



Removal of heavy metals from pharmaceutical effluents using (Activated Charcoal) rice husk

Moronkola Bridget Adekemi, Alegbe Monday John, Alege Justinah Racheal, Asimi Awonike Julianah, Dosunmu Olufemi Michael

Department of Chemistry, Lagos State University, Ojo Campus, Lagos-Badagry Express Way, Lagos State, Nigeria

Abstract

Pharmaceutical effluents are significant sources of environmental pollution, due to their complex composition and toxic heavy metal content, which threaten the aquatic ecosystems and human health. The aim of the study was to remove zinc (Zn), chromium (Cr), and iron (Fe) from pharmaceutical effluents, using activated rice husk charcoal derived from *Oryza glaberrima* as a low-cost and sustainable adsorbent. The rice husk charcoal was chemically activated and characterised using Fourier Transform Infrared Spectroscopy (FTIR), X-Ray Diffraction (XRD), Thermogravimetric analysis (TGA), Brunauer-Emmett-Teller (BET), Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM). The results for FTIR of the rice husk ash before adsorption showed the spectrum at 3227.88 cm^{-1} (O-H stretching), 2877.51 cm^{-1} (C-H stretch), 2072 cm^{-1} (C≡C stretch) or (C≡N), 1591 cm^{-1} (N – H bending), 1379.35 cm^{-1} (C – H bending), 1017.52 cm^{-1} (c – O stretching), or (Si – O stretching). X-ray diffraction (XRD) identified a mineral composition of Silicon dioxide 86.9%, Osmilite 10.41%, Graphite 2.63%, and Hanksite 0.09% for the rice husk ash sample. The Brunauer-Emmett-Teller (BET) for the pore size of the rice husk ash indicates an average pore of 7 diameter of 2.647 nm, which classifies the material as mesoporous, its cumulative surface area is $7.805\text{ m}^2/\text{g}$ -which is relatively moderate for materials with mesopores, and the pore volume of $0.953\text{ cm}^3/\text{g}$ suggests that the material has a significant amount of internal space for storing gas or liquid. In the thermogravimetric analysis (TGA), it was discovered that the rice maintains its stability, up to 250°C before significant degradation occurs. The batch adsorption experiments were conducted to assess removal efficiency under varying operational parameters, including adsorbent dosage, contact time, and pH. Heavy metal concentrations were determined using Atomic Absorption Spectroscopy (AAS). Removal efficiency increased with higher adsorbent dosage, with an optimum of 1.0 g per 50 mL. Maximum adsorption occurred at 60 minutes. Optimal pH values were 2.0 for Zn and Fe, and 4 for Cr, indicating pH-dependent adsorption. The removal demonstrates that activated rice husk charcoal is an effective, economical, and environmentally-sustainable material for heavy metal removal from pharmaceutical wastewater, while also promoting agricultural waste valorisation.

Keywords: Pharmaceutical effluents, heavy metal, activated rice husk, adsorption

Introduction

Pharmaceutical production industrialisation has increased significantly and contributed to environmental pollution. (Tchounwou *et al.*, 2015) ^[25]. Pharmaceutical effluents are complex mixtures containing organic compounds, active pharmaceutical ingredients, and inorganic contaminants, such as heavy metals, which pose serious environmental and public health risks (Patel *et al.*, 2019). Heavy metals are of particular concern, due to their non-biodegradable nature, toxicity, and ability to bioaccumulate in living organisms (Tchounwou *et al.*, 2015) ^[25].

Pharmaceutical manufacturing processes utilise various raw materials, catalysts, and additives that introduce heavy metals, such as lead (Pb), cadmium (Cd), chromium (Cr), and nickel (Ni) into wastewater streams (Aydin & Aydin, 2017). When discharged into aquatic environments, these metals can persist for long periods and enter the food chain, resulting in adverse health effects, including neurological damage, kidney dysfunction, and carcinogenic outcomes (Jaishankar *et al.*, 2015) ^[16].

