



## Removal of Pb(II) ions from Tigris river wastewater in Mosul city by using modified commercial activated carbon

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### Abstract

**Background:** The water pollution of Tigris River is considered the main increasing problem in Mosul city, Iraq. The divalent lead ions are direct or indirect toxic heavy metals ions contaminant confirmed as non-biodegradables and have a long half-life due to corrosions and sedimentation of these metal ions in storm sewer trunk mains, the critical issue is eliminating these heavy metal ions by treating wastewater via commercial activated carbon (CAC), which is the most effective used adsorbent in the adsorption process.

**Aim:** This study aimed to eliminate divalent lead ions by treating wastewater using commercial activated carbon (CAC).

**Methodology:** The surface of commercial activated carbon (CAC) was modified by using concentrated nitric acid. For this purpose, nitric acid in different concentrations (2, 4, 6, 8, and 10 M) and different temperatures (25, 50, 75, and 100) °C were used.

**Results and Discussion:** The best results were observed at 50 °C in which it was used as a standard temperature. A 10 M nitric acid concentration was noticed as the best one. It could be observed that the high pore volume observed in modified activated carbon samples (estimated as the highest adsorption capacity towards iodine, using iodine number determination method) was (707.2 mg/g). This result was supported by methylene blue (190 mg/g). The modified activated carbon (MAC) at the chosen acid concentration was a useful filter to remove the methylene blue organic dye. The modified activated carbon (MAC) was used to adsorb Pb<sup>2+</sup> ions from the Mosul Tigris river wastewaters, the percentage of mean changed activated carbon (MAC) surface adsorption removal efficiency of divalent lead ions were increased by 219% compared with the commercial activated carbon (CAC)

**Conclusions:** Using the methods for iodine number, ash content, methylene blue and density determinations, it was proven that the excellent sorption properties, strong microporous structure, large pore volume, surface of the acidic character, and porosity are the most critical chemical characterization parameters

**Keywords:** nitric acid, commercial activated carbon, adsorption, removal Pb<sup>2+</sup> ion, wastewater purification.

## 1. INTRODUCTION

When contaminants diminished the water, it becomes polluted and cannot be used by humans. Water pollution is the main cause of diseases or even death (about 1.8 million people in 2015) [1,2]. The dangerous problems for the existence of life on earth in the next decades are water pollution. About 70% of oxygen is produced by phytoplankton, and this phytoplankton removes a large part of carbon dioxide from the earth [3]. Organic and inorganic materials may be the primary contaminants, and many are toxic [4]. Among the organic materials, it can be described herbicides (organo-phosphorous or organohalides), organic insecticides, detergents, fats and grease,

processing food leftovers that have oxygen-demanding elements, hydrocarbons - including fuels (diesel fuel, fuel oil, gasoline, and jet fuels) and motor oil lubricants - and burning of the fuel by-products. All of them can come from runoff of stormwater [5]. While the examples of inorganic water pollutants include the industrial discharges acidity (e.g., sulfur dioxide), ammonia by-products from food processing waste [6], in addition to copper, mercury, uranium, and zinc [7].

Plumbing materials lead pipes are the sources of contamination caused by lead (Pb); this contamination caused significant health and

