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Effects of radical scavengers on humic acid removal by electron beam irradiation

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ABSTRACT

Despite many efforts to remove humic acid (HA) in water resources, this problem is not overcome, especially in underdeveloped countries. The aim of this study is to evaluate the effects of inorganic anions (Cl^-, HCO_3^-, SO_4^{2-}, and NO_3^-) as radical scavenger on the efficacy of electron beam (EB) irradiation for the removal of HA from aqueous solutions. In this study, stock HA solution was prepared in alkaline condition; furthermore, different concentrations of anions were added to all HA samples. Here, the effect of different electron beam absorbed doses (1, 3, 6, 9, and 15 kGy) on the removal of HA is studied. HA concentration was measured using UV–vis spectrophotometer at 254 nm. The results showed that by increasing absorbed dose from 1 to 15 kGy, HA removal efficiency increased from 28.2 to 77.7%, respectively. While, by increasing different inorganic anion concentrations from 25 to 100 mg/L, the removal efficiency decreased. According to the results, nitrate has more inhibitory effect on the hydroxyl free radicals and HA removal efficiency in the presence of EB irradiation compared with chloride, bicarbonate, and sulfate anions. Furthermore, the kinetic model fitted the pseudo-second-order kinetics well.

Keywords: Humic acid; Electron beam; Inorganic anion; Radical scavenger; Water treatment

1. Introduction

Many world water resources, particularly surface waters, contain plenty of natural organic matters (NOMs) [1,2]. NOMs are a group of organic macromolecules which cause different problems in the water treatment processes, including the formation of disinfection byproducts (DBPs) such as trihalomethanes (THMs), known as carcinogenic and mutagenic compounds. Approximately, 50% of natural organic matters in water are humic substances (HSs) [3].

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In general, HSs are subclassified to humic acid (HA), fulvic acid, and humin [2]. HAs are a part of HSs which are not soluble in water under acidic conditions ($pH < 2$); however, they are dissolved in higher pH [4]. Due to their adverse effects, HSs should be removed before water treatment disinfection processes [1].

In recent years, several methods, including electromicrofiltration (EMF) [5], adsorption on activated carbon [6,7], ion exchange [8], enhanced coagulation [9], membrane separation [10], precipitation, filtration, and advanced oxidation processes (AOPs) have been applied for removing NOMs from water resources [4]. Among these methods, AOPs appear more appropriate for water resources treatment that contain natural organic matters [2] and cause oxidizing of NOM into harmless end products such as carbon dioxide and water [4].

Hydroxyl radicals ($OH^{\cdot}$) are formed during AOPs [11] and react with organic compounds rapidly owing to their high oxidation potential and non-selective performance [12,13]. Radical scavengers (RSs) such as chloride (Cl^-), nitrate (NO_3^-), sulfate (SO_4^{2-}), and bicarbonate (HCO_3^-) can react with hydroxyl radicals and then decrease organic pollutants [14].

Ionizing irradiation such as UV, X, gamma rays, and accelerated electrons are efficient energy forms which can remove the organic contaminants from water and wastewater [15]. Irradiation efficiently depends on the factors such as irradiation energy, absorbed dose, concentration, and molecular structure of contaminant, temperature, and interactions between different factors. Using energetic electron beams is one of the advanced oxidation processes for water and wastewater treatment [15]. Electrons break the water molecules; consequently, highly reactive chemical species including hydroxyl radicals, aqueous electrons, and hydrogen radical will be regenerated, EB irradiation does not need disinfectant chemicals, while no toxic byproducts are traced during short contact time [16].

To the best of our knowledge, there is no available paper on the effects of RSs on HA removal by EB irradiation. In the present work, an attempt was made to develop a methodology for removing HA from aqueous solution by EB irradiation process in the presence of common inorganic anions (Cl^-, HCO_3^-, SO_4^{2-}, and NO_3^-) as RS and to evaluate the kinetics of these inorganic anions.

2. Materials and methods

2.1. Materials

In this study, HA powder was purchased from Sigma–Aldrich Co., USA. H_3PO_4 and sodium hydroxide 0.1 N for pH adjustment were supplied by Merck Co., Germany. NaCl, $NaHCO_3$, Na_2SO_4, and $NaNO_3$ were provided from Merck Co., Germany. All materials used in this study were pure analytical.

