Reusing of furnace bottom ash as an adsorbent for phosphate removal from water

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Abstract. Phosphate is a common chemical element that resides extensively in the Earth's crust, and its presence in water results in eutrophication of water. Therefore, many studies were devoted to study the ability of phosphate removal from water and wastewater using different treatment methods. Recent studies have suggested that filtration treatment techniques are effective for phosphate removal with one disadvantage which is the high cost of industrial filtration materials used. As a result, recent studies are concentrating on minimizing the operational costs of the filtration method by using cost-effective alternatives. This study is focusing on the efficiency of using the wastes of the iron industry (furnaces bottom ashes (FBAs)) for phosphates elimination from synthetic water. Several operational conditions which are detention times, FBAs doses, and phosphate concentrations, were studied to obtain the best conditions which ensure the best possible removal rate and a reasonable operational cost. The results indicated that FBAs are an efficient alternative for phosphate removal with an optimum removal rate of 88.9% with 31 min retention time, phosphate concentration of 5 mg/L, and 550 mg/L FBAs doses. The results obtained were used to construct a model with high reliability at R² = 0.967.

1. Introduction

Water demand is continuously increasing around the world due to the vast population growth with limited clean water resources [1, 2]. This high population growth has additional negative effects regarding wastewater quantities as the discharged wastewater volume is calculated in direct proportion with the population [1, 3]. Therefore, direct contaminants dumping into freshwater should be prohibited and it is necessary to treat wastewater correctly whether it was domestic, agricultural, or industrial effluents in order to eliminate or decrease the negative impact of these pollutants [4, 5]. Phosphates contamination of water results in an alga blooming which in turn influences the water quality. Phosphate is one of the most common contaminating factors for all types of effluents as it is considered as prevalent forms of nutrients [6-9]. The main issue caused by phosphates contamination is that the number of algae would highly increase resulting in blocking the sunlight and consuming the dissolved oxygen and hence, a large number of organisms and plants would die within a short period [10-12]. As a result, the taste, odour, and chemical composition of the water would be highly influenced due to the rapid
increment in the organic matter concentration. The elevated rate of algal blooms may lead to disastrous results regarding human health, industry, agriculture, and tourism. Just to name a few, many researchers found that phytoplankton species have the ability to secrete toxic materials that cause many dangerous diseases and in some cases death [13-16]. When considering the limitations of the allowable pollutant concentrations in water, both the Environmental Protection Agency (EPA) and the European Union Water Framework have their strict instructions, especially for the nutrients. For example, EPA has limited the allowable phosphate concentration to 0.1mg/L for drinking water while it was limited to 0.05 mg/L in the lakes and reservoirs. Removing phosphate using traditional methods normally require costly and complex treatment procedure [10, 17]. For instance, the traditional filtration method with some modifications may be considered as a promising method in terms of treatment efficiency but with using expensive filters, it is considered a non-economical method. Accordingly, this study was devoted to examining the ability to use the wastes of the iron industry (furnaces bottom ashes (FABs)) as an alternative affordable adsorption material for phosphates removals from water.

2. Methods used for phosphate removal

In general, phosphorus removal from water is achieved by converting the phosphorus ions to solid fractions, after that by floatation or precipitation, the generated fractions are eliminated from the solution [10, 18]. Different techniques are used for phosphate ions conversion, for instance, chemical additives, wetlands, biological digestion, and adsorption on the solid surface. The methods are used for phosphate treatment as physical, chemical, and biological. Phosphate biological treatment may be accomplished by utilization of the natural activities of different microorganisms such as microalgae, bacteria, and fungi, depending on the type of microorganism, these methods may be aerobic and anaerobic and the treatment efficiency depends on different factors such as initial pH, initial phosphate concentration, and temperature [19-22]. There are many disadvantages for using biomedical treatment methods for phosphate removal such as the microorganisms require sufficient biodegradable carbon quantities, rapid microorganism growth need to be avoided by controlling the concentration of the nutrient, and pH value should be controlled as an adequate pH change may kill the bacterial colonies [23-30]. Chemical treatment methods, on the other hand, include converting the phosphate precipitates, which is separated by floating or settled precipitation, by adding divalent or trivalent metallic salts to phosphate polluted solutions. In general, chemical methods are considered as simple methods and hence, widely used for the treatment of effluent containing phosphate [31-35]. The most common chemical additives that are used for phosphate treatment are the precipitation of calcium due to its availability, relatively low cost, and simplicity of addition. Aluminium hydroxide (Al(OH)₃) is also known for its effectiveness on phosphate removal from water especially with relatively low initial pH values. On the other side, the chemical treatment methods are generally having the disadvantage of producing toxic sludge as chemical additives are used. Another point is the chemical usage makes the process costly in comparison with other methods [36, 37]. Physical treatment methods are including methods that are used for phosphate treatment without affecting the chemical composition and they are normally reversible which means that the eliminated phosphate can be separated later from the removers. The most common physical treatment methods for phosphate removal are filtration and membrane methods [38, 39].

