International Journal of Environmental Sciences
ISSN: 2229-7359
Vol. 11 No. 8, 2025
https://theaspd.com/index.php

Single/Competitive Adsorption Removal Of $Pb^{2+}/Cu^{2+}/Ni^{2+}$ By Oxygenated Activated Carbon From Synthetic And Industrial Wastewaters

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Abstract
Industrial wastewaters contain heavy metals that should be safely removed before disposal. Adsorption removal by activated charcoal is commonly used because of its cost-effectiveness and high adsorption capacity. Competition between metals requires raising the charcoal adsorption capacity.

The objective of the current study is to use wood from Salix trees to produce Activated Charcoal and then use H_2O_2 *reactivation treatment to raise its adsorption capacity by developing Oxygenated Activated Charcoal (O-AC). O-AC is then used to remove* Pb^{2+}, Cu^{2+} *&* Ni^{2+} *from synthetically polluted solutions, and in steady-state fixed-bed two-column system for removing these metals and other salts from industrial wastewater to concentrations below the Maximum Permissible Limits (MPLs).*

Oxygenation treatment increased the EC of O-AC by +110%, surface charge by +177.7%, specific surface area by +73.9% and pore size by +31.4%. Best adsorption capacity was obtained at contact time of 40 minutes, O-AC dose of 0.1 g/500 ml, and solution pH 5 – 7. For initial concentrations around 40 mg/l for each of the studied metals, the net overall average adsorption capacity for the studied metals in single-, bi- & tri-metal solutions was 173.71 mg/g, which is almost four times higher than published studies. Fixed-bed system removed Lead, Copper, and Nickel & TDS concentrations from 22.0, 28.0, 18.62 & 1350.0 mg/l, down to 0.01, 1.0, 0.1 & 138.54 mg/l; respectively, which are below the MPLs. It is recommended to use the developed O-AC fixed-bed two-column system for competitive removal of the studied metals from industrial wastewaters with similar conditions.

Keywords: *Industrial wastewater treatment; Lead–Copper–Nickel; Oxygenated Activated Charcoal/Carbon; Adsorption Removal.*

1-INTRODUCTION

Industrial wastewaters usually contain heavy metals, which can cause serious hazards to the biota, and should be removed before disposal to the public drainage system (He et al., 2023; Ighalo et al., 2020). Removal of pollutants by adsorption on the charged surfaces of activated charcoal/carbon that is obtained from Salix wood is used because of its cost-effectiveness and high adsorption capacity (Elsaid et al., 2024; Elsaid and Anwar, 2022). Salix wood is abundant and is considered as waste material that can be used instead of being dumped. Competition between co-existing heavy metals on charcoal surface adsorption sites may result in the removal of only some of these metals, which requires higher adsorption capacity for the adsorbent material (Zhao et al. 2023). So, the current study prepared Activated Charcoal/Carbon (AC) from Salix wood and reactivated it using treatment with Hydrogen Peroxide (H_2O_2) that inserted Oxygen atoms in the AC crystal lattice structure (Sánchez et al., 2006). This produced Oxygenated Activated Charcoal (O-AC) with largely increased adsorption capacity. The current study focuses on the removal of Lead (Pb^{2+}), Copper (Cu^{2+}) & Nickel (Ni^{2+}) that coexist in the wastewater obtained from El-Ekhtiar Textile Factory in Riyadh; Kingdom of Saudi Arabia.

In addition, the Maximum Permissible Limits (MPLs) for heavy metals (including Pb^{2+}, Cu^{2+} & Ni^{2+}) are very low (WHO 2022), whereas the pollutants' residual concentrations in the treated solutions are affected by the initial concentrations (Elsaid et al., 2024; Elsaid and Anwar, 2022). This requires the adsorption removal treatment to be done on two steps, which allows for lower initial concentration into the 2nd step in order to allow for pollutants' removal below the MPLs.

The objectives of this work are (1) to develop the Oxygenated Activated Charcoal/Carbon (O-AC), (2) to examine its physico-chemical properties, (3) to test its performance in competitive adsorption removal of Pb^{2+}, Cu^{2+} & Ni^{2+} from synthetic solutions in batch experiments and (4) to examine the use of fixed-bed dual-stage two-column system for steady-state removal of these metals and other salts from industrial wastewater to concentrations below the MPLs.

2- MATERIALS & METHODS

Experimental works of the current study were done at Technical Colleges' Laboratories, and Chemical Engineering Department Laboratories at Onizah Colleges; Al-Qassim; Kingdom of Saudi Arabia.

The current study used wood from Salix trees to develop activated Charcoal/Carbon (AC), reactivate after treatment with Hydrogen Peroxide (H_2O_2) that inserted Oxygen atoms in the AC crystal lattice structure to develop Oxygenated Activated Charcoal/Caron (O-AC). Properties of AC and O-AC are then studied. Synthetically-polluted stock solutions with Lead (Pb^{2+}), Copper (Cu^{2+}) & Nickel (Ni^{2+}) are prepared in laboratory for use in preparation of single- bi- & tri-metal polluted solutions for use in batch experiments. Batch experiments are then used to examine the effects of solution pH, O-AC dose and contact time in single-, bi- & tri-metal polluted solutions on competitive adsorption removal.

The current study also obtained industrial wastewater from a textile factory for testing the removal of the studied metals using a fixed-bed two-column steady-state treatment system.

2-1- Development and Characterization of Adsorbent (AC & O-AC)

1000 g from Salix wood tree was carbonized at 800 °C with heating rate of 5 °C/min under Nitrogen (N_2) flow (100 ml/min) for 4.0 h to ensure total carbonization, and then was washed with deionized water to produce granule Activated Charcoal/Carbon (AC), as described by Deng et al. (2010-a).

The AC was then added to a 1000 ml of Nitric acid (HNO_3, 70%), Ammonium Persulfate ($(NH_4)_2S_2O_8$), Sulfuric Acid (H_2SO_4, 97%), and Hydrogen Peroxide (H_2O_2, 30%), and was shaken for 6.0 hours, and was washed again with deionized water. The AC was then carbonized again at 800 °C with heating rate of 5 °C/min under Nitrogen (N_2) flow (100 ml/min) for 4.0 h to produce the Oxygenated Activated Charcoal/Carbon (O-AC). The Hydrogen Peroxide (H_2O_2, 30%) was used in the second carbonization step to oxidize the surface of activated charcoal/carbon in order to improve its ability to adsorb the studied metals Murtazaoğlu et al. (2023).

Experiments were done to obtain the physico-chemical properties for the AC & O-AC following the methodology described by Murtazaoğlu et al. (2023). The SEM (Scanning Electron Micrograph) test was done for both the AC & O-AC using Cambridge S-360 Scanning Electron Microscope (SEM), which was operated at 16kv and 2.5 Å (0.00025 micrometers). It performed x-ray magnification of 1000X, and computer software visual magnification of 25X, with the total magnification of 25000X. This test makes it possible to visually compare the surface morphology of AC to that of O-AC after the oxygenation treatment.

2-2- Preparation of Synthetically Polluted Wastewater Solutions

Synthetically polluted wastewater solutions were prepared for use in batch experiments to determine the optimal values for contact time, activated charcoal/carbon dose and solution pH for adsorption removal from single-, Bi- & tri-metal polluted solutions. Synthetically polluted wastewater solutions were prepared by dissolving each of the studied metals (Lead – Pb^{2+}, Copper – Cu^{2+} & Nickel – Ni^{2+}) in distilled water to make stock solutions. Each of the stock solutions was at concentration around 40.0 mg/l. Single-metal polluted solutions were made directly from the corresponding stock solution for each metal. Bi-metal wastewater polluted solutions were made using 1/2 to 1/2 aliquots by volume of the corresponding metal-polluted stock solutions. Meanwhile, tri-metal wastewater polluted solutions were made using 1/3 to 1/3 to 1/3 aliquots by volume from the stock solutions of the corresponding three metals. Samples were taken from the prepared solutions and were analyzed to determine the actual polluting metal concentrations (Lead – Pb^{2+}, Copper – Cu^{2+} & Nickel – Ni^{2+}).

2-3- Collection of Industrial Wastewater Sample

Industrial wastewater sample was collected during the morning hours of August 2023 from El-Ekhtiar Textile Factory in Riyadh; Kingdom of Saudi Arabia. The sample was kept in the dark and was transported to the laboratory, where the chemical investigations were conducted, and wastewater properties and pollutants' concentrations were determined.

2-3- Batch Experiments

Batch experiments were done as described by Elsaid et al. (2024) to determine the optimal values for contact time, O-AC dose and solution pH that achieve best adsorption removal of the studied metals (Pb^{2+}, Cu^{2+} & Ni^{2+}) from polluted solutions. Batch experiments were also used to study single-metal versus competitive behavior in bi- & tri-metal solutions.

500 ml of the synthetically polluted solution (single, bi- or tri-metal solution) was put in a conical flask and was shaken at steady rate of 1000 rpm for up to 2.0 hours. Samples of 1.0 mm^3 were taken every 10 minutes and at the end of the 2.0 hours for analysis of residual polluting metal(s) concentration(s) in solution using an Elmer-Berkin UV spectrophotometer. In order to study the effect of O-AC dose (weight), the charcoal dose was varied in the experimental runs from 0.01 to 2.0 g. For studying the effect of solution pH on adsorption, the pH was adjusted from 2.0 to 10.0 by adding 0.1M HCl or 0.1M NaOH to the polluted solution.

