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Treatment on synthetic effluent by using limestone and granular activated carbon for removal of ammoniacal nitrogen

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Abstract

Discharging of excessive wastewater nutrient in the form of ammoniacal nitrogen (N-NH_4) into the receiving water causes environmental problems such as eutrophication, corrosion and fouling. Presently N-NH_4 is removed from wastewater in tertiary treatment. The purpose of the present study was to investigate the removal of N-NH_4 from synthetic wastewater using limestone (LS) as low cost media and granular activated carbon (GAC). The physio-chemical treatment adopted in this study is preferred over the other methods because of its simplicity, easy maintenance and quality control. The batch experiments were investigated to evaluate the combine mixture ratio of limestone and activated carbon at pre-examined optimum conditions on the removal of both parameters. Results indicated that 58% of N-NH4 was successfully removed at optimum media ratio of 25:15 (LS: GAC). Results indicated that the smaller particle size of media was more effective than larger particle size because small size particle causes a higher adsorption capacity due to great available surface area. The equilibrium adsorption data is fit and followed Freundlich isotherm (Kf=1875, n=0.42 and R2=0.9827). The experimental data agreed with the pseudo second-order kinetic model (R^2>0.90). This shows that the usage of GAC can be reduced by combining the media with limestone. The study indicated the potential use of LS and GAC for the treatment of N-NH4 and PPO4 from wastewater at lower cost as compared to the use of GAC alone.

Keywords: Adsorption; Ammoniacal nitrogen; Granular Activated Carbon (GAC); Limestone, (LS); Adsorption kinetics

1. Introduction

Water is a precious natural resource, and due to this realization it has become a prevalent source of concern; more attention is placed on its maintenance and purification. Nutrient compounds such as ammoniacal nitrogen (N-NH_4), nitrite and nitrate, often present in different types of waters and wastewaters (domestic, agricultural and industrial), can find their way to lakes, rivers and drinking water reservoirs. Nitrogen is an essential nutrient to all living organisms. It is a fundamental building block of plant and animal proteins. Typically proteins contain about 16% nitrogen. Animal wastes too have substantial amounts of nitrogen incorporated into organic molecules. Organic substances such as urine have nitrogen mostly in the form of urea [$(NH_2)_2CO$], which releases ammonia on biological degradation by microorganisms. Wastewater N-NH_4 is becoming more important in the alleviation of environmental problems including eutrophication, corrosion and fouling (Rozic *et al.,* 2000) [16]. It can promote the growth of algae and decrease the dissolved oxygen required for aquatic life if the concentration of N-NH_4 exceeds 0.3-0.5 mg/L (AWWA, 1990) [3]. N-NH_4 removal is one of the fundamental aims in wastewater treatment. It can be carried out by biological, physical, chemical or a combination of these methods.

Available technologies include adsorption, chemical precipitation, membrane filtration, reverse osmosis, ion exchange, air stripping, breakpoint chlorination and biological nitrification and denitrification (Metcalf and Eddy, 1991) [14]. Conventional wastewater treatment technologies, however, are expensive to build and also have associated operational and maintenance problems. By using air-stripping method, for example, it is possible to decrease ammonia level to 1 mg/L but special requirements of the process such as water temperature of over 15°C and carbonate deposition are the disadvantages of this system. Physico-chemical treatment or ion exchange/adsorption is preferred over other methods because it is stable, easy to maintain and reliable. Aguilar *et al.* (2002) [1] investigated physiochemical removal of N-NH_4 by coagulation-flocculation using activated silica, powdered activated carbon and precipitated calcium carbonate. They found very low ammonia removal of around 3-17% but albuminoid nitrogen (nitrogen in the form of proteins) removal was appreciable (74-89%) and the addition of coagulant aids reduced the sludge volume to 42%. Ion exchange usually employs organic resins, which are very selective but expensive. However, there are cheaper alternative natural and waste materials that can be used to replace high cost materials.

Various researchers have studied the effectiveness of a variety of low cost materials for ammonia removal such as clay and zeolite (Sarioglu, 2005; Demir *et al*., 2002; Celik *et al*., 2001; Rozic *et al*., 2000) [17, 11, 10, 16]; limestone (Aziz *et al.,* 2004a); natural and waste materials such as waste paper, refuse cement and concrete (Ahsan *et al.,* 2001) [2] and sepiolite and activated sepiolite (Ozturk and Bektas, 2004; Balci and Dincel, 2002). In previous studies, Aziz *et al.* (2001; 2004a) [4, 6] found that limestone, as a low cost adsorbent, was very effective in leachate treatment for metal and ammonia removal. The aim of the present study is to investigate the effect and adsorption behavior of limestone (LS), granular activated carbon (GAC) and mixture of both adsorbents as filtering media for ammoniacal nitrogen removal from domestic wastewater. This study also focuses on the establishment of essential parameters for the design of LS or mixture of LS and GAC filter for the post-treatment of wastewater before its release into the environment. Both batch and column experiments were conducted in this research.

