsorbent dosage (mg)	15	50	3.07	61.9
Temperature (°C)	40	60	33	66
Contact time	10	30	3	37

Table 2. Experimental design matrix for the adsorption of Pb, Cd and Ni

Runs	A: Adsorbent Dosage (mg)	B: Temperature (°C)	C: Contact Time (minutes)	Cd removal X_{Cd} (%)	Ni removal X_{Ni} (%)	Pb removal X_{Pb} (%)
1	15.00	60.00	30.00	50.1	57.8	61.3
2	15.00	60.00	10.00	36.2	40.9	43.1
3	32.50	50.00	20.00	70.1	82.1	75.3
4	50.00	60.00	30.00	40.2	42.2	40
5	15.00	40.00	30.00	67.1	64.3	75.1
6	32.50	50.00	20.00	79.8	76.3	78.6
7	32.50	50.00	20.00	79.5	83.6	79.8
8	32.50	50.00	20.00	77.7	75	71
9	15.00	40.00	10.00	30.1	45.4	55.1
10	32.50	50.00	20.00	78.6	76.4	80.7
11	32.50	33.18	20.00	67.3	80	92.2
12	32.50	50.00	3.18	48.1	59.1	52.2
13	50.00	60.00	10.00	31.2	33.6	48.7
14	32.50	66.82	20.00	41.4	45.2	49.6
15	61.93	50.00	20.00	56.8	62.4	65.4
16	32.50	50.00	36.82	88	92.4	80.8
17	3.07	50.00	20.00	30	37	39.6
18	50.00	40.00	30.00	91	92.7	95.5
19	50.00	40.00	10.00	86	79.8	78.1
20	32.50	50.00	20.00	86.5	77.5	76.3

2.4 BATCH ADSORPTION EXPERIMENTS

According to Abbas, et al., (2014) factors which affect the removal of heavy metals include but are not limited to the effect of temperature, contact time and adsorbent dosage. Similarly, Lakherwal, (2014) reported that adsorption parameters such as contact time, adsorbent dosage and temperature have immense effect on the removal efficiency. Each of the parameters was studied with each setup stirred continuously with the aid of a magnetic stirrer at an agitation speed of 190 rpm.

2.4.1 EFFECT OF CONTACT TIME

The effect of contact time on percentage removal was studied by transferring 50 ml of the wastewater into a 100 ml flask. The setup is left to stand on a magnetic stirrer at ambient room temperature for a suitable time at time ranges of 5, 10, 15, 20, and 30 mins at optimum adsorbent dosage and temperature (Akinyeye, et al., 2016; Adewoye, et al., 2017).

2.4.2 EFFECT OF ADSORBENT DOSAGE

The effect of the adsorbent weight on the percentage removal was studied by transferring 50 ml ofthe wastewater into different 100ml flask. The adsorbent at different dosages (10 mg, 20 mg, 30 mg, 40 mg, 50 mg and 60 mg) was then added to the flasks and the setup was maintained at optimum conditions and left to stand on a magnetic stirrer (Akinyeye et al., 2016).

2.4.3 EFFECT OF TEMPERATURE

The adsorption process is setup in a 100 ml flask containing 50ml of the wastewater and the adsorbent. The setup is exposed to temperatures of 32, 40, 45, 50, 60 °C respectively. Data from here helps in estimation of the thermodynamic behavior of the adsorption process whereby a decrease in the adsorption rate as the temperature increase would denote an exothermic system and vice versa. The setup was left to stand on a magnetic stirrer at optimum condition (Lakherwal, 2014).

2.5 EQUILIBRIUM STUDIES

The optimum parameters determined from the batch adsorption studies was used to carry out the adsorption of the heavy metals in the pharmaceutical waste water.

2.5.1 REMOVAL EFFICIENCY DETERMINATION

The removal efficiency of the adsorbents on the metal ion adsorption was determined using equation (1)

$$\text{Removal efficiency (\%)} = \frac{(C_o - C_e)}{C_o} \times 100 \quad (1)$$

Where Co and Ce are the initial and final concentrations of metal ions.

3 RESULTS AND DISCUSSION

Table 4 shows the XRF elemental analysis of African land giant snail Shell (Achatina maginata) this was carried out to determine the chemical composition of the snail shell. Which reveals that the bulk of the African land giant snail Shell is composed of CaO with other elements like SiO_2, Fe_2O_3, Al_2O_3 and ZnO occupying a minute fraction of the bulk of the African land giant snail Shell.