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environmental problems. This contamination is mainly found in cities and homes built older than in 1986 [8]. The lead material is considered highly toxic and harmful to human health because of its ability to be stored inside the human body over time, even if the person exposed to low levels, so its percentage is set to zero in water [9]. Exposure of kids to low lead leads to poor blood cell function and hearing, also caused short stature and damage to the nervous systems [10]. Some techniques, such as ultrafiltration, reverse osmosis, chemical precipitation, and ion exchange, have been advanced to treat lead wastewater contamination [11]. Taking, for instance, the Ion exchange, it is high cost and needs resin regeneration or replacement. However, it is a highly effective technology in depressing specifically charged contaminants. The significant advantages of using this technology are sustainability, simplicity, safety, selectivity, economy, and applicability. This study's central observation was the selectivity and efficiency to remove  $Pb^{2+}$  using cation exchange resin Purolite C100E. Using  $Ca(NO_3)_2$  utilization for regeneration and the purification treatment of regeneration total recycling effluents for  $Ca(NO_3)_2$  [12]. Reverse osmosis and unpolluted ultrafiltration techniques are also costly. Whereas chemical precipitation is a simple procedure, but a high volume of sludge could be generated. The electrocoagulation procedure could be the choice that meets the necessities of cleanliness, operation simplicity, and low costs. The principle depends on electrolytic processes that include disrupting, emulsified, dissolved, or postponed pollutants in an aqueous medium using an electric current [13]. The using of nitric acid with irradiation under a microwave oven raises the temperature and produces a good chance of the functional groups on the pore surface because this temperature rising helps carbonization and divalent lead ions to changes in the pore structure [14,15]. The adsorption of contaminants enormously depends on electrostatic and dispersion interaction between the impurity and the surface property [16], in addition to the result that polycyclic aromatic hydrocarbons can be removed by microwave irradiation [17]. In its use for modifying the surface of commercial-grade carbon, a potent oxidizing agent nitric acid showed that this surface modification increased to adsorb the copper ion from aqueous solutions and helped in the regeneration of carbon [18]. In 2008, and at the examination of modified activated carbon (MAC) was capacity properties of tailored adsorption, Stavropoulos and co-workers made activated carbons with preferred properties accomplished by alteration of functional surface groups and introduction of acidic/basic properties using the nitric acid treatment, partial oxygen gasification, and urea impregnation followed by pyrolysis in the stream of saturated urea [19]. Many studied nitric acid modified activated carbon

(MAC) as a good technique for getting rid of heavy metal divalent lead ions from wastewater. Many influences affect the efficiencies of these, such as the concentration, acidity, ionic strength, and adsorbent modification procedure [20-22]. The adsorbent properties of MAC are used to adsorb many heavy metals from wastewaters such as the divalent lead ions which are the most toxic, non-biodegradable and have long half-life metal which increases in wastewater directly or indirectly in the Mosul Tigris river [23], these divalent lead ions increase in Mosul Tigris river due to corrosions and sedimentation in storm sewer trunk mains. Elimination of Pb (II) has become an essential issue by treatment of wastewater via commercial activated carbon (CAC). This is the widely used and most effective adsorbent in the process of adsorption Acidic modification of commercial activated carbon (CAC) by its treating with concentrated nitric acid as oxidizing acid has been employed to adsorb this metal [26]. The most effective adsorption method between all the cited procedures, this adsorption process has been broadly explored because adsorption based methods are simple to plan, easy to run, cheap and show greater efficiency towards the removal of various toxic pollutants including metals, in addition to that activated carbon is commercially available adsorbent and has been widely used for the treatment of wastewater [24,26]. Besides, the CAC is shown to be a tremendous operative adsorbent and this is because of the excess capacity of CAC to adsorb contaminants. This system design provides a worthy-quality output mostly because of its features characteristics and excellent surface area due to its porous texture. It can be easily modified by chemical treatment to vary their properties, and the processes that use these standard adsorbents are frequently carried out in a batch mode [30]. All these with the ability of activated carbon to the removal of both cationic and anionic metal contaminants from water [27]. As part of a continuous program directed toward the study, preparation, and development of activated carbons [28], it becomes of interest to investigate preparative routes for acidic modification of commercial activated carbon (CAC) by treating concentrated nitric acid as oxidizing acid; this oxidation may produce acidic surface oxides and evolving of carbon monoxide after high-temperature heating. These results increase the hydrophilicity of the acidic groups of the surface. The treatment with nitric acid affects the surface area and porosity of commercial activated carbon (CAC); this treatment fixed the largest number of oxygen complexes that evolved as carbon dioxide [29]. Also, because it is one of the heavy elements that enter the freshwater environment and disturb the ecological balance, the reason for the high concentration of liquid and industrial wastewater is discharged [30]. The divalent lead is one of the essential contaminants that pollute

the Tigris river (the only drinking source river that crosses the Mosul city) with a primary maximum contaminant level (MCL) of (15  $\mu\text{g}\cdot\text{L}^{-1}$ ) (treatment technique) [31,32]. Therefore, this study aimed to use the modified commercial activated carbon (MAC) to remove divalent lead ions from the Tigris river wastewaters, Mosul.