2. Materials and Methods

2.1 Wastewater Sampling and Analysis

The oxidation pond receives a mixture of domestic wastewater from hostels, offices, schools and cafeterias. For the purpose of this study, influent wastewater to the pond was collected and analyzed. Ammonium chloride stock solution (500 mg/L N-NH_4) was prepared by dissolving 1.91 g of NH_4Cl in distilled water. Synthetic test samples were prepared to yield ammoniacal nitrogen concentrations of 2.5, 5, 10, 15, 20, 30, 40, 50 and 100 mg/L by diluting the ammonium chloride stock solution with distilled water. pH of wastewater was determined using a Hanna portable pH meter (Hach, Sens ion1, USA). Ammoniacal nitrogen concentration was measured by Nesslerization Method (4500-NH_3) using a HACH DR/2010 Spectrophotometer at 425 nm wavelength of light. All tests were conducted in accordance with the Standard Methods for the Examination of Water and Wastewater (APHA, 1992) [3].

2.2 Properties of Limestone and Granular Activated Carbon

The limestone used in the experiments was obtained as by-product of a marble industry. Its composition was determined by acid digestion followed by metal determination using an atomic absorption spectrophotometer (model Shimadzu AA 660). The composition of limestone contains 95.5% $CaCO_3$, 3% $MgCO_3$ and 1.5% others. Limestone grains were sieved to obtain a uniform size between 2.36 to 4.75 mm, the size suggested in previous studies (Aziz and Smith, 1992, 1996; Aziz *et al*., 2001, 2004a) [8, 7, 6, 4]. The density of limestone, determined conventionally i.e. weight/volume, was 2598 kg/m^3. Commercially available granular activated carbon (GAC) was used. The average particle density of the GAC was 1317 kg/m^3 (particle size, 0.65-2.36 mm) and 1265 kg/m^3 (particle size, 2.36-4.75 mm). General properties of the GAC are shown in Table 1.

Table 1: Properties of Granular Activated Carbon

Specification	Value
Ash content	5% maximum
Grade	K15060
Hardness	90 minimum
Iodine	1000 mg/g minimum
Moisture	5% maximum
Particle size	0.65-2.36 mm
pH	9-10

2.3 Batch Study

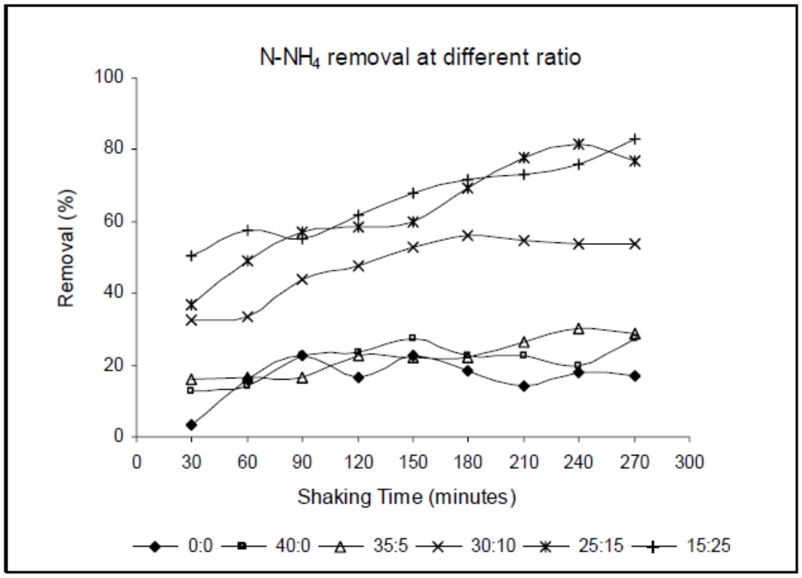
The batch study was conducted to establish the removal pattern of ammoniacal nitrogen using limestone (LS), granular activated carbon (GAC) and the mixture of both materials. The influence of shaking time, and settling time was determined prior to the main experiment. Settling was allowed in order to examine the effect of sedimentation after shaking. The shaking speed was adopted as 350 rpm based on earlier studies (Aziz *et al*., 2004a) [4]. To determine the optimum shaking time, 250 mL conical flasks containing 40 mL of media mixture (LS:GAC) ratios of 40:0, 35:5, 30:10, 25:15 and 15:25 and 120 mL of synthetic wastewater sample (N-NH_4=14.3 mg/L) were agitated on an orbital shaker. They were removed from the shaker one after the other after 30, 60, 90, 120, 150, 180, 210, 240 and 270 minutes. A settling period of 120 minutes was allowed prior to withdrawing the supernatant for N-NH_4 analysis. The optimum settling time was determined in a similar fashion; with the shaking time obtained from optimum shaking experiment and the supernatant withdrawn for N-NH_4 analysis after 30, 60, 90, 120, 150, 180, 210, 240 and 270 minutes of settling. Samples were adjusted to pH 2, 5, 7, 11 and 13 (with 0.1 M HCl or NaOH) to determine the effect of pH on N-NH_4 removal at different mixture ratios of LS:GAC i.e. 0:0, 40:0, 25:15, 20:20 and 0:40. A graph was plotted to relate the percentage removal of N-NH_4 with pH at optimum conditions. Three ranges of particle size of both adsorbents were used i.e. 0.75-1.14 mm, 1.14-2.0 mm and 2.36-4.75 mm and the mixtures were agitated on an orbital shaker at room temperature. The results were examined by plotting a graph of percentage removal against the ranges of particle size. The effect of initial concentration of ammoniacal nitrogen on the removal efficiency was also investigated by using different concentrations of N-NH_4 i.e. 2.5, 5, 10, 15, 15, 20, 30, 40, 50 and 100 mg/L. Data for Freundlich isotherm generation was obtained by varying the amount of adsorbent (5, 15, 20, 25, 30, 35 and 40 mL). The isotherm constants and least squares correlation coefficients (R^2) were studied to determine the best-fit isotherm model for this study. Adsorption kinetics model were studied to compare best fit among pseudo firstorder and pseudo second-order. The experiments were repeated two to three times to obtain a consistent average. All experiments were conducted at room temperature (25±2 ^oC).