Table 4. XRF result showing composition of snail shell sample (Achatina maginata).

Composition	Weight (%)
CuO	0
NiO	0
Fe_2O_3	0.066
MnO	0
Cr_2O_3	0
TiO_2	0
CaO	54.565
Al_2O_3	0.67
MgO	0
ZnO	0.011
SiO_2	1.35
LOI	43.338

From figure 1 it was observed that at temperature ranges of 27.7 °C–320 °C there was no significant degradation of the snail shell. Hence, this implies that the snail shell is thermally stable between temperature ranges of 27.7 °C–320 °C. As the temperature approaches 525 °C, a drastic loss of weight in the snail shell is observed which can be attributed to moisture losses and loss of volatile content as well. Further down the curve as the temperature approaches 825 °C, thermal decomposition sets in indicating that the calcium carbonate in the snail shell is converted into calcium oxide. This is accompanied with the liberation of carbon (iv) dioxide. Therefore, carbonization of snail shell at a minimum reaction temperature of 600 °C is required. According to Kolawole et al., (2017) the major un-degraded constituent contains calcium oxides and carbon residues.

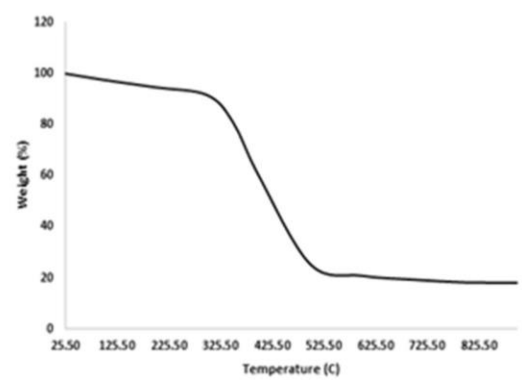

Figure 1: TGA curve for African land giant snail Shell (Achatina maginata) powder.

*Heating rate of 10 ^{0}C/min and 40cc/min N_2 flow rate

From table 5 below, it was observed that surface area and pore volume increased with increase in temperature while pore size reduced and increased in sinusoidal manner. This can be attributed to expansion as a response to heat treatment. Continuous expansion of the surface area and pore volume redefines pore sizes and shapes differently each time. A significant increase of 15.4271 % in surface area, 1.778 % in pore volume and a marginal decrease of 0.28 % in pore size was recorded at 600 ^{0}C. At the maximum temperature of 900 ^{0}C, 36.99 % surface area increase, 3.62 % pore volume increase and 0.468 % decrease in pore size were observed. This implied that snail shell has a shock response to heat considering changes in pore and surface structures.

Table 5. BET analysis result for African land giant snail shell (Achatina maginata)

Samples	Surface area (m^2/g)	Pore volume(cc/g)	Pore size (nm)
Raw Snail Shell	305.379	0.1839	2.138
S – 600	352.490	0.2166	2.132
S – 700	361.32	0.2182	2.231
S – 800	392.508	0.2111	2.100
S – 900	418.33	0.2201	2.128

Table 6,7 and 8 indicate the physiochemical, metal content and adsorption effect of adsorbent on pharmaceutical wastewater. It was observed that the adsorption process brought wastewater within standard simultaneously as heavy metals were removed. Notable among others were conductivity reduction by 76.14 %, COD reduction by 84.1 % and TDS reduction by 76.1 %. Highest removal efficiency of 95 % was recorded for Pb while Cd and nickel were removed at 90.89 and 90.06 % respectively. This shows the viability of carbonised snail shell as an adsorbent for pharmaceutical wastewater.