The filtration process is considered as solids – liquids separation methods that utilize porous materials for the treatment of as many pollutants as possible. After that, different methods may be used for separating pollutants from the fluid such as flocculation, precipitation, impaction, straining, adsorption, and adhesion. The filtration process required different porous materials for pollutant removal, for instance, a composite adsorbent consisting of mesoporous silica particles and ligand was used for phosphate removal. It was found that the adsorption capacity was the most with a pH value of 7 with (159.13 mg/g) adsorption capacity [40, 41]. In general, the filtration process costs are determined by the price of the materials used for filtration which are considered normally high especially for synthesized filters. According to the previous studies about the membrane methods, they showed a good ability for phosphate removal. The
most widely used membrane method is the reverse osmosis in which the phosphate concentration could be minimized as low as 0.008 mg/L in a relatively short time [42]. The reverse osmosis mainly depends on the passage of the polluted water through a semipermeable membrane by pressure which separates the pollutants from the fluid. However, this method is not widely used due to its high operational costs and energy consumption, specific equipment and expertise are required, and the difficulties of disposing of the membrane concentrators. Therefore, the recent studies focused on using industrial by-products or natural materials which are cost-effective and efficient alternatives [43-45]. A good example of using industrial by-products for wastewater treatment; calcined paper mill sludge and fly ash for ammonium nitrogen and phosphate removal. The results, in the literature, demonstrated that this sludge is efficient in the removal of ammonium nitrogen and phosphate at a neutral pH of wastewater and temperature of 20 ± 2°C. It should be highlighted that the recent increase in the average weather temperature (global warming) resulted in an increase in the rainfall in some parts of the world [46-49], which could results in washing out more of phosphate into water sources.

3. Methodology

This study was devoted to studying the ability to use furnace bottom ashes (FABs) as an industrial by-product material for the treatment of phosphate-containing effluent trying to provide low-cost adsorbent. In the beginning, it is important to study some physical properties of the FABs such as particle size, specific gravity, porosity, and surface area. The significance of the particle size test is that it specifies the total surface area which is directly proportional to the adsorption per unit mass. The test was carried out using the sieve analysis with sieves number (4,6,10,16,30, and 50), and the returned mass on each sieve was measured to calculate the particle size.

On the other hand, the specific gravity test was carried out using the gas jar method using the following formula:

\[
\text{Specific Gravity} = \frac{M_2-M_1}{(M_4-M_1)-(M_3-M_2)}
\]

Where

\(M_1\): Mass of the jar + plate
\(M_2\): Mass of the jar, plate, and dry sample together
\(M_3\): Mass of the jar, plate, dry sample, and water together
\(M_4\): Mass of jar + water

After that, using an X-ray fluorescence analyser, a chemical composition test was carried out for the sample to specify the components of the FABs. This test is essential before the removing process to assess the removing efficiency according to the components of the FABs as it was found that materials with iron, calcium, aluminium, or magnesium could be used for phosphate adsorption. After that, synthetic water samples were prepared and the FABs were used for water treatment. The central composite design was used for optimizing the process in terms of the temperature of the water, the dosage of FABs, and the retention time. The adsorption experiments were carried out using analytical chemicals provided by Sigma-Aldrich, UK. Different phosphate concentrations were used in the experiment, they were adjusted and controlled by mixing potassium diphosphate \( \text{KH}_2\text{PO}_4 \) in deionized water and different doses of the FBAs in 1.25 L container.

The polluted water and the FBAs were kept in the same container in contact for different periods to study the effect of the time factor. The operational factors were adopted to agree on the ranges studied in the literature. The retention time was studied between 4 to 60 minutes whereas the FBAs doses were from 100 to 1000 mg/L, and the phosphate concentration was between 5 and 20 mg/L. the concentrations were measured using a Hach Lange spectrophotometer (Model: DR 2800). All the experiments were carried out in Liverpool John Moores University labs at a constant pH value of 7 and 20±2°C.