3. Results and Discussion

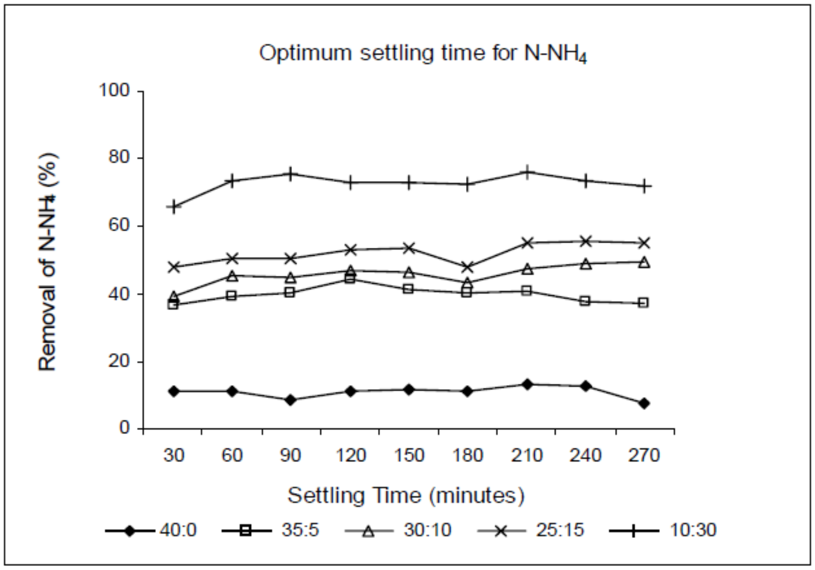
3.1 Preliminary Experiments

3.1.1 Effect of Shaking and Settling Times

Figs. 1 and 2 show the results obtained in the determination of the optimum shaking and settling times at different ratios of LS and GAC (40:0, 35:5, 30:10, 25:15 and 15:25). Without media (ratio, 0:0), N-NH4 removal was less than 23% and the maximum removal (23%) was achieved at shaking time of 90 and 150 minutes. The removal may occur due to air stripping (volatilization of NH_4). Also ratio 40:0 gives maximum removal (28%) at 150 and 270 minutes. The results show that limestone alone (40:0) has a very low adsorption capacity. Fig. 1 indicates that shaking time influences the performance of N-NH_4 removal, after 150 minutes shaking time. It was also observed that for mixture ratios 30:10, 25:15 and 15:25, N-NH_4 removal increased at high shaking time. However, from the results of Fig. 1 for N-NH4, as the shaking time with quite good removal 150 minutes was chosen for further experiments. Fig. 2 showed that the optimum settling time for N-NH_4 was observed to be 120 minutes for mixture ratios 40:0, 35:5, 30:10 and 25:15 at optimum shaking speed (350 rpm) and shaking time of 150 minutes obtained from the previous experiment. The main purpose to evaluate the optimum settling time was to simulate the sedimentation process after shaking.



(Ratio 40:0 means 40 parts of limestone per 0 parts by volume of GAC)
Figure 1: The effect of shaking/contact time versus NH_4 removal (Conditions: LS, 2.36-4.75 mm; GAC, 0.65-2.36 mm; shaking speed, 350 rpm)



(Ratio 35:5 means 35 parts of limestone per 5 parts by volume of GAC)
Figure 2: The effect of settling time versus NH_4 removal (Conditions: LS, 2.36-4.75 mm; GAC, 0.65-2.36 mm; shaking time, 150 minutes; shaking speed)

3.1.2 Effect of pH

The effect of pH on the removal of N-NH_4 is shown is Fig. 3. The agitation of the synthetic wastewater with no media and with limestone (LS) alone at different pH showed similar patterns and levels of N-NH_4 removal. It can therefore be inferred that LS does not have a direct effect on ammonia removal. Rather in these cases the principal removal mechanism was stripping. The role of LS was limited to raising the pH of the solution. The most effective pH for ammonia removal was 13 at which 40% of ammonia was removed due to agitation of the synthetic wastewater.

