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Ammonium Removal in Aquatic Conditions Using Different Levels of Calcium Bentonite

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ABSTRACT: Ammonia, necessary for life cycles is a natural substance that is available in all forms of life from plants to animals and human beings. Ammonia is toxic at high levels, however low concentrates can be tolerated by the living forms. Especially in mass production of fish under intensive culture conditions, significant amount of ammonia excretion deteriorates the water environment. Hence, removal of ammonia from water environment may support the re-use of freshwater in aquaculture facilities, and benefit for sustainable utilization of water resources. Compared to clay minerals, bentonite has a high usability due to its high adsorption capacity and low cost. In this study, it was aimed to determine the effects of two different concentrations of calcium bentonite on ammonium adsorption in a freshwater environment at constant temperature (17°C). Two different concentrations of calcium bentonite (7g/l B7 and 14g/l B14) were tested in triplicate and experiments were performed in chambers with 2 liter water and NH_4Cl incorporated to obtain 10 mg/l NH_4^+ in each of the water chamber. Water quality parameters such as temperature, pH, dissolved oxygen (DO) and total ammonium nitrogen (TAN) were determined during the course of the experiment. Ammonia adsorption effects of calcium bentonite were tested and at the end of the trial, NH_3 values were recorded as 0.461±0.01mg/l and 0.463±0.01 mg/l for the B7 and B14 groups, respectively. There were difference in TAN values between the experimental groups ($P<0.05$). As a result, it has been concluded that bentonite of 14 g/l might be ideal for ammonium removal under aquatic conditions applied.

Keywords – Adsorption, Ca-bentonite, Ammonium, Aquaculture

1. Introduction

Nitrogen (N) is essential for all life forms. Barely, high concentration of N increases oxygen demand and eutrophication in surface and groundwater (Huang et al., 2010). Ammonium (NH_4^+) is the inorganic ion form of N included in domestic and industrial wastewater, municipal sewage or decomposed from organic N compounds in those wastewaters (Zheng et al., 2008). Nitrogenous compounds are important for life-sustaining components to various aquatic organisms and these conclude ammonia, nitrites and nitrates which are the most common dissolved inorganic nitrogen forms in aquatic ecosystems (Oluwaseyi, 2016). Fish produce nitrogenous waste materials by catabolism of amino acids. Nitrogen compounds are important for fish health and profitability of production (Oluwaseyi, 2016). Ammonia exists in water in two forms (un-ionized ammonia NH_3 and ionized ammonium NH_4^+), especially ammonia (NH_3) is highly toxic to fish. In order to remove ammonium from wastewater, several technologies have been tested, namely adsorption and ion exchange (De Luna et al., 2018; Nazari et al., 2018), biological processes (Patroescu et al., 2015), air stripping (He et al., 2015), supercritical water oxidation (Asselin et al., 2008), breakpoint chlorination (Huang et al., 2015), chemical precipitation (Tulaydan et al., 2017), reverse osmosis (Kosutic et al., 2015) or microwave radiation (Huang et al., 2016). Among them, ion exchange and adsorption are preferred method due to their economic advantages, low energy input, attainability, reusability and easy operation.

Adsorption is the period in which molecules from solution accumulate in the external or/and internal surface of the porous solid (Gupta and Suhas, 2009; Kammerer et al, 2011). Adsorption and ion exchange participates of some basic characteristics. Adsorption and ion exchange are both diffusion processes, they are grouped together for a unified treatment and called as sorption process, that is a mass transfer of molecules form aqueous to the solid phase (Gupta and Suhas, 2009), and several different materials such as silica, cellulose, active carbon, natural or synthetic zeolite (Ergün et al., 2008) or clay bentonite (Rožić et al., 2000; Eturki et al., 2012) In this study, bentonite which is a kind of clay mineral was used because of its low cost and availability in the market and higher selectivity for ammonium removal. Clay minerals have large surface areas with a net negative charge, therefore, the inorganic and organic cations (NH_4^+, Pb^{+2}, Cu^{+2}, K^+ etc.) can hold in the water (Babel and Kurniawan, 2003; Konig et al., 2012). The use of clay minerals is especially preferred for waste water treatment in the removal of nutrients, organic compounds and heavy metals in the environment (Abdelaal, 2004). Bentonite is a phyllosilicate mineral formed by natural ways, respectively, with high cation exchange and ion adsorption capacity (Booth, 1999). There are two types of bentonites, namely sodium bentonite and calcium bentonite. Calcium bentonite is a low-swelling type, which evolved from volcanic ash deposited in freshwater environments, while sodium bentonite is usually a high-swelling type, derived from volcanic ash that is deposited in marine environments (Dwairi and Al-Rawajfeh, 2012). For the ammonium adsorption process in freshwater, the pH and temperature are two important water quality parameters. For ammonia adsorption in sustainable aquaculture applications, optimum results are obtained between values approximately 6.0-8.5 for pH and 10-27°C for temperature (Jorgensen, 2002; Iskander et al., 2011; Ismadji et al., 2016). In this study, it was aimed to determine the effects of two different amount of calcium bentonite on the ammonium adsorption in freshwater at constant temperature (17 °C) and pH (8.0) levels.