2-4- Adsorption Capacity Calculations

Adsorption capacities of the Oxygenated Activated Charcoal/Carbon (O-AC) for the studied metal(s) were calculated as (Ullah et al., 2020):

$$Adsorption\ Capacity\ = (C_o - C_e) \times \frac{V}{W} \quad \text{......................} \text{ Eq. (1)}$$

Where;

Adsorption Capacity (mg/g): is the capacity of O-AC to adsorb the studied metal(s) in the studied solution (single-, bi- or tri-metal solutions).

C_o (mg/l): is the initial concentration of metal(s) Pb, Cu, Ni in the polluted solution.

C_e (mg/l): is the treated equilibrium concentration of metal ion(s)in the treated solution.

V (l): the total volume of polluted solution.

W (g): adsorbent dose/mass (Oxygenated Activated Charcoal/Carbon).

2-5- Fixed-Bed Dual-Stage Two-Column experiment

Fixed-bed columns are used for adsorption removal of pollutants from wastewaters under steady flow, in order to lower pollutants' concentrations in treated water to values below the Maximum Permissible Limits (Elsaid et al., 2024; Elsaid and Anwar, 2022). Fixed-bed dual-column two-stage experiment is used in the current study for adsorption removal of the metals (Pb^{2+}, Cu^{2+} & Ni^{2+}) from industrial polluted wastewater that is obtained from El-Ekhtiar Textile Factory in Riyadh; Kingdom of Saudi Arabia.

Figure (1) shows a schematic description of the dual-stage two-column treatment system. Industrial wastewater obtained from Riyadh Textile Factory was stored in the reservoir tank (to the left). Wastewater was then pumped to the top of the column using a peristaltic pump at the steady rate of 30 ml/min, which was maintained throughout the entire experiment. Flow-control & sampling valves at the inlet & outlet of each of the two columns are used to maintain the steady flow rate and to collect water samples for analyses. Each of the two treatment glass columns has an internal diameter of 5.5 cm and a height of 90 cm and holds 10 grams of activated charcoal, equivalent to a height of 15 cm inside the column. The bottom and top of the cylindrical column were covered with 5 mm thick glass wool and a layer of glass beads respectively.

The fixed-bed experiment was run for 160 minutes (2.0 hours and 40 minutes). The first sample of treated water was taken after 40 minutes to allow for sufficient contact time. Samples were taken in 5 replicates every 10 minutes and continued for the duration of 2.0 hours, with sample volume of

volume 1.0 mm^3. Chemical analysis of wastewater samples was conducted to determine the concentrations of the studied metals (Pb^{2+}, Cu^{2+} & Ni^{2+}) and other dissolved salts at the outlets of the two columns.
Insert Figure (1) here.

3- RESULTS AND DISCUSSION

3-1- The Characteristics of Oxygenated Activated Carbon (O-AC)

Treatment of the Activated Charcoal/Carbon with Hydrogen Peroxide (H_2O_2) as an oxidizing solution in a second carbonization process leads to the penetration of oxygen atoms into the crystal lattice of activated carbon (Ang et al. 2020). Insertion of Oxygen atoms with a pair of lone electrons enhances the surface negative charge, improves its functionality, and raises its ability to adsorb positively-charged metals (Ang et al., 2020). This generates Oxygenated Activated Charcoal/Carbon (O-AC) which is used by the current study for adsorption removal of lead, copper & nickel from polluted wastewater. The modification in Oxygenated Activated Charcoal (O-AC) crystal lattice structure as compared to the Activated Charcoal (AC) is schematically illustrated in figure (2).
Insert Figure (2) here.
Insertion of Oxygen atoms in the Charcoal lattice structure fill the vacant locations in single & double bonds with Carbon and Hydrogen atoms within the Charcoal organic compounds (mainly Lignin, Pectin and Cellulose). This results in filling the atomic vacancies and in stable electric distribution, which in turn results in thermally stable Oxygenated Charcoal Lattice (Legrand et al., 2016).
Figure (3) shows the Scanning Electron Micrograph (SEM) of the Oxygenated Activated Charcoal (O-AC) as compared to Activated Charcoal (AC). In using the Cambridge S-360 Scanning Electron Microscope (SEM), the X-ray magnification is at1000X, and computer software visual magnification is at 25X, with the total magnification of 25000X. Comparison between (a) AC and (b) O-AC surface in figure (3) shows that the oxygenation treatment made the O-AC charcoal surface pores in (b) is much larger and more uniform than the non-oxygenated in (a). This results in larger number of adsorption sites on the surface with larger pores where heavy metals can fit in. The additional negative charges from inserted oxygen atoms in the O-AC lattice, together with the improved surface pores work together on enhancing the O-AC adsorption capacity for the studied metals.
Insert Figure (3) here.
In a previous study (Elsaid et al., 2024), we tested the surface functional groups of the Charcoal obtained from Salix wood. We used "Fourier Transform Infrared Spectra" - FTIR Test, and the thermal stability using "Thermo-Gravimetric Analysis" - TGA Test.
Elsaid et al. (2024) FTIR test of the Charcoal surface showed the existence of symmetric aliphatic amino group (N-H), alcohols, phenols, and carboxylic acids' groups (O–H), carboxylic acid and carbonylic functional branches' groups (O-H (COOH)), aldehydes, ester or ketones and carboxyl groups (carbonyl C=O), peptide amide groups (N- protein). Charcoal activation treatment uses these groups to enhance the charcoal surface charges and raise its adsorption capacity. This shows that activation of this charcoal will result in adsorption capacity that is suitable for use in adsorption removal of pollutants from solutions.
Meanwhile, TGA test reflects thermal stability/dissociation of the charcoal components, which reflects their stability against volatilization. Elsaid et al. (2024) test showed that hemo-cellulose was totally lost at 200°C, while cellulose and lignin sustained heating to 200°C with little weight loss, and that loss rate increased from 200 to 500°C, which became less from 500 to 700°C. However, pectin was more persistent and suffered little loss during the test range. Cellulose is responsible for high surface area and porosity, while lignin is the main source of negative charges, while pectin is a good adsorbent. These results indicate that the charcoal is quite stable will not dissociate during the adsorption removal process.
Table (1) shows the physico-chemical properties of Activated Charcoal/Carbon (AC), as compared to Hydrogen Peroxide (H_2O_2) "Oxygenated" Activated Charcoal/Carbon (O-AC). Oxygenation treatment raised the O-AC specific gravity from o.52 to 0.75 g/cm^3 (about 44% increase), and the

Electric Conductivity (EC) from 71.58 to 150.92 µ S/cm (about 110.8% increase), and the surface charge from 0.785 to 2.18 mmol^{c}/g (about 177.7% increase). Oxygenation treatment also raised the specific surface area from 524 to 911 m^{2}/g (about 73.9% increase), and the pore size from 51.32 to 67.41 Å (about 31.4% increase).

Higher specific gravity (+44%) reflects the significant increase in the O-AC mass by insertion of a large number of oxygen atoms in the crystal lattice structure. This resulted in the significant addition of negative charges to the O-AC surface, which was reflected on the large increases in the Electric Conductivity (+110%) and the surface charge (+177.7%). There is also a large increase in the specific surface area (73.9%) and pore size (31.4%). All these values participate in enhancing the O-AC adsorption capacity for the studied metals (Pb^{2+}, Cu^{2+} & Ni^{2+}).

Insert Table (1) here.

3-2- Batch Experiments

3-2-1- The Effects of Contact Time on O-AC Metal Adsorption Removal & Adsorption Capacities

Figure (4) Shows the effect of contact time on residual pollutant concentrations of the studied metals (Pb^{2+}, Cu^{2+} & Ni^{2+}) as indicators of adsorption removal in: (a) single-metal solutions, (b) bi-metal solutions, and (c) tri-metal solutions. In all solutions, metal residual concentrations deceased with increasing the contact time until 40 minutes, at which the residual concentration reached a minimum value and didn't decrease any further with additional contact time up to 2.0 hours. This is consistent with reported results of 40 minutes in other studies (Elsaid et al., 2024).

Contact time experiments were done with solution pH's =5.4, and the O-AC dose at 0.10 g/500ml solution and was run for 2.0 hours (120 minutes). The best contact time in all experiments was 40.0 minutes. As shown in figure (4), the initial metal concentrations were 41.32, 40.08 and 35.26 mg/l for lead, copper and nickel; respectively. The treated solutions' metal residual concentrations in single-metal solutions after 40 minutes were 0.04, 1.48 and 1.45 mg/l, which corresponded to adsorption capacities of 206.38, 192.97 & 169.05 mg/g for Pb^{2+}, Cu^{2+} & Ni^{2+}; respectively. For bi-metal solutions, residual concentrations & adsorption capacities were: for Pb/Ni solution: 3.57/8.84 mg/l & 188.75/132.08 mg/g, for Pb/Cu solution: 5.12/7.02 mg/l & 181.0/165.3 mg/g, for Cu/Ni solution were: 5.62/7.0 mg/l, 172.32/141.35 mg/g; respectively. For tri-metal solutions, residual concentrations & adsorption capacities for Pb/Cu/Ni were: 6.02/7.98/8.78 mg/l and 176.5/160.5/132.41 mg/g; respectively.

Insert Figure (4) here.