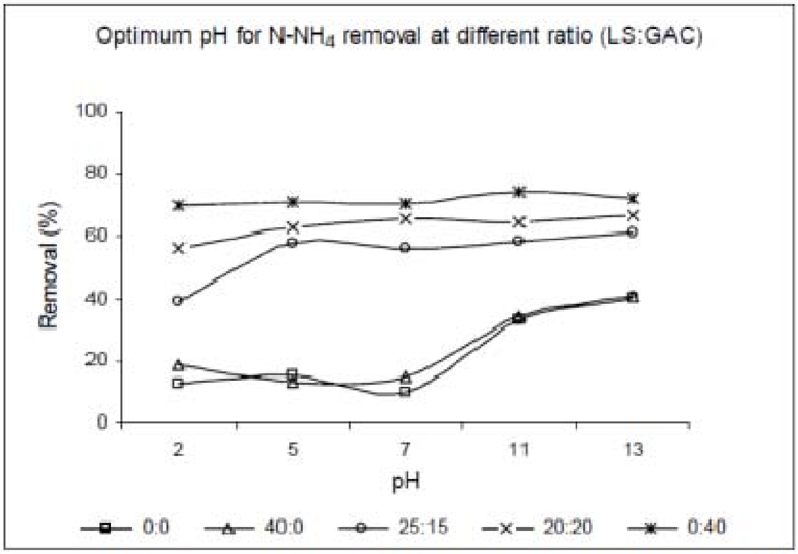


Figure 3: Effect of pH for N-NH_4 removal at different ratio of LS:GAC (Conditions: shaking speed, 350 rpm; shaking time, 150 min; settling time, 120 min)

Test with GAC as the sole medium, on the other hand, showed that GAC can be used as an effective adsorbent for ammonia removal. Over 70% ammonia removal was achieved over the entire range (2–13) of pH tested at ratio 0:40 (GAC). Media mixtures with LS: GAC ratios of 25:15 and 20:20 showed improved N-NH4 removal with alkaline pH (pH 13). At pH 7, comparable ammonia removal of 56.3% and 66% were obtained with LS: GAC ratios of 25:15 and 20:20, respectively. Subsequent studies were conducted at pH 7 to minimize the effect of stripping.

3.1.3 Effect of Particle Size

Fig. 4 shows the effect of particle size on N-NH_4 removal. It can be concluded that smaller particle size of media is more effective than larger particle size because small size particle causes a higher adsorption/ion-exchange capacity due to great available surface area. Particle sizes 0.65-1.14 mm and 1.14-2.0 mm showed a similar degree of removal at all ratios except 25:15, 15:25 and 10:30, and relatively have higher removal than 2.36-4.75 mm particle size. However, the overall mean removal efficiency of 0.65-1.14 mm, 1.14-2.0 mm and 2.36-4.75 mm particle sizes at all ratios of LS and GAC was 56%, 53.5% and 46%, respectively

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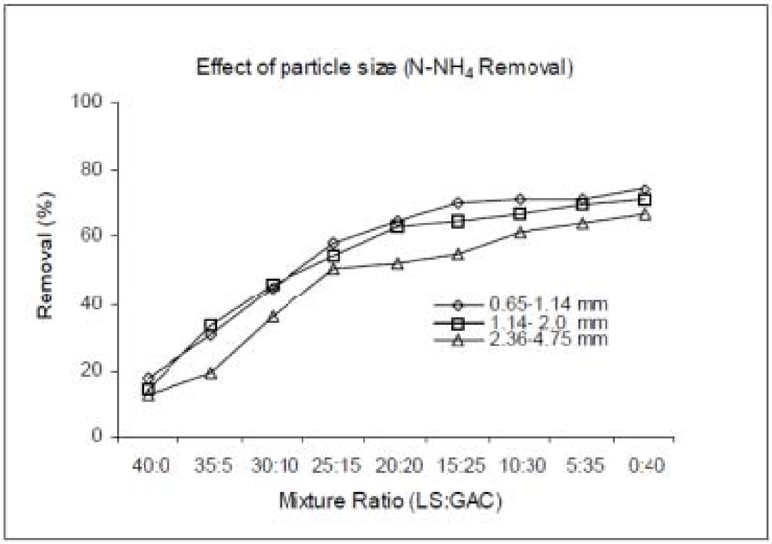


Figure 4: The effect of media size for N-NH_4 removal at different mixture ratios

