Research Article

Novel Application for Activated Carbon Pharmaceutical Formulation as an Efficient Adsorbent of Iron (III) from Contaminated Canned Food

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Additional medical indication has been realized for the active carbon commercialized in pharmaceutical stores for the first time. In this study, the active carbon was used to remove iron (III) from real canned food samples contaminated with iron by an adsorption process, in a new approach. Different parameters affecting the adsorption behavior of active carbon towards iron (III) have been optimized. Under the optimized conditions, namely, pH 4, 5 h contact time, 40 ppm iron initial concentration, and 2 g carbon, the study was applied to some of the real canned food samples. Black olive and green bean canned food samples containing relatively high iron levels of 698.0 and 1168 mg kg^{-1}, respectively, have been selected for this study due to their levels which were above the maximum permitted levels 48 mg kg^{-1} according to the Joint FAO/WHO Expert Committee on Food Additives (JECFA). These samples were digested and treated, and their iron concentration was determined using ICP-AES under the specified optimum conditions. The removal percentage of the real samples was ~91%. In a novel approach, this study simulates the adsorption behavior of active carbon towards iron (III) in the human stomach when canned food with relatively high iron levels has been digested, since the pH value of the human stomach (~3) comes in good agreement with the adsorption optimized pH range (2-4). Moreover, the overall tolerability and safety of active carbon are worldwide known and traditionally documented. These application studies realize the applicability of utilizing active carbon commercially available in pharmaceutical stores to eliminate the toxicity of canned food contaminated by high levels of the investigated elements.

1. Introduction

The problems of the ecosystem are increasing with the development of technology. Heavy metal pollution is one of the main problems [1]. Toxic metal compounds coming from the earth's surface not only reach the earth's waters (seas, lakes, ponds, and reservoirs) but can also contaminate underground water in trace amounts by leaking from the soil after rain and snow. Therefore, the earth's waters may contain various toxic metals. Drinking water is obtained from springs which may be contaminated by various toxic metals [2, 3]. One of the most important problems is the accumulation of toxic metals in food structures. As a result of accu-mulation, the concentrations of metals can be more than those in water and air. The contaminated food can cause poisoning in humans and animals. Although some heavy metals are necessary for the growth of plants, after certain concentrations, heavy metals become poisonous for plants, animals, and humans [4, 5].

Although the organic pollutants can be biodegraded, heavy metals do not degrade into harmless end products [6]. Iron is one of the major constituents of the lithosphere and comprises approximately 5% of it. It is routinely detected in municipal waste effluent, particularly in cities where iron and steel are manufactured. Iron readily complexes with sulphates in the sediments of many surface levels

of water. The primary concern about the presence of iron in drinking water is its objectionable taste. The taste of iron in drinking water can be easily detected even at low concentrations of about 1.8 mg L^{-1} [7, 8].

There are many problems that result from iron toxicity. These include anorexia, oliguria, diarrhea, hypothermia, diphasic shock, metabolic acidosis, and even death. In addition to these, the patient may experience vascular congestion of the gastrointestinal tract, liver, kidneys, heart, brain, adrenals, and thymus [9]. In acute iron poisoning, much of the damage happens to the gastrointestinal tract and liver, which may result from the high localized iron concentration and free radical production leading to hepatoxicity via lipid peroxidation and destruction of the hepatic mitochondria [9]. As a result of iron storage disease, the liver becomes cirrhotic. Hepatoma, primary cancer of the liver, has become the most common cause of death among patients with hemochromatosis [10, 11]. This iron storage disease results from the inability of the intestine to keep out unwanted iron. Instead, this iron accumulates in the liver causing siderosis and damaging the storage organs. Furthermore, when siderosis becomes severe in young people, it leads to myocardial disease which is a common cause of death. Impotence may also occur in young men and amenorrhea in young women. Both these problems related to reproduction are due to iron loading in the anterior pituitary [7, 12].