3-2-2- The Effects of Charcoal Dose on O-AC Metal Adsorption Removal & Adsorption Capacities

Figure (5-a, b, c) shows the effect of O-AC dose on single-, bi- & tri-metal solutions; respectively. Metal residual concentrations also decreased with increasing the O-AC dose until the value of 0.1 g/500 ml solution, where there was no further decrease in treated solution residual concentrations with higher O-AC doses for all solutions. Semerjian (2018) reported that increasing the AC dose can cause aggregation of adsorbent grains which results in reduction of exposed surface area and in lower adsorption.

Charcoal dose experiments were done with solution pH's =5.4, and the O-AC dose was varied from 0.01 g to 2.0 g /500ml solution, and was run for 40 minutes. As shown in figure (5), the initial metal concentrations were 39.25, 40.26 and 43.25 mg/l for lead, copper and nickel; respectively. The treated solutions' metal residual concentrations in single-metal solutions at 0.1 g/500ml were 0.04, 1.51 and 0.64 mg/l, which corresponded to adsorption capacities of 196.04, 193.73 & 213.07 mg/g for Pb^{2+}, Cu^{2+} & Ni^{2+}; respectively. For bi-metal solutions, residual concentrations & adsorption capacities were: for Pb/Ni solution: 4.64/9.39 mg/l & 173.05/194.32 mg/g, for Pb/Cu solution: 3.64/6.03 mg/l & 178.07/171.15 mg/g, for Cu/Ni solution were: 5.43/8.34 mg/l, 174.17/174.55 mg/g; respectively. For tri-metal solutions, residual concentrations & adsorption capacities for Pb/Cu/Ni were: 5.74/8.0/9.58 mg/l and 167.55/161.3/168.33 mg/g; respectively.

Insert Figure (5) here.

3-2-3- The Effects of Solution pH on O-AC Metal Adsorption Removal & Adsorption Capacities

Figure (6-a, b, c) shows the effect of solution pH on adsorption removal in single-, bi- & tri-metal solutions, respectively. The maximum adsorption of lead, copper and nickel was reached at pH values of 4.6, 5.4 and 5.6, respectively, and then remained unchanged up to pH 7.0, which is consistent with the results reported by Jain et al. (2016). For lower pH values from 2.0 to 5.0, there was higher residual metal concentrations in treated solutions (lower adsorption removal). This is because abundant hydrogen ions in batch solutions at lower pH values competed against metal ions on the O-AC surface adsorption sites (Deng et al., 2010b). On the other hand, at higher pH values from 8.0 to 12.0, lower residual pollutant metal concentrations are attributed to forming mineral complexes that cause minerals' precipitation out of the solution (Amarasinghe & Williams, 2007).

Experiments for pH effects were done by varying the solution pH's from 2.0 to 12, with the O-AC dose at 0.1 g /500ml solution, and was run for 40 minutes. As shown in figure (6), the initial metal concentrations were 41.32, 40.08 and 35.26 mg/l for lead, copper and nickel; respectively. The treated solutions' metal residual concentrations in single-metal solutions in the flat range (5.0 to 7.0) were 0.04, 1.48 and 1.43 mg/l, which corresponded to adsorption capacities of 206.39, 193.0 & 168.10 mg/g for Pb^{2+}, Cu^{2+} & Ni^{2+}; respectively. For bi-metal solutions, residual concentrations & adsorption capacities were: for Pb/Ni solution: 5.12/8.51 mg/l & 181.0/133.75 mg/g, for Pb/Cu solution: 3.52/7.03 mg/l & 189.02/165.25 mg/g, for Cu/Ni solution were: 5.51/6.97 mg/l, 172.85/141.47 mg/g; respectively. For tri-metal solutions, residual concentrations & adsorption capacities for Pb/Cu/Ni were: 6.06/7.65/8.73 mg/l and 176.3/162.15/132.65 mg/g; respectively.

Insert Figure (6) here.

3-2-4- Average Oxygenated Activated Charcoal/Carbon (O-AC) Metal Adsorption Capacity

Adsorption capacities for single-metal solutions in batch experiments for each of the three metals (Pb^{2+}, Cu^{2+} & Ni^{2+}) were: 206.39, 193.0 & 168.10 mg/g in the contact time experiments, and 196.04, 193.73 & 213.07 mg/g in the O-AC dose experiments, and 206.39, 193.0 & 168.10 mg/g in the solution pH experiments; respectively. The overall O-AC average adsorption capacity for one of the three metals in single-metal solutions is 193.1 mg/g.

Adsorption capacities obtained in contact time batch experiments for each of the three elements in bi-metal solutions batch experiments were: for Lead: (Pb/Ni) 188.75, (Pb/Cu) 181.0; for Copper: (Cu/Pb) 165.3, (Cu/Ni) 172.32; and for Nickel: (Ni/Pb) 132.08, (Ni/Cu) 141.35 mg/g. Meanwhile, adsorption capacities obtained in O-AC dose experiments were: for Lead: (Pb/Ni) 173.05, (Pb/Cu) 178.07; for Copper: (Cu/Pb) 171.15, (Cu/Ni) 174.17; and for Nickel: (Ni/Pb) 194.32, (Ni/Cu) 174.55 mg/g. In pH experiments were: for Lead: (Pb/Ni) 181.0, (Pb/Cu) 189.02; for Copper: (Cu/Pb) 165.25, (Cu/Ni) 172.85; and for Nickel: (Ni/Pb) 133.75, (Ni/Cu) 141.47 mg/g. The overall O-AC average adsorption capacity for one of the three metals in bi-metal solutions is 168.30 mg/g.

Adsorption capacities for tri-metal solutions in batch experiments for each of the three metals (Pb^{2+}, Cu^{2+} & Ni^{2+}) were: 176.5, 160.5, 132.41 mg/g in the contact time experiments, and 167.55, 161.3, 168.33 mg/g in the O-AC dose experiments, and 176.3, 162.15, 132.65 mg/g in the solution pH experiments; respectively. The overall O-AC average adsorption capacity for one of the three metals in tri-metal solutions is 159.74 mg/g.

This makes the net overall average adsorption capacity for O-AC for each of the three metals (Pb^{2+}, Cu^{2+} & Ni^{2+}) is 173.71 mg/g, for single-, bi- & tri-metal solutions with initial concentrations at 40 mg/l for each of the three metals.

3-2-5- Comparing O-AC Adsorption Capacity with Other Adsorbents

Table (2) shows average adsorption capacities (for each metal) of some adsorbents that were used for removing Pb(II) & Cu(II), together with one or two other pollutants (Ni(II), Cd(II), Fe(II) or Zn(II)) with initial concentrations of each pollutant either at 100, 50 or 40 mg/l. Tsai et al. (2015) used Chitosan-Coated Montmorillonite Beads to remove Pb(II), Cu(II), Ni(II) & Zn(II) with the initial concentrations (I.C.) of each metal at 100 mg/l, and the average adsorption capacity was 17.14 mg/g. Cheng et al. (2018) used Fly Ash-Based Linde F(K) Zeolite for removing Pb(II), Cu(II), Ni(II) & Cd (II) with I.C. at 100 mg/l, and the adsorption capacity was 25.42 mg/g. Ighalo et al. (2020) used Biochar from Oil Palm for removing Pb(II), Cu(II), Fe(II) & Zn(II) at I.C. of 100 mg/l, and the average adsorption capacity was 16.65 mg/g. Zhao et al. (2023) used Coal Fly Ash-Red Mud Modified Composite Material to remove Pb(II), Cu(II) & Cd(II) with I.C. also at 100 mg/l, and the average adsorption capacity was 44.0 mg/g.

Zhu et al. (2012) used Xanthate-modified Magnetic Chitosan to remove Pb(II), Cu(II) & Zn(II) with I.C. at 50 mg/l, and the adsorption capacity was 34.0 mg/g. Bourliva, et al. (2015) used Natural Bentonite to remove Pb(II), Cu(II), Ni(II) & Cd(II) with I.C. at 50 mg/l, and the adsorption capacity was 43.83 mg/g. Sahin et al. (2023) used Silver Nanoparticles and Magnetic Nanoparticles/Nanocomposites to remove Pb(II), Cu(II), Ni(II) & Cd(II) with I.C. at 50 mg/l, and the adsorption capacity was 15.85 mg/g. He et al. (2023) used Magnetic Nano-Chitosan to remove Pb (II), Cu (II), Zn (II) & Cr (VI) also at I.C. 50 mg/l, and the adsorption capacity was 5.13 mg/g.

Niamlang and Supaphol (2014) used Aminated Polyacrylonitrile Nanofiber Mats to remove Pb (II), Cu (II), Fe (II) & Ag(I) with I.C. at 40 mg/l, and the adsorption capacity was 2.04 mg/g. Meanwhile, the current study used Hydrogen Peroxide-Oxygenated Activated Charcoal/Carbon (O-AC) that was obtained from Salix wood to remove Pb (II), Cu (II) & Ni (II), with I.C. at 40 mg/l, and the average adsorption capacity was 173.71 mg/g.

The Oxygenated Activated Charcoal/Carbon (O-AC) of the current study showed much higher average adsorption capacity for the studied metals at 173.71 mg/g. This is at almost 4 times the highest adsorption capacity of adsorbents in other published studies. This recommends the use of the O-AC of the current study in removing the studied metals of Pb (II), Cu (II) & Ni (II) from polluted wastewaters.

Insert Table (2) here.