Conventional Methods for Heavy Metal Removal

Conventional wastewater treatment methods, such as chemical precipitation, membrane filtration, and ion exchange, are often costly and generate secondary pollutants. Consequently, there is increasing interest in adsorption-based methods due to its low cost and its being

environmentally-friendly (Bhatnagar *et al.*, 2015) ^[8]. Agricultural wastes have emerged as promising alternatives, due to their availability, renewability, and adsorption potential. Rice husk, an abundant agricultural by-product in rice-producing regions in Nigeria, has gained attention, as a precursor for activated charcoal. When properly carbonised and activated, rice husk charcoal exhibits a high surface area, with functional groups capable of binding heavy metals from aqueous solutions (Kumar *et al.*, 2018) ^[17]. This study, therefore, investigates the use of activated rice husk charcoal as an efficient and sustainable adsorbent, for the removal of heavy metals from pharmaceutical effluents.

Heavy Metals in Pharmaceutical Wastewater

Heavy metals are elements with relatively high density and atomic weight that exhibit toxicity at low concentrations. In pharmaceutical wastewater, metals such as Pb, Cd, Cr, and Ni originate from synthesis processes, equipment corrosion, and formulation additives (Zhang *et al.*, 2020) ^[28]. Their presence in water bodies can impair aquatic life and contaminate drinking water sources, making their removal a critical environmental concern.

Sources and Toxicity of Heavy Metals

The primary sources of heavy metals in pharmaceutical effluents include raw material impurities, catalysts, and industrial equipment. Lead exposure affects neurological

development, cadmium damages kidneys and bones, chromium (VI) is highly carcinogenic, and nickel exposure may result in respiratory and dermatological disorders (Tchounwou *et al.*, 2015; Jaishankar *et al.*, 2015) ^[16, 25]. Because heavy metals cannot be degraded biologically, their accumulation poses long-term environmental and health challenges.

Rice Husk as an Agricultural Waste Material

Rice husk constitutes approximately 20 % of rice grain weight and is often discarded as waste. It contains high carbon and silica content, making it suitable for conversion into activated charcoal (Kumar *et al.*, 2018) ^[17]. Utilising rice husk for wastewater treatment, not only reduces environmental waste, but also promotes sustainable resource management.

Activated Rice Husk Charcoal as an Adsorbent

Activated rice husk charcoal is produced through carbonisation, followed by physical or chemical activation, which enhances porosity and surface functional groups. Studies have demonstrated its effectiveness in removing heavy metals, such as Pb, Cd, and Cr from aqueous solutions (Hossain *et al.*, 2016; Singh *et al.*, 2019) ^[14, 24]. Adsorption data often conform to Langmuir and Freundlich's isotherm models, indicating favourable adsorption behaviour.

Aim and Objectives of the Study

Aim

The aim of the research is to remove toxic elements from pharmaceutical effluents using activated rice husk charcoal.

Objectives

- To prepare and activate rice husk charcoal.
- To Characterise the activated rice husk using the following Analytical Instruments, such as Fourier Transform Infrared (FTIR), Scanning Electron Microscope (SEM), Thermogravimetric Analysis (TGA), Buaner Emitte Teller (BET), X – ray diffraction (XRD), X-Ray Fluorescence (XRF), and Transmission Electron Microscopy (TEM).
- The study of the physicochemical properties of the activated adsorbent.
- To determine heavy metal concentrations in pharmaceutical effluents before and after treatment.
- To optimise the effects of pH, contact time, and adsorbent dosage on adsorption efficiency.

Materials and Methods

Description of the Study Area

This study was conducted under laboratory conditions in the Department of Chemistry, Lagos State University (LASU), Nigeria. Lagos State is a major industrial and pharmaceutical hub in Nigeria, hosting numerous pharmaceutical manufacturing companies whose effluents contribute significantly to environmental pollution. The choice of this location is justified by the availability of pharmaceutical effluent sources, laboratory facilities, and its relevance to local environmental challenges.

