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REMOVAL OF AMMONIUM NITRATE FROM AQUACULTURE BY SORPTION USING ZEOLITE

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ABSTRACT

Zeolite 4A was found to be useful and feasible method for removal of Ammonium nitrate from aquaculture waste water. These zeolites showed excellent sorption by ion-exchange. They can be successfully regenerated by using 0.1 M NaCl. The regenerated zeolites showed similar sorption capacity as virgin upto 3-4 cycles. During the following cycles their surface gets saturated with permanent sorption of nitrogen which reduces their sorption capacity. Modification of zeolite surface with strong acids leads to an increase (0.2602 gm of ammonium nitrate / gm of zeolite) in sorption capacity for ammonium nitrate. This shows that zeolites are excellent materials for removal of nitrogenous material from aquaculture waste water.

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INTRODUCTION

Concentration of nitrogenous material in aquaculture is highly important for proper growth and survival of aquatic animals [1]. The nitrogenous material is released by excretion of aquatic animals (viz., urine, faecal material), food residue, decaying of dead, etc [2]. Accumulation of ammonium, nitrate and nitrite materials leads to depletion of dissolved oxygen in water which results in growth of harmful algae in aquaculture [3]. The presence of unionized ammonia at concentration level of 0.06 ppm and oxygen level below 5.0 ppm could damage gill tissues (hyperplasia), lead to gills diseases, other tissue lesions and reduced growth rate in aquatic animals [4]. This would lead to death of fishes and shrimps in aquaculture. As the concentration of nitrogen increases, growth of aquatic animals slows down [5]. The aquatic nitrogen is commonly present in the forms of ammonia/ammonium ($\text{NH}_3/\text{NH}_4^+$), nitrate (NO_3), nitrite (NO_2), and organic nitrogen [6].

Efficient recycle of aquaculture water requires removal of ammonia and nitrogenous compounds [7]. The range of nitrogen in aquaculture varies from 20 to 70 mg N/L [8]. This is composed of 60-70 % ammonium nitrogen and 30-40% of organic nitrogen [9]. This concentration is very high and would have harmful effect on aquatic life. This makes removal of these nitrogenous material and purification of water highly important for aquatic life. Various technologies have been developed for TAN removal like biological nitrification,

chemical de-nitrification, catalytic liquid phase oxidation, air stripping, membrane separation, selective ion exchange [10]. Biological nitrification has been preferred for aquaculture treatment uptill now due to its low cost as compared to physical/chemical process [11]. Other most common procedures for removal of nitrogenous waste and water purification are water aeration treatment, sorption using zeolites and other components, use of formalin and bacterial products for the removal of aquatic nitrogen [12]. Aeration showed lower removal rates for total ammonium nitrogen (TAN) [13]. The nitrogen compounds from water can be removed by adsorption using artificially prepared zeolites which acts as ion exchange media [14].

Treatment of nitrogen containing water by adsorption with zeolites is an important due to ease of treatment and low cost processing. Though natural zeolites are available, but their low sorption capacity makes them unacceptable for real life application [15]. Artificially prepared zeolites can be economical as ion exchange resin in aquaculture waste water treatment [16]. Zeolites are three dimensional micro porous crystalline solids. Their ion exchange property and thermal stability make them highly useful in waste water treatment [17]. Treatment of aquaculture waste water using zeolites is a standard practice in south east Asia [18]. Modification of these zeolites will make them more attractive by increasing their ion exchange property and ability of selective removal of ammonium, nitrate and nitrite ions [19]. The ion exchange capacity in Zeolite is due to

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deficiency of positive charge generated by presence of aluminum (Al^{3+}) and substitution for silicon (Si^{4+}) [20]. Negative charge on the zeolites is balanced by NH_4^+ , Na^+ , K^+ , Ca^{2+} or Mg^{2+} . Depending upon the interaction capacity; cations in the Zeolite can be exchanged with those present in liquid solutions. Current study is focused on removal of nitrogenous compound (ammonium nitrate) by using zeolites. The parameters (natural Zeolite and modified Zeolite such as initial ammonium nitrate concentration, pH, NaCl dose) and operating conditions affecting removal of ammonium nitrate by selected zeolites and modifications (Modification of Zeolites with strong acid) will be optimized.