3-2-6- Comparison between Competitive Behaviors of Lead (Pb^{2+}), Copper (Cu^{2+}) & Nickel (Ni^{2+}) in Single-, Bi- & Tri-metal Solutions

Competitive behavior of metals on surface adsorption sites is affected by the electronegativity and the ionic radius of each metal (Neris et al., 2018). A metal with higher electronegativity and lower ionic radius has a greater ability to occupy active sites in the pores of activated charcoal than others (Neris et al. 2018). The ionic radii of lead Pb^{+2}, copper Cu^{+2}, and nickel Ni^{+2} are 0.154 Å, 0.145 Å, and 0.149 Å, respectively. As these values are quite near to each other, the effect of this factor on adsorption capacity cannot be considered. Meanwhile the values of electronegativity are Pb^{+2} (2.2 ps) > Cu^{+2} (1.90 ps), ≈ Ni^{+2} (1.91 ps); where ps is Pauling scale (Manickam and Muthusamy; 2022).

Taking this aspect into account, lead outperforms the other two elements, copper and nickel, due to the considerable difference in lead's value, whereas copper and nickel have negligible electronegativity values. As a result, Pauling's factor only explains why lead has a larger adsorption capacity than the other two metals; it does not explain why copper has a higher adsorption capacity than nickel. In batch experiments, studying the effects of Oxygenated Activated Charcoal/Carbon (O-AC) elaborated the competitive behavior of the studied metals. This is because the adsorption sites at lower O-AC doses (< 0.1 g/500 ml) were not sufficient for all metal ions in the solution (figure 5-a, b, c). Figure (5-a) for single-metal solutions, shows that Lead (Pb^{+2}) had the lowest residual metal concentrations (i.e. highest adsorption removal), followed by Copper (Cu^{+2}), and then followed by Nickel (Ni^{+2}), which is consistent with the explanation provided by Manickam and Muthusamy (2022).

In figure (5-b), Lead (Pb^{+2}) in bi-metal solutions had the lowest residual concentrations (highest adsorption removal), followed by Copper (Cu^{+2}), and then followed by Nickel (Ni^{+2}). Adsorption removal of Lead (Pb^{+2}) in the Lead & Copper solution had lower Lead residual concentrations (higher removal) than that in the Lead & Nickel solution because of the weaker competition of Ni^{+2} with Pb^{+2} on the insufficient adsorption sites. Copper residual concentration in (Cu^{2+} & Pb^{2+}) solution was slightly higher (less removal) than in (Cu^{2+} & Ni^{2+}) solutions because of stronger Pb^{+2} and weaker Ni^{2+} competition with Cu^{2+}. Similarly, Ni^{2+} residual concentrations were lower in (Ni^{2+} & Cu^{2+}) solution than that in in (Ni^{2+} & Pb^{2+}) solution because of the stronger Pb^{2+} and weaker Cu^{2+} competitions with Ni^{2+} ions.

Figure (5-c) also shows that the adsorption removal in tri-metal solution followed the same order of Pb^{+2} > Cu^{+2} > Ni^{+2} . It also shows that above the optimal O-AC dose of 0.1 g/500ml, residual concentrations will not get any lower with higher O-AC doses up to 2.0 g/500 ml, with metal residual concentrations around 5.74/8.0/9.58 mg/l for Pb^{+2}/Cu^{+2}/Ni^{+2}; respectively (results in figure are only shown up to 0.2 g/500 ml).

However, competitive metal behavior is not similarly pronounced in batch experiments for the effects of contact time (figure 4-a, b, c), nor for the effects of solution pH (figure 6-a, b, c).

3-2-7- Comparing the Behavior of Each Metal in Single-, Bi- & Tri-metal Solutions

Figures (7: for Pb^{2+}, 8: for Cu^{2+} & 9: for Ni^{2+}) compare the behavior of each of the three studied metals in a- contact time, b- charcoal dose & c- solution pH, in single- bi- & tri-metal solutions. For each of the three metals (Pb^{2+}, Cu^{2+} & Ni^{2+}), adsorption removal was highest in single-metal solutions, and lowest in tri-metal solutions, while in bi-metal solutions' values were mostly between these two values. It is important to note again that, contact times and O-AC doses above the optimal values of 40 minutes and 0.1 g/500 ml, respectively, didn't lower the residual pollutant metal concentrations in the treated solution any further in all batch experiments.

Insert Figure (7) here.

Insert Figure (8) here.

Insert Figure (9) here.

3-3- Fixed-Bed Dual-Stage Two-Column experiment

Elsaid et al. (2024) reported that pollutant concentrations in treated waters depend upon the initial concentrations, and that increasing the activated charcoal dose and contact time above certain values won't lower the treated residual concentrations. Maximum Permissible Limits (MPLs) for the studied metals are very low because of their potential harmful effects to the biota. Table (3) shows that the MPLs in drinking water are 0.01, 1.0 & 0.1 mg/l for Pb, Cu & Ni; respectively (WHO, 2022).

Thus, it was necessary to use dual-stage two-column treatment system in order to remove the studied metal pollutants to values below the Maximum Permissible Limits (MPLs). This is because when the smaller concentrations of the outflow from the first treatment column are input to the second column, the final outflow concentrations are lowered below these small values of the MPLs.

Table (3) shows analytical results of pollutant concentrations in industrial wastewater that was obtained from El-Ekhtiar Textile Factory in Riyadh; Kingdom of Saudi Arabia, before treatment and after outflow from first and second column treatments. Sampling the treated solution outflow from the first and second treatment columns started after 40 minutes of steady flow to satisfy the contact time requirement, which the input solution takes to flow across each of the two treatment columns. Samples were taken every 10 minutes, and continued for to two hours. Measured concentrations and other variables in the treated water remained almost unchanged during the sampling duration of two hours. Table (3) shows that average measured values with the standard deviations values that are very small, which indicate that stability of the treatment process.

Initial concentrations for Lead, Copper & Nickel (in the industrial wastewater tank) were at about 22.0, 28.0 & 18.62 mg/l, and outflow concentrations from the first treatment column were at about 3.01, 4.12& 1.12 mg/l; respectively. Meanwhile, final treated outflow concentrations from the second column were at about 0.008, 0.14& 0.046 mg/l, which are safely below the Maximum Permissible Limits (MPLs) of 0.01, 1.0 & 0.1 mg/l (WHO, 2022); respectively. This clearly shows the benefit that using a two-stage two-column treatment system in cleaning the treated wastewater from the studied metals resulted in very low concentrations below the MPLs.

Previous works showed that the adsorption removal breakthrough stabilized for only one hour (Maged et al., 2020). Thus, measurement of treated outflow from the fixed-bed column was conducted for up to 2-hours in order to verify the treatment stability. The fixed-bed experiment was operated at the flow rate of 30 ml/min to achieve balance between speed and the adsorption removal process (Busetty, 2023).

In addition, the two-stage two-column O-AC treatment system could successfully remove other pollutants in the industrial wastewater. As shown in Table (3), the industrial wastewater contained other pollutants, which were also removed to values below the MPLs. Total Dissolved Solids (TDS) initial concentration (IC) was at 1350.0 mg/l, which was lowered out of the 1st stage (Column 1) to 426.0 mg/l, and out of the 2nd stage (Column 2) to 138.54 mg/l, which is blow the MPL of 500.0 mg/l (Adeogun et al., 2011). IC, 1st stage & 2nd stage concentrations for Ammonia were 32.54, 1.04 & 0.05 mg/l; for Hardness ($CaCO_3$) were 425.0, 188.57 7& 35.87 mg/l; for Sulfate were 312.0, 115.0 & 32.58 mg/l, for Nitrate were 88.63, 56.84 & 18.65 mg/l and for Chloride were 287.61,

152.62 & 72.52 mg/l; respectively. The 2nd stage treated concentration values are all below the MPLs of 0.2, 100.0, 250.0, 50.0 and 200.0 mg/l for Ammonia, Hardness ($CaCO_3$), Sulfate, Nitrate, and Chloride; respectively (WHO, 2022). Thus, the Oxygenated Activated-Charcoal/Carbon (O-AC) two-stage two-column system is recommended for use in removing Lead, Copper & Nickel and other listed pollutants from industrial wastewater within the ranges of tested concentrations. In case of higher concentrations in wastewater, further studies will be needed. It is also recommended to study the operational duration that the O-AC will take to reach saturation of the charcoal surface adsorption sites, and the number of regeneration cycles for acceptable efficient reuse of the O-AC. In case of plans to use it on a large scale, environmental and economic issues regarding large scale production of Salix wood, and safe disposal of used O-AC should also be studied.
Insert Table (3) here.

4- CONCLUSION AND RECOMMENDATIONS

In the current study, Salix wood was treated to produce Activated Charcoal/Carbon (AC), then was treated with Hydrogen Peroxide (H_2O_2) and was activated again to produce Oxygenated Activated Charcoal (O-AC). Hydrogen Peroxide treatment was done in order to insert Oxygen atoms in the charcoal crystal lattice structure and to increase its surface negative charge and its adsorption capacity of the studied metals of Lead, Copper & Nickel. Oxygenation treatment made the O-AC charcoal surface pores in (b) much larger and more uniform than the non-oxygenated AC, as shown in Scanning Electron Micrograph (SEM). Oxygenation treatment caused large increases in the Electric Conductivity (+110%), surface charge (+177.7%), specific surface area (+73.9%) and pore size (+31.4%), which all participate in increasing the O-AC adsorption capacity for the studied metals.

