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# Chromium Removal from Wastewater Using Phosphorylated Fish Bone Char

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## Abstract

Hexavalent chromium is a toxic and highly leachable contaminant associated with several environmental hazards. A phosphoric acid-functionalized fish bone char has been synthesized by chemical impregnation and researched for the treatment of hexavalent chromium from water. The surface area of the synthesized material is found to be 19.79 m<sup>2</sup>/g by nitrogen adsorption-desorption isotherm having pore volume and diameter of 0.049 cm<sup>3</sup>/g and 13.7–14.9 nm, respectively. The XRF results have showed the presence of significant mineral content in the synthesized material. The major mineral content is found to be CaO and P<sub>2</sub>O<sub>5</sub> of 19.55 and 21.25 wt.% respectively. Maximum percentage of removal is 98.04% at low concentrations of hexavalent chromium and the maximum adsorption capacity is 5.77 mg/g at 25 mg/L. The obtained results are compared among Langmuir, Freundlich, and Temkin isotherms where the Freundlich isotherm has the highest R<sup>2</sup> value of 0.99882. The synthesized material, which is a combination of pore diffusion and surface complexation, has shown potential as a sustainable and low-cost adsorbent for the removal of hexavalent chromium from wastewater.

**Keywords:** Bio-adsorption, Phosphorylated fish bone char (PFBC), Hydroxyapatite, Wastewater.

## 1. Introduction

Heavy metals in water bodies have been recognized as a major environmental issue due to rapid industrialization and the increase in human activities. Pollutants are defined as metals or metalloids in amounts or chemical forms not desired in human health and the environment (R. Singh *et al.*, 2011). Major industries like metallurgy, electroplating, tanning, mining, pharmaceutical industries, pesticide industries, and chemical industries are the primary causes for the pollution of water bodies like rivers, lakes, and streams with heavy metals. Heavy metals are long-lasting pollutants in the environment since they are unable to degrade and makes them continue

to build up in water. This is because they are highly soluble in water and carcinogenic in nature (P. Singh *et al.*, 2021). Arora *et al.* demonstrate that hexavalent chromium has been implicated in damaging the kidneys, liver, gastrointestinal system, and in lung cancer (Arora, 2019). For drinking water, WHO has set a limit is 0.05 mg/L (Vaiopoulou & Gikas, 2020). For wastewater, the limit is 0.005 mg/L. In reality, however, hexavalent chromium compounds are often exceeded in wastewater from industries such as electroplating plants, pigment production plants, aluminum finishing plants, leather tanning plants, and from mines (Razzak *et al.*, 2022). This makes it a challenge to find an effective and affordable way to

remove hexavalent chromium from water. Among the conventional techniques used in removing chromium from wastewater are ion exchange, electro-coagulation, membrane filtration, electro-dialysis, reverse osmosis, electro-winning, and precipitation (Sangamnere *et al.*, 2023). Despite the fact that these techniques have been successful in removing chromium from wastewater, they have been associated with disadvantages such as high energy requirements, high operational costs, and the production of secondary sludge (Ramírez Calderón *et al.*, 2020). Consequently, cheaper and environmentally friendly alternatives have been explored. One of the promising techniques is biosorption for removing heavy metals from the wastewater (Muddada *et al.*, 2018). This has been made possible through the simplicity of the technique.

Park *et al.* have reviewed the development and potential of biosorption to remove heavy metals from the wastewater (Park *et al.*, 2010). Abdi *et al.* have discussed the advantages associated with the use of biosorbents in wastewater treatment (Abdi & Kazemi, 2015). Suzuki *et al.* demonstrated that hydroxyapatite-based adsorbents have been recognized as promising adsorbents to remove heavy metals. This is due to the calcium phosphate structure in hydroxyapatite, which has a strong tendency to bind metals through ion exchange (Suzuki *et al.*, 1981). Fish bone waste has abundant and cost-effective source of hydroxyapatite, particularly in coastal regions. Several studies demonstrated the potentiality of this waste in the adsorption of heavy metals. Ozawa *et al.* evaluated the adsorption capacity of hydroxyapatite obtained from fish bone waste (Ozawa *et al.*, 2003). Brunson *et al.* mentioned the adsorption capacity of this waste depends on the charring temperature, phosphorus content, and surface properties in the adsorption of arsenic and fluoride; the mineral composition and surface properties play a vital role in the adsorption capacity of the waste (Brunson & Sabatini, 2009). Research demonstrated that at optimum conditions the fish bone char has a maximum adsorption capacity of 32.7 mg/g. It also indicated the adsorption capacity of this waste followed Freundlich adsorption, while the adsorption kinetics followed the pseudo-first/second-order model (Rasheed, 2022). Rasheed *et al.*

