THE POTENTIAL APPLICABILITY OF NATURAL MINERALS AS FILTER MEDIA FOR MODULATING WATER QUALITY IN AQUATIC ECOSYSTEMS

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(Received 19th Apr 2022; accepted 11th Jul 2022)

Abstract. The optimal water quality requirement varies among species, and natural filtration materials can be used in aquatic systems to provide and maintain species-specific water quality parameters. Ammonia is one of the nitrogenous compounds originating from the metabolic wastes of aquatic organisms in aquatic ecosystems. Toxic substances and ammonia can be controlled in various ways by ion exchange and adsorption. In this study, the effects of natural clinoptilolite and diatomite on fresh water parameters were determined. This investigation was conducted by trial groups with 3 replicates for 16 days in two experimental systems. For the first experimental group, 3 g of raw zeolite (Z) was directly placed in a 500 ml freshwater aquarium, and the second experimental group was arranged with 3 g of raw diatomite (D) under the same conditions. The third experimental group was described as the control group (C) without zeolite and diatomite. Water parameters (such as pH and ammonium) were determined daily during the experiment period (8 days). After experiment 1, when zeolite and diatomite reached saturation, a desorption system was created with 3 groups containing 3 replicates, and this period was named experiment 2. During the 8-day period, water parameters were determined 7 times. At the end of the study, it was found that the NH_4^+-N concentrations different statistically ($P < 0.05$). pH, temperature and oxygen values did not vary among the experimental groups ($P > 0.05$). The results suggested that zeolite and diatomite have good adsorption performance for NH_4^+-N removal from the aqueous environment.
Keywords: *zeolite, diatomite, aquatic environment, ammonium, adsorption, freshwater*

Introduction

Optimum water quality parameters should always be constant to ensure the growth and health of fish in aquaculture. Ensuring optimum water quality is a critical factor, especially in aquarium conditions (Devi et al., 2017). In aquaculture systems, water quality parameters tend to deviate from a suitable range due to the metabolic waste of live animals and waste materials originating from uneaten feed (Hlordzi et al., 2020). In fish culture systems, it is known that the main wastes are excessive fish feed and feces. Various studies have reported that the amount of waste resulting from feces and unconsumed feed in intensive culture is approximately 10-30% (Kibria et al., 1997). In the ammonium adsorption process, water temperature and pH are efficient water quality parameters in fresh water. In sustainable aquaculture, pH values are slightly acidic for some species, while some species live in more alkaline waters. The water temperature values vary between 10-23 °C for warm water fish and 23-28 °C for tropical fish species. Freshwater culture medium and ambient conditions thought to be suitable for

http://www.aloki.hu ● ISSN 1589 1623 (Print) ● ISSN 1785 0037 (Online)
DOI: http://dx.doi.org/10.15666/aeer/2005_41454155

ammonia removal by natural adsorbents were created in this study. To maintain aquarium water conditions, proper filtration techniques and materials must be used (Öz et al., 2016).

Biological and chemical treatment is one of the methods used to remove ammonia. Materials such as marine and freshwater sand, shellfish, and activated carbon are used to prepare a substrate in the bacterial ammonia removal process (Aly et al., 2016). Ion exchange is a reversible chemical reaction in which an ion bound to a solid is exchanged for an ion in solution. While the ions on the solid surface pass into the solution, the ions in the solution are bound to the surface of the solid by electrostatic forces. This exchange process continues until the concentrations of the two types of ions on the surface and in the solution reach an equilibrium (Zain et al., 2018; Aly et al., 2016).

Natural adsorbent materials, such as zeolite, bentonite, and diatomite, can be used as filtration materials in aquarium filtration systems or as substrate materials on aquarium floors (Öz et al., 2021). Determining the application properties of these natural materials as filtration, decoration and plant ground materials in aquariums will make a multifaceted contribution to the regulation of aquarium ecosystems. Especially for recirculatory systems for intensive cultivation, these natural materials have some advantages to ensure and maintain the necessary balance between water parameters and aquatic species. These are economy, physico-chemical properties, easy availability, increasing efficiency by processing, sizing, not causing dispersion or turbidity in water, and use with different decoration features (Eroğlu et al., 2017; AbuKhadra et al., 2020).