2. Material and Methods

Ca-bentonite material was bought from a company, Samaş Industrial Materials. It was 0.075 mm particle size and chemical composition of bentonite was given in Table 1.

Table 1. *Chemical Composition of Ca-bentonite*

Component	Amount (%)
SiO_2	61.28
Al_2O_3	17.79
Fe_2O_3	3.01
CaO	4.54
Na_2O	2.70
MgO	2.10
K_2O	1.24

The present study was conducted with two experimental groups in a triplicate design. Two different levels of calcium bentonite were tested at rates of 7.0 and 14 g/l, and these two experimental groups were named as B7 and B14, respectively. Experimental groups were prepared by adding 2 liter of tap water and NH_4Cl to each of the experimental chamber to provide NH_4^+ at a rate of 10 mg/l. The initial solution of 10 mg/l NH_4^+ was prepared via dilution from a concentrated ammonium solution. Thereafter, experimental chambers of the two experimental groups (B7 and B14) were supplied with two different levels of bentonite in triplicate chambers (Fig. 1).

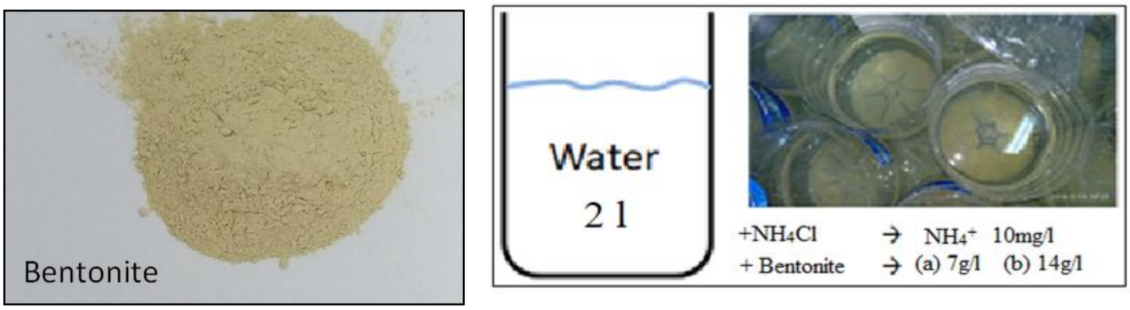

Figure 1. *Bentonite and experimental design*

Aeration, heating, lighting or agitation procedures were not applied during the course of the trial. Natural bentonite were used and no preconditioning was applied. Water temperature, pH, dissolved oxygen and NH_4^+ values were measured during the experimental period (345 min). Water parameters were measured using a YSI Professional multi-probe water quality analyzer (Jorgensen, 2002). In an earlier study, Prajapati (2014) reported that the results obtained from Nessler method and those of the conventional electrode method gave very similar and reliable values. Therefore, in the present study carried out in a water condition suitable for aquaculture practice, ammonia measurements in the water were performed via electrode method as reported by Prajapati (2014). Another reason for the preference of the electrode method was the easy-to-use, fast and high reliability of the method in laboratory conditions as well as for farmers in aquaculture production facilities.

The removal efficiency (RE, %) of ammonium (NH_4^+) for the two test groups were calculated using the following equations (Alshameri et al., 2017):

RE (%) = [(ACi – ACe) / ACi] x 100

where, ACi is the initial ammonium concentration in mg/l, and ACe is the equilibrium ammonium concentration in mg/l.

During the study NH_3 and TAN levels were calculated from NH_4^+, water temperature and pH values. The calculation of the ammonium concentration is formulated below (Emerson, 1975; Chow et al., 1997):

The dissociation constant, K_a, of ammonium ion is expressed as

$$K_a = \frac{[NH_3][H^+]}{[NH_4^+]}$$

Above equation can be further arranged as

$$\frac{[NH_3]}{[NH_4^+]} = \frac{K_a}{[H^+]}$$

Thus, the relationship between ammonia and ammonium concentrations may be described by

$$log_{10}\frac{[NH_3]}{[NH_4^+]} = pH - pK_a$$

pK_a varies with solution temperature. This temperature dependence is given as follows:

$$pK_a = 0.09018 + \frac{2729.92}{(273.2+T)}$$

Where T is the solution temperature in °C. Also

$$[NH_4^+] = [NH_3]_T - [NH_3]$$

$[NH_3]_T$ being the total concentration of ammonia forms.