demonstrated that the activated fish bone materials chemically and reported significant heavy metal removal with optimized conditions (Rashed *et al.*, 2024). Hyder *et al.* examined the adsorption isotherm and kinetic models for the removal of Cr(VI) using bone char. The results reported nearly complete removal at low pH values and the Langmuir model fit the results well (Hyder *et al.*, 2015). Chemical modification of the fish bone char enhanced the performance of the adsorbent. Wang *et al.* reported that the surface area of the NaOH-treated fish bone char was higher than that of the untreated material (Wang *et al.*, 2018). In a similar context, Yang *et al.* showed that the preparation of chitosan and Fe<sub>3</sub>O<sub>4</sub>-functionalized fish bone char. The results reported improved adsorption of Cd(II) due to surface complexation and ion exchange (W. Yang *et al.*, 2022). Recently, Castillo *et al.* showed that the application of bone char in the water treatment, emphasizing the ecological and economic benefits of using this adsorbent material, such as low production costs, waste reuse, and low ecological impact compared to other adsorbents (Medellín Castillo *et al.*, 2023).

Despite the progress in this research area, there has not been a thorough investigation of the application of fish bone char treated with phosphoric acid to remove hexavalent chromium from aqueous solutions. Phosphoric acid treatment of the char surface has the potential to introduce additional surface phosphate groups, enhance the surface acidity, and improve the electrostatic interaction with hexavalent chromium. In this research, the fish bone char is chemically activated using phosphoric acid and then calcined to produce phosphorylated fish bone char (PFBC). It is characterized to determine its texture and composition using BET surface area analysis and XRF. Batch adsorption tests are conducted to investigate the influence of initial concentration of Cr(VI) on the adsorption efficiency. Furthermore, to understand the adsorption mechanism Langmuir, Freundlich, and Temkin isotherms models are analyzed.

## 2. Materials and Methods

### Chemicals and Instruments

The chemical used in this experiment were analytical grade potassium dichromate, phosphoric acid, HCl,

NaOH, UV-VIS Spectrophotometer DR-6000 were used to measure the final concentration of the solution.

### Preparation of the bio-sorbent

Fishbones were collected from local market and clean the thoroughly to remove the remaining impurities. The bones were washed with tap water then washed deionized water and placed it an oven at 100°C for remove moisture. The fish bone was pulverized using grinder and treated the phosphoric acid (85%) solution, 6 ml acid for 10 gm powder stirring it 250 rpm and kept in overnight, then placed the acid treated fishbone in oven at 100°C for removing moisture. Heated the dried bones in a closed container and placed it in muffle furnace at high temperatures (400°C) for 2 hours. This process converts the organic matter in the bones into calcium rich fish bone char and the phosphate groups reacted with the surface of the fish bone char, forming phosphorylated fish bone char. The phosphorylated char was rinsed to get rid of extra phosphate using distilled deionized water. The materials were dried in an oven at 105°C and the chemically activated fishbone char was stored in air tight containers.

### Batch adsorption tests

We prepared a 1000 mg/L stock solution of chromium (VI) to serve as our adsorbate. Then, to create the range of test solutions, we diluted this stock solution to various concentrations using distilled, deionized water. A certain amount of phosphorylated fish bone char added to 50 ml beaker containing 50 mL of chromium solution. Using a magnetic stirrer fitted with a heated plate, the mixture stirred for a certain amount of time while being kept at room temperature. After this process, the liquid separated from the adsorbent by filtration using a Whatman filter paper (No. 42). The solution containing the residual metal concentration collected in a sample container and a total of five samples prepared and submitted for analysis using an UV-VIS Spectrophotometer DR-6000. The experimental data applied to decide the percentage removal and adsorption capacity of Phosphorylated fish bone char by using equation 1 and 2.

$$\text{Percentage Removal} = \frac{(C_0 - C_e)}{C_0} \times 100 \text{ -----(1)}$$

$$\text{Adsorption Capacity} = \frac{(C_0 - C_e) \times V}{m} \text{ -----(2)}$$

Where  $C_0$  and  $C_e$  are the initial and the final metal concentrations (mg/L) in the liquid phase respectively,  $m$  is the weight of adsorbent and  $V$  (L) is the volume of the liquid phase.