Zeolite is a naturally occurring rock that has a fairly unique structure with large internal cavities and entry channels that are easily filled with water, air, and other molecules. They have strong capacities to adsorb and desorb molecules that allow for rapid uptake and loss of charged particles. Zeolite is relevant to aquaculture (Ramesh et al., 2011; Aly et al., 2016).

Clinoptilolite has a limited capacity to adsorb ammonia, and when it reaches saturation, it can be made ready for reuse by soaking in a suitable solution. This process can be repeated several times (Aly et al., 2016).

Diatomite is a low-cost silica material of soft sedimentary rock that is abundantly available. It is a porous material with low density and constitutes mainly silicon dioxide. Although diatomite has a unique combination of physical and chemical properties, its use as an adsorbent in wastewater treatment has not been extensively investigated (Ahmad et al., 2019; Bakr, 2010). As a result, diatomite is nontoxic and odorless, present naturally in large quantities with high purities, and subsequently available at low cost (Bello et al., 2014). In this study, which will be one of the first studies made with diatomite in aquaculture, primarily a study on ammonia originating from fish feed was planned. This research aimed to determine the potential applicability of natural adsorbents in aquaculture environment conditions by examining the effects of natural adsorbents [zeolite (clinoptilolite) and diatomite] on important parameters, such as ammonium, pH and dissolved oxygen.

Materials and methods

Adsorbent materials

Raw (no preconditioning was applied) zeolite and diatomite were tested for their capability of adsorbing ammonia or effects on dissolved oxygen and pH in a freshwater

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aquarium. Zeolite (Clinoptilolite) and diatomite were provided by the Gordes Mining Company, Manisa, Turkey and Nanotech inşaat Kimya Maden ve Lojistic San. Tic. A.Ş., respectively. In this study, Anatolian zeolite (clinoptilolite) and diatomite were used, and their chemical compositions are shown in *Table 1*. The clinoptilolite and diatomite used in the experiment were characterized by SEM/XRF/BET (*Fig. 1*). These analyses were performed in the Kastamonu University Central Research Laboratory. pH values were calculated according to Tokat (2019) and Güneş (2017).