2.2. HA/inorganic anions solution preparation

For preparing different concentrations of HA (10, 25 and 50 mg/L), a stock solution of this compound was prepared by dissolving HA powder in 0.1 N sodium hydroxide. To investigate the effects of different concentrations of RSs such as Cl^-, HCO_3^-, SO_4^{2-}, and NO_3^-, the sodium salt of these compounds was separately added to HA solution and was mixed well. Prepared samples were transferred to Petri dishes with 40 ml sample volume, and then irradiated by energetic electron beams.

2.3. Electron-beam irradiation

Electron-beam irradiation was produced by an electron accelerator, TT 200 model was manufactured by IBM, Belgium in Radiation Application Research Center, Yazd, Iran. The EB energy was 10 MeV. The study was conducted in different absorbed doses of 1, 3, 6, 9, and 15 kGy and different pH of 4, 6, 7, 8, and 10. The pH was adjusted by 1 N phosphoric acid by pH meter HACH, model HQ40d, USA.

Concentration of HA solution in the samples was measured at 254 nm, before and after irradiation by UV–vis spectrophotometer, SP-3000 PLUS Model, Japan. In this study, the removal efficiency of HA is calculated using the following formula:

$$R\ (\%) = \frac{\mathrm{Abs}_0 - \mathrm{Abs}}{\mathrm{Abs}_0} \times 100 \tag{1}$$

where Abs_0 and Abs are initial and final absorbencies of HA, respectively, and R is removal efficiency of HA [17]. In this study, using EB irradiation, the HA removal kinetic was also investigated. The second-order kinetics equation was represented in Eq. (2):

$$\frac{1}{C} - \frac{1}{C_0} = KD \tag{2}$$

where C is the residual concentration of HA, C_0 is the initial concentration of HA, D is the absorbed dose in kGy, and K is the second-order rate constant in mg^{-1}/kGy [18,19].

3. Results and discussion

3.1. Effect of pH

In wastewater treatment, the pH value of a solution is an important factor. The removal efficiency of

HA at different pH values is shown in Table 1. As shown, HA removal in solution without RSs, in the presence of bicarbonate and sulfate at pH 8 was higher than removal efficiencies at other pH values. There is no significant difference between HA removal results at different pH values ($p > 0.05$).

Wang et al. [20] worked on the effects of UV light radiation on the coagulation of HA. Their study determined that UV light radiation could effectively improve the HA removal at pH above 6.0 [20]. Oskoei et al. studied the removal of HA from aqueous solution using zinc oxide nanoparticle irradiated by the ultraviolet light (ZnO/UV) and found that an increase in the pH of the environment decreased the HA removal efficiency [21]. Yazdanbakhsh et al. [22] investigated the performance of the peroxi-electrocoagulation process (PEP) for HA removal from aqueous solutions. They observed that the highest removal efficiency was at pH 3.0, while with increasing pH, the process efficiency decreased [22]. Jebali et al. [23] evaluated the adsorption of HA by amine-modified nanocellulose and found that the adsorption decreased with increase in pH. They also found that there were significant differences between HA adsorption at pH 9 vs. pH 7, 5, and 2 ($p < 0.05$) [23]. In another study, Ghaneian et al. [24] worked on HA degradation by the synthesized flower-like Ag/ZnO nanostructure and found that faster degradation of HA was achieved at pH 7 [24]. Degradation of marine HA was done using an ozone treatment technique by Imai et al. [25]. Their results revealed that after the ozone treatment, the absorbance of HA decreased; furthermore, more than 80% degradation was achieved under neutral pH [25]. Study of Zhang and Bai [26] on mechanisms and kinetic of HA adsorption onto chitosan-coated granules indicated that the adsorption was strongly pH dependent. For instance, high amount of HA was adsorbed at neutral and acidic conditions, while the adsorption reduced remarkably by increasing pH [26]. In another study, Anirudhan et al. studied adsorption of HA on amine-modified polyacrylamide–bentonite composite. They found that 99.9% adsorption was observed at pH range 4–7 [27].

3.2. Effect of HA concentration

The results of this study revealed that with increasing HA concentration, removal efficiency decreased. Oskoei et al. also confirmed that an increase in the initial concentration of HA decreased the removal efficiency using UV/ZnO nano-photocatalysis [21]. The study by Ghaneian et al. [24] on HA degradation by the synthesized flower-like Ag/ZnO nanostructure revealed that as the concentration of the HA increased, the rate of photodegradation decreased. For instance, for 10 and 25 mg/L HA concentrations, more than 90% of HA was degraded, while for 50 mg/L almost 70% of degradation occurred [24]. The results of these studies were consistent with the results of our study. Table 2 shows the HA removal efficiency, while Fig. 1 shows the correlation coefficient values (R^2) of second-order kinetics in the presence of different HA concentrations and different irradiation doses without inorganic anions.