Rearrangement of this equation of this equation yields

$$log_{10}\frac{[NH_3]}{[NH_3]_T-[NH_3]}$$
$$= pH - \left[0.09018 + \frac{2729.92}{(273.2+T)}\right]$$

Statistical analyses of the obtained data were performed using "Minitab Release 17 for Windows" software program. In terms of water parameters statistical significance between experimental groups were evaluated by one-way analysis of variance (ANOVA) and means have been compared using Fisher's range test at 5% level of significant (Zar, 2010).

3. Results and Discussion

In this study, it was found that bentonite was particularly effective in reducing ammonia and TAN levels. Water quality parameters analyzed at regular intervals throughout the course of the trial are given in Table 2.

Table 2. *Mean water quality parameter values during experiment (mean ± SE).*

Experimental Groups*	Water Quality Parameters					
	Water Temperature (°C)	Dissolved Oxygen	pH	NH_3 (mg/l)	Initial NH_4^+ (mg/l)	TAN (mg/l)
Bentonite 7g/l	17.43±0.03[a]	7.84±0.03[a]	8.25±0.00[a]	0.461±0.00 [a]	10.0±0.00	9.44±0.23 [a]
Bentonite 14 g/l	17.43±0.03 [a]	7.84±0.03[a]	8.30±0.01[a]	0.463±0.01 [a]	10.0±0.00	8.58±0.19 [b]

*[a,b] Different superscripts within column represent significant differences (P<0.05).

Over the course of the trial, TAN concentrations were significantly different between the two experimental groups (B7 and B14) (P<0.05). Ammonia concentrations were calculated using NH_4^+, water temperature and pH levels measured throughout the study and daily NH_4^+ levels of trial groups are given in Fig. 2.

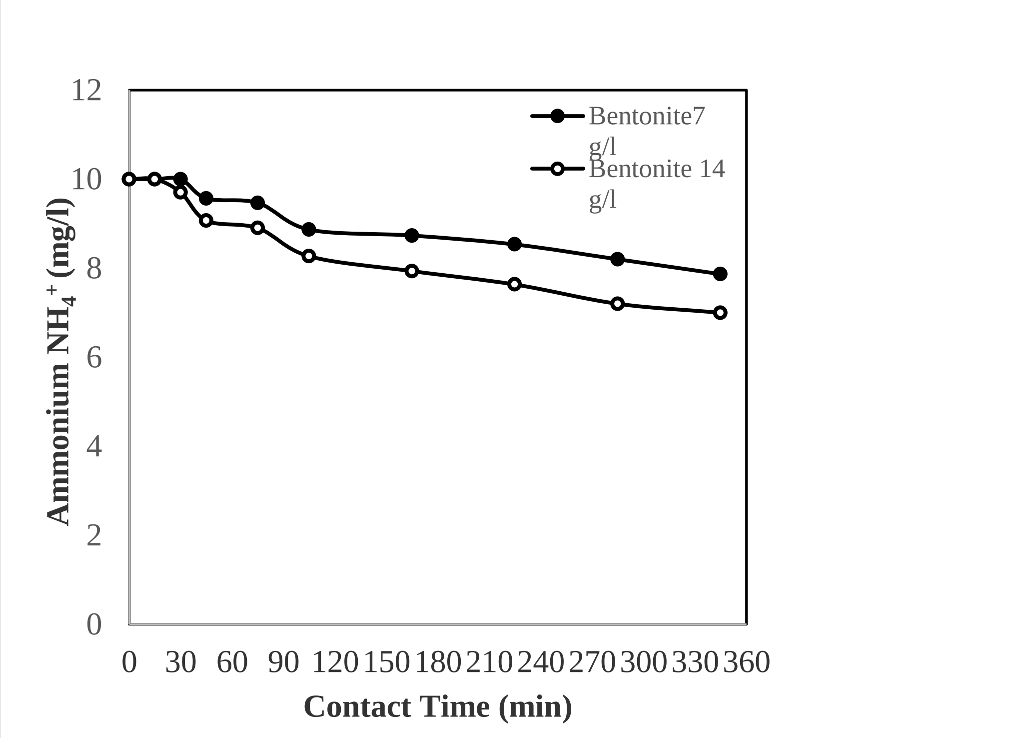

Figure 2. *Effect on ammonium removal on the two different amount of bentonites during the 345 min. at pH 8.0, 17 °C*