Estimates of the minimum daily requirement for iron depend on age, sex, physiological status, and iron bioavailability [13]. However, there is no statutory limit specified in the Food Act 1983 for the iron concentration in canned food. The concentration of Fe in canned food generally reflects that—in some instances—the concentration has been increased by corrosion of steel in the can. The average lethal dose of iron is 200–250 mg kg^{-1} of body weight, but death has occurred following the ingestion of doses as low as 40 mg kg^{-1} of body weight [14]. As a precaution against the storage of excessive iron in the body, the Joint FAO/WHO Expert Committee on Food Additives (JECFA) established a provisional maximum tolerable of 48 mg kg^{-1} [15]. In our previous study [16], the iron level not only was found relatively high in canned food compared to fresh food samples but also exceeds the international tolerance levels in some investigated real canned food samples. The results obtained showed that the mean value of iron in canned food (164.1 mg kg^{-1}) was higher than that detected in fresh food (34.35 mg kg^{-1}). It is desirable, therefore, to undertake investigations on the removal of iron metal from aqueous media and applied to such samples. On the other hand, different cheap adsorbents like peat, marine algae, clay, maize cob, bagasse, palm fruit bunch, and *Saraca indica* leaves are used in the removal of heavy and toxic metals from different samples in environmental, food, and industrial strategies [17, 18].

As a case study, this work represents an attempt to demonstrate the behavior of activated carbon in the human stomach towards iron (III) based on its high concentration in some canned foods as reported in our previous work [16]. In a new approach and very promising strategy, a new application of drugs containing activated carbon was reported for the first time. For this propose, the commercialized activated carbon available in local pharmaceutical stores has been used in the removal of iron from some canned food real samples. This sorbent has been selected for the present study based on its availability as well as its well-known safety for human intake. Prior to this process, the adsorption behavior of active carbon has been characterized and optimized.

2. Experimental

2.1. Sampling. The investigated canned food samples (black olives and green beans) were collected from different local markets of the western province of Saudi Arabia. The samples were transported to the laboratory and stored in a clean dry place prior to digestion and analysis.

2.2. Reagents. All reagents used were of analytical reagent grade unless otherwise stated. Deionized water with conductivity < 0.2 μS/cm obtained from a Milli-Q water system (Millipore, France, Elix 10) was used to prepare standard samples, dilute the digested food samples, and wash all glassware throughout. All plastics and glassware were cleaned by soaking in dilute nitric acid, washed with distilled water, rinsed with deionized water, and air dried before use. Iron (III) standard solutions were prepared by appropriate stepwise dilution of certified stock atomic spectroscopy standards (5% HNO$_3$, 3-500 mg kg^{-1}, PerkinElmer, USA) and used for ICP-AES validation measurements. Ferric chloride and nitric acid (69%) were purchased from Riedel-de Haën and Sigma-Aldrich (Germany), respectively. Commercialized activated carbon used in the adsorption studies was obtained from the local drug stores.

2.3. Apparatus. A PerkinElmer (Optima 2100 DV, Norwalk, CT, USA) inductively coupled plasma atomic emission spectrometer (ICP-AES) instrument connected to an AS 93 Plus autosampler was used in this study. The 40 MHz free-running generator was operated at a forward power of 1300 W; the outer, intermediate, and Ar carrier gas flow rates were 15.0, 0.2, and 0.8 L/min, respectively. The pump flow rate was 1.5 mL/min. The carrier gas flow rate was optimized to obtain the maximum signal-to-background ratio.

The microwave digestion system (Closed Vessel Acid Digestion-MARS System-CEM) procedure was chosen for the digestion of all investigated food samples, because of the shorter required time, smaller deviations, and excellent recovery and precision than other procedures [19]. The optimum digestion condition of the microwave digestion system (maximum power 1600 watts, maximum pressure 800 psi, and maximum temperature 300°C) equipped with a closed vessel (Easy Prep) of Teflon reaction vessels was used in all digestion procedures of food samples. The reaction vessels were cleaned using 5 mL of concentrated nitric acid and thoroughly rinsed with deionized water before each digestion.

The pH measurements were performed at 20°C with a HANNA microprocessor pH/ion analyzer (model pH 211) using a combination pH cell immersed in a stirred test solution.

2.4. Sample Preparation. The canned food samples were directly put in Petri dishes without washing and were then oven dried at 120°C for 6 hours. The dried samples were

stored in a fresh plastic bag after cooling and ready for digestion.