Materials, Reagents, and Chemicals

The materials used in this study included raw rice husk, pharmaceutical effluent samples, distilled water, and

laboratory glassware. Analytical-grade reagents were used throughout the experiment to ensure accuracy and reproducibility.

Reagents used included nitric acid (HNO₃), hydrochloric acid (HCl), sodium hydroxide (NaOH), phosphoric acid (H₂PO₄) or potassium hydroxide (KOH) for activation, and standard metal solutions for calibration. All chemicals were of analytical grade and sourced from reputable suppliers.

Equipment and Instruments Used

The equipment and instruments employed in this study included:

- Muffle furnace
- Hot air oven
- Analytical balance
- pH metre
- Mechanical shaker
- Atomic Absorption Spectrophotometre (AAS)
- Desiccator
- Sieves of different mesh sizes
- Filtration apparatus

Sample Collection of Pharmaceutical Effluents

Pharmaceutical effluent samples were collected from the discharge outlet of a selected pharmaceutical manufacturing company in Lagos State. Samples were collected in clean, acid-washed polyethylene containers to avoid contamination. The containers were rinsed with the effluent prior to final sample collection. Samples were transported to the laboratory and stored at 4 °C prior to analysis (APHA, 2017) ^[5].

Sample Preservation and Preparation

Immediately after collection, effluent samples were acidified with concentrated nitric acid to a pH below 2, to prevent metal precipitation and microbial activity. The samples were then filtered to remove suspended solids before heavy metal analysis. Preservation and preparation were carried out in accordance with standard methods for water and wastewater analysis (APHA, 2017) ^[5].

Preparation of Rice Husk Charcoal

Raw rice husk was collected from a local rice mill, washed thoroughly with distilled water to remove dirt and impurities, and air-dried for several days. The dried rice husk was then oven-dried at 105 °C to remove moisture. Carbonisation was carried out by heating the dried rice husk in a muffle furnace at temperatures between 400–500 °C for 2 hours in the absence of oxygen. The resulting charcoal was cooled in a desiccator, crushed, and sieved to obtain uniform particle size (Hossain *et al.*, 2016) ^[14].

Chemical Activation

Chemical activation was carried out by impregnating the rice husk charcoal with phosphoric acid (H₃PO₄) or potassium hydroxide (KOH) in a specific ratio. The mixture was allowed to soak for 24 hours, followed by heating in a furnace at 500–600 °C. After activation, the charcoal was washed repeatedly with distilled water until a neutral pH was attained, and then oven-dried (Hossain *et al.*, 2016; Singh *et al.*, 2019) ^[14, 24].

Characterisation of Activated Rice Husk Charcoal pH Determination

The pH of the activated rice husk charcoal was determined by mixing a known mass of the adsorbent with distilled

water and measuring the pH using a calibrated pH metre (APHA, 2017) [5].

Surface Area and Porosity

Surface area and porosity were estimated using standard adsorption techniques, as these properties influence adsorption capacity. Increased surface area enhances the availability of adsorption sites (Foo & Hameed, 2018) [11].

Moisture and Ash Content

Moisture content was determined by oven-drying a known mass of the adsorbent at 105 °C, while ash content was determined by heating the sample at 750 °C in a muffle furnace. These parameters provide information on adsorbent quality (Kumar *et al.*, 2018) [17].

Determination of Heavy Metals in Effluent Samples

Heavy metal concentrations (Pb, Cd, Cr, and Ni) in effluent samples were determined using Atomic Absorption Spectrophotometry (AAS) after acid digestion. Calibration curves were prepared using standard solutions, and metal

concentrations were quantified, following standard analytical procedures (APHA, 2017) [5].