## 2. MATERIALS AND METHODS:

### 2.1. Materials and Equipment's

The commercial activated carbon (CAC) was obtained from Seimahoshi B1 Development Moto fujisawa Pharmaceutical Industry Co., Ltd.; Japan. All reagents and solvents were obtained from commercial suppliers and were used without further purification. Atomic absorption Spectrophotometer (Perkin elmer /USA) was used to analyze the filtrate for residual  $\text{Pb}^{2+}$  concentration in mg/L. The shaker used was (HamburG 20 shau, Germany), and the electrical balance was (And. Ser. Acapap Ter De12 v, 0.3n).

### 2.2. Methods of Activation

The commercial activated carbon (CAC) was crushed and was screened using a 1.18 mm sieve to obtain granules of the same size. Then, it was washed with distilled water to remove impurity, desiccated for 72h, and used in the following methods [28].

#### 2.2.1. Methods for Iodine number determination

Iodine number tests were conducted based on the American Society for Testing and Materials method

A total of 1g of activated carbon was placed in a 250 ml dry flask. Into it, 10 ml solution of hydrochloric acid (5%) was added. The resulting mixture was heated for 30-60 minutes, then this mixture was cooled to room temperature. A total of 100 ml of iodine solution (0.1M standard) was added to the result mixture and the flask was shaken for 30 minutes. The content was filtered and the result filtrate was flushed with (0.1M standard) solution of sodium thiosulphate until the color of this solution became pale yellow.

A starch index solution of starch (1ml) was added to this result yellow solution, then the correction was completed until the blue color of the starch guide disappeared according to the size measurement.

The thiosulfate used, then according to the weight of the adsorbed iodine, the following equation was applied:

$B \times \text{ml of thiosulfate solution used}$

$X = A - 12693 \times N1 = A$

$\times 126.93 N2 = B$

Weight of the activated carbon model used = M

#### 2.2.2. Methods for ash content determination

A total of 1g of activated carbon in a ceramic vessel was placed in an electric oven at (1000°C) for 3 hours. Then, the product mixture was cooled down and weighed by a sensitive balance. The difference between the weights gives the percentage of ash in prepared activated carbon models

#### 2.2.3. Methods for calculations of $\text{Pb}^{2+}$ ions concentrations

Sealed plastic small bottles carefully cleaned was used for river wastewater samples collection in September 2019. The selection samples site was located at the Tigris River in Mosul in the north of Iraq. These samples, which are clean and odorless, were concentrated and lead ions are ionized using atomic absorption spectrophotometer (AAS) [31].

##### 2.2.3.1. Method (A)

A total of 3 ml of 1M Nitric acid was added to the beaker containing 2g of the commercial activated carbon (CAC). The mixture solution was transfer into crucibles and stirred for 25, 50, 75, and 100°C, respectively. After the mixture was converted to paste, the oxidizing product samples were then leached by washing with distilled water (250 ml) several times until neutral pH. The MAC samples were then dried overnight at 105°C and stored in a desiccator. The properties of modified activated carbon (MAC) samples after oxidation by 1M nitric acid with different temperatures are shown in Table1.

##### 2.2.3.2. Effecting of Concentration of nitric acid, Method (B)

A total of 3 ml of different nitric acid concentrations (2M, 4M, 6M, 8M, and 10M) were added to 2g of commercial activated carbon (CAC) (sample 2). The oxidation was started by stirring the mixture with fixing the temperature at 50°C, and the experiment was worked up as in method (A). The properties of modified activated carbon (MAC) samples after oxidation by different concentrations of nitric acid at 50°C temperatures are shown in Table 2.

