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Removal of pesticides from water using zeolites

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Abstract

Zeolites are aluminosilicate minerals that have extremely high adsorption capacity and high cation-exchange ability. Due to this, natural zeolites have been widely used as adsorbents in water purification processes in the past decades. The aim of this study was to investigate the adsorption capacity of zeolite and possibility for removing pesticides from water using zeolite. Response surface methodology Box-Behnken design with seventeen runs, three independent variables (pH, particle size and amount of zeolite) and five replicates at a center point was used to achieve the optimum adsorption conditions - pH 10, zeolite particle size of 1-2 mm and zeolite content of 15 g. Purification with zeolite is environmental friendly, relatively cheap and is a readily available technique.So these investigations can be applied in the area of water purification wherever pesticides appear as pollutants of ground and surface waters.

Keywords: Atrazine; pesticides; water purification; Zeolite.

1. Introduction

Atrazine is a selective systemic herbicide, and belongs to the chlorotriazine group (chloro-s-triazine herbicides) (Figure 1)

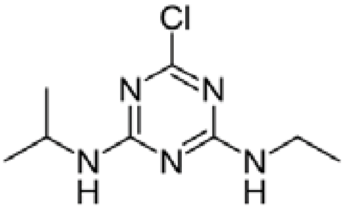

Fig. 1. Structural formula of atrazine

It is colorless, solid, sparingly soluble in water (0.030 g/l), but well soluble in organic solvents (chloroform - 52 g/l, ethyl acetate - 27 g/l ...) and with a half-life ranging from several days to several months (Pesticide Manual, 2003). For example, in surface water it remains for more than 200 days, in soil from 13 to 261 days and in the air about 14 hours (U.S. EPA 2003). It is used in agriculture for the removal of annual broadleaf and grass weeds,which grows along with the plant, as well as for controlling weeds in the vicinity of highways and railways, by stopping the process of photosynthesis (Legrand *et al.,* 2006). After use, a certain amount of atrazine remains in the air, some falls to the ground and under the influence of atmospheric precipitation enters in the surrounding area, including surface water. A part of atrazine penetrates to the lower layers of the soil and pollute the groundwater (Lazić *et al.,* 2013). Atrazine and some of its degradation products (diethyl atrazine (DEA),diisopropyl atrazine (DIA) and diaminochlorotriazine (DACT)) can often be found in drinking water wells in some agricultural regions (Wu *et al.*, 2010). People who live near agricultural areas, where atrazine is used may be exposed to its harmful effects through contaminated drinking water and air, during treatment of crops with atrazine, or if they live or work near landfills where used atrazine packing is disposed. Children can also be exposed by playing on clay surfaces contaminated with atrazine (Watts, 2013). Atrazine can mimic the action of sex hormones and also may alter or modify production of the natural hormones levels (Kucka *et al.,* 2012, Soto *et al.,* 1995, Damastra *et al.*, 2002)

In America, the Agency for environmental protection has determined the maximal residue level (MRL) for atrazine in drinking water (EPA 2009) to be 3 μg/l. The European Union directive for the quality of water intended for human consumption, prescribes the MRLs for single pesticide from 0.1 μg /l and 0.5 μg/l for total pesticide content (98/83/EC 1998). In the EC directive on water, the European Union has prescribed MRLs of atrazine in surface water to be 2 μg/l (2008/105/EC). In Serbia, there are regulations for hygienic quality of drinking water, which states that the allowable amount of atrazine in drinking water is 0.1 µg/l, and for total pesticides the MRLs is 0.5 µg/l (Official Gazette of SRJ 42/98).

In order to reduce exposure to the effects of atrazine, some scientists suggest its removal by using different physical and chemical techniques, such as incineration, reverse osmosis (Tepuš *et al.,* 2009), electrodialysis (Van der Bruggen *et al.,* 2003), chemical degradation (Campos *et al.,* 2000, Zhang *et al.,* 2011), phytoremediation, ultrasonic (Collings & Gwan, 2010) and photocatalytic degradation (Dong *et al.,* 2010; Baranda *et al.,* 2012; Chen *et al.,*2015). Some of these techniques may lead to the formation of toxic and other undesired products (Pathak & Dikshit, 2011) and some of them are very expensive. For these reasons, the goal is to find a simple technique, safe for the environment. One such technique is the adsorption of atrazine using zeolite.