Experimental Set Up

Materials

Zeolite (chemical formula: $\text{Na}_{16}(\text{Al}_{16}\text{Si}_{32}\text{O}_{96}) \cdot 16\text{H}_2\text{O}$, purity: 84%, Surface charge is negative, particle dimension: 4A^0) grade was obtained from Chemicals India Ahmednagar Maharashtra India. H_2SO_4 98% pure synthesis grade was obtained from Fisher Scientific. NaCl (extra pure AR grade) was obtained from Sisco Research Laboratories Pvt. Ltd. Ammonium nitrate was obtained from Lobo Chemie Pvt. Ltd. Mumbai (M.W 80.04 purity 98.5%).

Sorption studies

An experimental setup was consisting of cylindrical column (1 cm in diameter, 20 cm in height) was prepared, as shown in Figure (1)

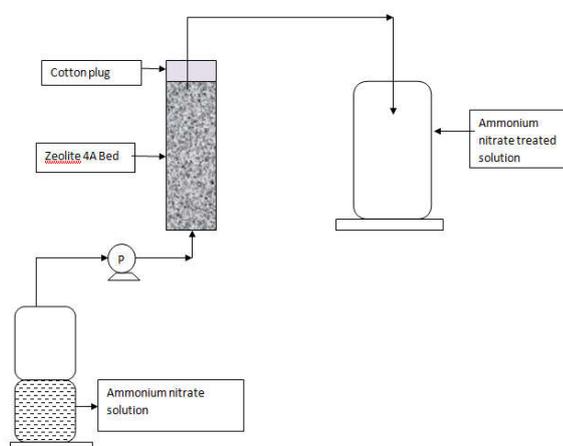


Fig1 Schematic diagram for Experimental set up

Zeolite regeneration

Zeolite column fed with 0.1 M NaCl solution for three hours. The performance of Zeolite 4A was analyzed for repetitive use, and the sorption capacity is measured.

Zeolite modification

25 gm of Zeolite was kept in H_2SO_4 solution of predetermined concentrations for 16 hr. Treated Zeolites filtered and washed thoroughly with distilled water so as to obtain water with neutral pH. Obtained Zeolites were dried in vacuum oven for 6 hr at 80 C and used in sorption studies.

RESULT AND DISCUSSIONS

Selection of material

Zeolite 4A is like a clinoptilolite, which has a high cations exchange capacity [21]. It possesses crystalline structure, high porosity, and three dimensional structures. It contains aluminosilicates from the alkali (mainly Na and K) and alkaline-earth (mainly Ca) metals [22]. Their crystal structure is based on a three dimensional framework of $(\text{SiAl})\text{O}_4$ tetrahedral with all four oxygen shared by adjacent tetrahedral [23]. As the result, they have a channel structure with molecular dimensions of 4A° [24]. Some of the Si^{4+} are substituted by Al^{3+} , the total net negative charge is balanced by framework containing exchangeable cations mainly Na^+ , K^+ , Ca^{2+} or Mg^{2+} [25]. These cations are loosely held within the central cavities and surrounded by water molecules from sides [26]. These water molecules are loosely held in the pores. The Zeolite 4A can be reversibly dehydrated and their cations can be readily exchanged [27]. Unlike most other tectosilicates, Zeolite 4A has large vacant spaces or cages in their structures that allow space for large cations and even for relatively large molecules or cations groups such as water, ammonia, carbonate and nitrate ions [28]. Due to these properties, Zeolite 4A is an ideal material for sorption based separation of nitrogenous material. It would provide superior sorption capacity and would be perfect material for separation of nitrogenous material from aquaculture waste water.

Ammonium nitrate contains both ammonium and nitrate group of nitrogenous material in single molecule. Its toxicity is one of the critical factors, contributed by enormous demand for high quality water by hatcheries and other freshwater phases of aquaculture. In practice there is a potential initial build up of ammonia, nitrite and nitrate during biological treatment of aquaculture water. Concentration levels of nitrate-nitrogen in excess of 0.2 mg/l (ppm) lead to methemoglobinemia and heavy mortalities [29]. Hence we thought it would be necessary to study the sorption of ammonium nitrate as a representative of nitrogenous compounds using zeolites.