3.2 Optimum Mixture Ratio (LS: GAC)

Ammoniacal nitrogen removal efficiency of different ratios of limestone and activated carbon was investigated as shown in Fig. 5. It was observed that about 58% removal was achieved by using the mixture ratio of 25:15 (LS:GAC) by volume. However, the use of limestone alone (ratio, 40:0) did not yield a good result, with only about 19% removal. Ahsan *et al*. (2001) [2] reported that limestone has a poor removal efficiency of N-NH_4 about 7-12% but maximum NH_4 + ion removal (75%) was achieved using nitrolite. This is probably due to the fact that the adsorption capacity of limestone is lower than the activated carbon, as indicated by the sharp increase of ammoniacal nitrogen removal when the ratio of activated carbon ratio was increased. It was observed that final pH of wastewater sample after shaking was about 9.5. This is mainly due to activated carbon media, which has the initial pH between 9 and 10 in Table 1. Limestone is also an alkaline media. However the results indicate that the removal of ammoniacal nitrogen was influenced by the media and not solely by the pH. In addition, the increase of pH due to limestone could decease the toxicity of un-ionized ammonia. Therefore, in the present study the ratio (25:15) was adopted as the optimum mixture ratio on economic considerations (cost of GAC, RM 8/kg and cost of LS, RM 0.12/kg). It can be concluded from the experiment that limestone has the potential to be used in conjunction with GAC to reduce the treatment.

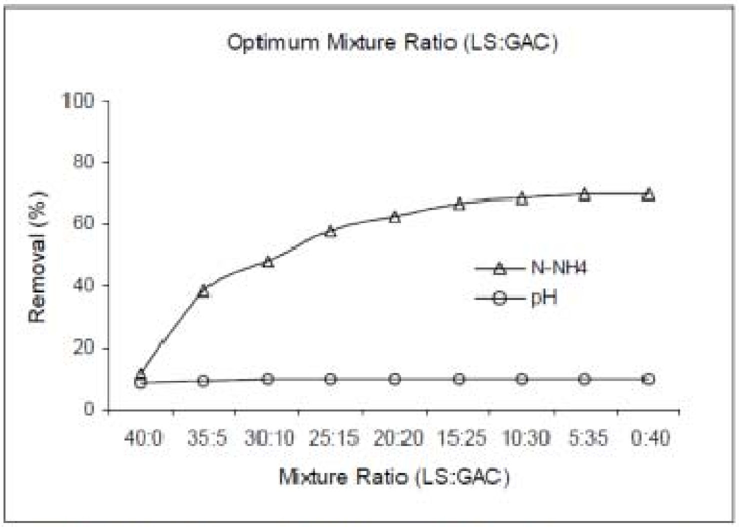


Figure 5: The effect of mixture ratio of limestone and activated carbon versus percentage removal of $N\text{-}NH_4$ at optimum conditions

3.3 Effect of Initial Concentration

Fig. 6 shows the effect of varying the initial concentration (2.5-100 mg $N\text{-}NH_4/L$) on $N\text{-}NH_4$ removal. It was observed that between 39-58% removal of $N\text{-}NH_4$ was achieved at concentration of $N\text{-}NH_4$ between 2.5 to 100 mg/L. The latter exhibited lower removal than the former. At lower initial concentration, the total number of moles of adsorbate to the adsorbent surface area is low and therefore the adsorption is independent of initial concentration of adsorbate. At higher concentration the number of moles of adsorbate can exceed the available sites of adsorption and therefore the adsorption of adsorbate decreases.

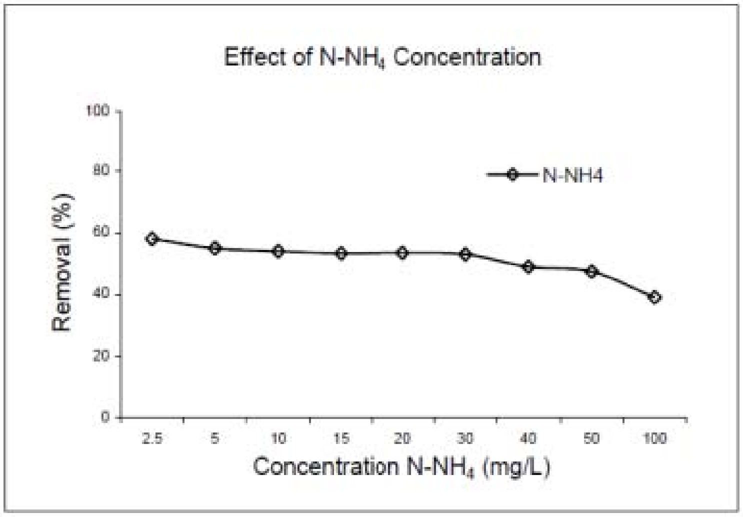


Figure 6: Different initial $N\text{-}NH_4$ concentrations versus percentage removal

3.4 Effect of Adsorbent Dosage

Fig. 7 shows the removal of $N\text{-}NH_4$ as a function of adsorbent dosage for LS: GAC ratio of 25:15. The increase in adsorbent dosage increased the percentage removal of $N\text{-}NH_4$, due to the increase in adsorbent surface area. However the increase in amount of GAC increases $N\text{-}NH_4$ removal while limestone doesn't affect on $N\text{-}NH_4$.The results indicate that 25 mL is the optimum dosage with a 45% removal.

