
MONITORING AND TREATMENT OF ATRAZINE IN KAFR ELSHEIKH REGION WATER

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ABSTRACT

Pesticides have negative impact on the human health and environment. They were classified as possible human carcinogens. World health organization (WHO) has set a limit for Atrazine (ATZ) at concentration 2 ppb. This work aims to monitor Atrazine in drinking and related raw water of Kafr El Sheikh governorate. Different treatment techniques for Atrazine removal like Fenton reaction and granulated activated carbon (GAC). The identification and quantification of Atrazine was explored using LC/MS/MS spectrometer. The study recorded the presence of Atrazine during summer spring and autumn in drinking and related raw water due to using it as herbicides in corn crops and the highest influent of atrazine was 0.15µg/l. The study showed that water purification steps have no effect on Atrazine treatment. Some trials were carried out to degrades and adsorb Atrazine using Fenton reaction and GAC. 99 % of Atrazine was removed after one hour by using Fenton oxidation, 97% was removed after two hours by using (GAC).

Key Words: Atrazine, Adsorption, Fenton oxidation and Kinetics.

INTRODUCTION

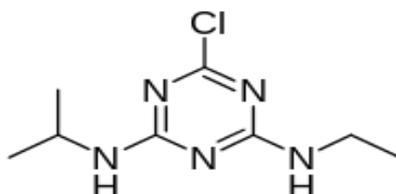


Figure 1: Atrazine (2-chloro-4-(ethylamino)-6-(isopropylamino) – s-triazine)

Agrochemicals constitute one of the most important synthetic chemical families that are bio-resistant and continuously released to the environment as a result of their industrial use and agricultural applications. The majority of these compounds either in their parent form or in other forms resulting from their participation in various biotic or abiotic processes in the environment can exhibit dangerous behavior towards organisms including among disruption activity and mutagenicity. Pesticides, insecticides, acaricides, herbicides, etc., are found in the environment all over the world, and numerous studies have focused on the presence of such compounds in fresh water (Calver *et al.*, 2006). (ATZ) with the chemical name 2-chloro-4-(ethylamino)-6-(isopropylamino) – s-triazine (C₈H₁₄ClN₅, MCIET = 215, 7 g/mol) which its solubility in water is approximately 30 mg/L and half-life in soil is 15–100 days (El Sebaï *et al.*, 2011) and (Ralebitso *et al.*, 2002). Atrazine belongs to the s-triazine ring herbicides and therefore was characterized as bio-recalcitrant. Its potential environmental hazard for polluting drinking water has led some countries to ban this herbicide (Chan and Chu, 2003). (ATZ) is mainly intended to control broad leaf and some grassy weeds (Guan *et al.*, 2013). For this reason it is extensively used in

agricultural applications to corn and rice, but substantial use is also made in the case of sorghum and sugarcane. Moreover, ATZ is widely applied on non-agricultural sites such as lawns and turf. As far as the mode of action is concerned, ATZ blocks electron transport in chloroplast's photo system II complex, thereby preventing CO₂ fixation and subsequent energy production. Plant death occurs mostly by desiccation following membrane damage, because of formation of reactive species (O₂ and triplet-state chlorophyll) that trigger a chain reaction of lipid per oxidation (Marchetti *et al.*, 2013). From among triazine pesticides, atrazine and its metabolites, Diethylatrazine and Deisopropylatrazine, can still be found in drinking water supplies throughout the EU, due to their usage as maize and sugar beet pesticide. Chromosome damage to Chinese hamster egg cells were observed if they were exposed to 0.005–0.080 µmol/L of atrazine, within two days. Two well-known atrazine metabolites, deethylatrazine and deisopropylatrazine, were found to be potentially carcinogenic, therefore the admissible levels for each pesticide individually in water are set at 0.1 µg/L, and the sum should not exceed 0.5 µg/L in EU (Thurman *et al.*, 1994). USEPA (US Environmental Protection Agency) set the total admissible levels for atrazine, deethylatrazine and deisopropylatrazine in groundwater at 3 µg/L (Richards *et al.*, 1995). A study by USEPA in 2003 showed that triazines – atrazine, simazine and propazine – as well as metabolites – deethylatrazine and deisopropylatrazine in deethyl-deisopropylatrazine – have the same mechanism concerning endocrine disruptions. A numerated compounds act the same way on human bodies, therefore, USEPA introduced the sum of all chloro-s-triazines. They are