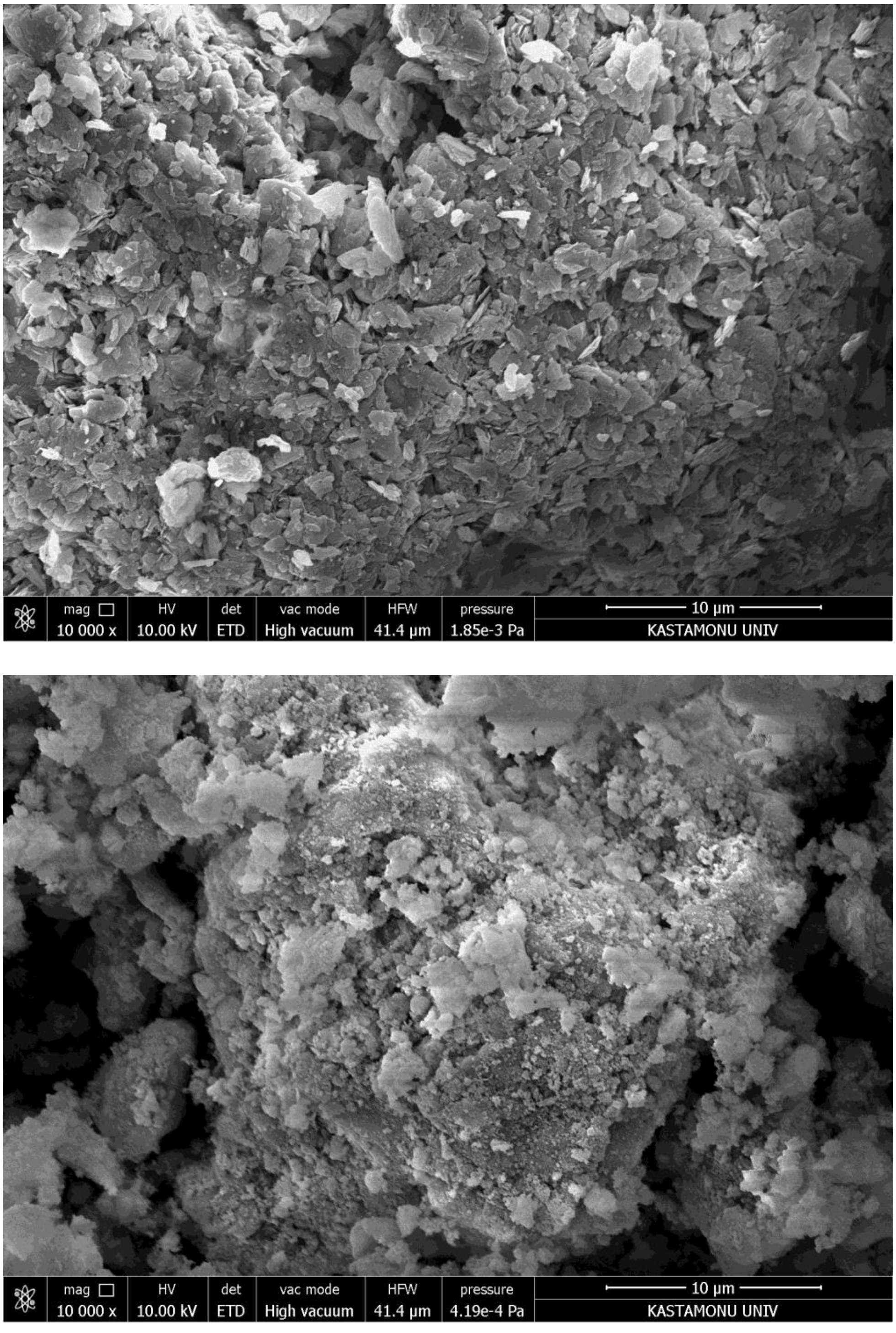

Figure 1. *SEM analysis of clinoptilolite (a) and diatomite (b)*

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Table 1. *XRF values of clinoptilolite and diatomite*

	Clinoptilolite composition	**Diatomite composition**
SiO_2 (%)	78.41	81.66
Al_2O_3 (%)	13.83	10.02
MgO (%)	1.646	3.839
K_2O (%)	2.372	0.99
CaO (%)	3.885	2.041
Na_2O (%)	1.042	1.261
Fe_2O_3 (%)	1.414	2.291
P_2O_5 (%)	0.058	0.243
SiO_2/Al_2O_3	5.67	8.149
BET Surface Area	34.316 m^2/g	174.698 m^2/g
pH	8.31	7.06

Experiment period 1

The main sources of ammonia in aquaculture are fish metabolic wastes and unconsumed feed. In this study, unconsumed feed was used as a source of ammonia. Tap water with the same characteristics was used in all groups. Experiments were conducted in plastic aquariums with 500 ml of water, 0.5 g fish feed (47.5% crude protein, 6.5% crude fat, 2% cellulose and 6% moisture 6%), and 3 g natural adsorbent. The experiment was carried out in 3 groups. Zeolite at 3 g/500 ml water was placed in the experimental aquariums for the first group (Z), while for the second group (D), diatomite was placed in the experimental aquariums at 3 g/500 ml water. Zeolite and diatomite were not used in the third group (control, C). The experiment was designed in triplicate (Zain et al., 2018) for each group without any fish or aeration. Water parameters values were determined in the beginning of this study, and all water parameter values were determined each day during the first experiment period.

Experiment period 2

After experiment 1, where zeolite and diatomite had reached saturation, a desorption system was created, and 3 groups were formed with 3 replicates; this period was named experiment 2. During this 8-day period, water parameters were determined 7 times. Saturated zeolite (3 g) and diatomite (3 g) in the first period were placed inside 500 ml tap water for the SZ (saturated zeolite) and SD (saturated diatomite) groups. Zeolite and diatomite were not used in the third group (control, C), and in experiment 2, a control group without feed was formed, and no further application was made until the end of the 2nd experiment (*Fig. 2*).

The physico-chemical quality of the water in the experimental aquariums was monitored at the same time daily. Experimental data were determined using the multi-parameter YSI Professional Instrument.

During the study, NH_3 and TAN (total ammonia nitrogen) ($TAN = NH_3 + NH_4$) levels were calculated using NH_4^+, pH and water temperature values were also determined (Purwono et al., 2017).