Batch experiments were done using synthetically-polluted solutions to examine the effects of contact time, charcoal dose & solution pH on O-AC adsorption capacity, and also to examine the competitive behavior of the studied metals in adsorption removal from polluted solutions. Best value for contact time was 40 minutes, and best O-AC dose was 0.1 g/500 ml solution, while residual concentrations were almost unchanged in the pH range of (5.0 to 7.0). The adsorption capacities of the Oxygenated Activated Charcoal/Carbon (O-AC) was 206.39, 193.0 & 168.10 mg/g for Pb^{2+}, Cu^{2+} & Ni^{2+}; respectively, while the overall average adsorption capacity for the three metals was 173.71 mg/g. This is almost 4 times higher than the highest adsorption capacity of adsorbents in other published studies for the same metals.

Fixed-bed dual-stage two-column experiment was done to examine steady-state adsorption removal of Pb^{2+}, Cu^{2+} & Ni^{2+}, in addition to Total Dissolved Solids (TDS), Ammonia, Hardness ($CaCO_3$), Sulfate, Nitrate & Chloride from industrial wastewater. The wastewater was obtained from El-Ekhtiar Textile Factory in Riyadh; Kingdom of Saudi Arabia. The fixed-bed experiment could remove the Lead, Copper & Nickel from the initial concentrations of 22.0, 28.0 & 18.62 mg/l, down to 0.008, 0.14 & 0.046 mg/l, which are safely below the Maximum Permissible Levels (MPLs) of 0.01, 1.0 & 0.1 mg/l; respectively. In addition, the other pollutants were also successfully removed to values below the MPLs.

Thus, the Oxygenated Activated-Charcoal/Carbon (O-AC) two-stage two-column system is recommended for use in removing Lead, Copper & Nickel and other listed pollutants from industrial wastewater within the ranges of tested concentrations. In case of higher concentrations in wastewater, further studies will be needed. It is also recommended to study the operational duration that the O-AC will take to reach saturation of the charcoal surface adsorption sites, and the number of regeneration cycles for acceptable efficient reuse of the O-AC. In case of plans to use it on a large scale, environmental and economic issues regarding large scale production of Salix wood, and safe disposal of used O-AC should also be studied.

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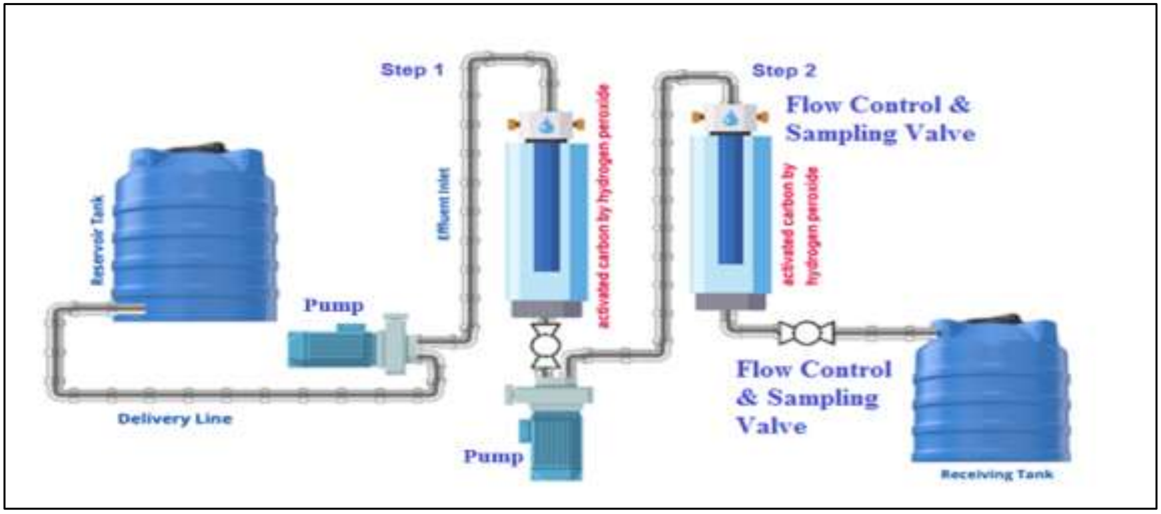

Figure (1). Design of a hydrogen peroxide activated charcoal (O-AC) Adsorption Two-Stage, Dual-Column System for multi-metal competitive removal from polluted industrial wastewater solutions.

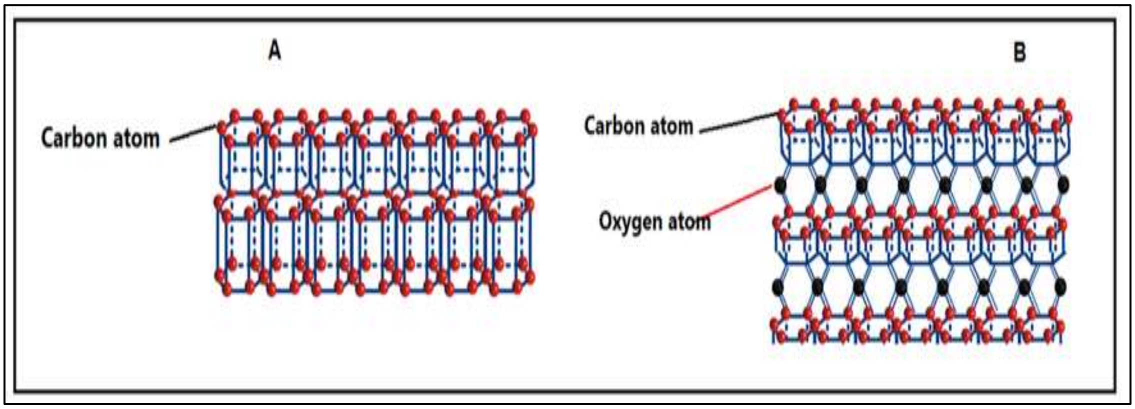

Figure (2) schematic diagram for lattice structure of: (A) Activated Charcoal/Carbon (AC), and (B) Oxygenated Activated Charcoal/Carbon (O-AC).

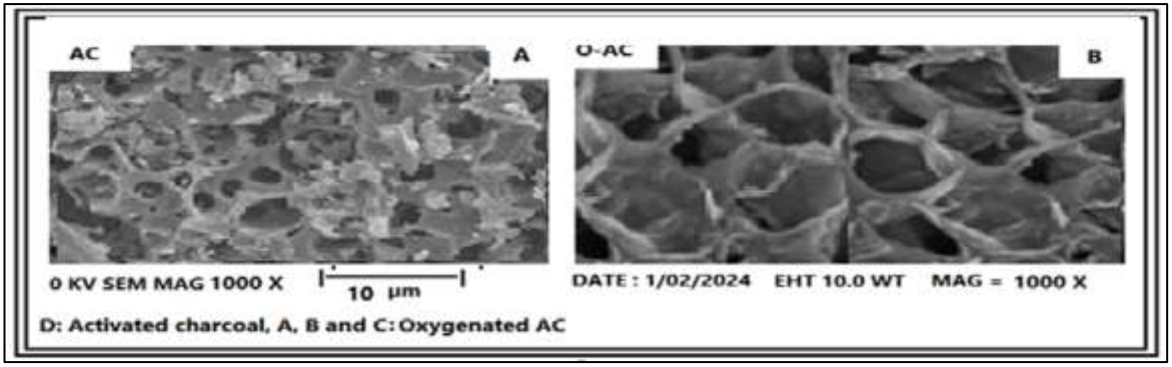

Figure (3): Scanning electron micrograph (SEM) of: (A) Activated Carbon, (AC) and (B) Oxygenated Activated Carbon (O-AC). Cambridge S-360 Scanning Electron Microscope (SEM), operated at 16kv and 2.5 A° (0.00025 micrometers). X-ray magnification at1000X, and computer software visual magnification at 25X, with the total magnification of 25000X.