2.5. Sample Digestion. Digestion of food samples for iron metal analysis involved a high-performance microwave-assisted digestion using (Closed Vessel Acid Digestion-MARS System-CEM). In this case, portions of 0.5 g of dried samples were weighed and deposited directly onto the base of clean, dry Teflon microwave digestion vessels (Easy Prep vessel). A total of 10 mL of concentrated nitric acid HNO_3 (69%) were added to the investigated food samples that were sealed and digested via one-stage temperature ramping (ramped to 200°C for 25 minutes, held for 15 minutes). Two blank samples containing only nitric acid were prepared in the same way. The solution was allowed to cool, quantitatively transferred into a 100 mL volumetric flask, and then diluted to the mark with deionized water. After dilution, all digested sample solutions were cleared before analysis by ICP-AES.

2.6. Iron (III) Metal Analysis. The iron metal was analyzed using ICP-AES under optimized plasma conditions. Using the auto-sampler, the measured samples were nebulized downstream to the plasma, and the concentrations were automatically determined using the standard calibration graph. The system was adjusted to measure the samples in triplicate, and the relative standard deviation was automatically calculated. The RSD was <2%, and the correlation coefficient was >0.99998.

2.7. Batch Adsorption Experiments. To assess the removal capability of high levels of iron ions, the adsorption behavior of such metals has been performed under optimized conditions. In this study, iron (III) and carbon have been selected in this study as examples for contaminant and adsorbent, respectively.

The stock solution of iron (III) was prepared by dissolving an appropriate amount of salt (0.3 g) in (1000 mL) of deionized water. The different working solutions were properly prepared by dilution of appropriate aliquots of the stock solution using deionized water.

Based on its availability in local drug stores as well as its safety as a treatment of some diseases, the active carbon obtained from these stores has been used as an adsorbent material. The active carbon-based drug was properly grounded using a laboratory jar mill before using in-batch experiments.

To optimize the removal process, many parameters that affect the adsorption behavior of Fe (III) on carbon materials have been studied. These parameters include the effect of Fe (III) concentration, the effect of contact time, the effect of carbon weight, and the effect of pH.

2.7.1. The Effect of Initial Concentration of Iron (III) Ions on the Removal Percentage. The effect of the initial concentration of Fe (III) on its adsorption behavior on carbon has been investigated. A series of multistandard solutions (with concentrations 0.62, 2.42, 5.87, 18.52, 40.26, and 72.61 mg L^{-1}) was prepared and used in this study. The pH of each test solution was adjusted at the value of 4. To 50 mL of each solution, 1 g of carbon (2.8 g of drug formulation) was added, and the mixtures were shaken in a shaker at about 600 rpm for 5 hours. After that, the mixtures were filtered using filter paper. The filtrate (after shaking with carbon) and the multistandard solu-

tions (before shaking with carbon) were analyzed using inductively coupled plasma atomic emission spectroscopy (ICP-AES).

2.7.2. The Effect of Contact Time on the Removal of Iron (III) Ions. To determine the equilibrium time, the effect of contact time on the adsorption behavior of carbon towards iron (III) has been studied. In this study, a standard solution whose concentrations were 40 mg L^{-1} was prepared. To different aliquots of 50 mL of the standard, 1 g of carbon was individually added, and the mixtures were shaken in a shaker at about 600 rpm for 5, 15, 30, 60, 120, and 300 minutes. Each solution was then filtered using filter paper. The filtrate was analyzed using inductively coupled plasma atomic emission spectroscopy (ICP-AES). The investigated contact time was selected in the range of 5 to 300 min to simulate the time that the food generally spends in the human stomach, since the food spends about 5 h in the human stomach [2].

2.7.3. Effect of Weight of Carbon on the Removal of Iron (III) Ions. The effect of adsorbent weight on the adsorption behavior of carbon towards iron (III) has been investigated. In this study, a standard solution containing (Fe^{3+}) ions whose concentrations were 40.97 mg L^{-1} was prepared. To different aliquots of 50 mL of standard solutions, 0.1, 0.5, 1, and 2 g of carbon were individually added. The different solutions were then shaken, filtered, and analyzed using the inductively coupled plasma atomic emission spectroscopy (ICP-AES).

2.7.4. Effect of pH on the Removal of Iron (III) Ions. The effect of pH of the test solutions on the removal percentage of iron (III) by carbon has been investigated. In this study, the pH values of the working solutions were adjusted using small quantities of concentrated nitric acid and sodium hydroxide. A series of standard solutions of iron (III) with a concentration of 40 mg L^{-1} was prepared at different pH values from 2 to 4. Then, to each solution, 1 g of carbon was added, and the solutions were shaken in the shaker for 5 hours. The solutions were then filtered and analyzed using inductively coupled plasma atomic emission spectroscopy (ICP-AES).