#### 2.2.4 Method for divalent lead ions removing

A conical flask containing eight wastewater samples from the Mosul Tigris river (100 ml) was shaken in an orbital shaker to agitate the flasks at room temperature for a scheduled period (8 hours). At the end of the expected agitation time, the flasks were removed from the orbital shaker and the mixture was filtered. Atomic absorption Spectrophotometer was used to analyze the filtrate for residual  $\text{Pb}^{2+}$  concentration in mg/L. The initial concentrations of  $\text{Pb}^{2+}$  ions on adsorption over commercial activated carbon (CAC) (using 10 g) and modified activated carbon (MAC) (sample 9), respectively, are shown in Table3.[32],

### 2.5. Atomic absorption Spectrophotometer Analysis

Describe here how the AAS analysis were performed. What parameters, solution preparation, all features, procedures, steps of this analysis.

### 2.6. Metal Uptake

The metal uptake per unit weight of adsorbent ( $q_e$ ) and the abstraction qualification (%) of the adsorbent preparations were calculated using the freundlich equation explaining the adsorption of solutions on heterogeneous surfaces by the following equations [33]:

$$q_e = (C_o - C_e) / m * v \quad (\text{Eq. 1})$$

where  $C_o$  and  $C_e$  are the concentrations (mg/L) of  $Pb^{2+}$  before and after adsorption respectively,  $V$  (ml) is the volume of the  $Pb^{2+}$  and  $m$  (g) is the mass of the adsorbent [34].

$$q_e (\text{CAC}) = (C_o - C_e) / m * v \quad (\text{Eq. 2})$$

$$q_e = (19.3 - 10.56) / 20 = 8.74 / 20 = 0.437 \text{ g/l}$$

$$q_e (\text{MAC}) = (C_o - C_e) / m * v \quad (\text{Eq. 3})$$

$$q_e = (19.3 - 0.125) / 20 = 19.175 / 20 = 0.9587 \text{ g/l}$$

$$\text{Removal efficiency (\%)} = [(C_o - C_e) / C_o] * 100. \quad (\text{Eq. 4})$$

$$\text{Removal efficiency of (CAC) (\%)} = [(19.30 - 10.56) / 19.30] * 100 = 45\%$$

$$\text{Removal efficiency of (MAC) (\%)} = [(19.30 - 0.125) / 19.30] * 100 = 99\%$$

#### 2.6.1 Isotherm Models

The best corporate sorption models used to fit the experimental data applied for lead metal are Temkin, Freundlich, DRK, and Langmuir. The Freundlich model, which is used in this work, repeatedly represents the initial surface adsorption followed by condensation, resulting from robust solute-solute interaction. Freundlich model is of the form[11].

$$Q_e = K_F (C_e)^{1/n} \quad (\text{Eq. 5})$$

where:

$K_f$  = The Freundlich constant.

$n$  = The correction factor denotes the adsorption capacity

$C_e$  = concentration of the solution at equilibrium (mg/L)

The values of  $K_f$  and  $n$  were obtained by plotting  $Q_e$  versus  $C_e$  and fitting the data to the power-law equation (i.e.,  $y = kX^n$ ) using Microsoft excel applications [35,36].

### 3. RESULTS AND DISCUSSION:

The Pb (II) concentration (contaminate level) in wastewater found in the province of Mosul city was (19 mg.L<sup>-1</sup>). This concentration exceeds the maximum Pb (II) acceptable (15 µg.L<sup>-1</sup>) according to the US Environmental Potential Agency (US EPA)

[37]. The structure and surface chemical properties of activated carbon after nitric acid modification were examined. The surface of commercial activated carbon (CAC) was modified by using a cheap concentrated nitric acid. These activating processes were obtained by method (A) in which wet oxidizing nitric acid is used with varying the temperatures of the solution in the range of (25, 50, 75 and 100) °C as shown in Table (1).