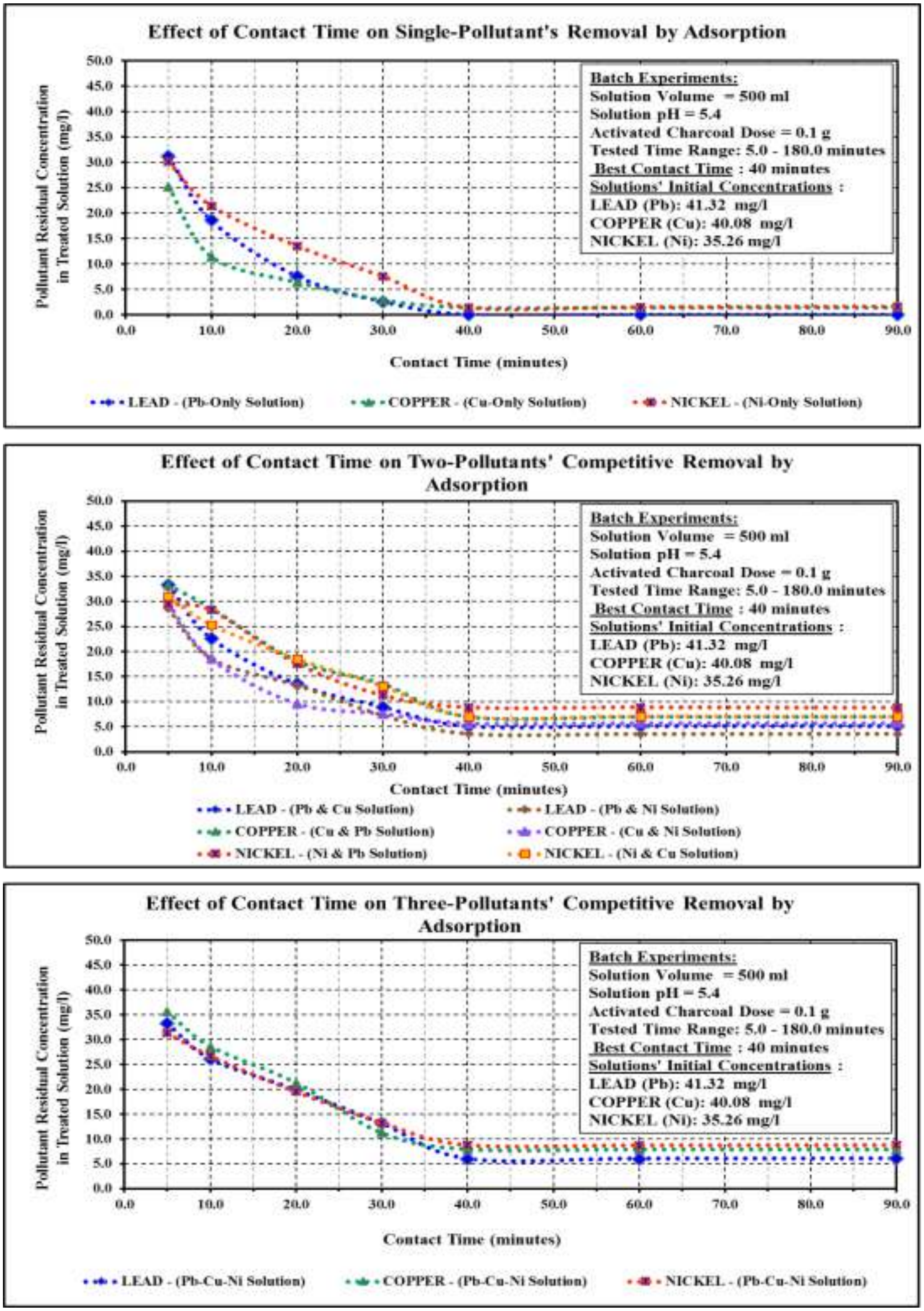

Figure (4-a, b and c). Batch experiments for the effect of contact time on single- two- & three-element removal from polluted solutions by adsorption on Oxygenated Activated Carbon (O-AC).

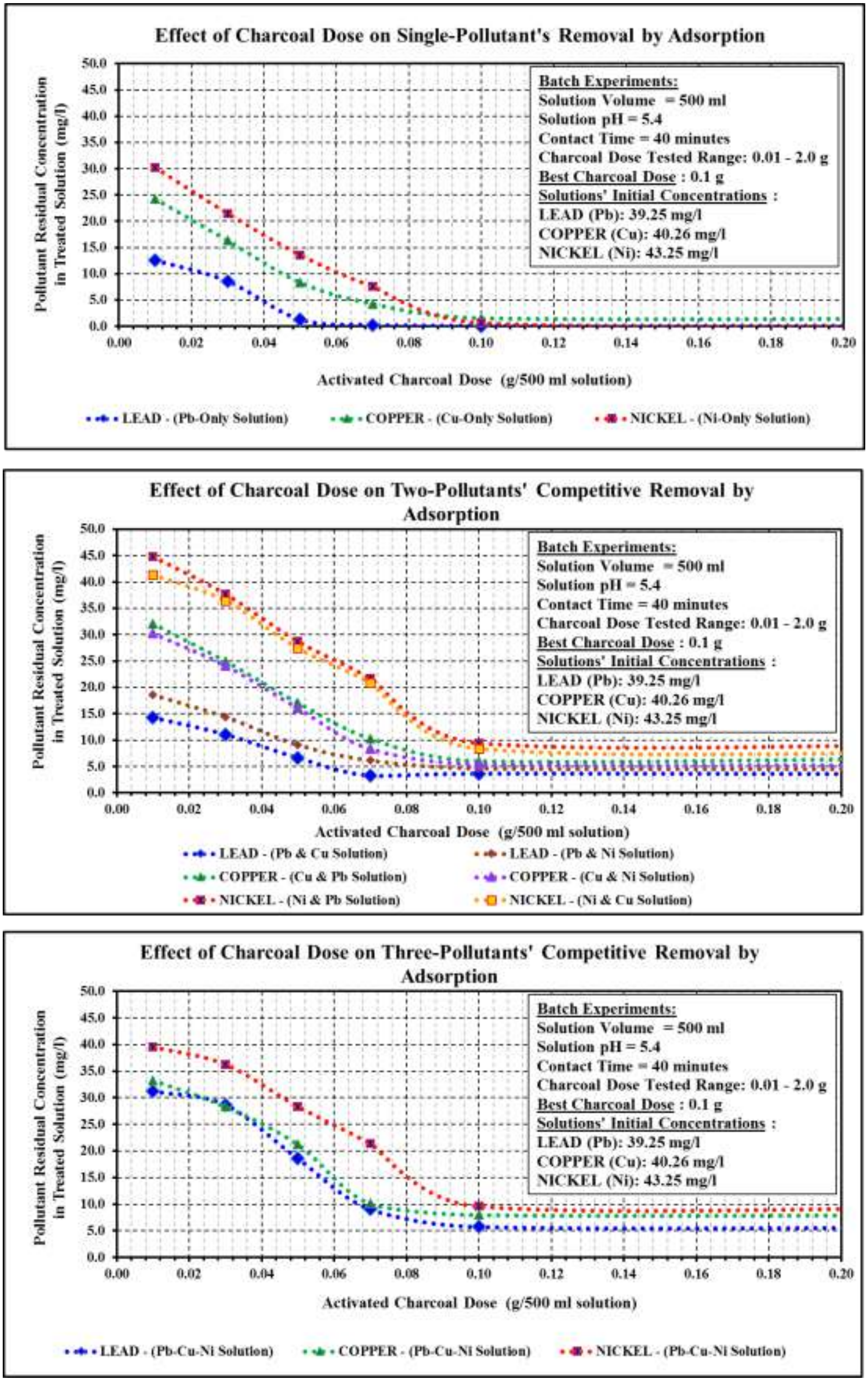

Figure (5-a, b and c). Batch experiments for the effect of activated charcoal dose on single- two- & three-element removal by adsorption on Oxygenated Activated Carbon (O-AC).

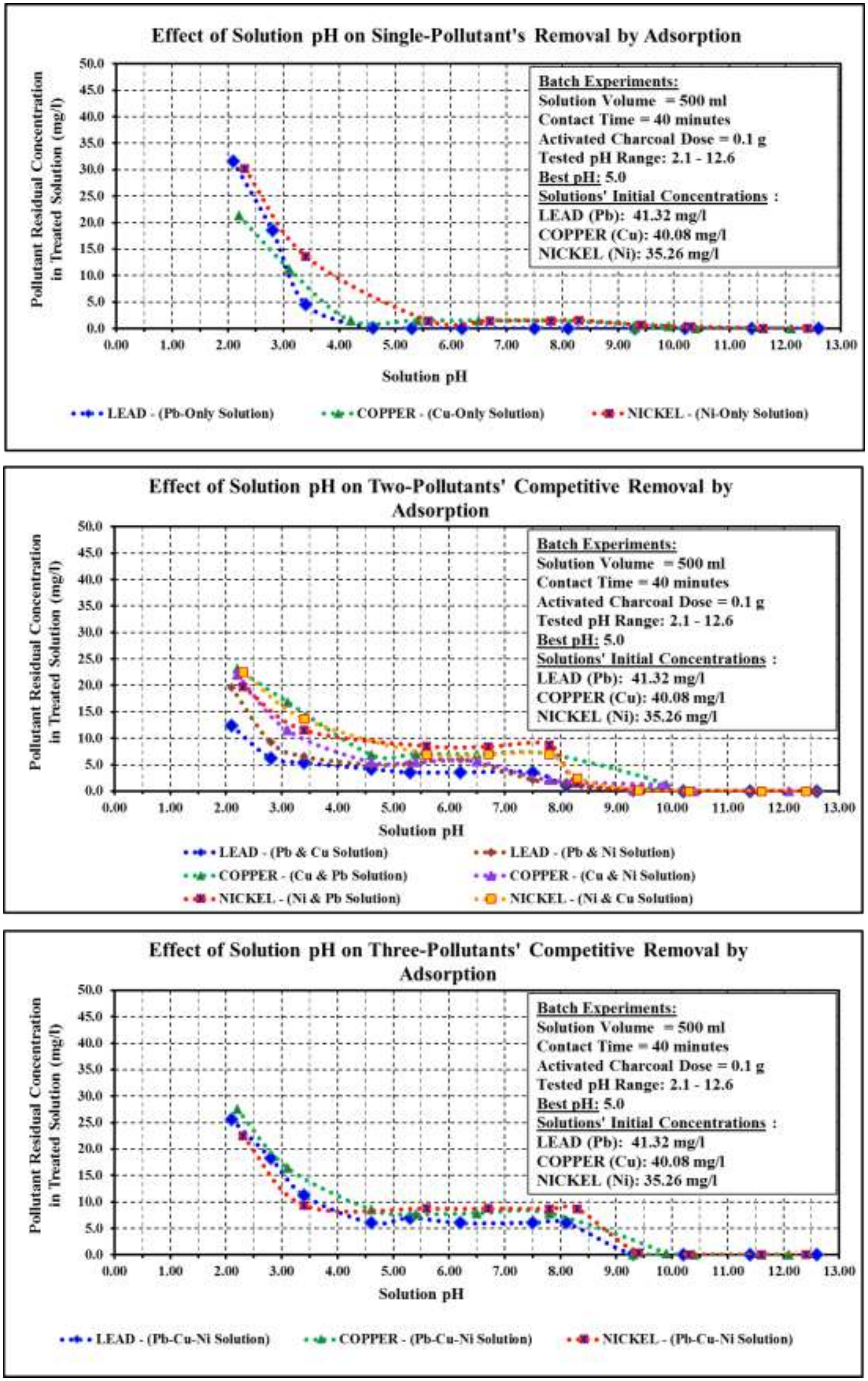

Figure (6-a, b and c). Batch experiments for the effect of solution pH on single- two- & three-element removal by adsorption on Oxygenated Activated Carbon (O-AC).