Zeolites are aluminosilicate minerals, that have extremely high adsorption capacity and good catalytic properties with excellent chemical and thermal stability. They are thermally stable and have good resistance to most chemicals. Results of the exploratory investigation of a group of scientist (Salvestrini *et al.,* 2010) indicate that acid-activated clinoptilolitic tuff could be considered a suitable material for removing atrazine and similar chemical compounds from water. Systematic adsorption tests were carried out by Lemic *et al.* (2006) to determine the efficiency of organo-zeolite for removal of atrazine, lindane and diazinone from water. Chmielewski *et al.,* (2008) designed and developed more efficient and cost effective water purification process using natural clinoptilolite based separation and reaction media. Experiments have also been performed to investigate how the concentration of some organic compounds may be reduced using natural zeolites as sorbents (Salvestrini *et al.,* 2015).

In Serbia several deposits of zeolites, are found near Zlatokop, Toponica, Igroš, Jablanica, Lužnica, Mionica, Slanci, Veliko Selo and Beočin (Fruška Gora). The aim of this study was to investigate the adsorption capacity of zeolite from Fruška Gora and its potency to purify atrazine contaminated water.

2. Experimental

2.1. Chemicals

The analytical standard of atrazine used in the experiment was supplied by Sigma-Aldrich-Riedel de Häen. Methanol was used for conditioning of a C-18 column and for the extractions of the atrazine.

Apparatus and conditions of chromatographic determination

Analysis of atrazine was performed by gas chromatography with NP detector on GC-NPD (Hewlett Packard 5890 GC Series II with ALS). The 30m long capillary column Supelco SPB 608 with a diameter of 0,53 mm and a 0,50 µm thick film of stationary phase was used for atrazine separation. The working conditions of the instrument were set as follows: carrier gas was helium with column head pressure of 55 kPa and constant flow of 20 ml/min. The gas flows for the detector were: hydrogen 3.5 ml/min, air 110 ml/min and make up nitrogen 20 ml/min. Applied temperature conditions were: a column temperature of 200ºC maintained for 5 minutes, then a rise of 30ºC /min to 250ºC. The temperature of the injector and the detector were 250ºC. The total run time was 8 minutes. NPD Bead Power was adjusted to get signal level about 20 counts.

2.2. Method

For this research zeolite (clinoptilolite) from the area of Fruška Gora was dried for 24 hours at 105 °C. The experiment was set for three parameters at three levels based on particle size in accordance with the Box-Behnken surface response modelling scheme shown in Table 1.

Table 1. Box-Behnken scheme of the experiment

Factors	**Units**	**Levels**		
		-1	0	1
pH	/	4	7	10
Particle size^A	/	-1	0	1
The amount of zeolite	g	5	10	15

^{A} -1 (0.5-1 mm), 0 (1-2 mm), 1 (2-4 mm)

The experiment was performed using batch equilibrium adsorption test, which means that the zeolite was in contact with atrazine in water for 18 hours, while being shaken in a rotary shaker (Milojević-Rakić *et al.*, 2013). After shaking and decantation, supernatant was extracted in the apparatus for solid-phase disc extraction. The content of atrazine was determined by using capillary gas chromatography with nitrogen-phosphorus detector.

The extraction of atrazine from water

50 ml of the prepared standard solution of atrazine in deionized purified water at a concentration of 1 µg/ml was passed through a conditioned C-18 column (500mg octadecilsilica (ODS) packing). The elution of the analyte

from a disk was carried out with 3 x 1 ml of methanol into a flask and supplemented to volume of 5 ml with methanol.

Experimental Design and Analysis

Experimental design by response surface methodology (RSM) is a powerful statistical tool for building empirical models. By design of experiments, the objective is to optimize a response (output variable), which is influenced by input variables. RSM was developed to model experimental responses (Box & Draper, 1987) and then migrated into optimizing the experimental design results.

In this study, a Box-Behnken design was used with seventeen runs, three independent variables (pH, particle size and amount of zeolite) and five replicates at a centre point. The pH of the sample was adjusted to 4, 7, and 10. The particle sizes of the zeolite were between 0,5 – 1 mm; 1-2 mm and 2-4 mm. Amount of zeolite was 5, 10 and 15 g respectively. The statistical and correlation analysis of the response of the model was performed with analysis of variance (ANOVA), interaction plots and surface responses.