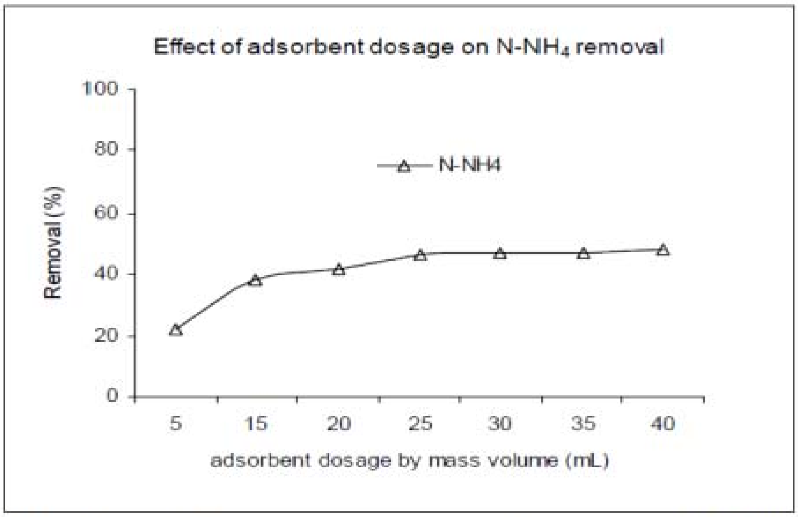


Figure 7: Effect of adsorbent dosage vs. percentage removal of N-NH_4

3.5 Adsorption Isotherm Studies

Adsorption isotherms are essential for the description of how N-NH_4 removal will be affected by limestone/activated carbon and are useful in optimizing the use of these adsorbents. The equilibrium adsorption isotherm is of fundamental importance in adsorption system design (Dogan *et al.,* 2000) [12]. In the present study, the Freundlich adsorption model, which has been successfully applied to many adsorption processes, was used to estimate the adsorption capacity of limestone and activated carbon at ratio of 25:15 (LS:GAC) for N-NH_4 removal from aqueous solution. The Freundlich model can be written as follows (Metcalf and Eddy, 1991) [14].

$x/m = Kf\ Ce^{1/n}$ (Eq. 1)

The above equation can also be written in the logarithmic form;

$\log x/m = \log Kf + (1/n) \log Ce$ (Eq. 2)

Where ‘x’ is the amount of ammonia adsorbed (mg), ‘m’ is the mass of adsorbent (mg) and ‘Ce’ is the equilibrium concentration (mg/L).

Kf and 1/n are Freundlich adsorption isotherm constants, affecting the adsorption capacity and intensity of adsorption. Linear plot of log x/m versus log Ce shows that the adsorption follows Freundlich isotherm. From the Fig.8; Kf=1875, 1/n=2.37 and R^2=0.9827 Hence, the Freundlich isotherm can be written as:

$x/m=1875\ Ce^{2.37}$ (Eq. 3)

The large Kf value shows that that limestone activated carbon mixture has a high adsorption capacity for N-NH_4. The constant 1/n is 2.37 (n=0.42) and it is a function of the strength of adsorbent. According to Treybal (1981), the values of n were between 1 and 10 representing beneficial adsorption. The smaller value of 1/n means that adsorption bond is strong and higher value of 1/n shows the adsorption bond is weak.

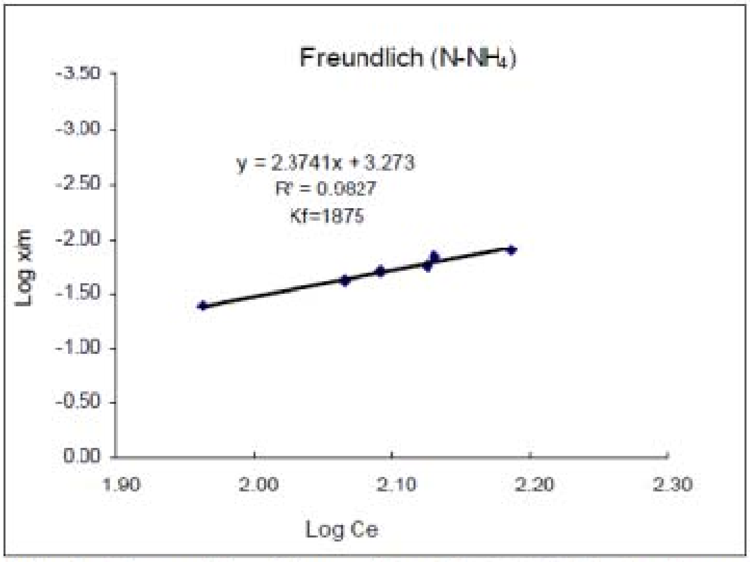


Figure 8: Freundlich Isotherm (Conditions: LS: GAC, 25:15 at optimum shaking speed, shaking time and settling time; pH neutral)