slowly biodegradable microbiologically (Reid *et al.*, 2003). ATZ removal from drinking water sources is impossible using chlorination, aeration, filtration or coagulation. Quite effective technologies include activated carbon, ozonation, membrane separation, and biofiltration. The most effective techniques for ATZ removal are reverse osmosis (RO) and Nano filtration (NF) membranes (Jiang and Adams, 2006). Pesticides can degrade in the environment through microbial decomposition, UV photolysis, chemical oxidation, or hydrolysis. Adsorption is well-established technique for the removal of low concentrations of organic pollutants from large volumes of potable water, process effluents, wastewater, and aqueous solutions. Adsorption onto activated carbon is often considered as the most economical and efficient process for the removal of organic compounds in dilutes aqueous solutions. Activated carbons are profusely used as adsorbents for decontamination processes because of their extended surface area, high adsorption capacity, microporous structure, and special surface reactivity (Hamdaoui *et al.*, 2005). Advanced oxidation processes (AOP) are well known for generating highly reactive and non-selective hydroxyl radical species, which are used to degrade (and mineralize into water, carbon dioxide and mineral salts) most of organics present in water and wastewater. The Fenton process as an AOPs usually involves four stages: pH adjustment, oxidation, neutralization, and (coagulation and precipitation) (Benatti *et al.*, 2006, Wadley and Waite (2004) and Bautista *et al.* (2007) Fenton process for wastewater treatment has many advantages compared with other AOPs., The Fenton reagents are easy-to-handle, inexpensive, abundant, non-toxic and

environmentally benign in diluted form, and they possess oxidative and coagulative properties, no additional energy input required to activate H₂O₂ and no special equipment required.

Our work aims to monitor atrazine in raw and drinking water of Kafr El Sheikh Governorate and its treatability.

2-MATERIALS & METHODS

2-1 Monitoring of Atrazine in Kafr El Sheikh Governorate:

A survey of pesticides was performed in raw water and produced treated water (drinking) of all water treatment plants in K. El Sheikh Governorate (18 plants).

- a) **Materials:** Atrazine 99.9% purity was obtained from Accustandard. Hydrogen peroxide 33% was purchased from Panreac. Ferrous sulfate, methanol (99.9%) purity, sodium thiosulfate and sodium hydroxide from Sigma Aldrich. HCl (37%) purity from Fisher. Granulated activated carbon from Chemviron-grade (207EA 12.5*40 US).
- b) **Instrument:** LC MS/MS instrument from waters, pH meter from Hack
- c) **Methods:** Samples were collected from water treatment plants were monitored and quantified for atrazine by (EPA 536, 2007) using LC/MS/MS. This method is applicable for the determination of atrazine in raw water, finished drinking water and drinking water during intermediate stages of treatment, using LC chromatographic equipped with MS detector with limit of quantification (LOQ) 0.005ppb, measurement uncertainty (MU) 20 and recovery criteria from 85% to 130%. Sample was

collected from field in glass container and drinking water sample preserved by adding sodium thiosulfate to stop the reaction of chlorine. Raw water sample is filtered by membrane filter (0.22 μm), and 20 μl aliquot is injected (direct injection) into a reverse phase UPLC column C8 (1.7 μm *2.1*50mm). Separation of the analytes is achieved using gradient eluent conditions with a mixture of (A) water and (B) methanol HPLC grade. Start by 90% (A) in the first 2.5 min. to 10% (A) from 2.5 to 7.75 min. then to 100% (B) from 7.75 to 8.50 min. then back to 90% (A) from 8.5 to 10 min., the flow rate was 0.450 ml/min. After elution from the UPLC column, the analytes were detected by a MS/MS detector (Xevo TQ-S) using Electron-Spray-ionization (ESI) in positive mode with the following conditions

Capillary volt=3.2 KV dessolvation temp. = 450C⁰

Desolvation=800L\Hr cone = 150L\Hr

Nebulizer= 7bar

Each batch (20 samples) controlled by calibration curve consist of 7 points from 0.005 ppb to 1.00 ppb. The calibration curve was checked by Lab control sample (LCS) at concentration 0.2 ppb. Also the batch was checked by organic free water (blank sample) which its result must be less than LOQ.

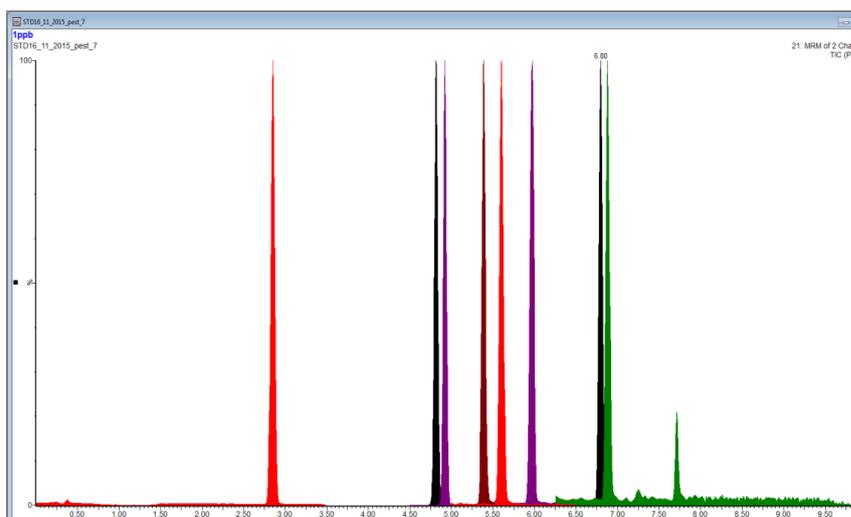


Figure (2): show pesticides compounds at concentration 1ppb

2.2 removal of Atrazine by different treatment methods

Removal of pesticides was performed using oxidation technique by Fenton oxidation and adsorption technique by (GAC)

2.2.1 Removal of Atrazine using Fenton ($H_2O_2+FeSO_4$)

Effect of H_2O_2 : Each sample (1000ml) was spiked by atrazine compound at concentration 2 ppb followed by adding different dosages of H_2O_2 at concentrations (0.1M, 0.2M, 0.4M and 0.8M) at constant concentration of $FeSO_4$ (0.89mM) to study the effect of H_2O_2 .