### BET and XRF Analysis

The texture of phosphoric acid-modified fish bone char was characterized with nitrogen adsorption-desorption at -195.8°C. The adsorption isotherm had a classical Type IV curve with a hysteresis loop at high relative pressures ( $P/P_0 > 0.8$ ). The composition of the oxide components of the PFBC was studied using X-ray fluorescence spectroscopy to determine which components are responsible for adsorption properties.

### Mechanism of biosorption

To evaluated the mechanism of biosorption process of phosphorylated fish bone char and understand the distribution of chromium (VI) ions between the liquid phase and the solid adsorbent, an equilibrium adsorption isotherm study was conducted. The experimental data were analyzed by fitting them to three well-known models: Langmuir, Freundlich, and Temkin. This was done to identify the most suitable model for the design of a practical treatment system and batch adsorption studies were conducted at the optimum conditions.

### Langmuir Isotherm

The adsorption efficiency of different adsorbents is evaluated and contrasted using Langmuir adsorption, which is usually used to explain gas-solid phase adsorption (Hyder *et al.*, 2015). A well-known model, the Langmuir isotherm, predicts a reduce in the number of accessible interaction sites as the metal ion concentration rises. According to this hypothesis, biosorption takes place on a homogenous surface by monolayer sorption, in which there is no engagement between the molecules that have been adsorbed. The equation 3 is the expression for Langmuir equation.

$$\frac{C_e}{q_e} = \frac{1}{q_m K_L} + \frac{C_e}{q_m} \text{ ----- (3)}$$

where  $C_e$  is concentration of adsorbate at equilibrium (mg/g),  $q_e$  is the amount of metal ion adsorbed on bio-sorbent (mol/g) at equilibrium time,  $K_L$  is the Langmuir constant related to adsorption

capacity(mg/g) and  $q_m$  is monolayer capacity of the bio-sorbent.

### Freundlich Isotherm

Adsorption processes that take place on heterogenous surfaces are described by the Freundlich isotherm (Hyder *et al.*, 2015). The linear form of the Freundlich isotherm is given by the equation 4.

$$\ln q_e = \ln k_F + \frac{1}{n} \ln C_e \text{----- (4)}$$

Where  $K_F$  is adsorption capacity (L/mg) and  $1/n$  is adsorption intensity.

### Temkin Isotherm

Temkin isotherm is applicable only an intermediate

range of ion concentrations (Hyder *et al.*, 2015). The following equation 5 provides the Temkin isotherm model's linear form.

$$q_e = \frac{Rt}{b} \ln K_T + \frac{RT}{b} \ln C_e \text{----- (5)}$$

Where  $b$  is Temkin constant which is related to the heat of sorption (J/mol) and  $K_T$  is Temkin Isotherm constant (L/g).

## 3. Results and Discussion

### Adsorption capacity

The data collected from the batch experiment and the associated parameters are listed in **Table 1**.

**Table 1:** Initial and equilibrium concentration of chromium.

| Sample | Initial conc.<br>$c_o$ (mg/L) | Equ. conc.<br>$c_e$ (mg/L) | Removal<br>$y$ (%) | Adsorption<br>(mg/g) |
|--------|-------------------------------|----------------------------|--------------------|----------------------|
| 1      | 05                            | 0.098                      | 98.04              | 1.226                |
| 2      | 10                            | 0.406                      | 95.94              | 2.399                |
| 3      | 15                            | 0.866                      | 94.23              | 3.534                |
| 4      | 20                            | 1.364                      | 93.18              | 4.659                |
| 5      | 25                            | 1.905                      | 92.38              | 5.774                |

### BET Analysis

This analysis indicates that the adsorbent has mesoporosity due to capillary condensation. The BET surface area of PFBC is found to be 19.79 m<sup>2</sup>/g which suggests that the surface area determination is reliable. The total pore volume of PFBC was found to be 0.049 cm<sup>3</sup>/g from the BJH desorption curve. The pore diameters are between 13.7 and 14.9 nm, which is in the mesoporous region of 2–50 nm. From the t-plot curve, the surface area of the micropore was found to be 5.09 m<sup>2</sup>/g, while the surface area of the exterior is found to be 14.70 m<sup>2</sup>/g. The result indicates that about 74% of the total surface area of PFBC is accessible to the adsorbate molecules. The mesoporous framework of PFBC provides an adequate pathway for the adsorbate molecules to travel to the active sites of the adsorbent without any significant intraparticle diffusion resistance.