The water parameters were statistically checked with one-way analysis of variance (ANOVA) and the means were compared at 5% ($p < 0.05$) significance level using the Tukey test. The results were analyzed statistically with the “Minitab Release 17 for Windows” software (Nanda et al., 2021).

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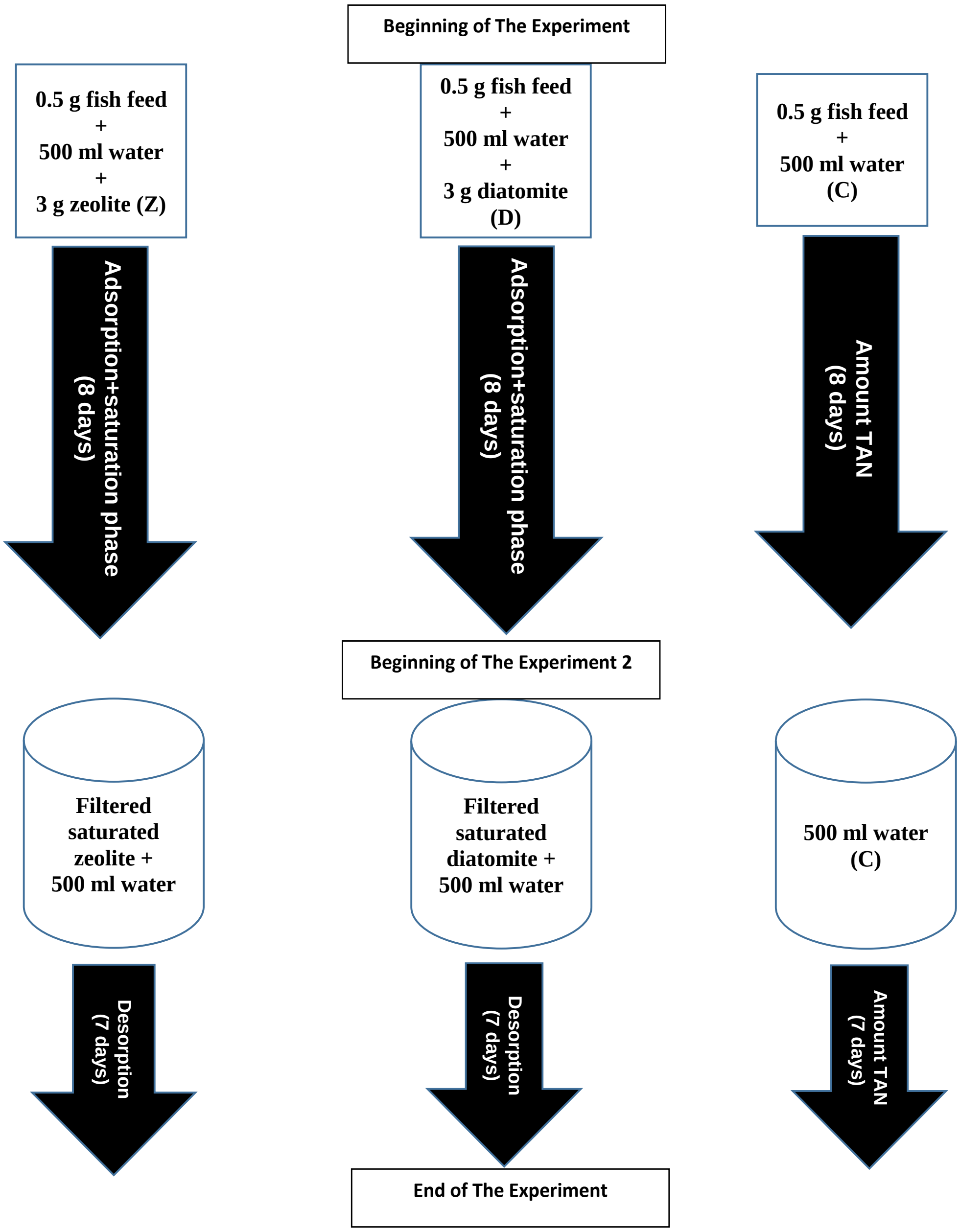

Figure 2. *Flowchart detailing experimental design*

Results

Tap water with the same characteristics was used in all groups. At the beginning of the study, water temperature, dissolved oxygen, pH and ammonium were determined to be 21.7 ± 0.01 °C, 2.84 ± 0.01 mg/l, 8.20 ± 0.01 and 0.4 ± 0.01 mg/l, respectively, in all groups.