Table 6. Result showing physiochemical characteristics of Pharmaceutical wastewater

Parameter	Raw	Treated	NSDWQ
pH	5.83	6.87	6.5-8.5
TDS	1196.1	285.4	500
Conductivity (µS/cm)	1869	446	1000
Turbidity (NTU)	3.45	2.16	5
Total Alkalinity (mg/L)	498	20	--
Total Hardness (mg/L)	156	76	150
Dissolved Oxygen (mg/L)	8.0	7.0	--
COD (mg/L)	144.68	23.6	30
BOD (mg/L)	6.0	2.0	6.0
Chloride (mg/L)	65.66	44.1	250
Magnesium (mg/L)	13.78	1.37	2
Calcium (mg/L)	37.84	12.62	--

Table 7. AAS analysis result for pharmaceutical wastewater sample

Metal	Raw water	Standard
Cd	0.0439	0.005
Ni	0.1034	0.02
Cr	BDL	-
Pb	0.09	0.01
Fe	0.0349	-

Table 8. result showing initial and final concentration of Cd, Ni and Pb before and after adsorption process

Heavy metal	concentration	(mg/l)	Removal	MCLS (mg/l)
	Initial (C_o)	Final (C_e)	Efficiency	
Cd	0.0439	0.004	90.89	0.01
Ni	0.2505	0.0249	90.06	0.20
Pb	0.09	0.0041	95.44	0.006

From the surface plot depicted in figure 2, it was observed that an increment in the adsorbent dosage lead to a similar increment in the percentage removal of Cd. However, an increase in the temperature resulted to a decrement in the percentage removal of Cd. In terms of the interaction effect between the adsorbent dosage and the temperature it was observed that an optimum percentage removal of Cd of 94.5 % was observed at an adsorbent dosage of 43 mg and a temperature 41 ^{0}C.

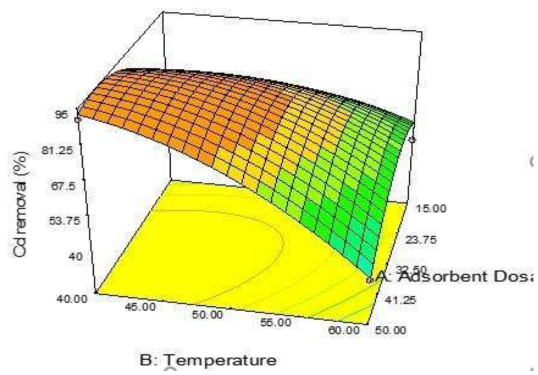

Figure 2: Response surface plot of the interaction effect of adsorbent dosage and temperature on the adsorption of Cd at a contact time of 30 minutes.

From the 3D surface plot represented in Figure 3, it was observed that an increment in the adsorbent dosage resulted in an increase in the percentage removal of Cd. Similarly, an increase in the contact time resulted in an increase in the percentage removal of Cd. Howbeit, in terms of the interaction effect of the Adsorbent dosage and contact time an optimum percentage removal of Cd of 93.2% was observed at adsorbent dosage of 50mg and contact time of 29 minutes.

From the 3D plot in Figure 4 it was observed that as the temperature dropped from 60 ^{0}C the percentage removal of Cd increased simultaneously. Similarly, an increase in the contact time also resulted in an increase in the percentage removal of Cd. In terms of the interaction effect of temperature and contact time an optimum percentage removal of Cd of 92.94 % was observed at a temperature of 40 ^{0}C and a contact time of 30 minutes.

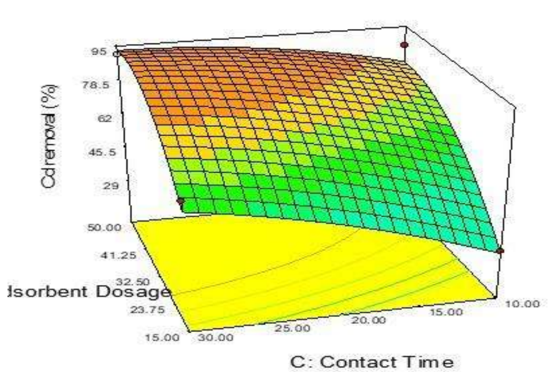

Figure 3: Response surface plot of the interaction effect of adsorbent dosage and contact time on the adsorption of Cd at a temperature of 40 ^{0}C.

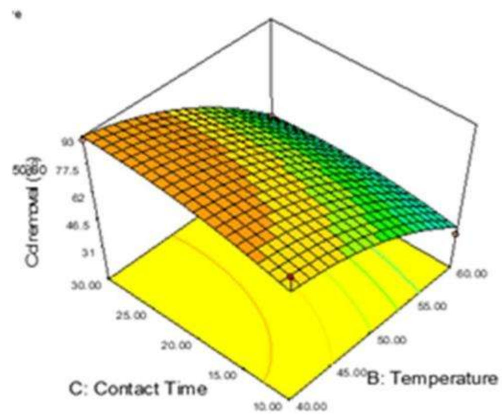

Figure 4: Response surface plot of the interaction effect of temperature and contact time on the adsorption of Cd at an adsorbent dosage of 50 mg.

