# RESEARCH OF THE EFFICIENCY OF ORGANIC MATTER REMOVAL FROM WATER USING SYNTHETIC ZEOLITE

**ORIGINAL SCIENTIFIC PAPER** 

Zoran Petrović<sup>1⊠</sup>, Marko Mitrović<sup>1</sup>, Aleksandra Radulović<sup>2</sup>, Begić Sabina<sup>3</sup>, Stevan Blagojević<sup>2</sup>, Kešeli Dragana<sup>1</sup>

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<sup>1</sup> University of East Sarajevo, Faculty of Technology Zvornik, Karakaj 34a, 75400 Zvornik, Bosnia and Herzegovina a <sup>2</sup> Institute of General and Physical Chemistry, Studentski trg 12/V, 11158 Beograd, Republic of Serbia

<sup>3</sup> Faculty of Technology, University of Tuzla, Urfeta Vejzagića 8, 75000 Tuzla

⊠ zoran.petrovic@tfzv.uers.ba

#### ABSTRACT:

The paper investigated the effectiveness of using synthetic zeolite as an adsorbent for the removal of organic matter from water. Characterization of zeolite was performed using advanced testing methods: XRD, FTIR, BET and SEM/EDS. Raw water was analyzed for physicochemical characteristics before treatment (turbidity, color, pH, conductivity, content of organic matter, ammonia, nitrate and nitrite nitrogen, iron, manganese and chloride) and after treatment with an adsorbent (content of organic matter). Research on the efficiency of adsorption was carried out under the conditions of water temperature 25°C, individual doses of zeolite of 1, 2 and 4 g/L, mixing 200 rpm and adsorption time 60 minutes. The obtained results showed that by applying synthetic zeolite in a dose of 1 g/L, it is possible to achieve the removal of organic matter of 71.2%, while increasement of that dose up to four times has an insignificant effect on the adsorption efficiency.

**KEYWORDS:** synthetic zeolite; organic matter removal

#### INTRODUCTION

Water has a multiple and irreplaceable role in industry [1], [2], serving either as a basic or auxiliary raw material, or as an energy source, which is why ensuring sufficient quantities of water of appropriate quality is an industrial imperative. In supplying water for their needs, industrial facilities most often rely on surface sources such as rivers and lakes [3], [4]. However, the quality of the raw water of those sources is usually lower than that required for its specific purpose [3], [5], which is why it needs to be corrected.

One of the quality parameters of raw water, whose values are often inconsistent with those in industrial water quality requirements, is the content of organic matter, which is usually expressed by the permanganate index, i.e. consumption of KMnO<sub>4</sub> required for their oxidation [6]. Organic matter consists of a variety of particulate and dissolved carbon-based compounds [7] originating from natural sources such as soil runoff [8], aquatic life [9], microbial activity [10], as well as from anthropogenic sources including agricultural and urban runoff [11], [12], industrial discharges, effluents from sewage treatment plants [13] and septic systems [14]. Organic substances are commonly more present in surface than ground waters [15], [16], and urban natural water streams typically receive higher organic loads [17]. Water with a high content of organic matter is generally undesirable for industrial use due to its multiple negative impacts such as biofouling and biocorrosion in industrial systems [18] and inefficiencies in water treatment processes [19]. In addition, high levels of manganese are known to occur in organic-rich surface waters, sometimes as organically-complexed which is difficult to remove during conventional treatment [20].

In order to meet the requirements of standards and regulations on water quality for use in industry a number of methods are available for removing organic matter, such as: chemical coagulation [21], electrocoagulation [22], adsorption [23], advanced oxidation processes [24], membrane filtration [25] and biological treatment [26]. Among the listed, adsorption has advantages due to simple design and low investment costs and space requirements [27], which is why it is also used for other applications besides water treatment, such as: oil bleaching [28] and adsorption of volatile organic compounds from air [29].