Batch Adsorption Experiment

Batch adsorption experiments were conducted by adding a known mass of activated rice husk charcoal to a fixed volume of pharmaceutical effluent in conical flasks. The mixtures were agitated using a mechanical shaker at room temperature. After agitation, samples were filtered, and residual metal concentrations were analysed using AAS (Foo & Hameed, 2018) [11].

Effect of Operational Parameters on Adsorption

Effect of pH

The effect of pH on adsorption was studied, by adjusting effluent pH between 2 and 10 using dilute HCl or NaOH. The pH influences metal speciation and surface charge of the adsorbent (Bhatnagar *et al.*, 2015) [8]. Adsorption efficiency was evaluated to determine the optimal pH for maximum heavy metal removal.



Fig 1: pH Samples and Electric shaker

Effect of Contact Time

Contact time experiments were conducted at various time intervals to determine the equilibrium time required for maximum metal removal. Samples were withdrawn at predetermined intervals and analysed (Foo & Hameed, 2018) [11].

A fixed mass of activated charcoal was added to a metal solution of known concentration. The mixture was agitated using an orbital shaker at 150 rpm. Samples were withdrawn at intervals of 20, 40, 60, 80, 100, and 120 minutes, filtered, and analysed for residual metal concentration.

Effect of Adsorbent Dosage

Different doses of activated rice husk charcoal (0.5 – 5.0 g per 100 mL solution) were tested, to determine the effect of adsorbent dosage on metal removal efficiency. Increasing dosage generally increases removal efficiency, due to the increased surface area (Kumar *et al.*, 2018) [17].

Results

Characterisation

Transmission Electron Microscopy (TEM) morphology of rice husk carbon

The FTIR spectra of Rice Husk Charcoal indicates the presence of several functional groups, based on the

characteristic absorption bands. The major peaks of the carbon rice husk were represented in figure 2.0. The absorption spectra, between 689 cm⁻¹ and 1650 cm⁻¹, represent C-O and the presence of alkene stretching. The band, at 1364 cm⁻¹, shows the band is amine, the peak at 1718 cm⁻¹, shows C=O stretch. While, the band at 3339 cm⁻¹, refers to O-H group. The peak, at 2850 cm⁻¹, represents the presence of alkanes.

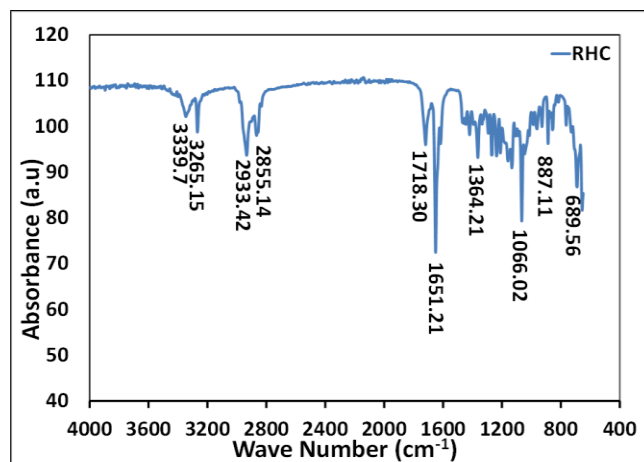


Fig 2: Fourier Transform Infrared Spectroscopy (FTIR) absorption band of rice husk carbon

Scanning Electron Microscope (SEM)

This image in fig 3.0 shows the scanning electron microscope (SEM) and the X –ray diffraction, which

provides detailed information about the surface topography and morphology of the RHC particles and rough surface made up of irregularly shaped particles.