From the plot represented in Figure 5 it was observed that there was an increment in the percentage removal of Ni when there was an increment in the adsorbent dosage. A similar increase in the percentage removal of Ni was observed when the reaction temperature dropped from 60 ^{0}C. The interaction effect of adsorbent dosage and temperature resulted in an optimum percentage removal of Ni of 95 % at an adsorbent dosage of 50 mg and a temperature of 40 ^{0}C.

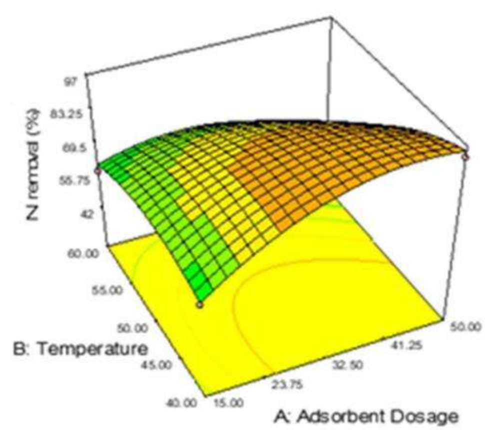

Figure 5: Response surface plot of the interaction effect of adsorbent dosage and temperature on the adsorption of Ni at a contact time of 30 minutes.

Figure 6 represents the Response surface plot of the interaction effect of Adsorbent dosage and contact time on the adsorption of Ni at a temperature of 40 ^{0}C. It was observed from the plot that an increase in the contact time brought about a similar increment in the percentage removal of Ni. A similar increment in the adsorbent dosage also led to an increment in the percentage removal of Ni. The interaction effect of the adsorbent dosage and the contact time resulted in an optimum percentage removal of Ni of 95.2 % at adsorbent dosage of 50mg and contact time of 30 minutes.

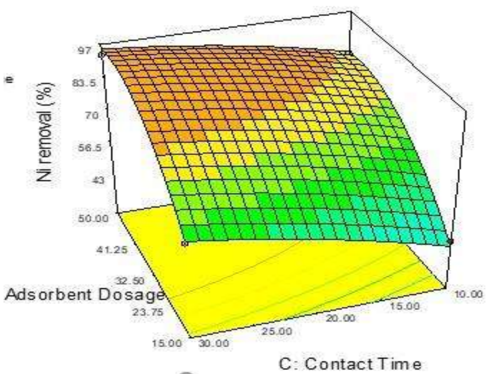

Figure 6: Response surface plot of the interaction effect of adsorbent dosage and contact time on the adsorption of Ni at a temperature of 40 ^{0}C.

The plot represented in Figure 7 represents the Response surface plot of the interaction effect of temperature and contact time on the adsorption of Cd at an adsorbent dosage of 50 mg. it was observed from the plot that an increment in the contact time resulted to an increase in the percentage removal of Ni. Similarly, a decline in the temperature from 60 ^{0}C to 40 ^{0}C resulted to an increment in the percentage removal of Ni. The effect of the temperature and the contact time resulted in an optimum percentage removal of Ni of 95 % at a temperature of 40 ^{0}C and a contact time of 30 minutes.

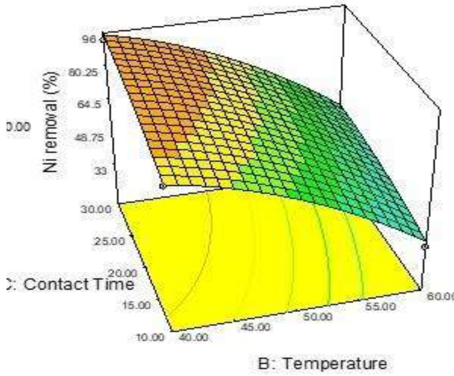

Figure 7: Response surface plot of the interaction effect of temperature and contact time on the adsorption of Ni at an adsorbent dosage of 50 mg.