According to the results presented in similar articles, pseudo-first-order and pseudo-second-order kinetics models have been applied to express the removal rate of different contaminants during irradiation with energetic electron beams [28]. In most studies, the pseudo-first-order kinetics is common [29,30]. In this study, the kinetic data was obtained from batch studies and analyzed using pseudo-first order and pseudo-second order models and showed that the R^2 values for the pseudo-first-order kinetics with 10, 25, and 50 HA concentrations have been 0.8768, 0.8547, and 0.9752, while the R^2 values for the pseudo-second-order kinetics for different HA concentrations were greater than 0.95; therefore, the results

Table 1
Removal efficiency (%) of HA at different pH values (irradiation dose = 3 kGy, HA = 25 mg/L, inorganic anions = 50 mg/L)

	Inorganic anions				
pH	None	SO_4^{2-}	HCO_3^-	Cl^-	NO_3^-
4	46.88 ± 2	45.32 ± 2	38.21 ± 2	45.77 ± 2	26.36 ± 2[a]
6	48.01 ± 3	45.23 ± 2	44.56 ± 1	47.13 ± 4	23.17 ± 1[a]
7	47.2 ± 3	46.68 ± 3	42.84 ± 2	46.43 ± 2	22.91 ± 2[a]
8	48.23 ± 2	47.73 ± 2	46.7 ± 2	46.34 ± 1	24.93 ± 1[a]
10	46.04 ± 2	42.38 ± 2	44.51 ± 2	45.53 ± 2	25.31 ± 2[a]

[a] $p < 0.05$ compared with other groups at same pH.

Table 2
Removal efficiency (%) of HA in the presence of different HA concentrations and different irradiation doses (anions concentration = 0 mg/L, pH 8)

	HA concentration (mg/L)		
Dose (kGy)	10	25	50
1	38.4 ± 2	28.2 ± 1	19.2 ± 1
3	62 ± 2	51 ± 2	35.7 ± 2
6	76.9 ± 2	67.2 ± 2	43 ± 2
9	84.3 ± 4	74.1 ± 3	51.2 ± 2
15	87.4 ± 4	77.7 ± 2	62.5 ± 3

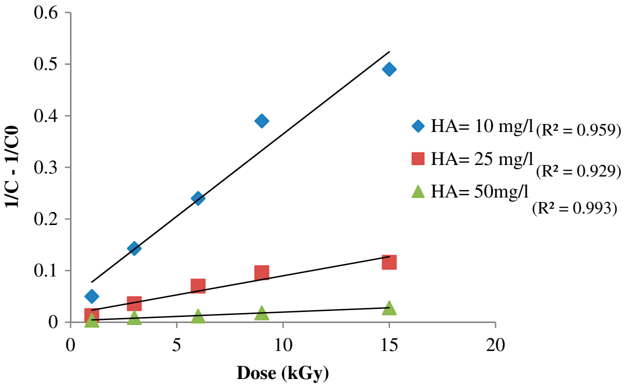

Fig. 1. R^2 second-order kinetics in the presence of different HA concentrations and different irradiation doses (anions concentration = 0 mg/L, pH 8).

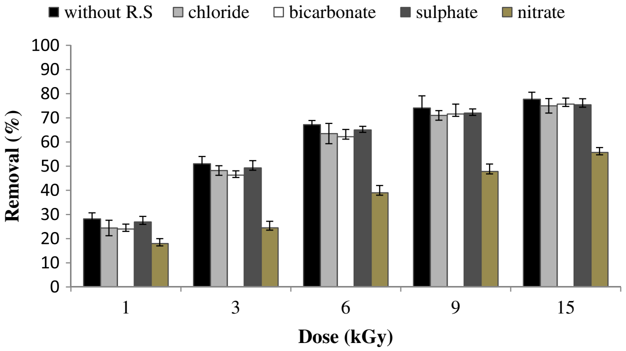

Fig. 2. The effect of EB absorbed dose on HA removal efficiency (HA concentration = 25 mg/L, anions concentration = 50 mg/L, pH 8).

of this study seem to fit the pseudo-second-order kinetics.