### XRF Analysis

The PFBC has a high amount of inorganic matter, with CaO at 19.55 wt.% and P<sub>2</sub>O<sub>5</sub> at 21.25 wt.% including other components like SO<sub>3</sub>, MgO, Na<sub>2</sub>O, Fe<sub>2</sub>O<sub>3</sub>. The high amount of P<sub>2</sub>O<sub>5</sub> is a clear indication UniversePG | [www.universepg.com](http://www.universepg.com)

that the phosphoric acid modification reaction has been successful. At the same time, the high amount of CaO also confirms that the hydroxyapatite structure of the fish bone char has been maintained. The fact that both components are present in the adsorbent clearly indicates that calcium phosphate is responsible for adsorption properties. It is known that calcium phosphate has strong adsorption properties towards oxyanions through electrostatic attraction.

### Effect of initial Cr (VI) ion concentration

**Table 1** shows how different initial Cr (VI) ion concentrations affect the biosorption of Cr (VI) ions onto phosphorylated fish bone char. As the initial concentrations increases, the removal percentage of Cr (VI) decreases. An inverse relationship is observed between removal efficiency and adsorption capacity. The system achieved its peak removal efficiency of 98.04% at a low initial Cr (VI) concentration (5 mg/L), whereas the adsorption capacity at equilibrium improved as the initial concentration has increased the percentage removal and removal capacity are compared with different initial metal concentrations in **Fig. 1** and **2**.

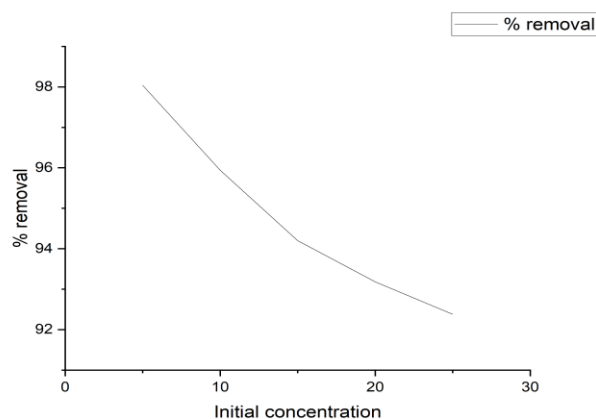


**Fig. 1:** Percentage of Removal Comparison.

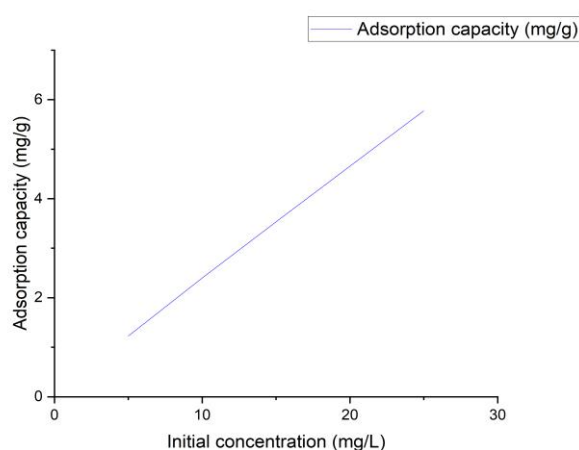


**Fig. 2:** Removal Capacity Comparison.

### Adsorption Isotherms

The respective linearized plots for the Langmuir, Freundlich, and Temkin equations applied to the biosorption of chromium (VI) onto phosphorylated fish bone char are presented in **Fig. 3, 4** and **5**.