Effect of $FeSO_4$: Each sample (1000ml) was spiked by atrazine compound at concentration 2 ppb followed by adding different dosages of $FeSO_4$ (0.05 mM, 0.1mM, 0.22mM, 0.44mM, 0.89mM and 1mM) at constant concentration of H_2O_2 (0.4M) to study the effect of $FeSO_4$.

Effect of pH: Each sample (1000ml) was spiked by atrazine compound at concentration 2ppb followed by adding constant dosages of H₂O₂ and FeSO₄ at different pH conditions (3, 5, 7, 9 and 11) to study the effect of pH

Effect of time: After sample treated by Fenton 1.00 ml of treated sample was taken at interval time (15min., 30min., 60min., and 120min.) to study the effect of time.

2.2.2 Removal of Atrazine using (GAC):

Effect of GAC amounts: Each sample (1000ml) was spiked by atrazine standard at concentration 2ppb followed by adding different amount of GAC (0.1g, 0.25g, 0.5g, 1g and 2 g) to study the effect of GAC amount.

Effect of pH: Each sample (1000ml) was spiked by atrazine standard at concentration 2ppb followed by adding constant amount of GAC (1g) at different pH conditions (3, 7 and 11) to study the effect pH condition.

Effect of time: For each sample was treated by Charcoal 1.00 ml of treated sample was taken at interval of time (15min., 30min., 60min., and 120min.) to study the effect of time

RESULTS AND DISSCUTIONS

Monitoring of Atrazine in different water plants at K. El Sheikh:

In the field study a survey was performed on the different 18 water treatment plants in Kafr El Sheikh Governorate.

Monitoring of atrazine in kafr El Sheikh:**Table (1):** Seasonal Monitoring of Atrazine in raw and Drinking water of water treatment plants at Kafr El Sheikh Governorate during 2014.

Atrazine					
Kafr El Sheikh Governorates regions	summer	Spring	Kafr El Sheikh Governorates regions	summer	spring
K.El Sheikh(1) Tap	0.052	0.017	Abo Ali Tap	0.028	0.009
K.El Sheikh(2) Tap	0.059	0.005	Abo Ali Intake	0.049	0.008
K.El Sheikh(2)Intake	0.037	0.017	El hamol Tap	0.082	0.009
Masser Tap	0.037	0.009	El hamol Intake	0.131*	0.015
Masser Intake	0.045	0.008	Baltem Tap	0.057	0.007
Qulene Tap	0.024	0.023	Baltem Intake	0.038	0.008
Qulene Intake	0.023	0.007	El Riade Tap	0.086	0.024
Abo Arafa Tap	0.028	0.007	El Riade Intake	0.145*	0.048
Abo Arafa Intake	0.109	0.030	Mahlet Mousa Tap	0.020	0.016
Haddadi Tap	0.047	---	Mahlet Mousa Intake	0.025	0.017
Haddadi Intake	0.076	---	Newaga Tap	0.141*	0.014
Qozo Tap	0.095	0.008	Newaga Intake	0.127*	0.016
Qozo Intake	0.089	0.009	Ebishan Tap	0.067	---
Diai Tap	0.032	0.008	Ebishan Intake	0.127*	0.016
Diai Intake	0.057	0.010	Greda Tap	0.079	0.010
El Mandora Tap	0.125*	0.007	Greda Intake	0.076	0.009
El Mandora Intake	0.12*	0.008	Fouh Tap	0.147*	-----
Shebas ElMahel Tap	0.113*	0.008	Fouh Intake	0.150	-----
Shebas ElMahel Intake	0.115*	0.014			

--- = \leq LOQ = 0.005 ppb

(*) = results don't comply with the European and National drinking water quality standards (0.1 μ g/l)