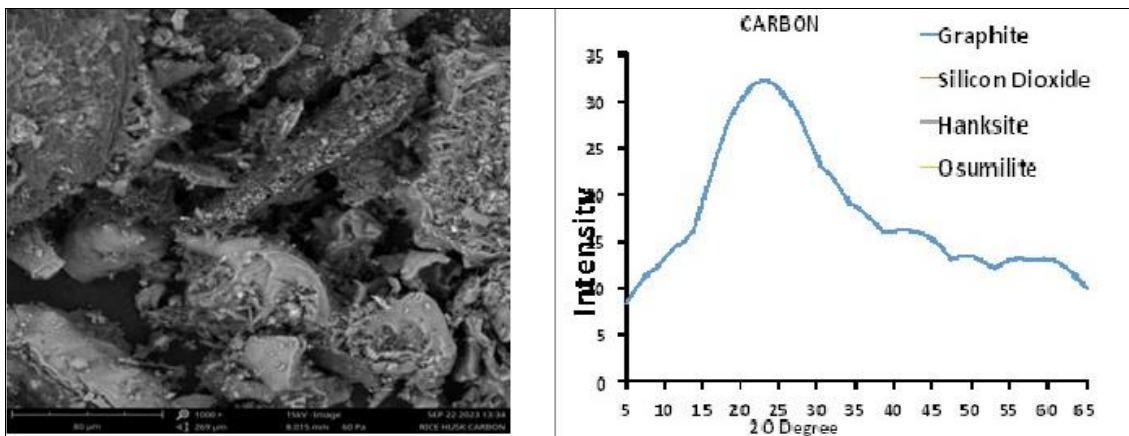


Fig 3: Scanning Electron Microscopy (SEM) images of rice husk carbon and X-Ray Diffraction spectrum of rice husk carbon

X-Ray Diffraction mineral phase composition of Rice Husk Charcoal

On the XRD Bar chart, graphite was the largest percentage of the minerals present in the rice husk, and this indicates the effectiveness of the activated charcoal of the rice husk, since graphite contributes to the high surface area and the adsorption capacity.

100°C, there is a slight weight loss in the blue line curve, corresponding to the initial red line peak around 70°C. This is typically attributed to the evaporation of moisture or adsorbed water from the sample. Between 350 - 450°C, there is a significant weight loss in the blue line and a large, sharp peak in the red curve line around 350°C. This may be due to the decomposition of some organic components and the blue line continues to show gradual weight loss, as the temperature increases.

Thermogravimetry Analysis (TGA)

The TGA in fig. 4.0 shows the study of the weight loss of the carbon rice husk; at 200°C, the particles were stable. At

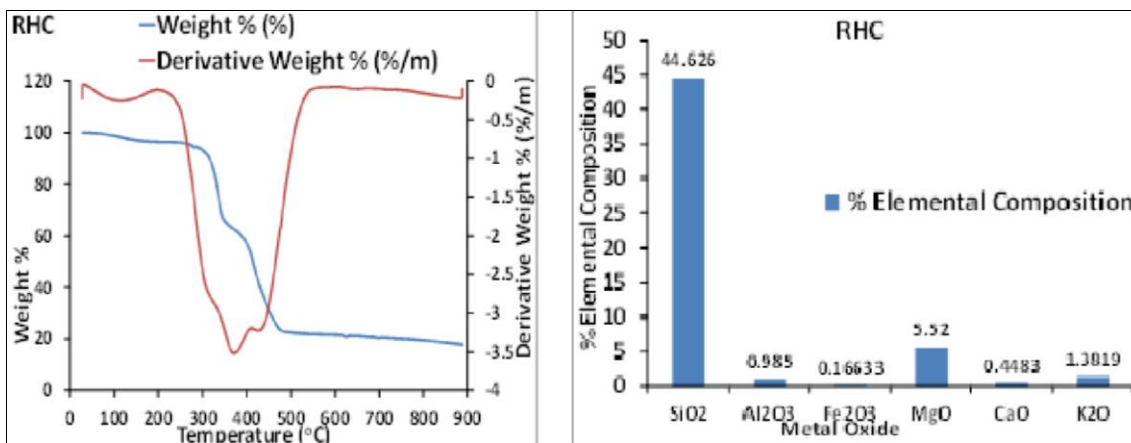


Fig 4: Thermogravimetric Analysis (TGA) of rice husk carbon and figure Brunauer-Emmett-Teller (bet) micropore of rice husk charcoal

