COMPARISON OF CLINOPTILOLITE WITH GRAVEL IN A RECIRCULATING

COURSE MEDIA BIOFILTER TREATING SIMULATED

SEPTIC TANK EFFLUENT

by

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ABSTRACT

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The main objective of the pilot scale study was to compare total inorganic nitrogen removal and COD removal in a recirulating clinoptilolite biofilter with a course gravel biofilter used to treat simulated septic tank effluent by simultaneous nitrification and denitrification. Both the biofilters were operated with a recycle ratio of 6 and a surface hydraulic loading rate of 7.5 gal/ft^2/day without recycle throughout the duration of the research. Results of the composite sample analysis establish that better nitrification of the influent was achieved in the clinoptilolite biofilter. Average total inorganic nitrogen removal of 80.4 % was achieved in the clinoptilolite biofilter as

compared to 73 % removal in gravel biofilter. Analysis of the results showed 77 % COD removal for the biofilters. The performance of both biofilters was affected by excessive biomass generation in the biofilter leading to biological clogging. However more severe clogging occurred in the clinoptilolite biofilter due to disintegration and breakage of clinoptilolite media.

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CHAPTER 1

INTRODUCTION

Based on a survey in 1999 approximately 23 percent of the 115 million U.S. homes use onsite systems for wastewater treatment and disposal. (See Figure 1.1) (USEPA 2002). The vast majority of single family homes use a septic tank, which is a large underground water tight, buried container where settling and anaerobic digestion lead to TSS and BOD removal (Minnesota Pollution Control Agency, 2001). Treatment in septic tanks can remove about 40 – 60 % of the BOD and TSS and 10-20 % of the nitrogen (Community Environmental Services Inc., 2001). Septic tanks are not efficient in nitrogen removal and septic tank effluent averages about 40 mg/L of nitrogen, of which 75 % is ammonium nitrogen and 25 % is organic nitrogen (Canter et al., 1985).
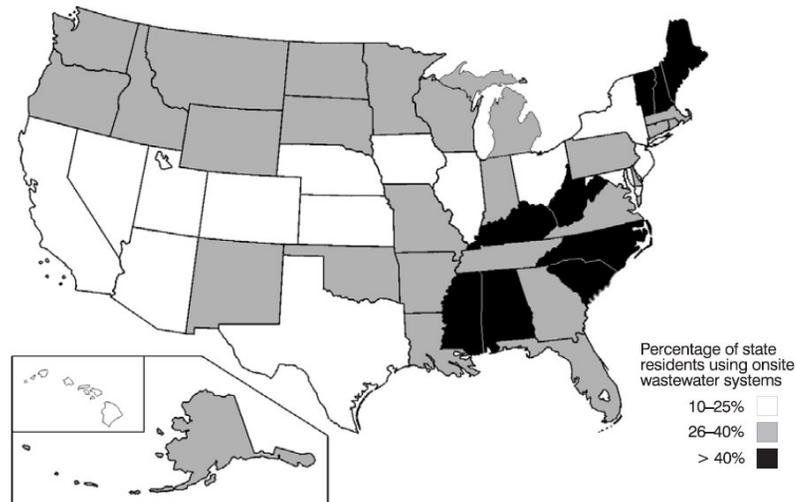
Figure 1.1 Onsite system distributions in United States (Source: US EPA, 2002)

The most common disposal system used in conjunction with a septic tank is a leachfield. In areas having a high density of septic tanks/leachfield systems, high amounts of nutrients are released to local ground waters. Nitrogen contamination from septic tank/leachfield systems can lead to health hazards and possibly contributes to the lake eutrophication. Researchers in Florida studied groundwater for nutrient levels downstream of three onsite wastewater treatment systems and found that nitrogen and phosphorus concentrations were elevated to 2 and 7 times respectively, above background levels (Corbett et al., 2002). The leachfield system converts the ammonia and organic nitrogen to nitrate by nitrification, but total nitrogen removal is relatively low. When nitrogen removal is necessary, there are several septic tank effluent treatment systems available which vary in capital, operation and maintenance cost and degree of nitrogen removal. Recirculating biofilters appear to be the most economical and lowest maintenance systems for removing nitrogen from a septic tank effluent. The term biofilter in this report refers to a filter treating wastewater as opposed to biofilter used for gaseous pollutant treatment. The initial goal of this research was to investigate ways to optimize nitrogen removal in a recirculating biofilter using gravel and clinoptilolite.

1.1 Research Objectives and Scope

Recirculating biofilters using sand, gravel and other media are used to treat septic tank effluent. Recirulating course media biofilters using gravel have the advantage of larger pore size, which helps in oxygen transfer and has less potential for clogging compared to recirculating sand biofilters. In this research clinoptilolite was

selected as a course media for the upper portion of one of the laboratory recirculating biofilters because of its affinity for ammonia adsorption and potential to achieve better nitrogen removal. The main objective of the research was to compare the ammonia and COD removal efficiencies of a recirculating clinoptilolite biofilter to a gravel biofilter. The pilot scale filters were operated and maintained over a period of six months with composite sample collection for performance analysis.