To design the simulation model trying to achieve optimum water treatment, a central composite design (CCD) utilized in this project as it is efficient and commonly used for water treatment.
experiments. The main variable in this experiment was the phosphate removal and it symbolized as (removal %). The other factors which are phosphate concentration, FBAs dose and time of retention are considered as independent variables and symbolized as Con, Dose, Tem. Minitab 19.2 was used to design the matrix with random order and for 5 levels. The used ranges of the independent variables are shown in table (1).

| Independent variables     | Code  | Low (Level) | Moderate (Level) | Elevated (Level) |
|---------------------------|-------|-------------|------------------|------------------|
| Time of retention         | Tim.  | 2.0         | 31.0             | 60.0             |
| FBAs dose                 | Dose  | 100.0       | 550.0            | 1000.0           |
| Phosphate concentration   | Con.  | 5.0         | 12.5             | 20.0             |

After that, the value of Alpha (α) was calculated as it provides a good prediction of orthogonality and rotatability, the next formula used:

\[ \alpha = \sqrt{2f} \]  

Where \( f \): the number of factors studied.

4. Results and discussion

4.1. Physical properties

The results indicated that the majority of particles of the FBAs were retained between sieve 4 and 16. This means that the majority of FBAs sample has consisted of small-diameter particles (1.180 – 4.750 mm). The sieve analysis results were used to calculate the degree of gradation as illustrated:

\[ UC = \frac{D_{60}}{D_{10}} \]  
\[ CC = \frac{D_{30}}{D_{10} \times D_{60}} \]  

Where UC: is the uniformity coefficient, CC: is the gradation coefficient. \( D_{10}, D_{30}, \) and \( D_{60} \): are calculated by plotting the passing rate with particle size. According to UC and CC values, the sample is considered as well-graded if the UC > 4.0 and 1.0 < CC < 3.0. According to the sieve analysis, \( D_{10}, D_{30}, \) and \( D_{60} \) values were 0.59, 1.48, and 3.09 respectively. Using equations (3) and (4), the UC value was 5.237 and CC was 1.201. The FBAs used are then considered as well graded.

To ensure that the FBAs would not float on the water during the experiment, the specific gravity was calculated and it was found 1.28 which is higher than the water-specific gravity and hence the FBAs would not float on water. The porosity was provided to be 0.66 m²/g while the surface area was 6.8 m²/g. according to the calculated physical properties, the FBAs are suitable to be used for phosphate treatment as it has a high surface area and acceptable porosity and it has a specific gravity higher than water.

4.2. Chemical composition

The chemical composition test showed that more than 20 % of the FBAs chemical composition was consisting of iron, magnesium, aluminum, and calcium oxides. According to the literature, the chemical composition of FBAs is suitable to be used for phosphate treatment.

4.3. Phosphate removal

Batch flow experiments were used to evaluate the FBAs efficiency for phosphate removal. The total experiments were 20 using different operational factors in each to specify the optimum
conditions that ensure a good removal rate with the minimum operational costs. The results are shown in Table 2.

Table 2. Batch flow experiments for phosphate removal.

| No. | Tim | Dose | Con. | Removal % | No. | Tim | Dose | Con. | Removal % |
|-----|-----|------|------|-----------|-----|-----|------|------|-----------|
| 1   | -1  | -1   | -1   | 68.8      | 11  | 0   | -1.68| 0    | 61.8      |
| 2   | 1   | -1   | -1   | 82.9      | 12  | 0   | 1.68 | 0    | 78.7      |
| 3   | -1  | 1    | -1   | 76.5      | 13  | 0   | 0    | -1.68| 88.9      |
| 4   | 1   | 1    | -1   | 84        | 14  | 0   | 0    | 1.68 | 67.3      |
| 5   | -1  | -1   | 1    | 59.8      | 15  | 0   | 0    | 0    | 75.4      |
| 6   | 1   | -1   | 1    | 60.2      | 16  | 0   | 0    | 0    | 79.3      |
| 7   | -1  | 1    | 1    | 71.8      | 17  | 0   | 0    | 0    | 75.1      |
| 8   | 1   | 1    | 1    | 77.3      | 18  | 0   | 0    | 0    | 77        |
| 9   | -   | 0    | 0    | 64.7      | 19  | 0   | 0    | 0    | 78.8      |
| 10  | 1.68| 0    | 0    | 79.9      | 20  | 0   | 0    | 0    | 77        |