2.8. Application. Under the optimized condition which extracted from the previous studies, the application of the removal of some investigated metal ions from some real canned food samples has been assessed. The optimum conditions were as follows: pH of 4, contact time of 5 hours, initial concentration of iron (III) of 40 mg L^{-1}, and weight of carbon adsorbent of 2 g (5.6 g of the drug). Iron (III) has been selected as an example for this study based on its high concentrations found in some of the canned food samples in comparison to their respective fresh food samples in our previous work [16]. The canned food samples underinvestigated in this study were treated similarly to the above-mentioned studies and under the optimized conditions. Thus, the removal percentage was also calculated in the same manner.

In all studies, the percent removal of iron (III) ions was calculated using the following equation [20]:

$$R\% = \frac{C_o - C}{C_o} \times 100,　　　　(1)$$

where C_o is the initial iron (III) ion concentration (before the addition of carbon) of the test solution, mg L^{-1}, C is the final iron (III) ion concentration (after the addition of carbon) of the test solution, mg L^{-1}, and $R\%$ is the removal percentage.

3. Results and Discussion

In a novel approach, the adsorption behavior of iron (III) on active carbon present in some local drugs as an adsorbent has been assessed for the first time in this study. Systematic studies of the adsorption of ferric ions on active carbon under different experimental conditions have been carried out to understand the nature of the adsorption process. It is worth mentioning here that iron has been selected in this study as an example of the investigated metals determined in the studied canned foodstuffs due to their levels being above the maximum permitted levels in about 60% of the investigated canned food samples [16]. However, the investigated drug containing about 60% charcoal has been selected in this study as a sorbent based on its availability in local drug stores. It has been used safely for a long time as a laxative and relieves gas pain by a lot of household people. Moreover, charcoal, which represents a high percentage of the drug, has been used in the removal of heavy and toxic metals from different media in environmental, industrial, and food strategies [21]. Activated carbons are widely used as adsorbents due to their high adsorption capacity, high surface area, porous structure, and high degree of surface, respectively. The chemical nature and pore structure of carbon usually determine the sorption activity [22, 23]. More specifically, the commercialized activated carbon was produced in different formulations based on its application. These revealed BET-specific surface values ranging from 500 to 2000 m^2/g. In addition, the superactivated carbons provided surfaces area higher than 3000 m^2/g. The macro-, meso-, and micropore volumes of the activated carbon magnitude are between 0.5 and 2.5 m^2/g [24–26]. Moreover, the nature of adsorption and its kinetics thermodynamics of versatile species on activated carbon have been reported [24–26]. Instead, this work oriented and reported a new application of the commercial activated carbon in a very promising and novel approach.

3.1. Effect of Carbon Weight on the Removal of Iron (III) Ions. The effect of carbon weight as a sorbent on the removal percentage of iron (III) from aqueous media has been assessed. The experiments were carried out with 0.1, 0.5, 1, and 2 g of carbon, which is equivalent to 0.3, 1.4, 2.8, and 5.6 g of drug tablets, respectively. Appropriate amounts of iron (III) chloride were individually used to prepare standard solutions of iron (III) of 40.97 mg L^{-1} concentration. The mixtures were vigorously shaken for 5 hours and then filtered. The concentrations of the standard iron solutions were determined directly before and after shaking using ICP-AES under the optimized condition mentioned in the experimental section. The removal percentages were then calculated using Equation (1). The results obtained are presented in (Figure 1). As can be seen, the removal percentage increases with increasing the weight of active carbon sorbent until it reaches a steady-state value. Increasing the removal percentage of iron (III) with increasing the weight of active carbon is attributed to the increase of the adsorption capacity and hence the active sites of the sorbent, which would increase the adsorption of the investigated metal ion [4, 6, 7].

Inspection of the data obtained showed that the maximum removal percentage was obtained for Fe^{3+} ions, which is nearly equal to 94.28%, and the minimum removal was obtained for Fe^{3+} at 38.44%. The removal percentage significantly increases as the weight of sorbents increases. A carbon weight of 2 g which gives the maximum removal percentage has been selected in the rest of the experiments and in the application, as well.