The Elemental Composition of the Carbon Rice Husk

Fig. 5.0 shows the X-ray fluorescence (XRF) analysis, which confirmed that carbon and silica were the major constituents of the rice husk carbon, with minor traces of metal oxides. The low presence of intrinsic heavy metals suggests that the adsorbent itself does not introduce secondary contamination into treated effluents. Additionally, silica contributes to mechanical strength and enhances resistance to attrition, which is beneficial for repeated adsorption cycles (Ioannidou & Zabaniotou, 2017) [15]. The % elemental composition of the activated carbon rice husk shows the presence of SiO₂ with 44.6%, MgO has 5.52%, K₂O 1.46%, Al₂O₃ is 0.98%, and CaO as 0.45%.

The graph displays the micropore size distribution for rice husk charcoal (RHC), indicating that the material primarily consists of micropores within a very narrow size range. According to IUPAC classification, pores with a diameter of less than 2 nm are micropores, and those between 2 nm and 50 nm are mesopores. The peak around 3 nm signifies that the material is predominantly microporous to small mesoporous. This narrow distribution suggests a relatively uniform pore structure. Therefore, the presence of a high volume of small pores and a high specific surface area are typical characteristics of activated charcoal derived from rice husks, making them effective for carrying out the application of adsorption.

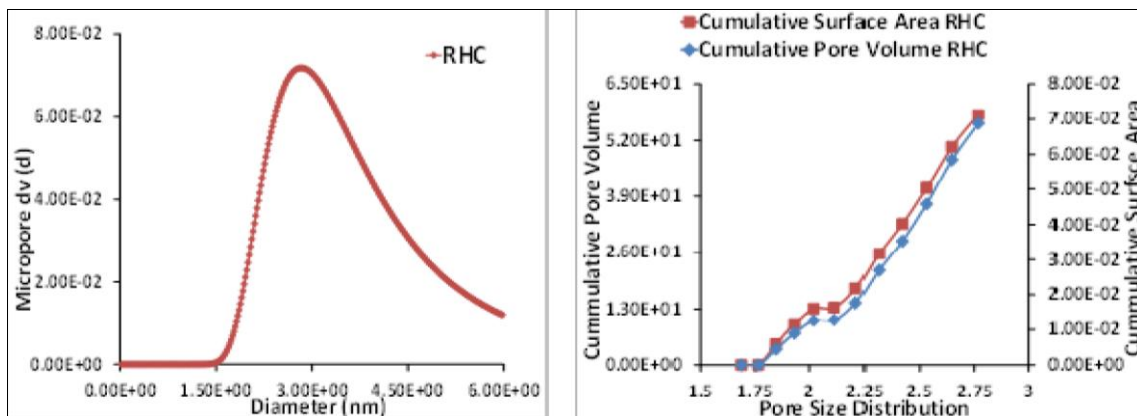


Fig 5: Brunauer-Emmett-Teller (BET) Micropore of rice husk carbon and BET Pore Size Distribution of rice husk carbon

BET Isotherm of Rice Husk Charcoal

The fig. 5.0 graph above displays a linear isotherm. The higher the relative pressure, the higher the volume at STP; which means the particles have various layers, and the first layer will be good for adsorption.

BET Langmuir of rice husk charcoal indicates a monolayer, because the graph is slightly curved, which is potentially an effective adsorbent for applications where a monolayer adsorption mechanism is desired, or applicable.

The Effect of Dosage

The effect of dosage on fig. 6.0: Depends on the quality of the adsorbent to the biosorption of metals in aqueous media. The adsorption experiment was varied using a 0.2g to 1.0 g dose of the rice husk, the removal efficiently increased with an increasing amount to 1.0 g and remained constant, as it reached equilibrium, due to the functional group on the adsorbent (Yu *et al.*, 2000). The percent removal efficiency was 96.32% with optimised dosage of 0.6 g of the activated rice husk charcoal.