From the plot represented in Figure 8 it was observed that as the adsorbent dosage increased so did the percentage removal of Pb. Howbeit, as the adsorbent dosage increased above 39 mg there was a slight dip in the percentage removal of Ni. A decrement in the temperature from 60 ^{0}C to 40 ^{0}C resulted in the increment in the percentage removal of Ni. In terms of the interaction effect of the adsorbent dosage and temperature an optimum percentage removal of Ni of 96 % was observed at an adsorbent dosage of 45 mg and a temperature of 40 ^{0}C.

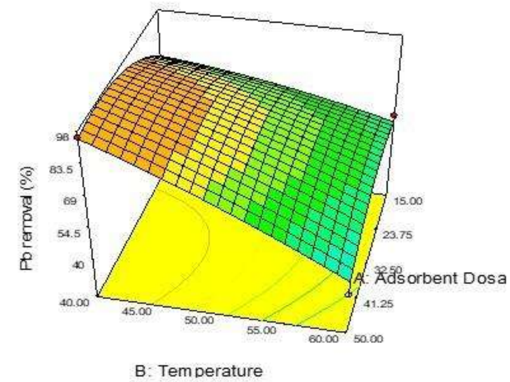

Figure 8: Response surface plot of the interaction effect of adsorbent dosage and temperature on the adsorption of Pb at a contact time of 30 minutes.

From the plot represented in Figure 9, it was observed that as the adsorbent dosage increased so did the percentage removal of Pb. Similarly, it was observed that as the contact time also increased so did the percentage removal of Pb increase. In terms of the interaction effect of the adsorbent dosage and the contact time an optimum of 95 % at an adsorbent dosage of 50 mg and a contact time of 30 minutes.

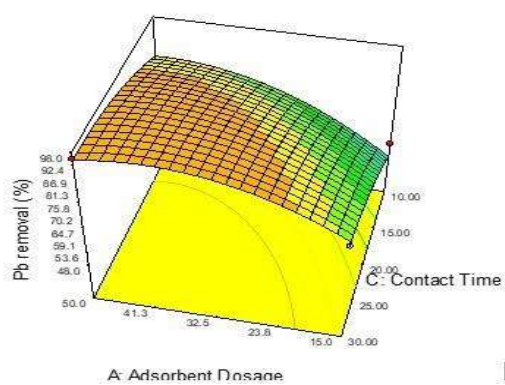

Figure 9: Response surface plot of the interaction effect of adsorbent dosage and contact time on the adsorption of Pb at a temperature of 40 ^{0}C.

The plot represented in Figure 10 represents the interaction effect of temperature and contact time on the adsorption of Pb at an adsorbent dosage of 50 mg. it was observed from the plot that a decline in the temperature resulted in an increment in the percentage removal of Pb. When the contact time is considered an increment in the contact time also resulted in an increment in the percentage removal of Pb. In terms of the interaction effect an optimum percentage removal of Pb of 94.9 % was observed at a temperature of 40 ^{0}C and a contact time of 30 minutes.

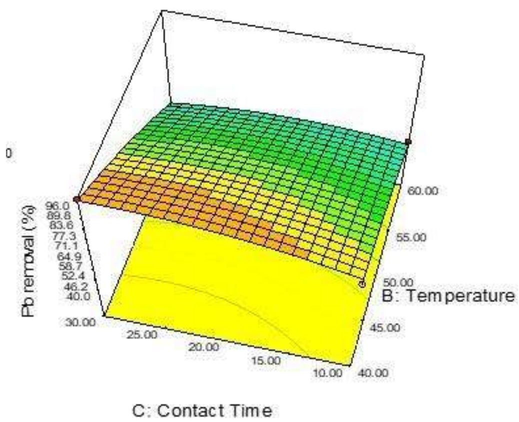

Figure 10: Response surface plot of the interaction effect of temperature and contact time on the adsorption of Pb at an adsorbent dosage of 50 mg.

From Figure 11 below, it was observed that as the adsorbent dosage increased so did the percentage removal increase. It is noteworthy that the percentage removal of cadmium, Nickel and Lead increased significantly from 67.2 to 90, 69.8 to 91.1 and 77.8 to 93.1% respectively as the adsorbent dosage was varied from 10 to 50 mg. Further increment in the adsorbent dosage resulted in a slight reduction in the removal efficiency. The reason for this may be attributed to overlapping of the adsorption sites which could lead to an overall decrease in the available binding sites (Adewoye, et al., 2017).

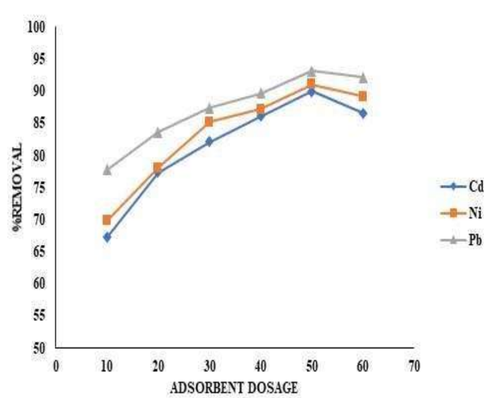

Figure 11. Effect of Adsorbent Dosage on Adsorption of Cd/Ni/Pb on Carbonized Snail shell

Figure 12 represents the plot of the effect of temperature against the adsorption of Cd/Ni/Pb on Carbonized Snail shell. It was observed that an increase in the temperature resulted in an increase in the adsorption of Ni and Pb while at the same time it resulted in a decrease in the adsorption of Cd. Further increment in the adsorption temperature above 50 °C resulted in a notable reduction in the adsorption of Cd/Ni/Pb. Optimum adsorption of Cd/Ni/Pb was observed at a temperature of 50 °C with adsorption rate of 89.4, 91.2 and 89.2 respectively.

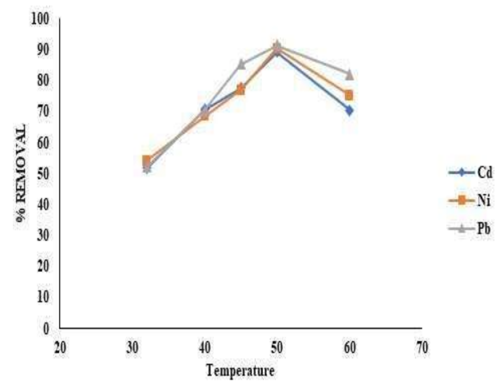

Figure 12. Effect of Temperature on Adsorption of Cd/Ni/Pb on Carbonized Snail shell

The effect of Contact Time on Adsorption of Cd/Ni/Pb on Carbonized Snail shell was studied and the data presented in Figure 13. From the graph, it was observed that an increase in the contact time resulted in a comparatively similar increment in the adsorption of Cd/Ni/Pb with the progression of time from 10 min to 40 min. Further increment in the contact time caused a decline in the adsorption rate and this was attributed to overlapping of the adsorption sites over time resulting in an overall decrease in the available binding sites (Adewoye, et al., 2017).

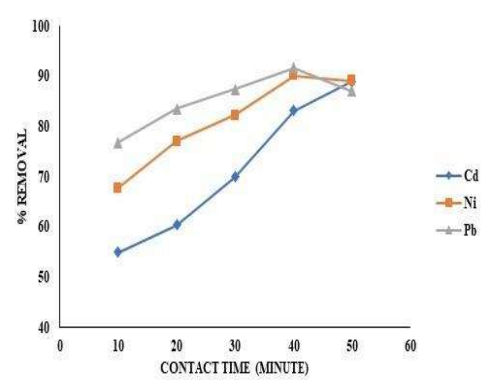

Figure 13. Effect of Contact time on Adsorption of Cd/Ni/Pb on Carbonized Snail shell

The analysis of the variance (ANOVA) for the response surface quadratic model for the adsorption of Cd, Ni and Pb was shown in Equation 2,3 and 4 respectively. The ANOVA analysis gives a model expression which relates the adsorption of Cd, Ni and Pb to the three process parameters (A, B and C). The ANOVA analysis for the adsorption of Cd and Ni shows that the significant process parameters that affect the adsorption process are A, B, C, AB, A^2 and B^2 while the ANOVA analysis of the adsorption of Pb shows that the significant process parameters that affect the adsorption of Pb are A, B, C, AB, A^2 and C^2 respectively. These terms are significant because their p-values are less than 0.05. Similarly, the quadratic model selected in the adsorption of Cd, Ni and Pb is significant because the models p-values are less than 0.05. In this study the value of the determination coefficient (R^2) for the adsorption of Cd, Ni and Pb are 0.9513, 0.9694 and 0.9598 respectively imply that 95.13 %, 96.94 % and 95.98 % of the total variation can be attributed to the independent variables while 4.8 7 %, 3.06 % and 4.02 % of the total variation is not explained by the model in the adsorption of Cd, Ni and Pb respectively.

The value of the coefficient of variation (C.V. %) gives the precision and reliability of the experiment carried out where a lower value of 10.64, 7.02 and 7.07 obtained in the adsorption of Cd, Ni and Pb respectively indicate a better precision and reliability of the experiment.

Similarly, the model equation representing the adsorption of Cd, Ni and Pb respectively is presented in coded form where:

$$\text{Cd removal (\%)} = 78.70 + 8.05A - 11.72B + 9.67C - 11.84AB - 4.61AC - 2.39BC - 12.45A^2 - 8.58B^2 - 3.74C^2 \quad (2)$$

$$\text{Ni removal (\%)} = 78.63 + 6.05A - 12.17B + 8.30C - 10.71AB - 1.79AC - 0.79BC - 11.16A^2 - 6.59B^2 - 1.94C^2 \quad (3)$$

$$\text{Pb removal (\%)} = 76.96 + 5.21A - 13.35B + 6.96C - 7.39AB - 3.69AC - 3.49BC - 8.72A^2 - 2.21B^2 - 3.77C^2 \quad (4)$$

From Figure 14, 15 and 16, it was observed that there was a close correlation between the actual values obtained from the study as they lie close to the regression line as such they correlated with the predicted values generated by design expert. This close correlation is indicative of the fact that the quadratic model selected for the adsorption of Cd, Ni and Pb is suitable.

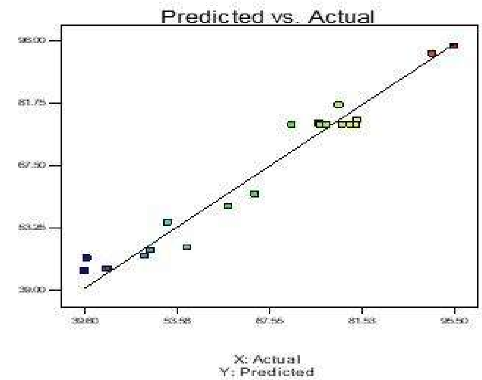

Figure 16. Parity plot of Predicted values (model) vs Actual values for the Adsorption of Pb

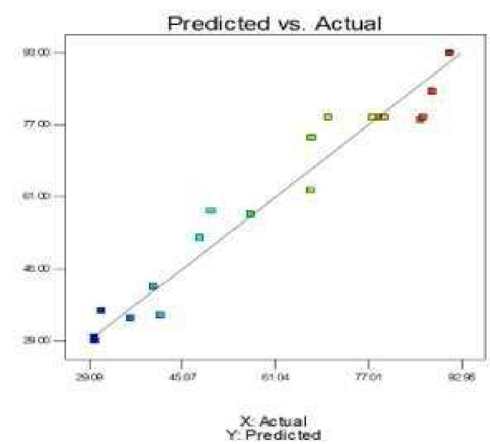

Figure 14. Parity plot of Predicted values (model) vs Actual values for the Adsorption of Ni

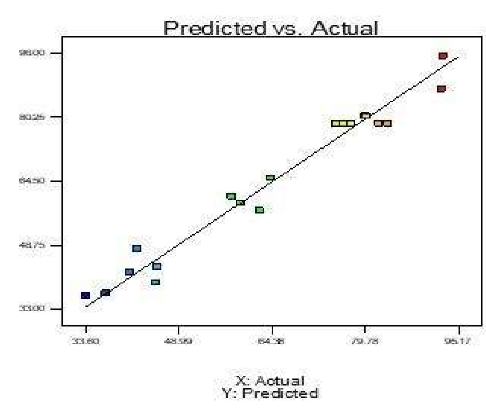

Figure 15. Parity plot of Predicted values (model) vs Actual values for the Adsorption of Ni

4 CONCLUSION

From the results obtained from the experiment, it can be concluded that African giant snail shell contains 54.565 % CaO thus, can be converted into an adsorbent with large surface area and pore volume when subjected to carbonization to burn off other components. Again, it can be concluded that temperature, time and absorbent dosage has a significant effect on the adsorption of Pb(ii), Ni(ii) and Cd(ii) respectively using carbonised snail shell. It can also be concluded that the removal efficiency of Cadmium, Lead and Nickel were 90.89, 95.44 and 90.06 % respectively when using carbonised snail shell as an adsorbent. It can be said that the lower value of 10.64, 7.02 and 7.07 % obtained as the percentage coefficient of variation of the adsorption models of Cd(ii), Ni(ii) and Pb(ii) respectively indicate a better precision and reliability of the experiment.

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REFERENCE

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Abbas, S.H., Ismail, I.M., Mostafa, T.M., Sulayman, A.H. (2014). Biosorption of Heavy Metals: A Review. Journal of Chemical Science and Technology, 3(4), 74 – 102.

Adewoye, L.T., Mustapha, S.I., Adeniyi, A.G., Tijani, J.O., Amoloye, M.A. & Ayinde, L.J. (2017). Optimization of nickel (ii) and chromium (iii) removal from contaminated water using sorghum bicolor. Nigerian Journal of Technology, 36(3), 960 – 972.

Adiotomre, K.O. (2015). Effectiveness of Snail Shell as An Adsorbent for The Treatment of Waste Water. International Journal of Innovative Environmental Studies Research, 3(3), 1 – 12.

Akinyeye, O.J., Ibigbami, T.B., Odeja, O. (2016). Effect of Chitosan Powder Prepared from Snail Shells to Remove Lead (II) Ion and Nickel (II) Ion from Aqueous Solution and Its Adsorption Isotherm Model. American Journal of Applied Chemistry, 4(4), 146 – 156.

Bolade, O.O. & Sangodoyin, A.Y. (2018). Adsorption and Equilibrium Studies of Textile Effluent Treatment with Activated Snail Shell Carbon. IOSR Journal of Environmental Science, Toxicology and Food Technology, 12(4), 26 – 33

Chen, H., Xie, A. & You, S. (2018). A Review: Advances on Absorption of Heavy Metals in the Waste Water by Biochar. International Journal of Material science and Engineering, 1(1), 1 – 5.

Czikkely, M., Neubauer, E., Fekete, I., Ymeri, P. & Fogarassy, C. (2018). Review of Heavy Metal Adsorption Processes by Several Organic Matters from Wastewaters. Journal of water, 10(1), 1 – 15.

Hossain, M.A., Hao Ngo, H., Guo, W.S. & Nguyen, T.V. (2013). Removal of Copper from Water by Adsorption onto Banana Peel as Bioadsorbent. International Journal of GEOMATE, 2(2), 227 – 234.

Khan, M. & Lo, M.C. (2016). A holistic review of hydrogel applications in the adsorptive removal of aqueous pollutants: Recent progress, challenges, and perspectives. Journal of Water Research, 1(1), 1 – 14.

Kolawole, M.Y., Aweda, J.O. and Abdulkareem, S. (2017). Archachatina marginata bio-shells as reinforcement material in metal matrix composites. International Journal of Automotive and Mechanical Engineering., 14(1), 4068 – 4079.

Odoemelam S. A. & Eddy N. O. (2009) Studies on the Use of Oyster, Snail and Periwinkle Shells as Adsorbents for the Removal of Pb^{2+} from Aqueous Solution. E-Journal of Chemistry, 6(1) 213 – 222.

Singh, N. & Gupta, S.K. (2016). Adsorption of Heavy Metals: A Review. International Journal of Innovative Research in Science, Engineering and Technology, 5(2), 2267 – 2281.

Radaideh, J.A., Abdulgader, H.A. & Barjenbruch, M. (2017). Evaluation of Absorption Process for Heavy Metals Removal found in Pharmaceutical Wastewater. Journal of Medical Toxicology and Clinical Forensic Medicine, 3(2), 1 – 12.

Sunday, E. A., & Magu, T. O. (2017). Determination of some metal contents in ashed and unashed snail shell powders. World news of Natural Science, 7(2017), 37 – 41.

Sani, J., Samir, S., Rikoto, II., Tambuwal, AD., Sanda, A., Maishanu, S.M., & Ladan, M.M. (2017) Production and Characterization of Heterogeneous Catalyst (CaO) from Snail Shell for Biodiesel Production Using Waste Cooking Oil. Innovative Energy Research, 6(2), 1 – 4.

Zhang, Y., Liu, S. & Wu, H. (2014). Experiment study on the decomposition properties of snail shell. Journal of Biotechnology, 9(8) 303 – 307.

Lakherwal, D. (2014) Adsorption of Heavy Metals:A Review. International Journal of Environmental Research and Development, 1, 41 – 48.