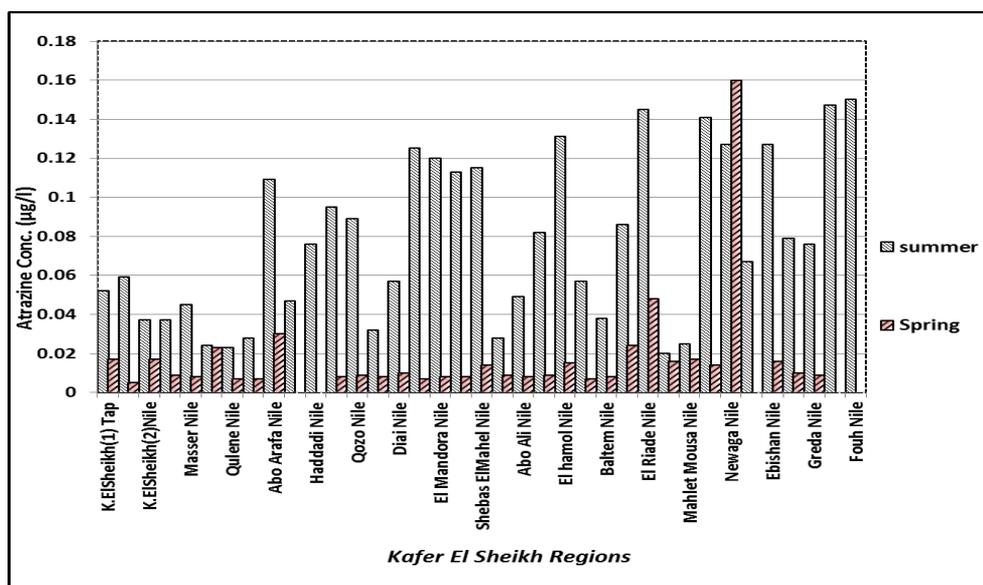


Figure (3): Monitoring of Atrazine in eighteen water treatment plants and their Nile in Kafr El Sheikh during four seasons of 2014.

Figure (3) showed that, the highest Atrazine concentration (0.15 µg/l) was found in the intake of Fouth Water treatment plant during summer due to the use of it as herbicides in corn and rice crops, otherwise atrazine disappeared during winter and autumn in all studied water treatment plants except Haddadi region, it was detected at (0.006 µg/l). Rodriguez *et al.*, 2004; also reported the presence of atrazine at concentration of 0.463 µg/l in Llobregat River during June. The levels of triazines found in the Llobregat River were of the same order as those reported by other authors in southern European rivers. In northern European countries, triazines are found in surface waters at lower levels, from 0.01 to 0.1µg/l (Van der Burggen *et al.*, 2003). Also a study on atrazine monitoring in Slovenia from 1993 to 1996 showed that Atrazine concentrations were up to 7.23 µg/L, and then they

started to decrease to 0.06 µg/l (Pintar and Lobnik, 2001). Haldik *et al.*, 2008, reported the presence of atrazine in Missouri River, Mississippi River and Vermillion River at median concentrations 0.78µg/l. So, from the represented data in Figure (3) all water treatment plants results for atrazine were under the most permissible level for WHO and Egyptian Minister of health regulations (2ppb). On the other hand, some regions like El Mandora, Shebas El Mahel, El Hamol, Elriad, Newaga , Ebishan and Fouh not comply with the European and National drinking water quality standards (0.1µg/l). From Figure (3), the conventional treatment process in water treatment plants has no effect in atrazine removal which agrees with Jiang and Adams, (2006).

Removal of Atrazine by different treatment techniques:

Fenton oxidation:

Effect of H₂O₂: As shown in *Figure(4)*, at concentration of 0.1M H₂O₂ about 83.4% of atrazine was removed after 2 hours and at 0.8M H₂O₂ concentration, the extent of atrazine removal increases to 99.3% within 1 hour so that as the concentration of H₂O₂ increases the atrazine removal from water increases. (Wang *et al.*, 2008) reported that increased H₂O₂ concentration accelerated the rate of dye degradation and (Benatti *et al.*, 2006) reported an overall COD reduction of 92% at the optimal conditions of H₂O₂: COD=9:1, Fe²⁺:H₂O₂= 1:4.5. The reaction rate constants were determined by simplifying the order of the reaction as pseudo first-order and by plotting -ln(C/C₀) versus time (t) *Figure (5)*, the highest R² is (0.979) at concentration 0.2M of H₂O₂ and it clearly demonstrated that the rate of Atrazine removal increased proportionally with increasing H₂O₂ concentration, and that the linear

relationship was not valid after 0.4M. This can be explained by the scavenging of OH^- by excess of H_2O_2 as suggested by (Gogate and Pandit 2004). By plotting the k' against the concentration of H_2O_2 show that; as the concentration of H_2O_2 increases the rate constant increases due to increasing the amount of OH^- so that the suitable point for atrazine removal is (0.8M) of H_2O_2 . Also (Muruganandham and Swaminathan, 2004) reported that in small concentrations, increase of the amount of peroxide causes an increase of the reaction rate since more hydroxyl radicals are produced.