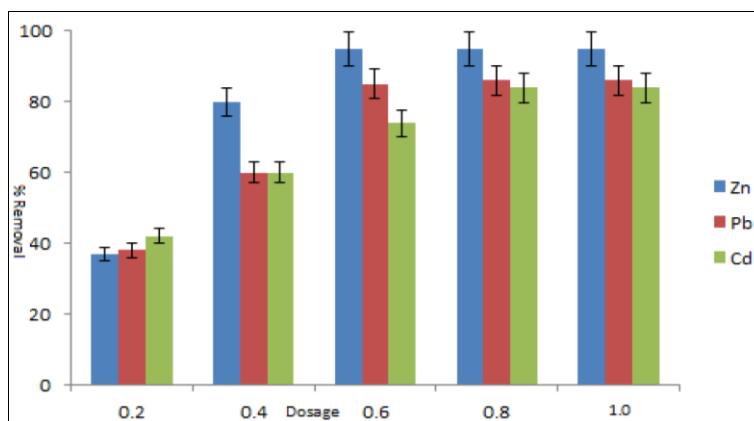


Fig 6: The effect of dosage on the removal of heavy metals from pharmaceutical effluents using (activated rice husk charcoal)

The effect of pH

The effect of the pH of metal ion depends on the solution, as shown in fig. 7.0 below. The study was between pH 2 and 10, in the presence of metals such as Zn, Pb, and Cd. The optimum pH adsorption was observed at pH 2, with a maximum removal of 79, 97, and 97 %, respectively,

due to the competition between the functional groups on the rice husk charcoal adsorbent. The degradation occurs between pH 6 and 10. This is due to the accumulation of metals Zn, Pb, and Cd, from the pharmaceutical effluent on the activated rice husk adsorbent (Igwe *et al.*, 2006).

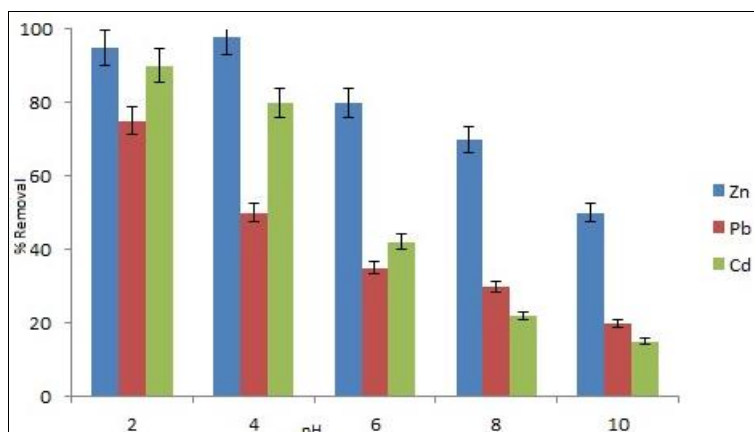


Fig 7: The effect of pH on the removal of heavy metals (Zn, Pb and Cd) from pharmaceutical effluents, using activated charcoal rice husk

Optimisation of time for heavy metals (Zn, Pb and Cd) onto from pharmaceutical effluents using (activated rice husk charcoal).

Time plays an important role in the percentage removal of heavy metals Zn, Pb, and Cd from pharmaceutical effluents, at a pH of 2.0; as shown in 6.0 c below, the heavy metal, Zn,

is the highest at 97.6 %, followed by Pb at 88.4 %, and Cd at 86.9 %, respectively at 60 minutes. The uptake of the heavy metals onto activated rice husk was due to the diffusion of metals from the bulk of the pharmaceutical solution onto the surface of the adsorbent, which is described as *instantaneous sorption*.