Sample (2) of the best results of 50 °C is used as a fixed sample temperature with different acid concentrations (2,4,6,8 and 10M), method (B), Table (2). Sample (9) of 10M acid concentration gives a well-developed surface area, large pore volume, and good sorption properties with a strongly microporous structure and the surface of acidic character. Study the porosity, which is the most crucial parameter of chemical characterization, the highest pore volume available in the modified activated carbon samples, i.e., the highest sorption capacity towards iodine is (707.2mg/gm), this result approves with methylene blue 190mgm/gm indicates that the treated modified activated carbon (MAC), i.e. sample (9) at 50 °C and 10M of acid concentration is the most attractive candidate for removing the organic dye of methylene blue which shows a significant reduction of the color[38-41]. This sample also gives the lowest ash percentages (1.9%), which indicates high carbon purity.

Thus, in this work, sample (9) of modified activated carbon (MAC) is used to adsorb  $Pb^{2+}$  ions from wastewaters (in Mosul Tigris River) compare with commercial activated carbon (CAC).

Two adsorption columns are used sample (9) of the commercial activated carbon (CAC), and nitric acid modified activated carbon (MAC) respectively to compare the adsorption behavior of divalent lead ions under constant conditions of pH and time. The results showed that the percentage of mean modified activated carbon (MAC) surface adsorption removal efficiency of divalent lead ions increases by 219% compared with commercial activated carbon (CAC). The removal efficiency of lead ions by various activated carbons is always the highest compared with heavy metal ions copper, chrome, and cadmium ( $Pb^{2+} > Cu^{2+} > Cr^{3+} > Cd^{2+}$ ) [38].

### 4. CONCLUSIONS:

1. Modified activated carbon is more effective than non-axial activated carbon.
2. The use of nitric acid as a modified agent was very good.
3. Removal of the lead ions using modified activated carbon was very effective.
4. sample (9) of modified activated carbon (MAC) is the best best specifications to adsorbed  $Pb^{2+}$  ions from wastewaters (in Mosul Tigris river) compare with commercial activated carbon (CAC).

**Table 1. The properties of modified activated carbon (MAC) samples after oxidation by 1M nitric acid with different temperatures.**

Sample	Temp. (°C)	Iodine No. (mg/g)	MB (mg/g)	Density (g/cm <sup>3</sup> )	Ash (%)
CAC	---	627	172.58	0.3112	3.27
1	25	654	161.7	0.332	3.6
2	50	668	190.3	0.315	2.45
3	75	599	188	0.337	4.33
4	100	627	187.59	0.326	2.68

**Table 2. The properties of modified activated carbon (MAC) samples after oxidation by different nitric acid concentrations at 50°C temperatures.**

Sample	Nitric acid concentration (M)	Iodine Number (mg/g)	Methylene blue (mg/g)	Density (g/cm <sup>3</sup> )	Ash (%)
5	2	644	175.5	0.292	2.29
6	4	487.5	177	0.316	2.31
7	6	515	185	0.32	2.33
8	8	529	187	0.342	2.33
9	10	702	190	0.356	1.90

**Table 3. The initial concentrations of Pb<sup>2+</sup> ions on adsorption over commercial activated carbon (CAC) and modified activated carbon (MAC) (10 g each) (sample 9), respectively.**

Sample no.	10	11	12	13	14	15	16	17	Mean (mg/l)
Concentrations of (mg/L) Pb <sup>2+</sup>									
Before adsorption.	19.1	19.2	19.4	19.2	19.8	19	19.6	19.5	19.3
After adsorption by (CAC).	10	11	10	10	11.1	11.9	10.5	10	10.56
After adsorption by (MAC).	0.10	0.13	0.15	0.13	0.09	0.10	0.11	0.11	0.125

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