3.6 Adsorption Kinetics

Figs. 9a and 9b show results of the adsorption kinetics studies. To investigate the kinetics of the nutrient removal process, the experimental data were applied to selected adsorption kinetic models, namely pseudo first-order and pseudo second-order models.

The adsorption kinetic data for N-NH_4 removal were analyzed using linear form of the pseudo first-order (Lagergren first-order) rate equation as follows:

$\log(qe-qt) = \log(qe) - k_1 t / 2.303$ (Eq. 5)

where qe and qt are the amount of adsorbate adsorbed (µg/g) at equilibrium and at time t (min), respectively and k_1 is the equilibrium rate constant of pseudo first-order adsorption, (g/µg min). The values of k_1, R^2 and qe were calculated from the intercept and the slope of the plots of log (qe-qt) vs. t and are shown in Table 2. It was found that the correlation coefficient (R^2) values of the pseudo first-order kinetic were rather low.

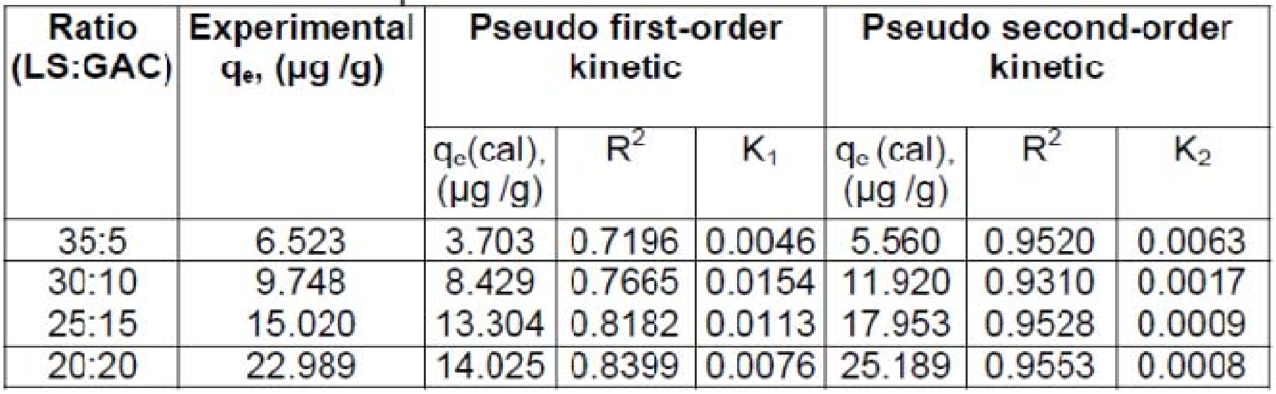
The pseudo second-order kinetic law in the linear form can be expressed as follows (Ho and McKay, 1999):

$(t/qt) = 1/k_2qe^2 + 1/qe\ (t)$ (Eq. 6)

where k_2 is the equilibrium rate constant of pseudo second-order adsorption, (g/µg min).

The calculated values of k_2 and qe from the plot of t/qe vs. t. The correlation coefficients (R^2) for the pseudo second–order kinetic model for N-NH_4 were higher than the pseudo first-order kinetic model. The data showed good compliance with the pseudo secondorder kinetic model ($R^2 > 0.90$). The values of the rate constant (k_2) were found to be in the range of 6.3 x 10^{-3}-8.0 x 10^{-4} g/µg min. The amount of N-NH_4 adsorbed from the sample solutions at equilibrium was in the range of 5.56 - 25.19 µg /g. A relatively high correlation coefficient (R^2) value indicated that the model successfully described the kinetics of ammonia adsorption. The data obtained separately for each of the kinetic models from the slopes of plots showed a good compliance with the pseudo secondorder equation, the high R^2 values (Table 2) obtained from the linear plots showed that the kinetic data fitted the pseudo second-order adsorption kinetic equation.