Common adsorbents are: activated carbon [30], zeolites [31], silica gel [32] and bentonites [33]. Zeolites have high surface area and porosity that provide ample active sites for the adsorption of organic molecules [34] and can be modified in order to increase their adsorption capacity [35]. Although they are available in nature, they are more often synthesized, due to the simplicity of the procedure and higher purity of the product [36]. In this paper, the research of the effects of synthetic zeolite as an adsorbent in the removal of organic matter from natural surface water was carried out.

## **MATERIALS AND METHODS**

In the experimental part of the research, the following materials were used: synthetic zeolite ZEOflair 110 (Zeochem, Zvornik), surface water of the Jala River sampled in the urban area of the city of Tuzla, and other reagents and chemicals required for zeolite characterization and physicochemical analysis of water.

The following methods were used to characterize the zeolite: X-ray diffractometry (XRD), infrared spectroscopy (FTIR), low-temperature nitrogen adsorption (BET) and scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM-EDS). X-ray diffractometry was performed on a Rigaku Smartlab X-ray diffractometer, and crystalline phases were identified using Rigaku PDXL 2.0 software with the ICDD PDF-2 2016 database. Infrared spectroscopy was performed on a Shimadzu infrared spectrophotometer, IRAffinity 1S, using the ATR method (MIRacle 10). With this method, spectra were recorded in a wavenumber range of 4000 - 500 cm<sup>-1</sup>. Low-temperature nitrogen adsorption was performed on the Micrometrics ASAP 2010 device, which determined the textural characteristics of the zeolite. Morphological characteristics determined with an electron microscope JEOL-JSM-6460LV (Japan) at a resolution of 3-4 nm and 500-3000 times magnification. The samples were sputtered with gold on a BAL-TEC SCD 005 device, with a current of 30 mA, from a distance of 50 mm for 80 s. Microelement analysis was performed with an energy dispersive spectrometer with a Noran System Six 200 analyzer (detection of elements  $Z \ge 5$ , detection limit  $\sim 0.1\%$  m/m, resolution 126 eV).

The physicochemical analysis of water before adsorption treatment included the determination of pH and electrical conductivity by potentiometric methods, content of organic matter, iron, manganese and chloride by volumetric methods, ammoniacal, nitrate and nitrite nitrogen by spectrophotometric methods. The content of organic matter in water was determined also after adsorption treatment.

The adsorption operation was carried out in laboratory conditions at room temperature, by adding a certain amount of zeolite  $(0.25, 0.5, 1.0 \, \mathrm{g})$  to a glass beaker with 500 mL of sampled water and simultaneously mixing the water with a magnetic stirrer at a speed of 200 revolutions per minute and a total mixing time of 60 minutes. After the specified time, the adsorbent was separated from the water by filtration on filter paper (blue strip), and then the content of organic matter was determined in the filtrate. The efficiency of removing organic matter from water  $(E_{om})$  was calculated using the following relation:

$$E_{om} (\%) = (OM_{rw} \text{ -}OM_{tw}) / OM_{rw}$$

where  $OM_{rw}$  and  $OM_{tw}$  are contents of organic matter in raw and treated water.

#### RESULTS AND DISCUSSION

#### RESULTS OF CHARACTERIZATION OF SYNTHETIC ZEOLITE

Figure 1 shows the diffractogram of synthetic zeolite ZEOflair 110. Based on the obtained values of intensity I (imp) and interplane distances d (Å), and by comparison with literature data and ICDD standards, it was determined that the examined zeolite sample has a crystal structure of ZSM-5 (MFI) zeolite.

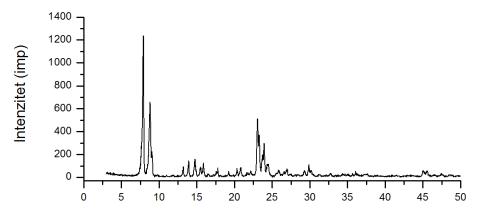


Figure 1. X-ray diffractogram of zeolite ZEOflair 110

The FTIR spectrum of zeolite ZEOflair 110 (Figure 2) shows bands that are characteristic of zeolite type ZSM-5. The bands at ~1225 cm<sup>-1</sup> and ~1070 cm<sup>-1</sup> originate from external and internal asymmetric stretching vibrations of Si-O-Si bonds, while the band at ~790 cm<sup>-1</sup> originates from symmetrical stretching vibrations of Si-O-Si bonds.