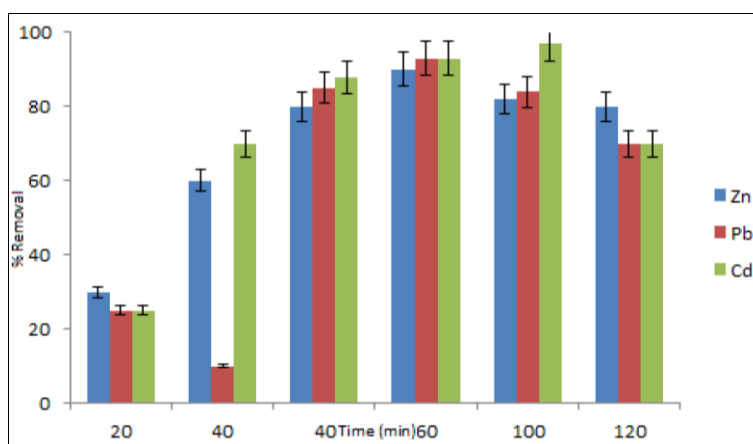


Fig 8: The effect of time on the removal of heavy metals (Zn, Pb and Cd) from pharmaceutical effluents using (activated charcoal) rice husk pH 2.0 at 60 min n =3

The re-usability of rice husk was shown in Fig 6.0 d: which was carried out to investigate the feasibility of the adsorbent. The activated rice husk can be reused for two times, with no significant change in the efficiencies of

adsorption and desorption. The decline in efficiencies after the second run of the activated rice husk charcoal could be attributed to the loss of the amount of the sorbent during usage (Chiban *et. al.*, 2006).

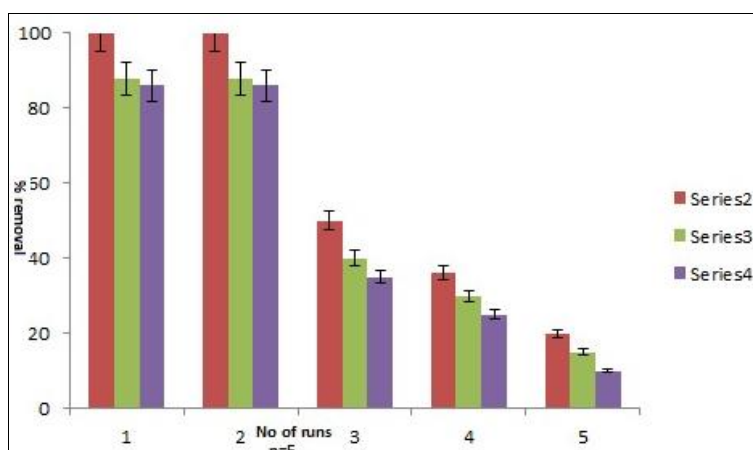


Fig 9: Shows the re-usability for heavy metals (Zn, Pb and Cd) from pharmaceutical effluents using (activated charcoal) rice husk. Optimized dosage 0.6 g, pH 2.0 and time, 60 mins

Conclusion

The characterisation results confirm that the prepared adsorbent possesses desirable properties, including high carbon content, well-developed porosity, abundant surface functional groups, good thermal stability, and a high surface area.

The XRD analysis confirms the formation of carbon-rich phases, while the SEM and TEM analyses revealed a porous structure suitable for adsorption. FTIR results showed the presence of functional groups responsible for metal binding, and BET analysis confirmed the availability of extensive adsorption sites. These properties collectively support the suitability of activated rice husk charcoal as a low-cost, efficient, and environmentally-friendly material for wastewater treatment. The batch adsorption experiment showed that both pH and the dosage, using activated rice husk charcoal, significantly influenced the efficiency of zinc

removal, as shown in the presence of metals, such as Zn, Pb, and Cd. The optimum pH for adsorption was observed at pH 2, with a maximum removal of 79, 97, 97 %, respectively, due to the competition between the functional groups on the rice husk charcoal adsorbent.

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