Table 2: Adsorption kinetics: Pseudo First & Second-orders



Ratio (LS:GAC)	Experimental q_e, (µg /g)	Pseudo first-order kinetic			Pseudo second-order kinetic		
		q_e(cal), (µg /g)	R^2	K_1	q_e (cal), (µg /g)	R^2	K_2
35:5	6.523	3.703	0.7196	0.0046	5.560	0.9520	0.0063
30:10	9.748	8.429	0.7665	0.0154	11.920	0.9310	0.0017
25:15	15.020	13.304	0.8182	0.0113	17.953	0.9528	0.0009
20:20	22.989	14.025	0.8399	0.0076	25.189	0.9553	0.0008

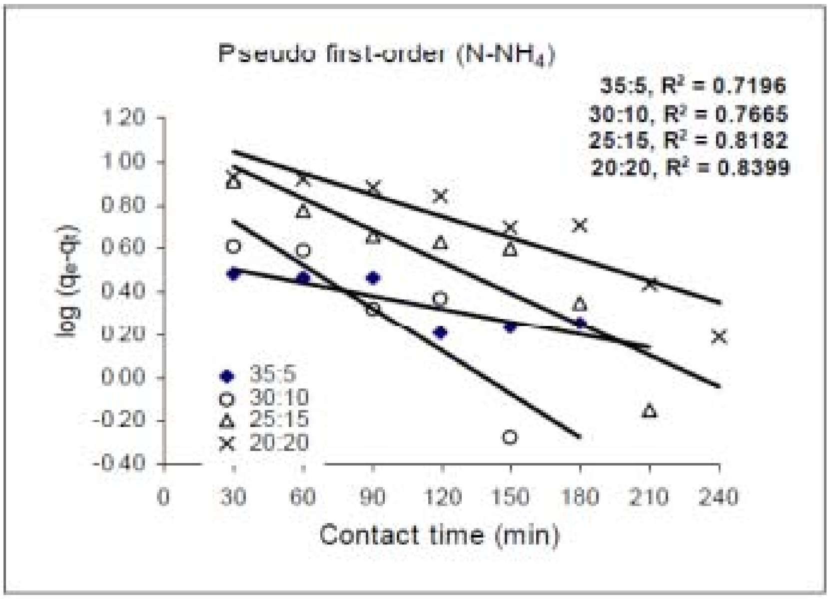


Figure 9 (a): Pseudo first-order for N-NH_4 removal (contact time, min versus log (q_e-q_t) at different ratios of LS:GAC

4. Conclusions

The batch experiments demonstrated that combine mixture ratio of limestone and granular activated carbon (LS: GAC) at 25:15 resulted about 58% of N-NH_4 removal efficiency at optimum conditions. In preliminary experiments optimum shaking and settling times were investigated as 150 minutes and 120 minutes, respectively. Limestone alone was not efficient to remove N-NH_4, with less than 23% removal. The increase in adsorbent dosage increased the percentage removal of N-NH_4 due to increase adsorbent surface area. Higher pH (alkaline) promoted higher removal of NNH_4 with limestone media and without media. The maximum removal with limestone and without media was observed about 40% at pH 13. The smaller particle size was more effective than larger size because small size particle causes a higher adsorption capacity due to great available surface area. The Freundlich isotherm provided the best correlation coefficient of N-NH_4 based onto combine mixture of LS and GAC. The kinetic models of the N-NH_4 adsorption for different ratio of LS and GAC adsorbents were studied. The experimental data was fit to selected kinetic models. The extremely high correlation coefficients (R^2) obtained to follow a pseudo second-order rate equation model which showed that the adsorption was chemisorption. This shows that the usage of GAC can be reduced by combining the media with limestone. The study indicated the potential use of LS and GAC for the treatment of N-NH_4 from wastewater at lower cost as compared to the use of GAC alone.

5. Acknowledgements

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Biography -

Dr. Sachin Madhavrao Kanawade was born in 11 March 1978 at Nashik, Maharashtra, India. His native place is Nimgaonpaga, Tal-Sangamneer, Dist-A'Nagar, Maharashtra, India. He received his Bachelor's Degree in Chemical Engineering from Pravara Rural Education Society's Pravara Rural Engineering College, Pravaranagar (Loni) which is affiliated to Pune University in India in Nov.2001. Then he worked as a Production Officer in different Multinational Chemical Industries in India (2001 to 2008) like M/S Watson

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Then he changes his field. He joined K. K. Wagh College, Nasik, MS, India in 2008 & worked as Lecturer for 2 years. At the same time he received his Master of Engineering in Environmental Engineering from Pravara Rural Education Society's Pravara Rural Engineering College, Loni in Dec.2010.Then he joined Pravara Rural Education Society's Sir Visvesvaraya Institute of Technology, Chincholi, Tal-Sinnar, Dist-Nasik, M. S. India in 2010 & worked as Assistant Professor in Chemical Engineering Department for 5 years. In the same period he completed his PhD Degree in Chemical Engineering in session 2011 – 2014 from Kumar Bhaskar Varma Sanskrit and Ancient Studies University Nalbari, Assam, India.

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His research topic includes & interested in Chemical Engineering, Environmental Engineering, Wastewater Treatment by Adsorption, Advanced Separation Process, Chemical Engineering Design, Mass Transfer, Chemical Process Synthesis, Chemical Engineering Thermodynamics etc.