The bands at ~590 cm<sup>-1</sup> and ~545 cm<sup>-1</sup> originate from double ring vibrations.

The adsorption isotherm of zeolite, shown in Figure 3 is of Type I, which is characteristic of microporous materials [37].

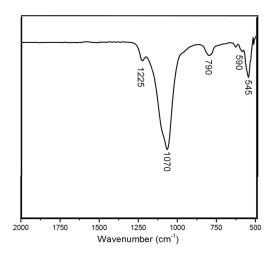


Figure 2. FTIR spectrum of zeolite ZEOflair 110

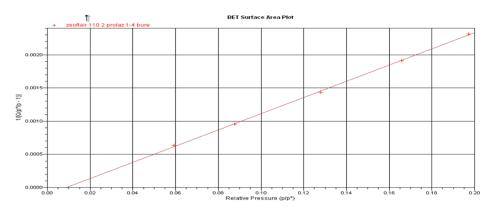


Figure 3. Adsorption isotherm of zeolite ZEOflair 110

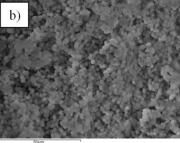
The results of the analysis of textural characteristics of zeolites (table 1) show that the specific surface is  $336.88 \text{ m}^2/\text{g}$ , which is within the typical range ( $300\text{-}2000 \text{ m}^2/\text{g}$ ) of their specific surfaces measured by gas adsorption [38]. From the given data, it follows that the share of micropore surface area in zeolite amounts to about 71.65% of the total specific surface area determined by the BET method.

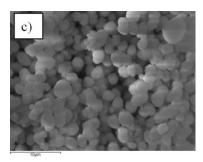
Table 1. Textural characteristics of zeolite ZEOflair 110

Characteristics	Value
Specific surface area (SP <sub>BET</sub> ), m <sup>2</sup> /g	336.88
Constant, C <sub>BET</sub>	-71.34
External specific surface area (SP <sub>ext</sub> ), m <sup>2</sup> /g	95.49
Micropore surface area $(S_{\mu p})$ , $m^2/g$	241.39
Micropore volume $(V_{\mu p})$ , cm <sup>3</sup> /g	0.1208
Mean pore diameter (d <sub>p</sub> ), nm	3.287

SEM micrographs of zeolite at magnifications of 500, 1000 and 3000 times are given in Figure 4, where it can be seen that the examined material has a regular spherical crystal grain shape.







**Figure 4**. SEM micrographs of synthetic zeolite ZEOflair 110 at magnifications: a) 500x, b) 1000x and c) 3000x

The SEM analysis was done in combination with X-ray energy dispersion (EDS) analysis (Fig. 4) revealing the elemental composition (wt%) of the zeolite sample, which represents silicalite: 59.10% O,

40.09% Si, 0.81% Na. Based on EDS analysis of the zeolite, high aluminosilicate module ( $SiO_2/Al_2O_3 > 500$ ) was calculated.

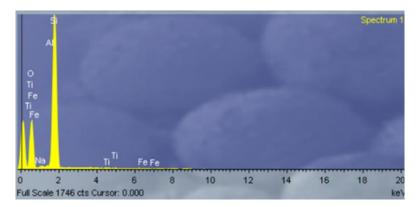


Figure 5. EDS spectrum of zeolite ZEOflair 110

#### **RESULTS OF WATER ANALYSIS**

The results of the physicochemical characteristics of the untreated water sample, shown in Table 2, are mostly within the range that is characteristic for urban surface watercourses. The electrical conductivity of rivers generally ranges from 50 to 1500  $\mu$ S/cm [39], and pH from 6.5 to 8.5 [40]. The Jala River has a high content of organic matter (table 3), which is expected considering the urban environment through which it

flows and the exposure of the water to the surrounding environment, therefore the origin of organic matter can be either from products of plant and animal life, or communal and industrial discharges. The high chloride content in the surface water of the Jala River in Tuzla, Bosnia and Herzegovina, probably originates from the underground salt deposits for which this geographic area is known, which can significantly affect chloride levels in local watercourses.