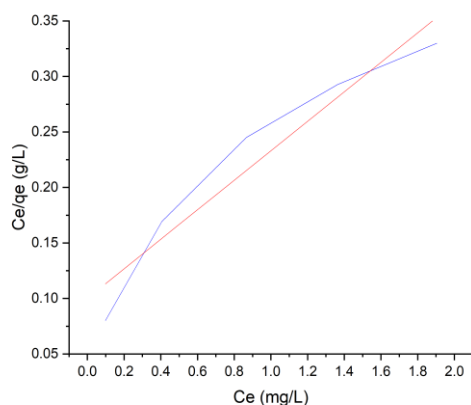


**Fig. 3:** Langmuir Isotherm.

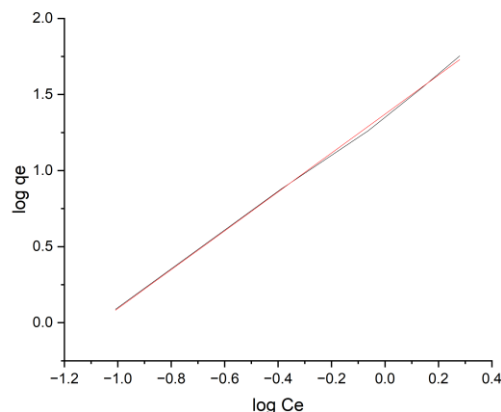


**Fig. 4:** Freundlich Isotherm.

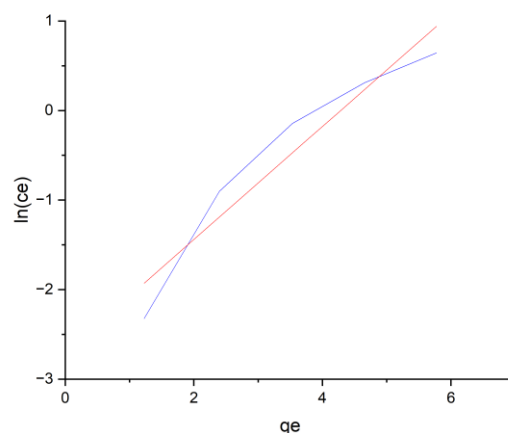


**Fig. 5:** Temkin Isotherm.

By analyzing the graphs in **Fig. 3, 4**, and **5**, the necessary parameters for the Langmuir, Freundlich, and Temkin isotherm models are obtained which are shown in **Table 2**.

The adsorption capacity of the bio-sorbent ( $q_m$ ) for the Langmuir isotherm was found to be 7.5273 mg/g with an  $R^2$  value of 0.90371, which suggests that the ideal assumption of monolayer adsorption does not hold perfectly for this case. On the other hand, the Freundlich equation gave the highest  $R^2$  value of 0.99882. This suggests that the adsorption process is strong and effective with the value  $K_F$  is 23.52. The value of  $n$  is 0.7823 indicates that the adsorption occurs on energetically heterogeneous surfaces and the adsorption is cooperative with decreasing adsorption energy as the surface coverage increases.

The Temkin equation gaive an  $R^2$  value of 0.89502, which is less than that of the Freundlich equation.

**Table 2:** Isotherm Parameters.

| Langmuir Isotherm |              |         | Freundlich Isotherm |       |         | Temkin Isotherm |               |         |
|-------------------|--------------|---------|---------------------|-------|---------|-----------------|---------------|---------|
| $q_m$ (mg/g)      | $K_L$ (L/mg) | $R^2$   | $n$                 | $k_F$ | $R^2$   | $b$ (J/mol)     | $k_T$ (L/mol) | $R^2$   |
| 7.5273            | 0.13265      | 0.90371 | 0.7823              | 23.52 | 0.99882 | 1696.94         | 2.8926        | 0.89502 |

This suggests that the assumption of decreasing adsorption energy with an increase in surface coverage does not hold perfectly for this case. The predominance of the Freundlich equation indicates that the adsorption of Cr(VI) occurs on energetically heterogeneous surfaces that have different adsorption energies. The presence of mineral surfaces and carbon surfaces supports the adsorption of Cr(VI). To assess

the performance of PFBC, its Cr(VI) adsorption capacity is compared in **Table 3** with the various adsorbents reported in the literature. Although adsorption capacities are strongly influenced by experimental parameters such as initial Cr(VI) concentration, pH, adsorbent dosage, temperature, and contact time, the comparison provides a useful indication of the relative performance of the developed PFBC.