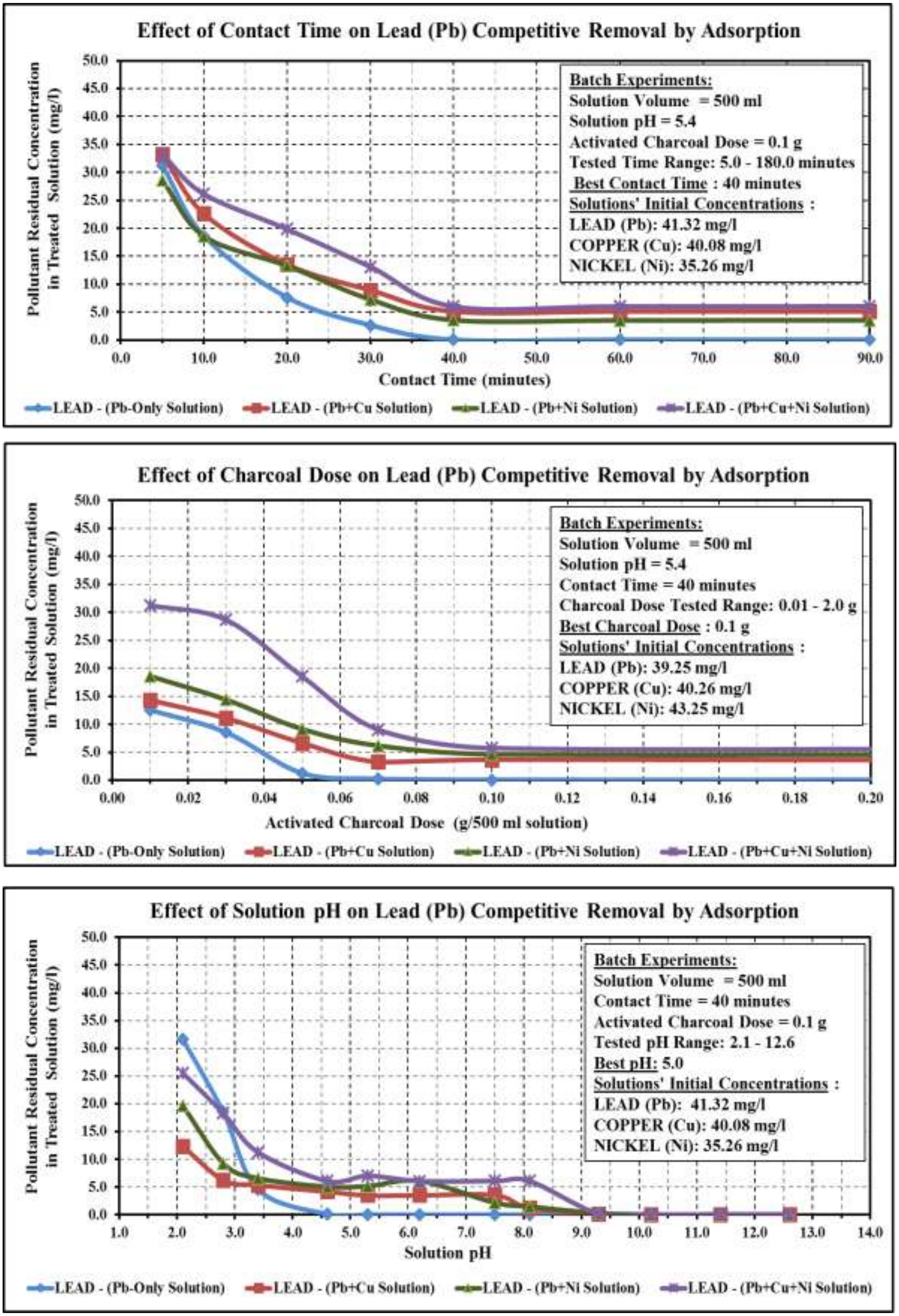

Figure (7-a, b, c). Batch experiments for competitive removal of Lead by adsorption on oxygenated activated carbon.

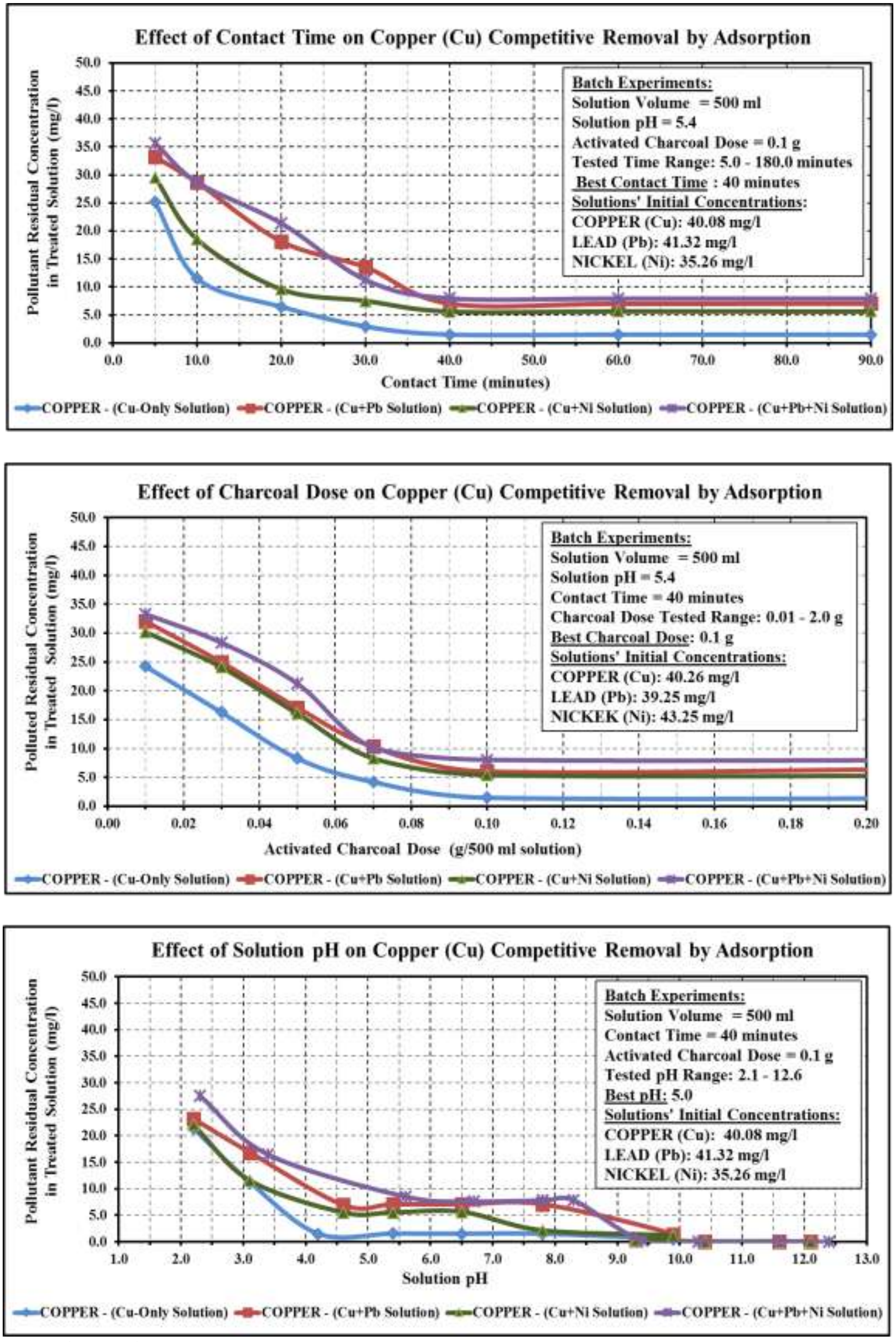

Figure (8-a, b, c). Batch experiments for competitive removal of Copper by adsorption on Oxygenated Activated Carbon (O-AC).