3. Results and discussion

Zeolite (clinoptilolite) from the area of Fruška Gora (Beočin) was used for the purposes of this research. The mineralogical properties and chemical composition of zeolite tuffs from Beočin – Serbia have been investigated by Mihajlovic *et al.* (2013). Qualitative mineralogical analyses were made by polarization microscopy in transmitted light. Mineralogical composition zeolite used in this study includes carbonates, plagioclase, quartz (Q) and biotite. Chemical analyses, made by classic wet-silicate analyses, gave the following results: SiO_2 – 56%, Al_2O_3 – 14,04%, Fe_2O_3 – 1,85%, CaO – 6,2%, MgO – 2,64%, K_2O – 2,32 %, Na_2O – 0,52% and Si/Al – 3,93 mmolM^+/100 g. Zeolite from Beocin was also investigated by Kolakovic *et al.* (2014). They tried to define the optimum operating parameters for forming adsorbents based on organo-zeolite as a filter for wastewater treatment. They have proven that larger grains have lower zeolite adsorption power of organic cations on the zeolite surface and that interference between adsorption of mixture and temperature has no significant impact on adsorption of organic cations on the surface of the zeolite.

Table 2 shows the experimental design of the three factors with the experimental and predicted values of the amount of adsorbed atrazine. The obtained results were statistically analyzed using the Design Expert 9.0. A mathematical model of third order polynomial relating the amount of adsorbed atrazine with the independent process variable was developed as shown in equation (1).

Table 2. Experimental design of the three factors by Box-Behnken

	Factor 1	**Factor 2**	**Factor 3**	**Amount of adsorbed atrazine, µg**	
Run	**pH**	**Particle size**	**Amount of zeolite (g)**	**Experimental**	**Predicted**
1	4	0	15	36	36
2	7	0	10	29	47
3	10	1	10	20	20
4	7	1	15	25	25
5	10	0	15	73	73
6	7	-1	5	66	66
7	7	0	10	56	47
8	4	1	10	27	27
9	4	-1	10	45	45
10	7	0	10	49	47
11	4	0	5	38	38
12	10	-1	10	49	49
13	10	0	5	52	52
14	7	-1	15	24	24
15	7	0	10	55	47
16	7	0	10	46	47
17	7	1	5	35	35

$$\text{Amount of adsorbed atrazine}= 47{,}00+12{,}75A-7{,}50B-13{,}00C-2{,}75AB+5{,}75AC+8{,}00BC+0{,}25A^2-12{,}00B^2+2{,}50C^2-4{,}25A^2B+17{,}75A^2C-13{,}50A \qquad (1)$$

where A, B and C are terms of the coded factors pH, particle size and amount of zeolite. The coefficients with two factors andthose with second order terms show the interaction between the two factors and quadratic effect, respectively.

The model can be used to predict the amount of adsorbed atrazine on zeolite as a response for given levels of each factor. The correlation coefficient R^2 between experimental and predicted values is 0,8722, indicating good fit. Model graph of dependencies between experimental and predicted values is shown on Figure 3.

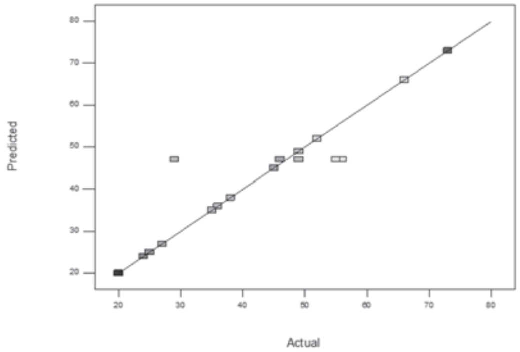

Fig. 3. Predicted value vs. actual value of the amount of adsorbed atrazine on zeolite (from 20 to 73 µg)

The results obtained by analysis of variance (ANOVA) are shown in Table 3. Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case, there are no significant model terms. Values greater than 0.1000 indicate the model terms are not significant. In this model the terms A, C, B^2 and A^2C have p-values between 0,05 and 0,1.

The 3D surface graphs and contour plots for the effect of independent process variables on the amount of adsorbed atrazine are shown in Figures 4, 5 and 6.