3.2. Effect of Contact Time on the Removal of Iron (III) Ions. The effect of contact time on the adsorption behavior of carbon towards iron (III) has been examined. In this study, amounts of 1 g of carbon (2.8 g of drug) were added to aliquots of 50 mL of standard iron (III) of concentration 40 mg L^{-1}. The mixtures were vigorously shaken for different time intervals and then filtered. The concentrations of the standard iron solutions were also determined directly before and after shaking using ICP-AES under the optimized condition mentioned in the experimental section. The removal percentages were then calculated using Equation (1). The investigated contact time was selected in the range of 5 to 300 min to simulate the time that the food generally spends in the human stomach, since the food spends about 5 h in the human stomach [2]. As shown in Figure 2, the removal percentage of carbon for iron (III) increases as the contact time increases, the maximum removal value for iron (III) was obtained with a contact time of 5 hours, and the value was equal to 72.02%. Thus, the contact time of 5 hours was used for the rest of the experiments.

3.3. Effect of pH on the Removal of Iron (III) Ions. The effect of pH is one of the most important parameters controlling the uptake of heavy metals from aqueous solutions [20]. The effect of pH of the test solution on the adsorption behavior of carbon presents in the drug towards iron (III) has been investigated. In this study, amounts of 1 g of carbon (2.8 g of drug) were added to aliquots of 50 mL of standard iron (III) solutions of a concentration of 40 mg L^{-1}. The pH of the solutions was adjusted to different values using small aliquots of concentrated nitric acid and sodium hydroxide solutions. As mentioned above, the concentrations of iron (III) in the different solutions were properly determined directly before and after shaking the mixture for 5 hours. The results obtained presented in Figure 3 show that the removal percentage of iron (III) was independent of the pH of the test solution in the investigated pH range (2-4), which indicates that the maximum adsorption affinities occur in moderately and slightly acidic medium. Experiments with higher pH values were
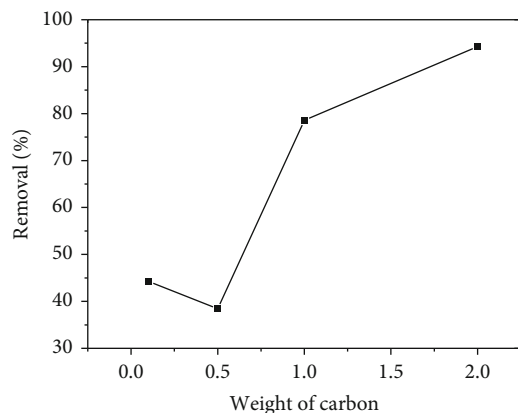
FIGURE 1: Effect of the carbon weight on the removal of iron (III).
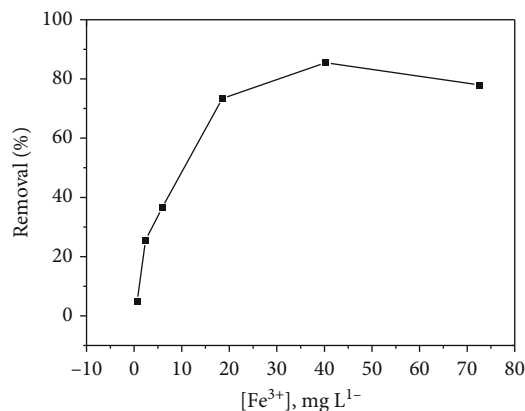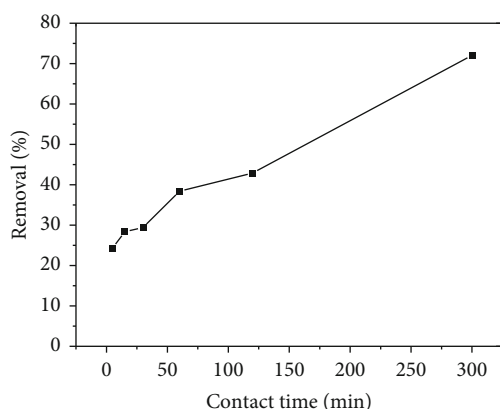

FIGURE 2: Effect of the contact time on the removal of iron (III).

FIGURE 3: Effect of the pH on the removal of iron (III).

FIGURE 4: Effect of the initial concentration of iron (III).

the human stomach when poisoned with canned food containing high levels of the investigated elements.