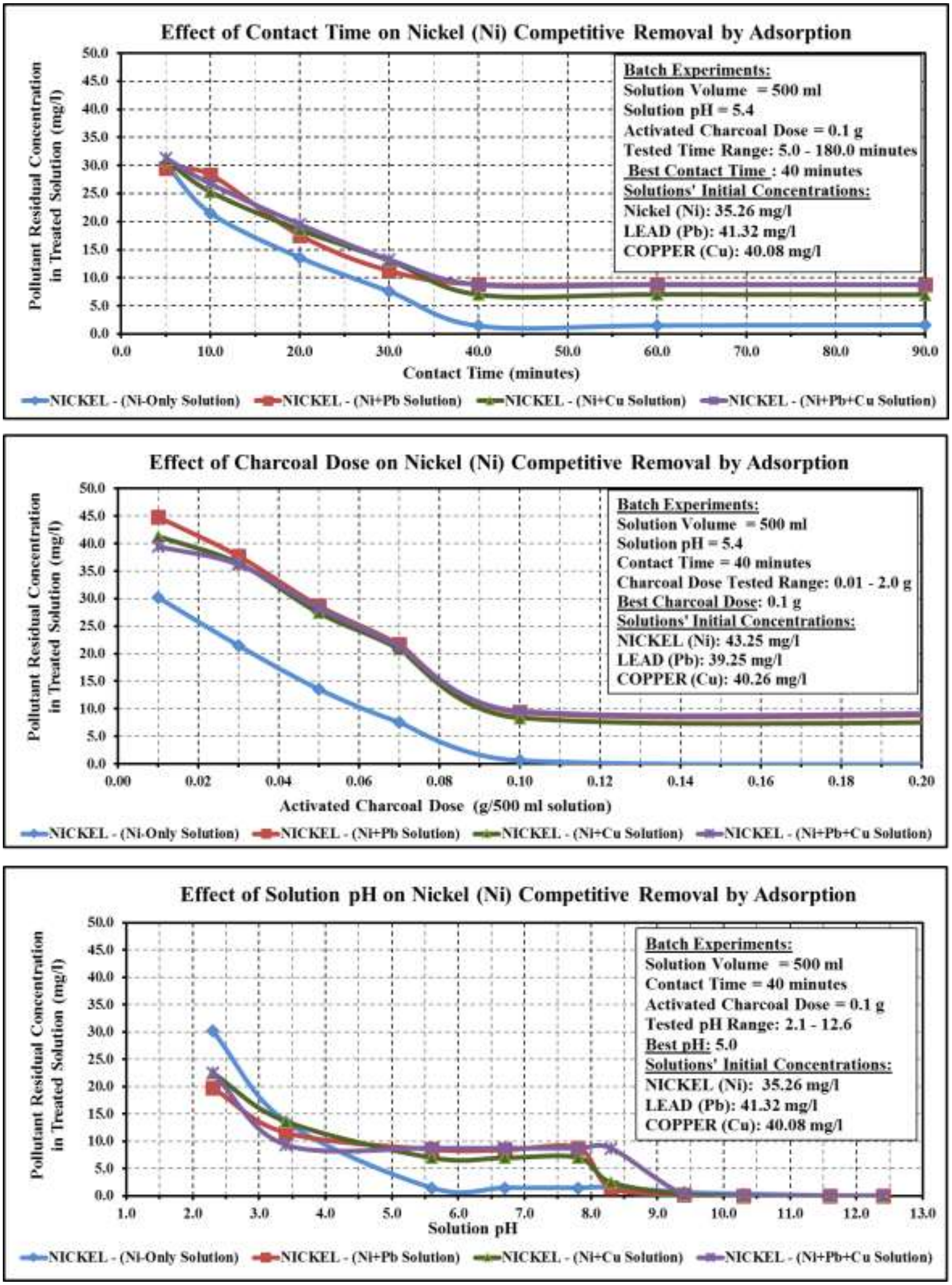

Figure (9-a, b, c). Batch experiments for competitive removal of Nickel by adsorption on Oxygenated Activated Carbon (O-AC).

Table 1. Physico-chemical properties of Activated Carbon (AC) & Hydrogen Peroxide "Oxygenated" Activated Carbon (O-AC).

Parameters	Unit	Activated Carbon (AC)	Oxygenated (O-AC)

Moister content %	%	5.32±.0.0121	8.54±0.000145
Specific gravity	g/cm^3	0.52±0.00012	0.75±0.000015
Electric Conductivity, EC	μ S/cm	71.58±0.1247	150.92±0.1987
Surface charge	mmol/g	0.785±0.000412	2.18±0.000457
Bulk density	g/ml	0.154±0.00054	0.621±0.0000145
Specific surface area	m^2/g	524±0.124	911±1.02
Pore size	A^o	51.32±0.1458	67.41±0.1987
Pore volume	cm^3/g	0.254±0.00085	0.872±0.00014
pH	--	8.1±0.00082	6.7±0.00024
Ash content	% by weight	7.84±0.00082	5.41±0.00142
Volatile matter %	% by weight	6.41±0.0014	9.84±0.000168
Carbon	% by weight	47.54±0.1258	32.71±0.1698
Hydrogen	% by weight	5.41±0.00752	7.58±0.00245
Nitrogen	% by weight	3.23±0.00147	1.07±0.0001876
Oxygen	% by weight	13.54±0.1287	24.51±0.009586

Table 2. Comparison of published adsorbents and their adsorption capacities for targeted pollutants, under the listed initial concentrations.

No	Adsorbent name	Target pollutant	Initial Conc. (mg/l)	Adsorption capacity (mg/g)	Reference
1	Chitosan-Coated Montmorillonite Beads	Pb(II), Cu(II), Ni(II) & Zn(II)	100	17.14	Tsai et al. (2015)
2	Fly Ash-Based Linde F(K) Zeolite	Pb(II), Cu(II), Ni(II) & Cd(II)	100	25.42	Cheng et al. (2018)
3	Biochar from Oil palm	Pb(II), Cu(II), Fe(II) & Zn(II)	100	16.65	Ighalo et al. (2020)
4	Coal Fly Ash-Red Mud Modified Composite Material	Pb(II), Cu(II) & Cd(II)	100	44.0	Zhao et al. (2023)
5	Xanthate-modified Magnetic Chitosan	Pb(II), Cu(II) & Zn(II)	50	34.0	Zhu et al. (2012)
6	Natural Bentonite	Pb(II), Cu(II), Ni(II) & Cd(II)	50	43.83	Bourliva, et al. (2015)
7	Silver Nanoparticles and Magnetic Nanoparticles/Nanocomposites	Pb(II), Cu(II), Ni(II) & Cd(II)	50	15.85	Sahin et al. (2023)
8	Magnetic Nano-Chitosan	Pb(II), Cu(II), Zn(II) & Cr(VI)	50	5.13	He et al. (2023)
9	Aminated Polyacrylonitrile Nanofiber Mats	Pb(II), Cu(II), Fe(II) & Ag(I)	40	2.04	Niamlang & Supaphol (2014)
10	Hydrogen Peroxide (O-AC: Oxygenated Activated Carbon)	Pb(II), Cu(II) & Ni(II)	40	173.71	Elsaid and Anwar; The current study

Table 3. Measured Physico-chemical parameter values and the Maximum Permissible Limits (MPL) of the discharged real polluted industrial wastewater before and after treatment in the Dual-Stage Two-Column fixed-bed system.

Parameters	**Real Industrial Wastewater**			**Maximum Permissible Limits (MPL)**	**References**
	Before Treatment	**After Treatment**			
		First stage (Column 1)	**Second stage (Column 2)**		
pH	5.8 ± 0.11	7.02 ± 0.03	7.4 ± 0.01	🟩 7.0 - 8.5	WHO,2011
Temperature (°C)	22.0 ± 0.32	23.30 ± 0.06	22.15 ± 0.03	🟩 10-22 °C	WHO,2011
TDS (mg/l)	1350.0 ± 11.34	426 ± 4.67	138.54 ± 0.8	🟩 500 mg/l	Aina et al., 2011
EC (µS/m)	857.82 ± 2.5	625.21 ± 0.8	175.23 ± 2.1	🟩 300 µS/cm	WHO, 2011
Pb (mg/l)	22.0 ± 0.31	3.01 ± 0.46	0.008 ± 0.0001	🟩 0.01 mg/l	WHO,2011
Cu (mg/l)	28.0 ± 0.154	4.12 ± 0.82	0.14 ± 0.001	🟩 1.0 mg/l	WHO,2011
Ni (mg/l)	18.62 ± 1.24	1.12 ± 0.00	0.046 ± 0.001	🟩 0.1 mg/l	WHO,2011
Ammonia (mg/l)	32.54 ± 0.14	1.04 ± 0.003	0.05 ± 0.001	🟩 0.2 mg/l	WHO,2011
Hardness; ($CaCO_3$ – mg/l)	425.0 ± 0.81	188.57 ± 1.0	35.87 ± 1.2	🟩 100 mg/l	WHO,2011
Sulfate (mg/l)	312.0 ± 1.16	115.0 ± 0.95	32.58 ± 0.82	🟩 250 mg/l	WHO,2011
Nitrate (mg/l)	88.63 ± 1.2	56.84 ± 1.15	18.65 ± 7.6	🟩 50 mg/l	WHO,2011
Chloride (mg/l)	287.61 ± 2.51	152.62 ± 4.1	72.52 ± 1.25	🟩 200 mg/l	WHO,2011

* Green-shaded cells indicate that treated wastewater is down to values below MPL.