The results showed that both FBAs dose and retention time has a directly proportional relationship with the removal efficiency. On the other hand, the phosphate removal rate was negatively impacted by the phosphate concentration increment. This can be attributed to the fact that the available surface area on the FBAs surface would be less sufficient to accommodate the total phosphate ions which resulted in less removal rate. Using higher FBAs will supply a more available surface for adsorption and with more time that results in a higher possibility of contact between FBAs and phosphate ions, which consequently result in higher removal. For instance, the removal rate was 88.9% with 550 mg/L FBAs dose, 5 mg/L phosphate concentration, and with 31 min retention time and it reduced to 60.2% with 60 min retention time, FBAs dose of 100 mg/L, and phosphate concentration of 12.5 mg/L. the obtained results are represented in figures 1 and 2.
The obtained results were used to estimate the removal depending on the values of \( F_1, F_2, \) and \( F_3 \) as illustrated below:

\[
\text{Removal} \% = 71.96 + 0.828 F_1 + 15.810 - 3 F_2 - 0.984 F_3 - 9.3810 - 3 F_1^2 - 0.01310 - 3 F_2^2 + 3.7610 - 3 F_3^2 - 0.0310 - 3 F_1 F_2 - 18.6910 - 3 F_1 F_3 + 0.75210 - 3 F_2 F_3
\]

\( F_1, F_2, F_3 \) (5)

To check the reliability of this equation, the same conditions were used theoretically to calculate removal rates and comparing it with the obtained ones. After that, and to check the suitability of the model equation obtained, the difference between the theoretical and experimental values...
was calculated and it was comparable with a very small difference except for two cases that ensure the reliability of the equation. The results illustrated in table 3.

Finally, to ensure the ability to use the model to simulate the removal of phosphate using FBAs, the value of R² was calculated. It was found to be 96.73% which ensures high reliability of using the model in comparison with laboratory outcomes. Finally, with the significant development in sensing technologies [50-53]; employment of a sensing system to monitor the depletion of the filtration media will significantly enhance the performance of the filtration unit.

Table 3. Differences between predicted and actual removals.

| Run | Removal (%) | Predicted removal (%) | Difference | Run | Removal (%) | Predicted removal (%) | Difference |
|-----|-------------|-----------------------|------------|-----|-------------|-----------------------|------------|
| 1   | 68.8        | 70.39                 | 1.59       | 11  | 61.8        | 64.08                 | 2.28       |
| 2   | 82.9        | 79.09                 | -3.81      | 12  | 78.7        | 79.17                 | 0.47       |
| 3   | 76.5        | 75.07                 | -1.43      | 13  | 88.9        | 92.99                 | 4.09       |
| 4   | 84          | 82.2                  | -1.8       | 14  | 67.3        | 66.36                 | -0.94      |
| 5   | 59.8        | 57.6                  | -2.2       | 15  | 75.4        | 79.07                 | 3.67       |
| 6   | 60.2        | 50.04                 | -10.16     | 16  | 79.3        | 79.07                 | -0.23      |
| 7   | 71.8        | 72.43                 | 0.63       | 17  | 75.1        | 79.07                 | 3.97       |
| 8   | 77.3        | 63.31                 | -13.99     | 18  | 77          | 79.07                 | 2.07       |
| 9   | 64.7        | 56.94                 | -7.76      | 19  | 78.8        | 79.07                 | 0.27       |
| 10  | 79.9        | 56.58                 | -23.32     | 20  | 77          | 79.07                 | 2.07       |

5. Conclusion
Within this research, the ability to use FBAs for the treatment of phosphate-containing effluent was studied to find the optimum operational factors. The results indicated that the FBAs are having appropriate chemical and physical properties to be used for phosphate treatment. Using 550 mg/L FBAs dose, 5 mg/L phosphate concentration, and with 31 min retention time, the removal efficiency was comparable to the literature results with about 89% removal rate. Both FBAs dose and retention time are in direct proportion with the removal rate. In contrast, the phosphate concentration has an inversely proportional relationship with the treatment efficiency. Finally, according to the suitable CCD value, there is a possibility to construct a simulation model for determining the phosphate removal at different FBAs doses, phosphate concentrations, and retention periods. [54-59]

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