3.4. Effect of Initial Concentration of Iron (III) Ions on the Removal Percentage. The effect of initial iron (III) concentration on the adsorption behavior of carbon present in the drug has been investigated. In this study, amounts of 1 g of carbon (2.8 g of drug) were added to aliquots of 50 mL of standard iron (III) solutions of different concentrations. After shaking vigorously for 5 hours, the solutions were separated, and the iron concentrations have been also determined. The variation of the removal percentage with the change in initial concentration of iron (III) ions is shown in Figure 4. The removal percentage of iron (III) increases as the initial concentrations increase up to $40.26 \, mg \, L^{-1}$, then decreased as the initial concentration increases. As can be seen, at a constant adsorbent dose (1 g), and a constant time (5 h), the removal (%) of iron (III) increases due to the increase in the sorbent capacity until the steady state reaches the maximum capacity. However, at a higher concentration, the removal (%) gradually decreases with increasing metal concentration which is attributed to the numbers of iron (III) ions relatively higher than the available active sites of the sorbent. Thus, after saturation, the initial concentration of the investigated metal ion has a restricting effect on the removal capacity whilst simultaneously the sorbent media has a limited number of active sites [4, 6, 7].

3.5. Application on the Adsorption Study. Different parameters affecting the adsorption behavior of carbon towards iron (III) have been optimized as previously discussed. These optimized conditions were found 2 g of carbon weight (5.6 g of drug), 5 hours contact time, pH range of 2-4, and $40 \, mg \, L^{-1}$ initial iron concentration. Under the optimized conditions, the adsorption behavior study of carbon towards iron (III) has been applied to real food samples. Based on our previous work [16], black olive and green bean canned food samples containing relatively high iron levels of 698.0 and $1168 \, mg \, kg^{-1}$, respectively, have been selected for this study. These samples were digested using a microwave system and treated, and their iron concentration was determined as described above using ICP-AES under the

avoided to prevent the precipitation of iron (III) as iron hydroxide. This independent pH range is almost fortunately the pH range of the human stomach [27].

Indeed, the adsorption behavior study of carbon present in drugs on the removal of relatively high concentrations of iron (III) simulates its adsorption behavior to such metals in

TABLE 1: Application studies on real canned food samples.

Sample type	Food type	The level of iron (III) pretreatment (mg kg^{-1})	The level of iron (III) after treatment (mg kg^{-1})	Removal (%)
Canned food	Black olives	698.0	66.00	90.5
	Green beans	1168	100.8	91.4

specified optimum conditions. The results obtained are presented in Table 1. The removal percentage of the real samples was ~91%. This study simulates, for the first time, the adsorption behavior of carbon towards iron (III) in the human stomach when canned food with relatively high iron levels has been digested, since the pH value of the human stomach (~3) comes in good agreement with the adsorption optimized pH range (2-4). Moreover, the overall tolerability and safety of active carbon drugs are worldwide known and traditionally documented [28–30]. These application studies realize the applicability of utilizing the active carbon drug to eliminate the toxicity of canned food contaminated by high levels of the investigated elements.

4. Conclusions

In a new and very promising application of drugs containing active carbon, the adsorption behavior of contaminant iron (III) on such drugs has been reported. The study simulates the behavior of the tested contaminant inside the human stomach to avoid the toxicity of the tested species in case of chemical poisoning, since the concentration of iron metal has been detected in black olives and green beans using ICP-AES under optimum conditions and after digestion using a closed vessel microwave digestion system in our previous work. The data obtained showed that the iron metal has exceeded the international tolerance levels in some investigated samples which were collected from the local markets of the western province of Saudi Arabia. In this context, the parameters affecting the adsorption behavior of carbon present in the active carbon drugs towards iron (III) have been investigated and optimized. Under these optimized conditions, namely, pH 4, 5 hours contact time, 40 mg L^{-1} iron initial concentration, and 2 g carbon (5.6 g of the drug), the study was applied to the real canned food samples (black olives and green beans) contaminated by relatively high concentrations of iron after digestion using microwave system. The results showed that there was about ~91% removal of iron (III) from the investigated real canned food samples. This study simulates the elimination of toxicity in the human stomach that comes from the digestion of foodstuffs contaminated by the investigated toxic heavy metal, since the pH value of the human stomach (~3) comes in good agreement with the adsorption optimized pH range (2-4). Moreover, the overall tolerability and safety of active carbon-based drugs are worldwide known and traditionally documented.

Data Availability

All data supporting this work are included within the article.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Acknowledgments

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