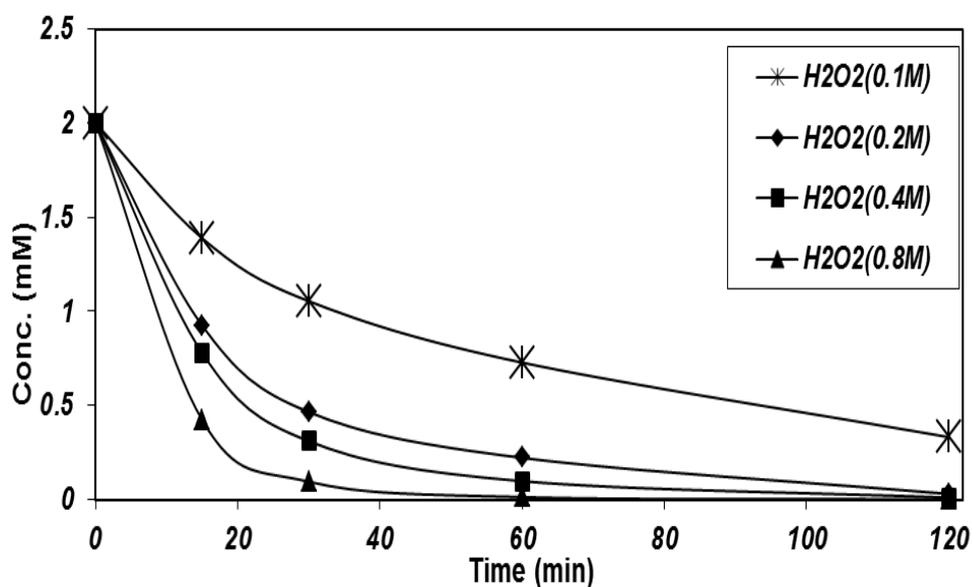


Figure (4): Atrazine removal by various concentrations of H_2O_2 at 0.89 mM of FeSO_4 . Atrazine concentration at zero time was 2.0 ppb).

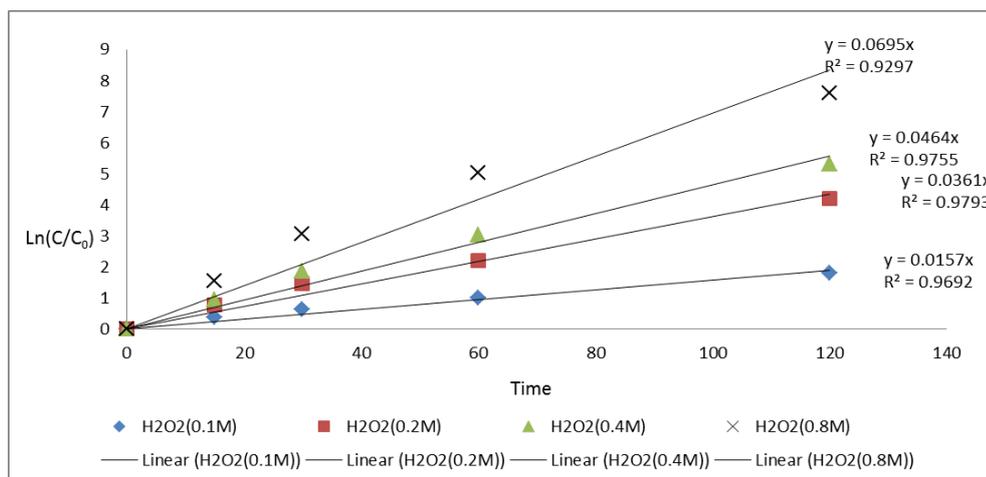


Figure (5): The relation between $\ln(C/C_0)$ and contact time

Effect of $FeSO_4$: As shown in Figure (6). At concentration of 0.05mM $FeSO_4$ no significant removal of atrazine observed, at concentration 0.1mM $FeSO_4$ only about 46.6% of atrazine was removed after 2 hours and at concentration of 0.89mM $FeSO_4$ the extent of atrazine removal increases to 99.5 within 1hour. This result confirmed by (Wang *et al.*, 2008) who found that increasing $FeSO_4$ dosages, the rate of dye degradation is accelerated and (Gulkaya *et al.*, 2006) They found that the optimal molar ratio of H_2O_2/Fe^{+2} fell within the range of 1:153 to 1:470 which achieved 90-95% COD removal. The reaction rate constants were determined by simplifying the order of the reaction as pseudo first-order and by plotting $-\ln(C/C_0)$ versus time (t), Figure (7). The best R^2 is (0.997) at concentration 0.89mM of $FeSO_4$, and By plotting the k' against the concentration of $FeSO_4$ this clearly demonstrated that as concentration of Fe^{+2} increases the rate constant increases due to increasing the amount of catalysts until concentration 1mM

the rate constant decrease. This may be explained by redox reaction that OH*(hydroxyl radical) is scavenged either by the reaction with hydrogen peroxide or by the reaction with Fe⁺², as expressed in the equations (Malik and Saha, 2003).