Table 3. The significance of the terms of the polynomial (1)

Source	Sum of Squares	df ^{A}	Mean Square	F Value	p-value Prob>F
Model	3235.88	12	269.66	2.28	0.2221
A	650.25	1	650.25	5.49	0.0792
B	225.00	1	225.00	1.90	0.2403
C	676.00	1	676.00	5.70	0.0753
AB	30.25	1	30.25	0.26	0.6400
AC	132.25	1	132.25	1.12	0.3504
BC	256.00	1	256.00	2.16	0.2156
A^2	0.26	1	0.26	0.22	0.9647
B^2	606.32	1	606.32	5.12	0.0865
C^2	26.32	1	26.32	0.22	0.6620
A^2B	36.13	1	36.13	0.30	0.6103
A^2C	630.12	1	630.12	5.32	0.0824
AB^2	364.50	1	364.50	3.08	0.1543

A=pH, B=Particle size, C=Amount of zeolite, df^{A}– degree of freedom

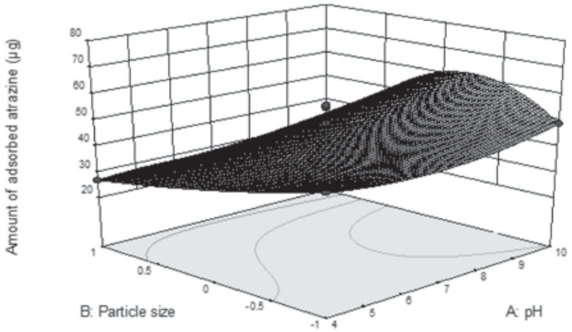

Fig. 4. Effect of pH and particle size on the amount of adsorbed atrazine

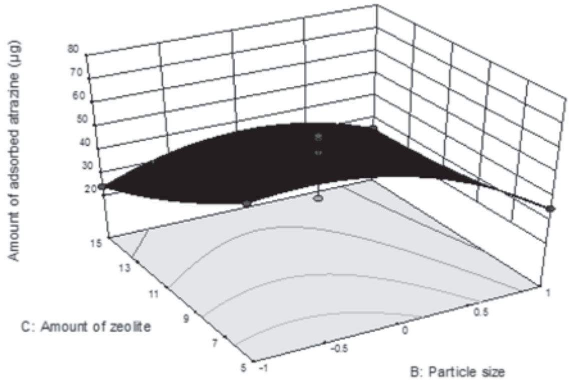

Fig. 5. Effect of amount of zeolite and particle size on the amount of adsorbed atrazine

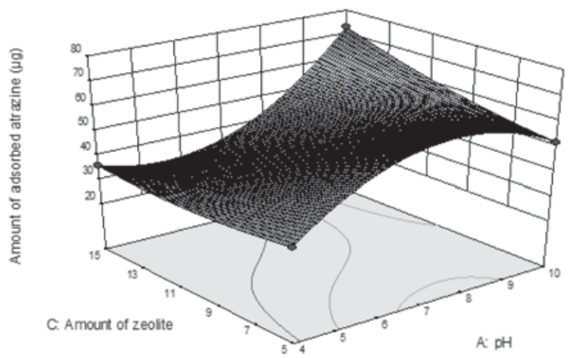

Fig. 6. Effect of pH and amount of zeolite on the amount of adsorbed atrazine

Because the model and terms were insignificant it was necessary to improve the model by reduction of terms. Hence, AB, A^2 and A^2B terms were omitted from the full model to obtain a reduced third order polynomial equation and to identify the significant terms. The form of this new polynomial is in equation (2).

$$
\begin{aligned}
&Amount\ of\ adsorbed\ atrazine = \\
&47.1053 - 0{,}75A - 9{,}625B + 4{,}75C + \\
&5{,}75AC + 8BC - 11{,}9868B^2 + \\
&2{,}51316C^2 + 13{,}5AC^2 - 17{,}75B^2C \qquad (2)
\end{aligned}
$$

The outcomes of the ANOVA test for the resultant response surface reduced cubic model are given in Table 4.

The model F-value is 4.56 and implies that the model is significant. There is only a 2.90% chance that an F-value this large could occur due to noise. As mentioned above, values of "Prob > F" less than 0.05 indicate that model terms are significant. In this case A, B, C, B^2, A^2C are significant model terms.

The lack of fit F-value of 0.19 implies that the lack of fit is not significant relative to the pure error. There is a 89.98% chance that a lack of fit F-value this large could occur due to noise. In fact no significant lack of fit is good because a model needs to fits.