The experiments show that natural bentonite presented positive effects on aquaculture water conditions in terms of reduced TAN concentrations. Our findings regarding the efficient use of bentonite in ammonia removal from water are in close agreement with the results report earlier by Ismadji et al. (2016). As shown in Fig.3, the removal efficiency of NH_4^+ in all trial groups increases with the increasing of the contact time. Similarly Aydın Temel and Kuleyin (2016), Tilaki and Kahe (2012) the adsorption of NH4+ by Turkish natural adsorbent increased rapidly with increasing contact time in the first 30-60 min. for all experimental groups and thereafter increased slowly till equilibrium time.

In order to increase the efficiency of adsorption process with bentonite, recent investigations were intensified on different methods such as conditioning or changing the physical and chemical properties of the solution or a combination of different adsorbents (Toor and Jin, 2012; Hank et al., 2014). At the end of this study, the removal efficiency of bentonite in regards to ammonia concentrations was investigated and ammonium was successfully removed up to 22% (B7)-33% (B14) from the aquatic environment.

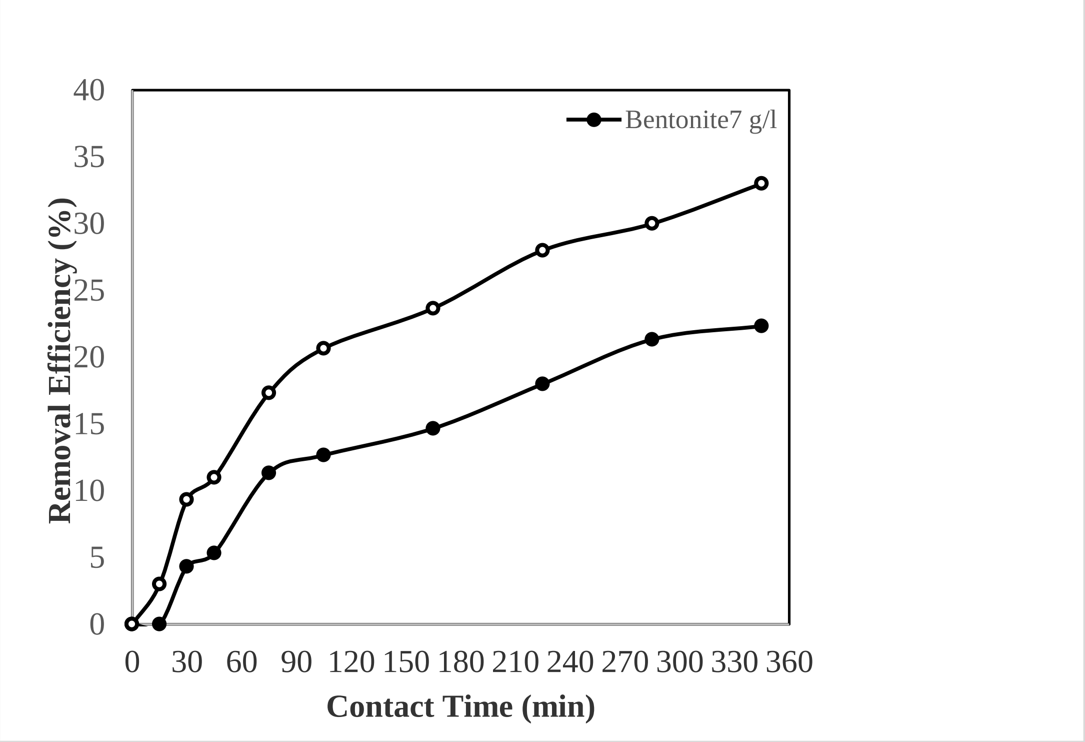

Figure 3. *Effect of contact time on NH_4^+ removal efficiency during the course of 345 min. at pH 8.0, 17 °C*

The pH was influenced during the experiment because of a small rate of increase in all the samples (approximately 8.0 prior to bentonite exposure) (Fig. 4). The increased pH values in the solution might be explained with the hydrolization of calcium and magnesium carbonate compunds by bentonite exposure (Mazeikiene et al., 2008; Oz et al., 2015; Ismadji et al., 2016).