It is obvious that the increase of iron concentration causes an increase in the reaction rate. As already mentioned, ferric ions are photo reduced to ferrous ions. The latter are the main species that can catalyze hydrogen peroxide to produce hydroxyl radicals. As a result, the amount of ferric ions added is directly proportional to the hydroxyl radicals produced and consequently to the photo catalytic degradation rate of the insecticides (Muruganandham and Swaminathan, 2004). So the suitable concentration for atrazine removal was (0.89mM) of FeSO₄

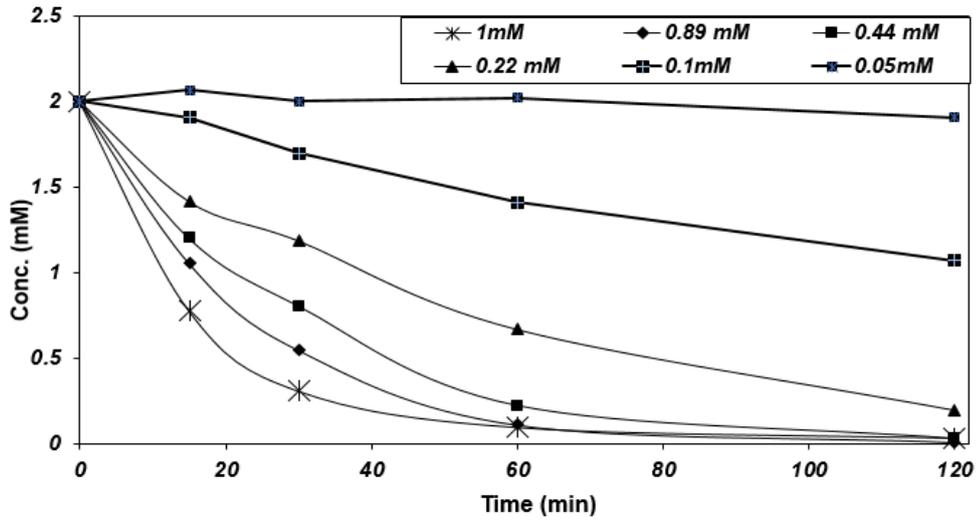


Figure (6): Atrazine removal by various concentrations of FeSO₄ at 0.4M of H₂O₂. (Atrazine concentration at zero time was 2.0 ppb)

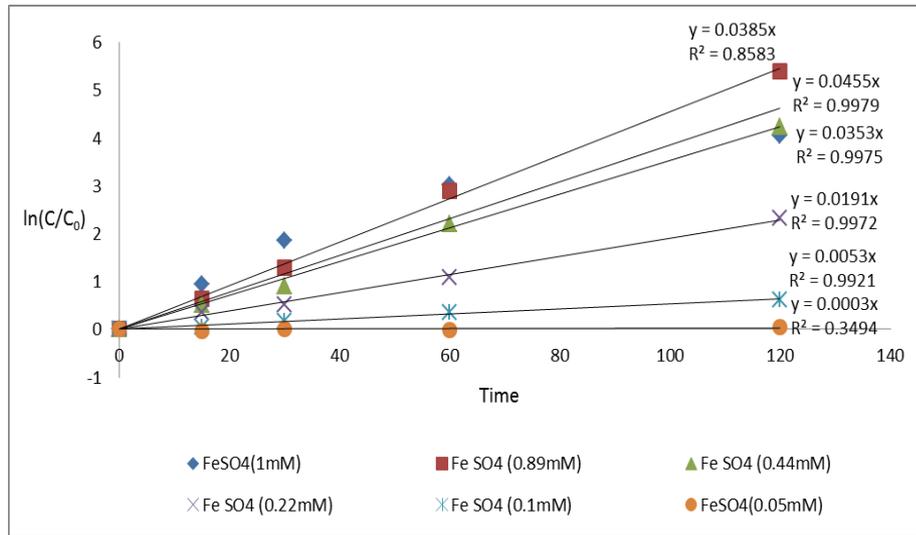


Figure (7): The relation between Ln(C/C₀) and contact time

Effect of pH: Fenton oxidation of atrazine was studied over pH range from pH 3 to pH 11 as shown in *Table (2)*

Table (2): Effect of pH on atrazine removal

Factors \ pH	pH3	pH5	pH7	pH9	pH11
R²	0.977	0.974	-3.79	-0.487	-2.82
k	0.053	0.044	0.033	0.026	0.014

As shown in table (2) the reaction rate constants were determined by simplifying the order of the reaction as pseudo first-order and by plotting $-\ln(C/C_0)$ versus time (t) the highest K constant and R^2 were at pH 3 so that the suitable pH condition for atrazine removal was at pH 3. pH plays a crucial role in photo Fenton reaction efficiency, because it strongly influences which complexes are formed. Thus, pH 2.8 has frequently been postulated as an optimum pH for photo-Fenton treatment, because at this pH there is no precipitation yet and the predominant iron species in the solution is $[\text{Fe}(\text{OH})]^{2+}$, the most photoactive ferric iron–water complex (Pignatello *et al.*, 2000). Also these results are in agreement with related studies of other researchers like (Wang *et al.*, 2008) reported that decrease in pH from 6.3 to 2.4 or 3.4 significantly improved the rate of dye degradation, (Gogate and Pandit 2004) reported that the optimum pH condition was found to be 3 – 4, and the highest oxidation efficiency was reported when acetic acid/acetate buffer was used instead of phosphate and sulphate buffers which encouraged the formation of stable Fe^{3+} complexes and (Bautista *et al.*, 2007) was found highest removal of total organic carbon (TOC) at pH 2.5 – 3.