1.2 Overview of the Report

A review of the literature related to nitrification, denitrification, selective ion exchange of ammonia and septic tank effluent systems is provided in Chapter 2. Chapter 3 describes the materials used, methodology, details of the laboratory setup and testing procedure related to and used in the research. Chapter 4 includes experimental results, observations and discussion on scale-up design recommendations. Chapter 5 contains conclusions and recommendation for future research.

CHAPTER 2

BACKGROUND AND LITERATURE REVIEW

2.1 Introduction

A literature review related to nitrification, denitrification, and effects of ammonia on the environment, clinoptilolite, properties of clinoptilolite, alternative and currently used onsite wastewater treatment systems and septic tank wastewater characteristics was conducted and is discussed in this chapter.

Various forms of nitrogen are present in the environment. Figure 2.1 is a general schematic representation of the nitrogen cycle of the movement and transformation of nitrogen compounds through the biosphere. Nitrogen gas which can be assimilated by higher plants at different oxidation states combines with hydrogen or oxygen to form
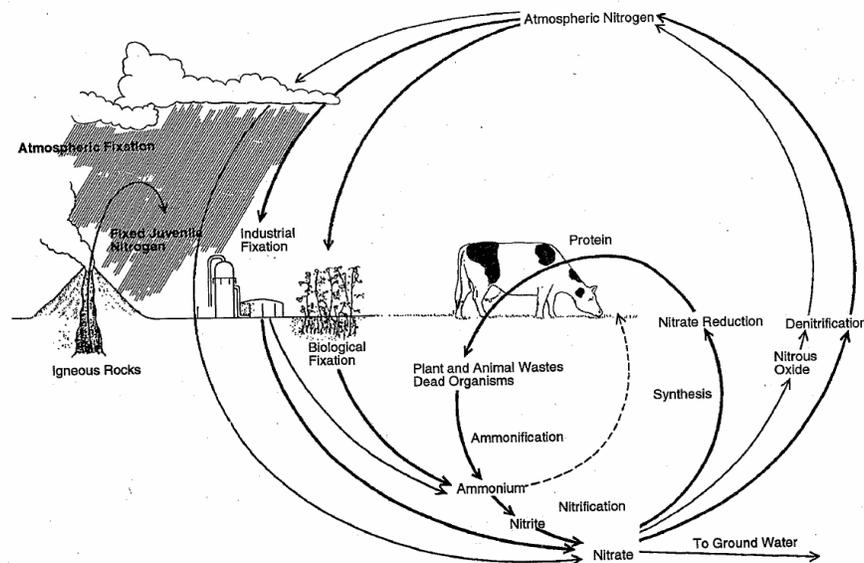
Figure 2.1 The nitrogen cycle (Source: U. S. EPA, 1993)

a variety of compounds such as ammonia, N_2O and NO. Mechanisms responsible for removing nitrogen from water and wastewater include nitrification/denitrification, ammonia volatilization and immobilization by microbial uptake. In wastewater treatment nitrification followed by denitrification is usually the primary process responsible for nitrogen removal. Nitrification and denitrification are discussed in sections 2.2 and 2.3, respectively.

<div align="center">2.2 Nitrification</div>

Nitrification is a two step process in which ammonium (NH_4^+) and ammonia (NH_3) are oxidized to nitrite (NO_2^-), and nitrite is oxidized to nitrate (NO_3^-). It involves two distinctive groups of autotrophic bacteria. Oxidation of ammonia to nitrite is achieved by ammonia oxidizing bacteria including *Nitrosomonas, Nitrosococcus, Nitrosospira, Nitrosolobus and Nitrosorobrio*. Oxidation of nitrite to nitrate is achieved by nitrite oxidizing bacteria such as *Nitrobacter, Nitrococcus, Nitrospira, Nitrospina and Nitroeystis* (Tchobanoglous et al., 2003). The limiting step in the nitrification process is the growth of nitrosomonas (Knoff, 1999). Nitrification can be typically described by the following reaction

$$NH_4^+ + O_2 \xrightarrow[\text{bacteria}]{\textit{Nitrosomonas}} NO_2^- + O_2 \xrightarrow[\text{bacteria}]{\textit{Nitrobacter}} NO_3^-$$
$$\text{ammonium} \qquad\qquad \text{nitrite} \qquad\qquad \text{nitrate}$$
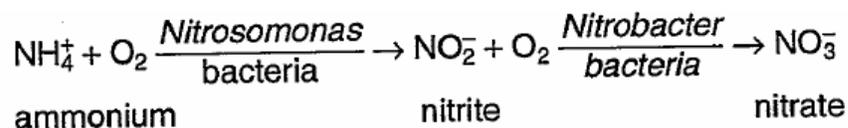
<div align="right">(Source: U. S. EPA 1993)</div>

Nitrification occurs in only aerobic environments and thus dissolved oxygen as the electron acceptor is a main limiting factor in aqueous environments. Other factors affecting nitrification are pH and temperature. It can be concluded from previous

<div align="center">5</div>

research that DO level of above 2 mg/l, pH in the range of 7.0-8.2 and temperature in the range of 30-35° C are optimum for nitrification (Knoff, 1999).

2.3 Denitrification

Denitrification, which is the biological of reduction of nitrate to nitrite and nitrogen gas, is an integral part of nitrogen removal in wastewater treatment. Denitrification is primarily performed by heterotrophic bacteria such as *Achromobacter, Acinebactor, Agrobacterium, Alcaligene, