Remediation of Heavy Metals from Galvanic Wastewater Using Cow Bone Char as Low Cost Adsorbent

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INTRODUCTION

Galvanized wastewater is one of the highly rated toxic wastewater because of its heavy metal contents, organic substances and surfactants, and can be considered among the most polluting wastewater difficult to treat. Therefore, requires special treatment in order to reduce the pollutants’ concentration before disposal [1–3]. Indiscriminate disposal of galvanized wastewater into the environment contaminates the soil, surface and groundwater and it has been reported to be one of the leading activators of cancer and cardiovascular diseases in man as well as contributor to other negative health conditions like brain damage, lethargy, neurological signs etc. [4, 5]. Galvanization or galvanizing is the process of applying a protective zinc coating to steel or iron, to prevent rusting, improve wear resistance or for ornamental purposes [3]. It consists of sequential electrochemical processes resulting in wastewater generation of different composition [3, 6]. There are two widely known processes; the hot-dip galvanization and the cold galvanization [6, 7]. The most common method is hot-dip galvanizing, in which the metal parts are submerged in a bath of molten hot zinc [6, 8]. Cold
galvanization has three main phases: surface preparation, galvanizing and post treatment, each phase involving the use of rinsing tanks with water and are often loaded with high concentrations of heavy metals which are often disposed indiscriminately [8]. One of the primary producers of toxic compounds into the environment is improper discharge of galvanic wastewater. More often focus has been placed on the galvanic sludge produced during treatment of the wastewater, however, if the wastewater is adequately treated, less of the sludge will be generated and the environment would remain safe.

Several techniques had been employed for the treatment of galvanized wastewater, these include chemical coagulation/floculation, microfiltration, ultrafiltration, nano-filtration, reverse osmosis, chelating, polyelectrolytes, ionic exchange - electric arc furnace [3–5, 9]. Most of these techniques are slow, associated with high consumption of reagents and generation of sludge that require disposal in a special engineered landfill which often result in high cost, considerable investment and operating costs, some require large processing areas [2]. The use of locally sourced agricultural by-products like moringa, orange peel, cassava peel, orange peel, banana peel, plum leaves, guava fruits etc. [10, 11], natural clay minerals [12], composite nano-fibrous material [13] and carbon as materials for adsorbent have gained wide application for the removal of pollutants and heavy metals.

Presently, there is paucity of research on the use of animal bone for remediation of heavy metals from real time galvanic wastewater. Among the various carbonaceous materials used as absorbent, animal/ cow-bone had been reported to be effective for removing a variety of pollutants, such as heavy metals and dyes from aqueous solutions due to its high surface area, well-developed internal micro-porosity, and a broad spectrum of surface functional groups [14]. Globally, increasing demand for meat has resulted in the generation of large amounts of cow-bone waste [15, 16]. One of the ways to manage this excessive bone wastage is conversion to a usable product as adsorbent because of its availability, low or no cost, versatility, low energy requirement and sustainability in usage. Previous studies had reported the effective use of cow bone for wastewater remediation. Cow bone had been adopted for the removal of hexavalent blue from aqueous solution [17]. Adsorption of hexavalent chromium from aqueous solution using cow bone had also been reported [18]. Adsorption of selected metal ions; iron, zinc, lead and manganese from cassava wastewater using cow bone had been reported to give excellent removal efficiency between 93% and 99% for Fe $^{2+}$, Zn $^{2+}$, Pb $^{2+}$ and Mn $^{2+}$ [5]. Little is known on the efficacy of cow bone for galvanic wastewater remediation.

Several work had been carried out on the treatment or remediation of galvanized wastewater using various techniques. Liquid surfactant membrane technique was utilized to reduce Cr (VI) and Zn (II) from galvanized wastewater [19]. Coagulants and flocculants in a system of Jar-test have been used to remediate galvanized wastewater [5], these techniques are slow with large amounts of sludge production. Natural adsorbents had been utilized to remediate iron and chloride from wastewater achieved removal efficiency of 72% and 54%, respectively [20]. Magnetic nanoparticles had been utilized to obtain 95% of Zn$^{2+}$ removal from galvanic wastewater [21]. Electric spark method using metal loading (Fe, Al) and low-voltage (up to 1000 V) equipment had been employed reduce the concentrations of Zn$^{2+}$, Cr$^{6+}$, Cr$^{3+}$, Cu$^{2+}$ from multicomponent galvanic wastewater [2]. These turned out to be economically inexpedient due to high cost and limited resource of high-voltage equipment, increased danger and low process productivity. Hence, most of the methods employed by these studies are expensive, requiring considerable investment and operating costs as well as large processing areas. Several commercially available activated carbons have also been identified for use as adsorbent as stated earlier especially in the removal of heavy metals from industrial wastewater. Nevertheless, these processes are also proven to be expensive. There is a growing demand to utilize alternative low-cost available adsorbents for the removal of heavy metals [4, 22] from galvanic wastewater. Cow bones are cheap and readily available locally from slaughterhouses and abattoirs. Therefore, their use as an alternative adsorbent for remediation of galvanic wastewater had to be assessed.

In this study, the efficacy of cow bone char was assessed for simultaneous heavy metal ions removal from real life galvanic wastewater in a competitive adsorption process and not from synthesized aqueous wastewater solution.

**MATERIALS AND METHODS**

All the chemicals and reagents used were of analytical grade. The experiments were performed following standard method procedures.

**Characterization of galvanized wastewater**

The galvanized wastewater was obtained from a Machine Tools factory located on 110 hectares industrial site, South Western Nigeria (7.839043° N, 4.602833° E). The raw wastewater was characterized for heavy metal content following standard procedures described in the standard method for examination of water and wastewater [23]. The initial pH of the galvanized wastewater was 5.9 and was adjusted to 7.0 using dilute solutions of NaOH throughout the experiments. Firstly, the digestion of the galvanized wastewater sample was carried out with the aim of breaking down the complexity of the samples before using the Atomic Absorption Spectrophotometer (No. FG6990). A 10 mL of the galvanized wastewater
Preparation of cow bone and characterization of cow bone char
Cow bones were obtained from the abattoir Market, in Ogbomoso (8.1447° N, 4.2426° E), Southwestern Nigeria. Fresh cow bones obtained were boiled in hot water to tenderize the meat for easy removal of leftover flesh on the bone and washed to remove impurities on the surfaces. The bones were sundried, crushed and were carbonized inside a muffle furnace at 800°C for 5 hours. The bone charred produced were further reduce by grinding using ceramic ball mill grinded and was sieved with 125 µm sieve to obtain the fine powdered form. The method for the preparation of the cow bone char is as stated in our previous work [14]. Characterization of the cow bone char was performed in accordance to procedures outlined in the Association of Official Analytical Chemist (AOAC) [24]. The adsorbents produced were characterized by physical and chemical properties. The elemental content of the adsorbent was determined with X-ray Fluorescence (XRF), Scanning Electron Microscope (SEM, model: JEOL JSM 6380LA) was employed to determine the surface morphology of the adsorbent while Fourier transform infrared spectrometer FTIR (Perkin Elmer) was used to determine the characteristics of the functional groups on the adsorbent surface as outlined in our previous work [14].

Batch experiment procedure
The batch experiment was performed in triplicates in accordance to the steps outlined in previous study [2, 14]. Variation of absorbent dose was performed using four conical flasks of 250 mL labeled 1 to 4. In each of the flasks 100 mL of the galvanized wastewater was added and 0.02, 0.04, 0.06 and 0.08 g of the CBC was added to the four respective conical flasks. The flasks were then placed in a rotary mechanical shaker and agitated at a speed of 150 rpm for 60 minutes. The filtrate from each flask was collected and subjected to AAS to determine the heavy metal concentrations. Contact time effect was performed using six conical flasks with 100 mL of the galvanized wastewater to which 0.02 g of adsorbent was added into each. The flasks were then placed in a mechanical shaker and agitated at 150 rpm for different selected contact times of 20, 40, 60, 90, 100 and 120 minutes. Filtrates from the content of each flask were subjected to AAS analysis. Effect of agitation rate was performed to determine its effect on the treatment process, 0.02 g each of the adsorbent was added to 4 conical flasks of 100 mL of the galvanized wastewater. The conical flasks were placed in a mechanical shaker in turns and rotated at the selected speed ranging from 50 to 200 rpm at intervals of 50 rpm at a constant time of 60 minutes. The percentage of metal removal efficiency was estimated using Equation (1):

$$Removal\ efficiency\ (%) = \frac{C_1 - C_2}{C_1} \times 100$$

where $C_1$ = Initial concentration of heavy metals in the wastewater
$C_2$ = Final concentration of heavy metals in the wastewater

RESULT AND DISCUSSION
Heavy metal content of the galvanized wastewater
The mean concentrations of Mn$^{2+}$, Pb$^{2+}$, Zn$^{2+}$, Fe$^{2+}$ and Cr$^{2+}$ in the raw galvanic wastewater are presented in Table 1. The observed mean concentration of Mn$^{2+}$, Pb$^{2+}$, Zn$^{2+}$, Fe$^{2+}$ and Cr$^{2+}$ in the wastewater were 0.185 mg/L, 0.079 mg/L, 0.153 mg/L, 0.31859 mg/L and 0.072 mg/L, respectively, which is higher than WHO and EPA permissible limits. High concentration of metal ions above the threshold was also reported by Beh et al. [4] for iron, zinc and manganese in galvanic wastewater. Similarly, high concentration for zinc and iron in galvanized wastewater was reported by Berradi et al. [5].

Adsorbent characterization
The physical and chemical properties of the cow bone char are presented in Table 2. This result is in line with cow bone charcoal reported in previous studies [25, 26]. Figure 1a and b presents different magnification of SEM images for the adsorbent. The adsorbent was crystalline in nature, its surface morphology revealed a significant distribution of well-developed porous and rough surface with crack for the efficient adsorption process. Adsorbent pores provide an effective surface for ions to be adsorbed in the wastewater. Figure 1 revealed that the adsorbent has good porosity which allows for the adsorption of heavy metals.

Table 1. Mean concentrations of the heavy metals in the galvanized wastewater

| Metals       | Concentration (mg/L) | WHO (mg/L) | EPA |
|--------------|----------------------|------------|-----|
|              | mean ± SD            |            |     |
| Manganese (Mn) | 0.185 ± 0.0092       | 0.05       | 0.05|
| Lead (Pb)    | 0.079 ± 0.0075       | 0.015      | 0.01|
| Zinc (Zn)    | 31.859 ± 0.0577      | 5          | 5   |
| Iron (Fe)    | 10.848 ± 0.069       | 0.3        | 0.3 |
| Chromium (Cr)| 5.23 ± 0.057        | 0.1        |     |
Table 2. Physicochemical properties of the absorbent

| Parameters          | Mean ± standard deviation of triplicate evaluation |
|---------------------|-----------------------------------------------------|
| Moisture (%)        | 3.3 ± 0.1                                           |
| Volatile matter (%) | 1.73 ± 0.004                                        |
| Fixed carbon (%)    | 24.71 ± 0.27                                        |
| Surface area (m²/g) | 1.92 ± 0.009                                        |
| Pore volume (cm³/g) | 0.013 ± 0.001                                       |
| Carbon (%)          | 25 ± 0.03                                           |
| Nitrogen (%)        | 1.5 ± 0.1                                           |
| Hydrogen (%)        | 3.27 ± 0.09                                         |
| CaCO₃ (%)           | 51.87 ± 0.19                                        |
| Sulphur (%)         | 0.9 ± 0.1                                           |

Source: Previous work of Olaoye et al. [14]

The FTIR spectra [14] revealed several peaks: 3981.6, 3645.5 - 3547.4, 3433.8 - 3321.5, 3237.1- 3049.4, 2944.8-2833.7, 2728.5- 2571.5, 2411.9 – 2150.4, 2065.5 - 1613.6, 1369.1 - 1280.2, 1066.2- 878.5, 761.3 cm⁻¹ etc. representing the presence of different functional groups like alcohol (O-H stretch, H-bonded, free), alkane (C-H stretch, -C-H bending) alkene (=C-H bending, C=C stretch) amides (N-H stretch) nitro compounds (N-O stretch) acid (O-H, stretch) ester (C=O, stretch). These functional groups play a vital role in the adsorbent property for adsorption process.

Adsorption process

It was observed that the concentration of metal ions reduces with adsorbent dose, contact time and speed of agitation as compared with the initial concentration of the raw wastewater as shown in the stacked column in Figure 2.

As the adsorbent dose was increased from 0.02 – 0.04 g the concentration of metal ions reduces. Further increase of adsorbent, slows down the reduction of metal ions in the solution. The average concentrations at adsorbent doses of 0.02 g were 0.002, 0.08, 1.10, 0.008 and 1.39 mg/L for Mn²⁺, Fe²⁺, Zn²⁺, Pb²⁺, and Cr²⁺ respectively. These concentration values reduced to 0.001, 0.035, 0.030, 0.007 and 1.29 mg/L at adsorbent dose of 0.04g before an increase was observed between adsorbent doses of 0.06 - 0.08 g with average metal concentrations between 0.002 – 0.003, 1.17 - 2.07, 0.32 - 0.33, 0.009 - 0.01 and 1.58 - 1.61 mg/L for Mn²⁺, Fe²⁺, Zn²⁺, Pb²⁺ and Cr²⁺, respectively.

The average concentrations between contact time of 20 – 60 minutes were 0.0009 - 0.004 mg/L for Mn²⁺, 0.30-0.28 mg/L for Zn²⁺, 0.04 - 0.02 mg/L for Pb²⁺, 1.71 -
0.077 mg/L for Cr\(^{2+}\) while Fe\(^{2+}\) was not detected. With further increase in contact time between 80 – 120 minutes, the observed average metal concentrations were between 0.0007 – 0.019, 0 – 1.08, 0.31 – 0.76, 0.02 – 0.025 and 0.775 – 1.02 mg/L for Mn\(^{2+}\), Fe\(^{2+}\), Zn\(^{2+}\), Pb\(^{2+}\) and Cr\(^{2+}\), respectively. Average concentration values between 0.0007 – 0.019, 0 – 1.08, 0.31 – 0.76, 0.02 – 0.025 and 0.775 – 1.02 mg/L for Mn\(^{2+}\), Fe\(^{2+}\), Zn\(^{2+}\), Pb\(^{2+}\) and Cr\(^{2+}\), respectively was observed at agitation speed of 50 – 200 rpm. Minimum concentrations were observed at a speed of 150 rpm with average concentration values of 0.002, 0.33, 0.21, 0.04 and 1.13 mg/L for Mn\(^{2+}\), Fe\(^{2+}\), Zn\(^{2+}\), Pb\(^{2+}\) and Cr\(^{2+}\), respectively.

**Effects of adsorbent dose**

The removal efficiency in percentage against adsorbent dose is presented in Figure 3(a). Adsorbent dosage was varied in the order of 0.02 to 0.08 g at an interval of 0.02 g for the 100 mL galvanized wastewater, agitated at 150 rpm for 60 minutes. It was observed that the percentage of metal ion removal increased with increasing dosage of adsorbent until peak adsorption was obtained at adsorbent dose of 0.04 g. Removal efficiency generally increases with increase in adsorbent dose until an equilibrium condition is reached at appropriate temperature after which gradual decrease in adsorption is observed. This trend is expected because as the adsorbent dose increases the number of adsorbent particles in the solution increases and thus more metal ions are attached to their surfaces [27]. All active sites were entirely exposed at lower adsorbent doses, while only a fraction of the active sites were exposed at higher doses [14, 28, 29]. Thus, a higher adsorbent dosage causes aggregation, which decreases the total surface area of adsorbent, leading to gradual decrease [14, 30 – 32]. Hence the adsorbent dose was fixed at 0.04 g. Maximum metal ion removal expressed as a percentage was 99% for Mn\(^{2+}\), 99.6% for Fe\(^{2+}\), 99.05% for Zn\(^{2+}\), 90.63% for Pb\(^{2+}\), and 75.23% for Cr\(^{2+}\), observed at an adsorbent dose of 0.04 g. Hence an optimum dosage of 0.04 g of Cow Bone Char was adopted for all the adsorption processes. It infers that effective adsorption sites increase with adsorbent doses until a decline after the dose of 0.04 g was utilized. All adsorbent doses used revealed adsorption efficiency above 95% except for Cr\(^{2+}\). Adsorbent dose of 0.4 g/100 mL was reported for Cd(II), Pb(II), Zn(II), Cr(III) and Cu(II) adsorption using milled adsorbents of mango peel and Alisma plantago aquatic [33]. Similar results were reported with 5 g increase of adsorbent dose for Mn\(^{2+}\) removal efficiency between 95.3% and 95.4% [34, 35]. High adsorption

![Figure 3](image-url)
efficiency for adsorbent dosage had been reported to be due to internal structure of the adsorbent and the availability of active sites as well as larger pore spaces [10, 34].

**Effects of contact time**
The effect of contact time with metal removal is presented in Figure 3(b). Contact time was varied between 20 - 120 minutes for an adsorbent dose of 0.04 g. Reduction of metal ion removal was observed with an increase in contact time for all the metal ions. It was observed that removal efficiency was rapid initially for Pb\(^{2+}\) and Cr\(^{6+}\) due to presence of available vacant sites which became used up as contact time increases and then decreases gradually until equilibrium time was reached beyond which there was no significant increase in removal rate of the metal ions.

For Mn\(^{2+}\), Fe\(^{2+}\) and Zn\(^{2+}\) as contact time increases, removal efficiency increases at a constant steady rate until an equilibrium time is reached at 60 minutes after which it decreases and remains constant up to 120 minutes except for Fe\(^{2+}\) which declines after 100 minutes. Removal efficiency increases with increasing contact time until an equilibrium time when it gradually declines. The time required for the equilibrium to be attained for all the metal ions was 60 minutes beyond this contact time; desorption occurs which releases ions that had already been absorbed. Similar results was reported, that at saturation point increasing contact time would reduce the effectiveness of the adsorbent [36]. In this study, removal efficiency obtained varied between 89.9 – 99.7% for Mn\(^{2+}\), 90 – 100% for Fe\(^{2+}\), 97 – 99.12% for Zn\(^{2+}\), 49 – 77.2% for Pb\(^{2+}\) and 67 – 85% for Cr \(^{6+}\). It was observed that increasing contact time has little effect on Fe\(^{2+}\) removal because at 20 minutes of contact the Fe\(^{2+}\) had been completely removed due to availability of active sufficient site for the adsorption process. Adsorption efficiency above 95% was observed for Mn\(^{2+}\), Fe\(^{2+}\) and Zn\(^{2+}\) while 77% and 85% was recorded for Pb\(^{2+}\) and Cr\(^{6+}\), respectively. Adsorption efficiency of 95% in less than 10 minutes for only Zn\(^{2+}\) removal from galvanic wastewater using magnetic nanoparticles had been reported by Sawalha et al. [21]. However, despite the simultaneous adsorption of metals in this study Fe\(^{2+}\) removal efficiency was almost 100%. Contact time of 60 minutes had been reported for Fe \(^{2+}\), Zn \(^{2+}\), Pb \(^{2+}\), and Mn \(^{2+}\) removal from cassava wastewater [14]. It was reported by Coelho et al. [19] observed removal efficiency of 95% and 70% for Cr\(^{6+}\) and Zn\(^{2+}\), respectively from galvanized wastewater between a contact time of 3 to 30 minutes, after which there was slight reduction. Kumar et al. [20] obtained maximum removal efficiency of 72% at a contact time of 90 minutes, adsorbent dosage of 60 mg/L for Fe\(^{2+}\) removal in galvanized wastewater. Maximum adsorption of Cr\(^{6+}\) at 35 minutes, 53 µm size of adsorbent, and 3 g of adsorbent dosage was also reported by Sunday et al. [18].

There was also a high increase in the rate of adsorption of Cr(VI) using tamarind wood activated carbon within 20 minutes, which decreased as the time was increased; thus, attaining equilibrium within 40 minutes [27].

**Effects of agitation**
The effects of rotating speed on the metal removal is presented in Figure 3(c). Agitation is an important parameter in adsorption phenomena because it influences the distribution of the solute in the bulk solution and the formation of the external boundary. The speed was varied from 50 rpm – 200 rpm at 50 rpm interval. Increase in removal efficiency of metal ions was observed between agitation speeds of 50 rpm – 150 rpm because as stirring speed increases, the diffusion and mass transfer also increases within the solution before a slight decline at 200 rpm. By increasing the stirring speed turbulence also increases and at the same time the adsorbent reacts more with the wastewater, which aids the adsorption process; however, beyond this contact time, slight decrease was attained. These agree with previous study on the removal of Cr (VI) using trioctylphosphine oxide (TOPO) and trioctylamine and Alamine 336 as adsorbent [37] where agitation speed was studied in the range of 200- 350 rpm. The extraction efficiency of Cr \(^{6+}\) increased with increasing speed, optimum mixing speed of 325 rpm resulted in the highest extraction efficiency beyond which leakage of the metal ions into the solution starts which results in gradual decline in metal removal. The decrease was attributed to rupture of the globules by shear forces and welling which led to loss of solute and removal agent due to the agitation of internal and feed phases thereby decreasing the removal efficiency [37–39].

In this study, mixing speed of 150 rpm was taken as the best speed for the adsorption of metal ions, beyond this leakages of metals that have already been adsorbed may result due to shearing of the solution thus causing gradual depletion in metal extraction [37]. Maximum adsorption efficiency of 98.7% was obtained for Mn\(^{2+}\), 96.9% for Fe\(^{2+}\), 99.3% for Zn\(^{2+}\), 45.6% for Pb\(^{2+}\) and 78% for Cr\(^{6+}\) was obtained in this study at agitation speed of 150 rpm. It can be seen that agitation has the least effect on Pb\(^{2+}\) removal as the active sites for adsorption processes were stable throughout, similar results was reported by Olaoye et al. [14] using Cow bone for metal ion removal.

**Adsorption capacity**
The average adsorption capacity for Mn\(^{2+}\) was 0.44 mg/g, 26.7 mg/g for Fe\(^{2+}\), 78.5 mg/g for Zn\(^{2+}\), 0.133 mg/g for Pb\(^{2+}\) and 10.36 mg/g for Cr\(^{6+}\) as shown in Figure 3(d). Adsorption capacity between 0.1403 – 1.551 mg/g for Pb\(^{2+}\) had been reported [40] using modified melon husk for tannery wastewater treatment while higher adsorption capacity of 85.4 mg/g for Pb\(^{2+}\) was reported by Rameznani et al. [41] using modified magnetic graphene oxide.
The coefficient of determination ($R^2$) obtained for the adsorption processes were; 0.94 for Mn$^{2+}$, 0.99 for Fe$^{2+}$, 0.91 for Zn$^{2+}$, 0.89 for Pb$^{2+}$ and 0.78 for Cr$^{2+}$. $R^2$ values obtained were indicative of sound correlation. $R^2$ values for Mn$^{2+}$, Fe$^{2+}$ and Zn$^{2+}$ metal ion removal were $>0.9$, subsequently for Pb$^{2+}$ and Cr$^{2+}$, $R^2$ values were $>0.78$. The best fit for metal ions removal efficiency at varying agitation speed as shown in Figure 4 reflect good adsorption properties of the adsorbent. The lines were smooth and continuous, the equations for the indeterminate variable for Mn$^{2+}$, Fe$^{2+}$, Zn$^{2+}$, Pb$^{2+}$ and Cr$^{2+}$ are displayed in the equations were second order polynomial equations for each of the metal ions.

**CONCLUSION**

Galvanic wastewater is toxic in nature and hence should be adequately treated before discharge. The concentrations of heavy metals Mn$^{2+}$, Fe$^{2+}$, Zn$^{2+}$, Pb$^{2+}$, Cr$^{2+}$ in the treated galvanic wastewater are significantly lower than their maximal permissible concentrations after remediation with CBC as adsorbent. The optimum adsorbent dose for all the metal ions was 0.04g/100 mL at an optimum contact time of 60 minutes except for Fe$^{2+}$ with optimum contact time of 20 minutes, and agitation speed of 150 rpm. The maximum metal removal efficiencies obtained were 99.7%, 100%, 99%, 90% and 85% for Mn$^{2+}$, Fe$^{2+}$, Zn$^{2+}$, Pb$^{2+}$ and Cr$^{2+}$, respectively. The average adsorption capacity for Mn$^{2+}$ was 0.44 mg/g, 26.7 mg/g for Fe$^{2+}$, 78.5 mg/g for Zn$^{2+}$, 0.133 mg/g for Pb$^{2+}$ and 10.36 mg/g for Cr$^{2+}$. CBC is an efficient and low cost adsorbent for remediating heavy metals from galvanic wastewater.

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326