Granulat47323+995547/*-ed Activated Carbon Adsorption(GAC):

As shown in Figure(8) at 0.1g of GAC 37.7% of Atrazine was removed after 2 *hours* contact time, at 0.25g of GAC 62.3% of Atrazine was removed after 2 *hours* contact time and at 2g of GAC the extent of atrazine was removed after 2 *hours* contact time. This result confirmed by (Amy *et al.*, 2012) reported that 92% removal was achieved with a mass ratio of activated carbon to atrazine of 18:1. Also (Sarkar *et al.*, 2006) was found that Activated carbon highly effective for removal toxic pesticides organic compound. The reaction rate constants were determined by simplifying the order of the reaction as pseudo first-order and by plotting $-\ln(C/C_0)$ versus time (t) Figure (9). The best R^2 is 0.973 at amount 0.25g of GAC and the linear relation was not valid as the amount of GAC decreases beyond 0.5g. By plotting k versus the amount of charcoal, as the amount of charcoal increases the rate constant increases so that the suitable amount for atrazine removal is 2g of GAC. This result was agreement with related study (Gupta *et al.*, 2011) concluded that pseudo first order reaction adsorption model is suitable for pesticides adsorption and (Hameed *et al.*, 2009) reported that pseudo first order reaction adsorption model is suitable for 2,4D pesticide adsorption on activated carbon.

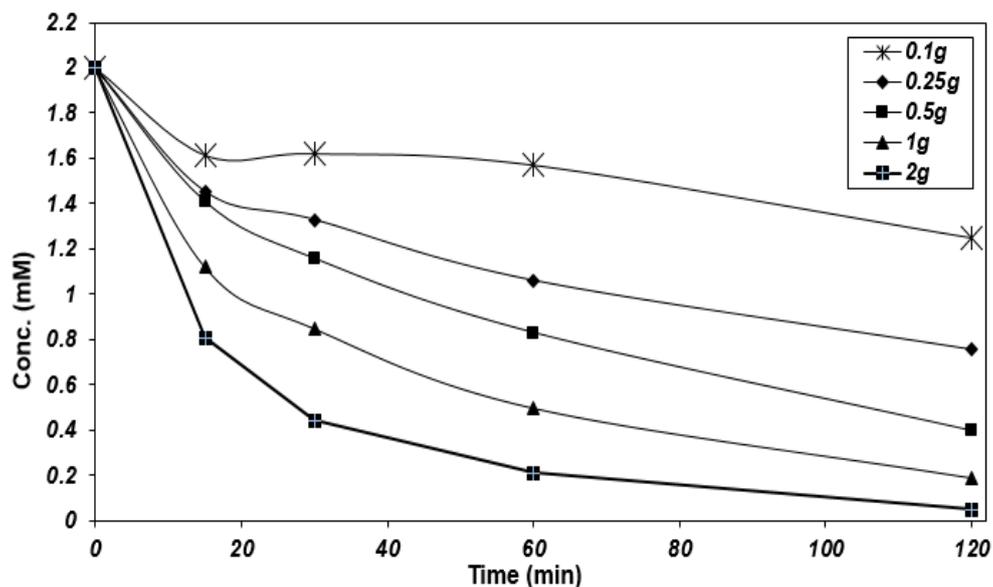


Figure (8): Atrazine removal by various Amounts of GAC. Atrazine concentration at zero time was 2.0 ppb

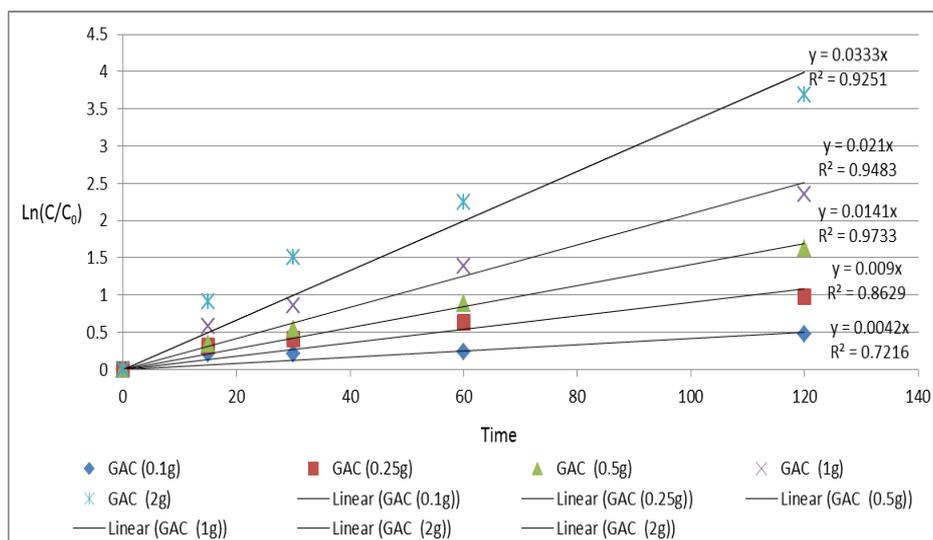


Figure (9): The relation between $\ln(C/C_0)$ and contact time

Effect of pH: The adsorption of the atrazine was studied at pH3, 7 and 11 the result plotted in Figure (10) showed that