The water quality parameters determined at the end of experiment 1 are summarized in *Table 2*. At the end of the first experiment, although the differences between groups (Z, D, C) regarding water temperature, pH and dissolved oxygen were statistically insignificant ($p > 0.05$), there was a difference among groups in values of NH_4^+-N levels (Z, C) ($p < 0.05$).

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Table 2. *Water parameters at the end of the first experimental period (adsorption) (mean ± SE)*

Experimental groups*	Temperature (°C)	Dissolved Oxygen (mg/l)	pH	NH_3 (mg/l)	NH_4^+-N (mg/l)	TAN (mg/l)
Z (Zeolite)	20.26 ± 0.28	0.44 ± 0.15	7.67 ± 0.07	0.22 ± 0.05	8.60 ± 1.50^{b}	8.83 ± 1.55^{b}
D (Diatomite)	20.08 ± 0.26	0.47 ± 0.15	7.70 ± 0.07	0.34 ± 0.09	11.73 ± 2.46^{ab}	12.07 ± 2.55^{b}
C (Control)	20.17 ± 0.25	0.47 ± 0.16	7.66 ± 0.08	0.44 ± 0.12	18.53 ± 3.58^{a}	18.97 ± 3.68^{b}

*Superscript letters in a column indicate significant ($p < 0.05$) differences between experimental groups. Means were tested by ANOVA and ranked by Tukey's multiple range test

At the end of the first experiment, TAN values in water were determined to be 8.83 ± 1.55, 12.07 ± 2.55 and 18.97 ± 3.68 for the Z, D, and C groups, respectively. When these values were examined statistically, it was found that the two groups were different ($p < 0.05$) compared to the control group. Likewise, using TAN data obtained from the three experimental groups, TAN concentrations decreased by 57.57 ± 0.41% in the Z group and 27.21 ± 3.34% in the D group compared to the control group. At the beginning of experiment 2, water temperature, dissolved oxygen, pH and ammonium were determined to be 19.2 ± 0.01 °C, 2.79 ± 0.01 mg/l, 8.60 ± 0.01 and 0.2 ± 0.01 mg/l, respectively, in all groups. The water quality parameters determined at the end of the second experiment are presented in *Table 3*. At the end of the second experiment, TAN values in water were determined to be 3.21 ± 0.30, 3.90 ± 0.29 and 0.30 ± 0.01 for the Z, D, and C groups, respectively. When these values were examined statistically, it was found that the two groups were different ($p < 0.05$) compared to the control group.

Table 3. *Water parameters at the end of the second experimental period (desorption) (mean ± SE)*

Experimental groups*	Temperature (°C)	Dissolved Oxygen (mg/l)	pH	NH_3 (mg/l)	NH_4^+-N (mg/l)	TAN (mg/l)
Z (Zeolite)	20.18 ± 0.21	0.55 ± 0.03	8.48 ± 0.02	0.37 ± 0.04^{a}	2.84 ± 0.26^{a}	3.21 ± 0.10^{a}
D (Diatomite)	19.87 ± 0.22	0.57 ± 0.03	8.47 ± 0.02	0.42 ± 0.04^{a}	3.48 ± 0.26^{a}	3.90 ± 0.29^{a}
C (Control)	19.75 ± 0.22	0.63 ± 0.03	8.52 ± 0.02	0.03 ± 0.00^{b}	0.24 ± 0.01^{b}	0.30 ± 0.01^{b}

*Superscript letters in a column indicate significant ($p < 0.05$) differences between experimental groups. Means were tested by ANOVA and ranked by Tukey's multiple range test

When the 1st and 2nd experiments were evaluated together, the ammonia concentration started to differ between the groups on and after the 4th day, and the adsorbent groups had lower concentrations than the control group. With the saturation of adsorbents on the 8th day of study, the 2nd trial period started, and when zeolite and diatomite, which reached saturation at the end of experiment 1, were taken into the fresh water in experiment 2, it was determined that some of the NH_3 adsorbed was released to the water as 0.51 and 0.49 mg/l, respectively (*Fig. 3*). The base ANOVA tables were given in *Table 4* for experiment 1 and 2.