The results of this study are consistent with Kwon et al. [18] in the removal of iopromide (IPM) and degradation characteristics in EB irradiation process. While the results of their study showed that IPM was removed by first-order kinetics ($R^2 = 0.85$), the second-order kinetics ($R^2 = 0.98$) was better fitted in the removal of IPM ($R^2 = 0.98$) [18]. The study by Anirudhan et al. on the HA adsorption by amine-modified polyacrylamide–bentonite composite also showed the kinetics of adsorption described by pseudo-second-order model [27]. Yoon et al. [19] evaluated the efficiency of acetone removal using EB irradiation in groundwater. Based on their results, the acetone removal reactions followed the pseudo-first-order kinetics model [19]. According to the results of Ghaneian et al. [24] in the photodegradation of HA study, R^2 of pseudo-first-order and pseudo-second-order models obtained were 0.99 and 0.91, respectively. Therefore, their results followed pseudo-first-order kinetics [24].

3.3. The effect of EB absorbed dose

According to the results of this study, with the increase in absorbed dose, HA removal efficiency increased while the presence of inorganic anions led to decreasing removal efficiency. As a result, the inhibitory effect of nitrate is much more than other RSs, while approximately similar effects were observed for chloride, carbonate, and sulfate. These results are shown in Fig. 2.

Irradiation of water with energetic EB leads to the formation of excited species, free radicals, and ions. Energetic electron beams are able to release the energy in water and start thousands of secondary reactions. During irradiation with energetic electron beams, water radiolysis occurs at a short time. The EB irradiation in water resulted in the production of several radicals, ions, and molecules [18].

The reactive species such as hydroxyl radical, hydrated electron (e^-_{aq}), and hydrogen atom, as well as reactive species H_2O_2, H_3O^+, and H_2 are produced as follows [3,31]:

$$H_2O \rightarrow OH^{\cdot} + e^-_{aq} + H^{\cdot} + H_2O_2 + H_3O^+ + H_2$$

The results of Palfi et al. study on the decomposition of HA and its derivatives by ionizing radiation (4 MeV electrons and ^{60}Co gamma irradiation) in aqueous solutions revealed that these compounds are effectively destroyed by hydroxyl radicals, when e^-_{aq} intermediates are formed during water irradiation [32]. Hydroxyl radicals in the irradiated process with energetic electron beams are also formed and led to the contaminant removal efficiency increase. In addition, with the increase in absorbed dose, more hydroxyl radicals are produced [33] which are associated with increasing HA removal efficiency. Change in irradiation dose is an important parameter for the evaluation of irradiation process since with increase in the radiation dose, the pollutant removal efficiency increases.

Ting and Jamaludin [31] studied on decolorization and decomposition of organic pollutants for reactive and disperse dyes using EB technology. In their study, it was found that the EB is very effective in removing highly colored resistant organic compounds. Experiments were conducted with irradiation of samples in Petri dishes. Dye removal was performed using 0.5, 2.4, 8, 18, 41, 53, 108, and 215 kGy doses. In this study, by increasing irradiation dose, the percentage of dye removal increased [31].

Abdou et al. [17] study showed the efficiency of EB and gamma irradiation for treatment of dye solutions. Their results revealed that by increasing the irradiation dose from 5 to 7 kGy, decolorization increased [17]. The results of these studies are consistent with the results of this study.

3.4. Effect of inorganic anions (Cl^-, HCO_3^-, SO_4^{2-}, and NO_3^-)

One of the problems in the operation of advanced oxidation processes is high concentrations of RSs in some wastewaters that can react with OH·, and reduce the efficiency of advanced oxidation processes. Furthermore, other factors such as suspended solids, pH, type and nature of residual TOC and other wastewater components affected treatment processes. In advanced oxidation processes, these factors caused the oxidation of organic compounds which do not completely oxidize organic matters into CO_2 and H_2O by oxidants. In other words, these factors led to the increase in the oxidant and reduction of degradation of pollutants. Products of the reaction between hydroxyl radicals and chloride, bicarbonate, sulfate, and nitrate ions are deactivating radicals of these agents which are effective in preventing the effective performance of hydroxyl radical. These factors are called RSs.

Table 3 shows the effect of different concentrations of bicarbonate, chloride, sulfate, and nitrate ions in the presence of various doses of electron beams on HA removal. Fig. 3 also shows R^2 second-order kinetics in different concentrations of HCO_3^- (Fig. 3(a)), Cl^- (Fig. 3(b)), SO_4^{2-} (Fig. 3(c)), and NO_3^- (Fig. 3(d)) ions in the presence of different irradiation doses.

The results revealed that by increasing EB absorbed dose from 1 to 15 kGy, the HA removal efficiency increased while increasing rate is great at the beginning from 1 to 6 kGy and then slightly increased up to 15 kGy. It can also be deduced from tables that the rate of HA decomposition in the presence of various doses of energetic electron beams reduced commensurate with the increase in RSs concentrations from 25 to 100 mg/L. The presence of bicarbonate ions significantly reduced the rate of HA decomposition.

Higher concentration of bicarbonate ions led to a further reduction in the rate of HA decomposition which is clearly due to the bicarbonate ions as inhibitors of RS, according to the following reaction:

$$HCO_3^- + HO^\cdot \rightarrow HCO_3^\cdot + OH^-$$

Table 3
The effect of different concentrations of bicarbonate, chloride, sulfate, and nitrate ions in the presence of different irradiation doses on the removal efficiency (%) (HA = 25 mg/L, pH 8)

Dose (kGy)	25	50	100
	HCO_3^- concentration (mg/L)		
1	27 ± 1[a]	24 ± 1	22.9 ± 1
3	47.7 ± 2	46.3 ± 2	45.4 ± 2
6	65.5 ± 2	62.2 ± 3	59 ± 2
9	73 ± 3	71.6 ± 3	69.3 ± 3
15	76.6 ± 4	75.7 ± 3	74.8 ± 4
	Cl^- concentration (mg/L)		
1	25.7 ± 1	24.4 ± 1	23.4 ± 1
3	49.8 ± 2	48.2 ± 1	46.7 ± 2
6	65.4 ± 2	63.5 ± 2	61.6 ± 2
9	72.2 ± 3	71 ± 2	69.8 ± 3
15	76.2 ± 3	75 ± 3	74.3 ± 3
	SO_4^{2-} concentration (mg/L)		
1	27.3 ± 1	26.9 ± 1	26 ± 1
3	50 ± 2	49.3 ± 2	47.9 ± 2
6	66.2 ± 2	65 ± 2	64.3 ± 2
9	73.2 ± 3	72 ± 2	70.5 ± 3
15	76.9 ± 3	75.4 ± 3	74.6 ± 3
	NO_3^- concentration (mg/L)		
1	21 ± 1	18 ± 1	12.8 ± 1
3	32 ± 1	24.5 ± 2	15 ± 1
6	43.8 ± 2	39 ± 2	36.6 ± 1
9	55 ± 2	47.8 ± 2	40 ± 2
15	62.8 ± 3	55.7 ± 2	51.6 ± 2

[a] In all treated groups the concentration of HA was 25 mg/L.

Nitrate, chloride, and sulfate ions have inhibitory effects similar to bicarbonate ions. The following equations show the reactions of chloride, sulfate, and nitrate ions with hydroxyl radicals:

$$Cl^- + HO^\cdot \rightarrow Cl^\cdot + OH^-$$

$$SO_4^{2-} + HO^\cdot \rightarrow SO_4^{-\cdot} + OH^-$$

$$NO_3^- + HO^\cdot \rightarrow NO_3^\cdot + OH^-$$

The results of Ma and Graham indicated that when ozone is used as an oxidizer, the presence of bicarbonate ions prevents the formation of hydroxyl radicals, something which results in lower degradation of pollutants [34]. In another study, Wang et al. reported that carbonate and bicarbonate ions prevent the HA