Table 4. The significance of the terms of the reduced polynomial model (2)

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob> F	Remark
Model	3169.24	9	352.14	4.56	0.0290	significant
A	650.25	1	650.25	8.42	0.0229	significant
B	741.12	1	741.12	9.60	0.0174	significant
C	676.00	1	676.00	8.75	0.0212	significant
AC	132.25	1	132.25	1.71	0.2320	
BC	256.00	1	256.00	3.31	0.1115	
B^2	606.67	1	606.67	7.85	0.0264	significant
C^2	26.67	1	26.67	0.35	0.5753	
A^2C	630.13	1	630.13	8.16	0.0245	significant
AB^2	364.50	1	364.50	4.72	0.0664	
Lack of Fit	66.64	3	22.21	0.19	0.8998	not significant

A=pH, B=Particle size, C=Amount of zeolite

For model validation, two experiments with three replicates in optimal conditions were performed. The amount of atrazine in the first experiment was 500 μg, while in the second was 10 μg. The other conditions (pH=7, particle size=1-2 mm and amount of zeolite=15 g) were the same in both experiments.

pH is an important factor in atrazine adsorption (Jamil *et al.,* 2011) since, in this study, adsorption of atrazine increases with increasing pH, suggesting that the adsorption mechanism was basically diffusion trough the pores of the zeolite, of the atrazine in molecular and not protonated form. This suggested that pores of Beočin-zeolite are smaller than protonated atrazine molecule.

The adsorption was shown to be 15.9 % and 76.4 % respectively. This confirms that the model explains quite well the influence of pH, particle size of and amount of zeolite on the amount of adsorbed atrazine and reflects how the amount of atrazine affects the adsorption potential. The higher impact on the adsorbed atrazine amount has a particle size of zeolite.

4. Conclusion

The effects of pH, particle size and amount of zeolite on the amount of adsorbed atrazine have been investigated. The highest achieved adsorption of atrazine was 73 % at high pH values with a zeolite mass of 15g (as shown in the Figure 6). It can also be seen that the largest amount of atrazine was adsorbed on a zeolite with particle size of 1-2 mm at high pH value (pH=10) (Figure 4). At low pH values, adsorption increases with decrease in particle size, with the highest values being achieved on the smallest particles (as can be seen on Figure 4).

It can be concluded that zeolite has the potential to be used for removal of atrazine and therefore other similar pesticides present in groundwaters and surface waters under optimal conditions of adsorption at pH 10 and zeolite particle size from 1 – 2 mm. The protonated zeolite at low pH could lead to decrease of atrazine adsorption (Salvestrini *et al.*, 2010)

Also, small amount of zeolite particles of smaller dimensions results in higher adsorption efficacy, which indicates the higher influence of particle size on the adsorption compared to the influence of the zeolite amount (Figure 5).

Research should continue in the direction of studying adsorption capacity of zeolites over other pesticides as well as conducting similar experiments using raw waters.

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خلاصة

الزيوليت ومعادن ألومينوسيليكات لديها قدرة امتصاص عالية للغاية وعالية القدرة الموجبة للتبادل. ونتيجة لهذا، قد استخدم الزيوليت الطبيعي على نطاق واسع كماص في عمليات تنقية المياه في العقود الماضية. وكان الهدف من هذه الدراسة هو التعرف على قدرة امتصاص الزيوليت وإمكانية إزالة المبيدات من المياه باستخدام الزيوليت. تم استخدام تصميم بوكس بينكين لاستجابة منهجية السطح مع سبعة عشر أشواط، ثلاثة متغيرات مستقلة (الرقم الهيدروجيني، وحجم الجسيمات وكمية من الزيوليت) وخمسة مكررات في نقطة مركزية لتحقيق الظروف المثلى للامتصاص - درجة الحموضة 10، الزيوليت حجم الجسيمات من 1–2 مم والمحتوى الزيوليت من 15 غرام. التنقية باستخدام الزيوليت هي صديقة للبيئة، ورخيصة نسبيا وهي تقنية متوفرة بسهولة. لذلك فان هذه الدراسة يمكن تطبيقها في مجال تنقية المياه في أي مكان تظهر فيه المبيدات كملوثات للمياه الجوفية والسطحية.

كلمات البحث: الأترازين. مبيدات حشرية؛ تنقية المياه؛ الزيوليت.