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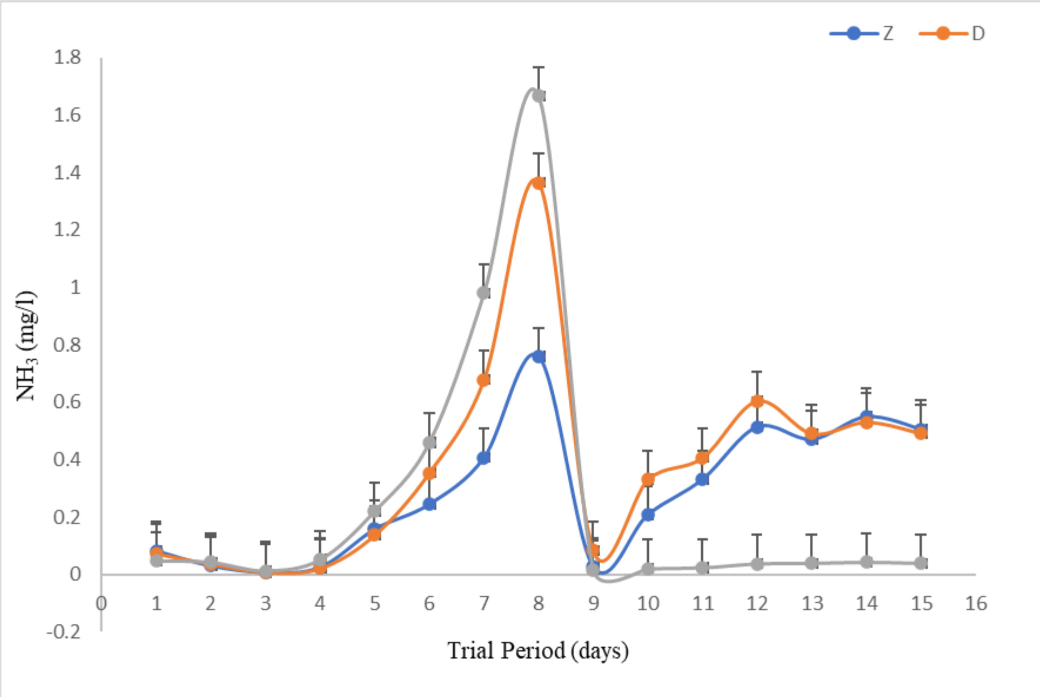

Figure 3. *NH_3 exchange during the experimental period (15 days)*

Table 4. *The base ANOVA tables for the first (adsorption) and second experimental period (desorption)*

Experimental groups*	Df	Adj SS	Adj MS	F	P
First experiment	2	1234	617.2	3.66	0.03
Second experiment	2	123.57	61.78	64.83	0.01

Discussion

This study focused on testing two different natural adsorbents, zeolite and diatomite, and their efficiency in removing ammonium cations from freshwater environments. TAN is the sum of NH_3 and NH_4. NH_3 values are important for fisheries. Therefore, in this study, TAN, which shows the total values, and NH_3 values, which are important for fisheries, were examined.

Metabolic wastes of aquatic organisms and unconsumed feeds are among the most important factors affecting water quality in aquaculture (Boyd, 1990).

The potential to use natural zeolite mineral, which is used as a filtration material, has increased rapidly in recent years due to a natural, economical and effective ammonia removal. Many studies have been conducted on the use of natural zeolite mineral in water treatment (Jorgersen and Weatherley, 2003; Mazeikiene et al., 2008; Öz et al., 2017).

Diatomite is a low-cost silica material of soft sedimentary rock that is abundantly available. It is a porous material with low density and constitutes mainly silicon dioxide. There are studies conducted on hydrogen sulfide (Ahmad et al., 2019), heavy metal (Şenol and Şimşek, 2020; ElSayed, 2018) or ammonium (Luo et al., 2011; Khadra et al., 2020) retention by raw or processed diatomite.