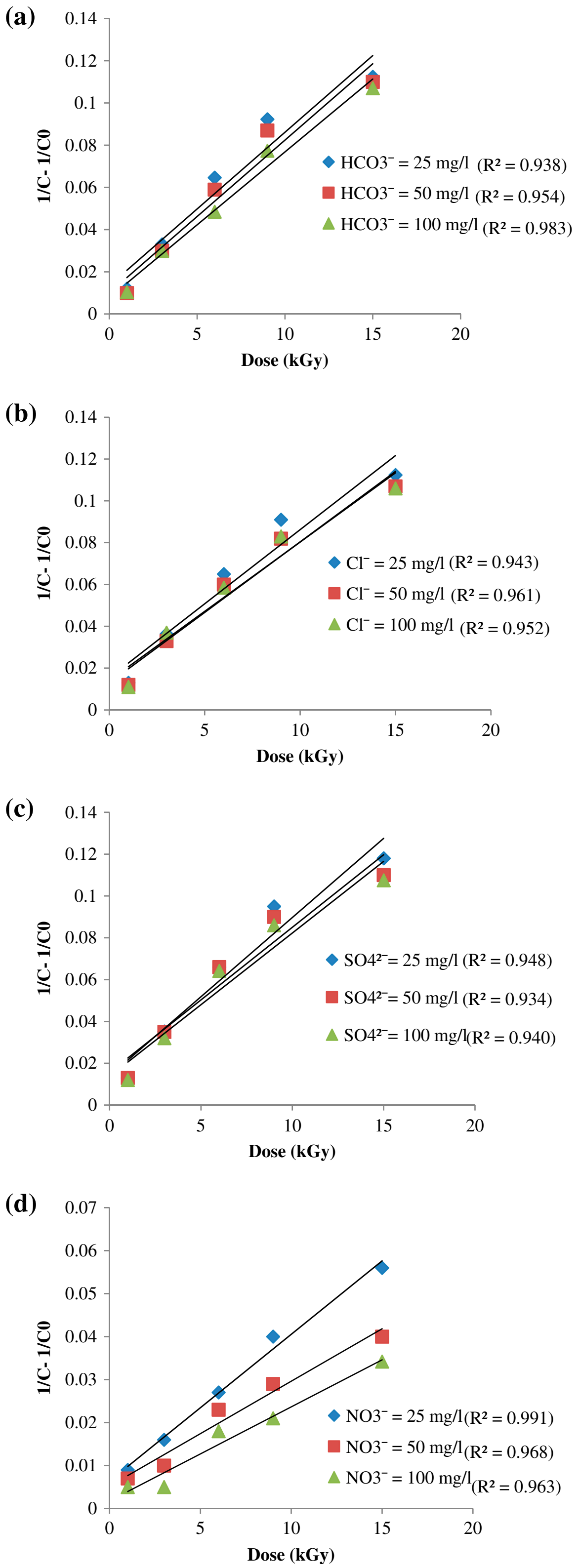

Fig. 3. R^2 second-order kinetic in different concentrations of bicarbonate (a), chloride (b), sulfate (c), and nitrate (d) ions in the presence of different irradiation doses (HA = 25 mg/L, pH 8).

decomposition in UV/H_2O_2 process and had a negative effect due to the scavenging of hydroxyl radicals, especially when their concentrations are high [35]. Gultekin and Ince [36] investigated the inhibitory impact of carbonate, bicarbonate, and chloride ions on H_2O_2/UV process performance using Reactive Red 141 dye. They concluded that in the absence of RSs, the complete removal of dye in 15 min under laboratory conditions is possible, while by increasing different concentrations of carbonate, bicarbonate, and chloride, decomposition of dye is greatly reduced [36].

Lu et al. [37] studied on HA removal from water by solvent solution. In their study, the effects of electrolytes (e.g. NaCl), non hydrophobic organics, and pH of the solution upon the process were studied. They concluded that the increase in NaCl concentration tended to decrease the removal rate and the removal efficiency. Under optimized condition, the treatment performance was found to be very efficient (almost 100%), indicating that the solvent can serve as a possible alternative technology for the removal of HAs [37]. Daifullah et al. studied the factors affecting the HA removal by activated carbon prepared from biomass material and concluded that by increasing nitrate concentration from 73 to 87.5 mg/L at pH 10, HA adsorption is reduced from 30 to 23 mg/g [38]. Nickelsen et al. [39] worked on the treatment of benzene and toluene in the presence of RSs using high-energy EB and showed that the dose required to remove 99% of benzene at 1.3 mg/L initial concentration was 0.95 kGy while in the presence of 3.3 mM methanol as a RS, a dose of 15.1 kGy was required to achieve the same removal. Their results also revealed that in the absence of methanol, toluene showed greater removal effects than benzene under similar conditions [39]. In the study by Yoon et al. on acetone removal by EB irradiation in groundwater, the presence of nitrate and sulfite as RSs decreased the acetone removal, while the inhibitory effect of nitrate was more than sulfite [19]. The same results are achieved in this study.

According to this study, HA removal in the presence of various concentrations of RSs and electron beams matches better with pseudo-second-order kinetics.

4. Conclusion

It can be concluded that by increasing absorbed dose and decreasing the initial HA concentration, removal efficiency increases. Furthermore, by increasing RSs' concentration such as chloride, bicarbonate, sulfate, and nitrate, removal efficiency reduces, while the inhibitory effect of nitrate is more than other anions. The investigation of results in this study about the kinetics of HA removal in the presence of electron beams revealed that HA removal by EB irradiation is more consistent with pseudo-second-order kinetics. Finally, it can be concluded that the electron beam is an effective method for removing natural organic matters such as HA from aqueous solutions.

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