**Table 3:** Comparison of Cr(VI) adsorption Performance of various adsorbents.

| Adsorbent                  | Modification/<br>Preparation                                 | adsorption<br>capacity (mg/g) | Experimental Condition                                  | Ref.                               |
|----------------------------|--------------------------------------------------------------|-------------------------------|---------------------------------------------------------|------------------------------------|
| Bone char                  | Calcined cattle bone                                         | 4.8                           | pH 1; 800 mg/L initial Cr(VI) concentration             | (Hyder <i>et al.</i> , 2015)       |
| Biochar                    | Biomass-derived                                              | 3.2                           | pH 2; 500 mg/L initial Cr(VI) concentration             | (Hyder <i>et al.</i> , 2014)       |
| PFBC                       | H <sub>3</sub> PO <sub>4</sub> -modified                     | 5.77                          | Acidic conditions; 25 mg/L initial Cr(VI) concentration | <b>This work</b>                   |
| Sn/HAp                     | Sn-functionalized hydroxyapatite                             | 10                            | Acidic condition; 25-50 mg/L Cr(VI) concentration       | (Campisi <i>et al.</i> , 2021)     |
| RGO-HAp                    | Reduced graphene oxide-hydroxyapatite hybrid                 | 45.24                         | Optimized Conditions                                    | (Karthikeyan <i>et al.</i> , 2021) |
| HAp-modified biochar       | Hydroxyapatite-modified <i>Mimosa pigra</i> biochar          | 67.68                         | pH 6; Batch adsorption                                  | (Loc & Phuong, 2025)               |
| N-functionalized bone char | NH <sub>3</sub> .H <sub>2</sub> O-assisted functionalization | 339.8                         | pH 5; 293 K                                             | (Z. Yang <i>et al.</i> , 2022)     |

In the **Table 3**, the adsorption capacity of PFBC (5.77 mg/g) is within the range reported for several conventional bone and biomass-derived adsorbents. The capacity is higher than that reported for the bone char studied by Hyder *et al.* (4.80 mg/g). Although direct comparison should be made cautiously because the experimental conditions are not same. More highly engineered materials, such as hydroxyapatite-based composites and functionalized bone char provide higher capacities Hasan *et al.*, (2023).

**4. Conclusion**

The synthesized phosphorylated fish bone char (PFBC) is used for hexavalent chromium removal UniversePG | [www.universepg.com](http://www.universepg.com)

from water. The mesoporous texture of PFBC is confirmed by textural analysis using the BET model. The surface area of BET is 19.79 m<sup>2</sup>/g, with a pore volume of 0.049 cm<sup>3</sup>/g and the pore diameter is around 14 nm. The mesoporous surface area facilitates mass transfer of hydrated chromate. XRF results have confirmed the presence of a significant amount of minerals where the major components of the sample are CaO and P<sub>2</sub>O<sub>5</sub>. The percentage of CaO is 19.55 wt%, and the percentage of P<sub>2</sub>O<sub>5</sub> is 21.25 wt% which suggests the presence of calcium phosphate. The mesoporous surface area and the minerals facilitate the adsorption process. Therefore, it can be

concluded that surface area is not the only factor in the adsorption process. In the batch mode of operation, the results have showed that PFBC is efficient to remove hexavalent chromium from wastewater where surface chemistry plays a vital role. The percentage removal of hexavalent chromium at a concentration of 5 mg/L is 98.04%, that is, at low concentrations removal rate is very high.

The maximum amount of hexavalent chromium adsorbed at equilibrium is 5.77 mg/g. Among the three models, Freundlich model fit the results very well and the Langmuir model has shown a poor fit with the results. The Freundlich model has shown that the results for a heterogeneous surface area and the adsorption energy vary as hexavalent chromium is not adsorbed on a single layer. The variation in adsorption energy may be due to the composite structure of the sample. PFBC is efficient in removing hexavalent chromium from water and it is an environmentally friendly material synthesized from waste fishbone. Further optimization adsorption conditions and surface chemistry of PFBC could improve Cr (VI) removal performance.

## 5. Author Contributions

F.H.J.: Conceptualization, Methodology, Investigation, Formal analysis, Writing - Original draft.  
M.A.I.: Resources, Supervision, Formal analysis, Fund accusation, Project administration, Writing-Review and Editing.

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## 7. Conflicts of interest

The authors declare that they have no conflicts of interests.

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