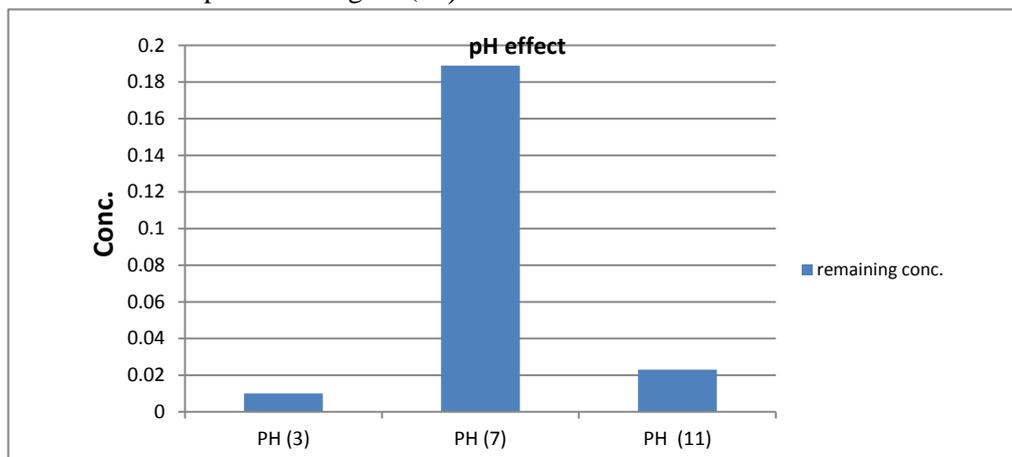


Figure (10): The effect of pH on atrazine removal

the adsorption capacity of atrazine was greater at pH3 this due to deprotonation increases with increases in pH, this deprotonation results in a more negative charged carbon surface at higher pH than at lower pH values. The negative charge developed at carbon surface due to deprotonation cause strong electrostatic repulsion for pesticides in solution while with decreases in pH value, deprotonation decreases (Gupta and Ali, 2006).

CONCLUSIONS

Samples were collected from eighteen water treatment plants in K.El Sheikh Governorate during one year (2014). The study recorded the presence of atrazine at concentration 0.1 ppb and the conventional treatment process ineffective on atrazine treatment. This study demonstrated the possibility of realizing a quasi-complete removal of atrazine in aqueous solution by means

of the photo-Fenton process and GAC. The degradation rate by photo-Fenton process was found to depend on the H_2O_2 and Fe^{3+} initial concentrations as well as pH and contact time, the highest degradation value (99%) was obtained after *one hour* of treatment. The adsorption of atrazine by GAC was found to depend on the amount of GAC, pH and contact time, the highest adsorption value (97%) was obtained after *2 hours* contact time.

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رصد ومعالجة مركب الأترازين في مياه محافظة كفر الشيخ

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المستخلص

المبيدات لها تأثير سلبي علي البيئة وصحة الإنسان ولذلك قد تم تصنيفه كأحد المسببات للسرطان. أقر كل من منظمة الصحة العالمية ووزارة الصحة المصرية (قرار وزير الصحة ٢٠٠٧/٤٥٨ "المواصفات القياسية لمياه الشرب) أن يكون تركيز الأترازين في مياه الشرب لا يزيد عن ٢ ميكرو جرام/لتر ولذلك تهدف الدراسة إلي رصد مركب الأترازين في مياه الشرب ومياه النيل في محافظة كفر الشيخ بمصر وأيضا تطبيق طرق معالجه أخرى مثل الأكسده باستخدام الفنتون والإمتزاز باستخدام الفحم النشط. تم تقدير والتعرف علي الأترازين باستخدام جهاز التحليل الكروماتوجرافي السائل المزود بكاشف الكتله من خلال الطريقه القياسيه EPA536 . الدراسة رصدت وجود الأترازين خلال شهر الصيف والربيع والخريف في مياه الشرب ومياه النيل نتيجة لاستخدامه كمبيد حشائش في زراعة الذره وكان أعلي تركيز تم رصده وهو ٠.١٥ ميكرو جرام/لتر. وأثبتت الدراسة ان مراحل المعالجه التقليديه ليس لها تأثير علي معالجه الأترازين. وباستخدام الفنتون والفحم النشط وجد أن ٩٩% من الأترازين تم معالجته بعد ساعه باستخدام الفنتون و ٩٧% تم معالجته بعد ساعتين باستخدام الفحم النشط.