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Ammonium adsorption of natural adsorbents changes with the amount, pore size, surface area, and mining area of minerals, initial concentration, pH, temperature, and presence of other cations in the solution (Huang et al., 2017).

Many factors are effective in ammonia removal using natural adsorbents, such as zeolite and diatomite. These factors include the amount of adsorbent, its size, preliminary applications, and the temperature of the environment, pH, other competitor ions or the concentration of the substance to be removed. At the end of the first experiment (adsorption), groups containing raw zeolite (Z) and raw diatomite (D) had lower ammonia values (8.6 ± 1.50 and 11.73 ± 2.46 mg/l, respectively), whereas the control group had a higher ammonia value (18.53 ± 3.58 mg/l). The final of the second trial period (desorption), the Z and D groups had higher ammonia values of 2.84 ± 0.26 mg/l and 3.48 ± 0.26 mg/l, respectively. The control group contained the lowest ammonia value (0.24 ± 0.01 mg/l) compared to the adsorbent-added groups.

In this study, the TAN removal percentages of crude zeolite and crude diatomite were found to be $57.57 \pm 0.41\%$ and $27.21 \pm 3.34\%$, respectively, compared to the control group. Similar to our study, Zhou and Boyd (2014) found a TAN removal percentage of 43% in zeolite. Clinoptilolite and diatomite exchange ions in their structures with ammonium ions in the aquatic environment. Thus, it reduces the amount of ammonia in the water and prevents it from rising to harmful levels (Ghiasi and Jasour, 2012; Khadra et al., 2020).

One of the most important water parameters affecting production in aquaculture is total ammonia nitrogen. In closed or circulating aquaculture systems, among the main causes of diseases and fish deaths, nitrogenous compounds produced by the deterioration of feces and uneaten fish feed (El-Gendy et al., 2015). It has been determined that the harmful efficients of ammonia (NH_3) for short-term effect is generally between 0.6 and 2.0 mg/L for aquaculture, and non-lethal effects can occur between 0.1 and 0.3 mg/L. It has been suggested that the tolerable ammonia limit for aquaculture should be lower than 0.2 mg/L (Bhatnagar and Devi, 2013). In general, it was determined that ammonium in the water was adsorbed by the adsorbents in the 1st experiment and that ammonia retained by the adsorbents was released into the water in the 2nd experiment. In this study, on the 5th day, the NH_3 level was approximately 0.1 mg/l in the zeolite and diatomite groups, while this value was found to be 0.2 mg/l in the control group. From the 6th day, the NH_3 values of the diatomite group were higher than those of the zeolite group but remained at lower levels than those of the control group. On the 8th day, it was determined that the adsorbents reached saturation and the adsorption efficiency decreased. In the second period, it was ascertained that diatomite and clinoptilolite could release some of the adsorbed NH_3, and this result was similar to the results of Henstrom and Amofah (2008) and Öz et al. (2017).

Conclusion

Controlling the data in the study revealed that when the zeolite and diatomite reached saturation, they desorbed as dramatically as the amount of ammonium they retained. As soon as clinoptilolite and diatomite reach saturation, they can be used again by keeping them in salt water and similar solutions. In intensive aquaculture systems or aquarium conditions, based on the present results, zeolite and diatomite should be used after being processed for the required period and ready for readsorption.

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It has been reported that zeolite can be used effectively for ammonia removal in aquaculture for a long time, studies on ammonia removal with diatomite are more recent, with limited information available. Even the number of studies related to aquaculture is deficient. In this regard, the results of this study indicated that diatomite can be used effectively in ammonia removal, especially for aquaculture. In future research, application processes, such as preconditioning or the use of mixed adsorbents, can be investigated to increase the efficiency of both zeolite and diatomite in conditions with ammonia, pH and water temperature suitable for aquaculture.

Acknowledgment. We would like to express our appreciation to the company, Gordes Mining Company, and Nanotech İnşaat Kimya Maden ve Lojistic San. Tic. A.Ş for providing us with the zeolite and diatomite trial materials.

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http://www.aloki.hu ● ISSN 1589 1623 (Print) ● ISSN 1785 0037 (Online)
DOI: http://dx.doi.org/10.15666/aeer/2005_41454155