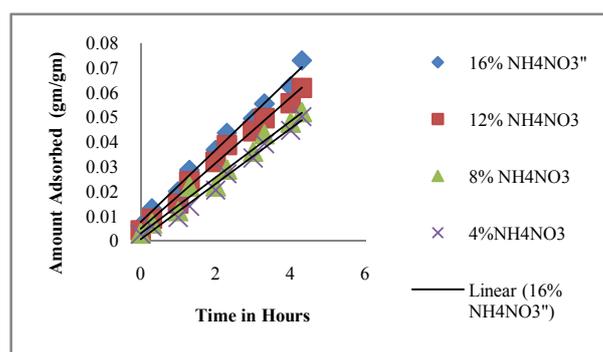


Fig2 Sorption of ammonium nitrate on Zeolite Vs Time

It can be seen from Figure that sorption increased linearly with increase in contact period. This is obvious that with increase in contact time, more and more amount of nitrogenous material. This would lead to interaction between zeolite and increase. Higher concentration lead to higher sorption, due to increase in interaction.

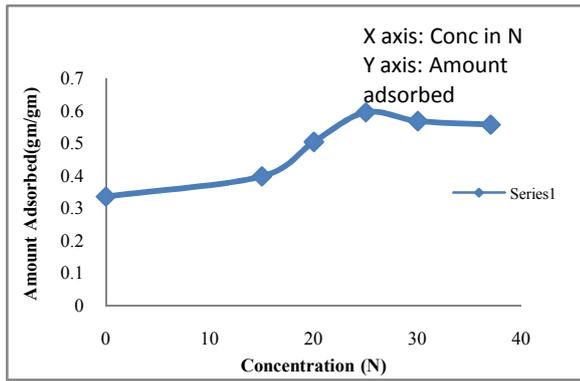


Fig 3 Graph of Amount Adsorbed(gm/gm) Vs Concentration H_2SO_4 (N)

This graph was obtained by taking amount adsorbed v/s concentration of H_2SO_4 . Bell like structure is observed adsorption increases at the 25N and at other concentrations the adsorption is decreased this is due to the change in the structure of Zeolite by acid treatment. The removal efficiency of ammonium ion decreases with an increase in concentration [30]. Adsorption isotherm for NH_4^+ ion exchange data on zeolites 4A fitted better to Langmuir model at the concentration of 15N to 25N. This might have occurred due to the change in the surface adsorption. The change in surface structure was seen due to the acidic treatment crystallization was occurred, due to this the adsorption capacity might have been changed [31].

Regeneration and reuse of zeolites: we have regenerated the Zeolite column by treating it with 0.1 M NaCl by passing through it for 3 hours. Regeneration of zeolites bed can be done by NaCl solution sodium ion gets exchanged with the ammonium ion. [32]. Regeneration of the zeolite is important because we cannot replace the zeolite by using it once so for the commercial purpose we have to regenerate it. The cost of regeneration of the zeolite is less than that of the replacement of the zeolite [33]. we have recycled it 7 times but the adsorption capacity decreases after 3rd time, so we can regenerate it only 3 times zeolites can be regenerated upto certain times [34].

Modification of zeolite: Adsorption of Ammonium nitrate is observed in zeolites if we want to increase more adsorption capacity it should be modified with acid treatment [35]. As we have to adsorb ammonium nitrate we have modified zeolite with strong acid i.e. H_2SO_4 . With modified zeolite we have got 0.2602 more adsorption rate than normal zeolite 4A. Adsorption in terms of gm of NH_4NO_3 /gm of modified zeolite was seen more in 25N (0.5951) than 37N (0.5570), 30N (0.5682), 20N (0.5039) and 15N (0.3914). Change in adsorption is there due to modifications done in the zeolite which increases its adsorption capacity [36]. Crystallization of zeolite structure have occurred due to treatment with the strong acid the colour of zeolite changed from white to yellowish brown. Structural changes occurs when zeolites are treated with different solutions [37].

crystallized and colour changed zeolite. To ensure a high-quality product, diagrams and lettering MUST be either computer-drafted or drawn using India ink. Figure captions appear below the figure, are flush left, and are in lower case letters. When referring to a figure in the body of the text, the abbreviation "Fig." is used. Figures should be numbered in the order they appear in the text.



a) zeolite just added to strong acid



b) Crystallization after treatment



c) Crystallized and colour changed zeolite

Fig 4

Table captions appear centered above the table in upper and lower case letters. When referring to a table in the text, no abbreviation is used and "Table" is capitalized.

CONCLUSION

Zeolite was found to be excellent materials for removal of nitrogenous material from aquaculture waste waters. They have excellent ammonium nitrate removal but the chemisorptions lead to permanent sorption of nitrogenous material on surface of reducing its sorption capacity. Modification of zeolite with concentrated acid lead to large (0.2602 times) increase in sorption capacity for ammonia. This makes the sorption based removal of ammonium components using zeolite an attractive method.

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