Table 2. Results of physicochemical characteristics of raw water

Parameter	Value
рН	7.49
Electrical conductivity (μS/cm)	453
Organic matter as KMnO <sub>4</sub> consumption (mg/L)	113
Ammoniacal nitrogen (mg/L)	2.26
Nitrate nitrogen (mg/L)	0.47
Nitrite nitrogen (mg/L)	0.098
Iron (mg/L)	0.00
Manganese (mg/L)	0.021
Chlorides (mg/L)	40.00

#### RESULTS OF ORGANIC MATTER REMOVAL EFFICIENCY

Table 3 shows the results of adsorption of organic matter from water by zeolite ZEOflair 110, depending on the adsorbent dose.

**Table 3**. Efficiency of adsorption of organic matter by zeolite ZEOflair 110

Dose of adsorbent (g/L)	Consumpti on of KMnO <sub>4</sub> (mL)	Content of organic matter in treated water (mg/L)	Organic matter removal efficiency (%)
1	10.3	32.55	71.2
2	9.7	30.66	72.8
4	10.9	34.45	69.5

Increasing the dose of zeolite generally increases the efficiency of adsorption of organic matter from water. This is because more zeolite provides more surface area and more adsorption sites for organic molecules to adhere to. However, once the optimal dose is reached, further increases may result in only improvements. Adsorption processes eventually reach an equilibrium in which the rate of adsorption is equal to the rate of desorption. The results in Table 3 show that by increasing the dose of synthetic zeolite, the efficiency of adsorption of organic matter in water increased to a certain value. after which it decreased. This phenomenon is generally due to several factors. First, higher doses of zeolite can lead to particle aggregation, which reduces the effective surface area available for adsorption. Aggregated particles have fewer available active sites for the adsorption process. Second, excessive amounts of zeolite can interfere with the mass transfer of organic molecules to the adsorption sites. This can create a situation where the diffusion of organic matter to the adsorbent surface is hindered, thus lowering the overall adsorption efficiency. Third, at higher doses,

most of the easily accessible adsorption sites are quickly saturated. Additional zeolite does not significantly contribute to further adsorption because the remaining sites are less accessible or the concentration of organic matter in the solution is too low for effective adsorption.

A study investigating the removal of humic acid using surfactant-modified zeolite [41] indicated that after reaching an optimal zeolite dose, further increases did not improve and could even reduce adsorption efficiency due to these reasons. Another study on the adsorption of Congo red dye [42] also observed that beyond an optimal dose, the adsorption capacity did not increase and could decrease due to similar factors like particle aggregation and reduced mass transfer efficiency.

### CONCLUSION

In this paper, the possibility of using the commercial synthetic zeolite ZEOflair 110 for the removal of organic matter from water was investigated. Characterization of zeolite was performed using advanced methods: X-ray powder diffractometry, infrared spectroscopy, temperature nitrogen adsorption and scanning electron microscopy with energy dispersive spectrometry. Based on the test results, it was determined that the examined zeolite sample has a crystal structure of ZSM-5 zeolite, and that it is a microporous material characterized by a type I adsorption isotherm. The zeolite has a regular spherical shape and is silicalite, in which the presence of aluminum was not detected, and it has a high modulus of SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> (>500).

Synthetic zeolite ZEOflair 110 is an effective adsorbent in the treatment of water with a high content of organic matter; in this research, the dose of adsorbent of 1 g/L enabled the reduction of organic matter from the initial concentration of 113 mg/L to 32.55 mg/L, while with a double dose of adsorbent (2) g/L), the treatment efficiency was only slightly increased (1.5%). However, increasing the dose of synthetic zeolite beyond an optimal point can lead to a decrease in adsorption yield for removing organic matter from water, due to particle aggregation, interference with mass transfer and saturation of adsorption sites. Thus, it is crucial to determine the optimal dose of synthetic zeolite for a given water treatment application to ensure maximum efficiency without unnecessary material usage or adverse effects on the adsorption process.

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