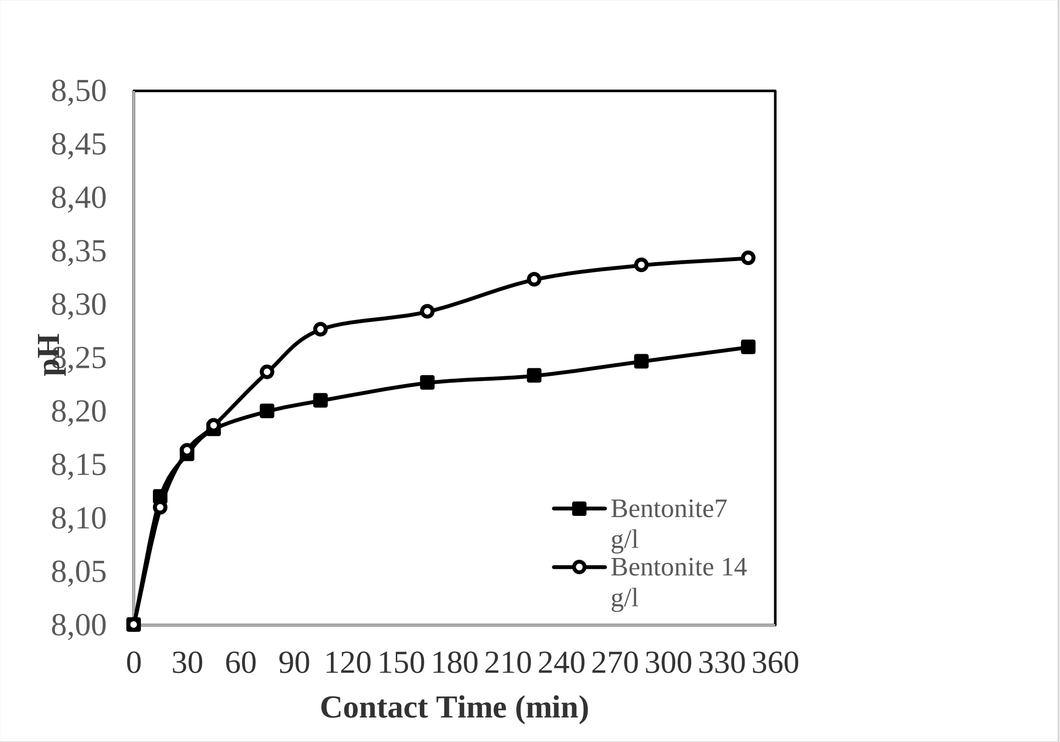

Figure 4. *pH variation during the course of 345 min. at 17 °C*

Keeping water conditions within the ideal range is one of the most important challenges in intensive fish culture. Metabolic waste from live animals and unconsumed feed particles are the most important factors affecting water conditions in aquaculture (Boyd, 1990). It was reported that toxicity of unionized ammonia (NH_3) began at 0.05 mg/l, while it caused death at 2 mg/l for many fish species (Floyd et al., 2015). In general, 0.02 ppm is considered as a limit for ammonia in aquaculture facilities (Swann, 1992). In this study, it was determined that the ammonia value is approximately 0.5 mg/l. This value obtained in the experiment is safe for fish because it remains within the range determined in the literature. In this study, the use of natural bentonite showed a remarkable performance as a promising filter material for the intensive aquaculture. Ammonium adsorption of clay minerals such as bentonite changes with the amount, pore size, surface area, and mining area of minerals; the initial concentration, pH, temperature, and the presence of other cations of the solution (Huang et al., 2017). Similarly, findings in this study revealed that inclusion of bentonite in the aquatic environment at 14 g/l level reduced further increasing of ammonium concentrations. As the amount of bentonite increased in our study the rate of ammonium uptake also increased. Further, the adsorption tests in the present study have demonstrated that the contact time and adsorbent dosage provided significant effects the NH_4^+ adsorption, which is in agreement with Eturki et al. (2012) and Angar et al. (2017).

4. Conclusion

The results from this study are presented below:
(1) Adsorption process is a strong alternative among the other removal technologies for NH_4^+, because it's easy-to use.
(2) Considering lower (7 g/l) and higher (14 g/l) bentonite levels tested in this study for ammonia removal, it can be concluded that the removal efficiency was remarkably higher in the 14 g/l (33%) bentonite group over the 7 g/l treatment group.
It is important to balance high ammonium concentrations in aquaculture. For this purpose, Ca-bentonite, which can be used as filter material, can be a good alternative to ammonium removal, given the results obtained in this study. In addition, it was determined that other water parameters (pH, dissolved oxygen, water temperature) in freshwater were not be adversely affected from ammonium removal with bentonite (P>0.05). Thus, it can be concluded that bentonite might be used as a natural filtration material in aquaculture facilities, supporting sustainable and environment friendly aquaculture industry in the future.

5. Acknowledgment

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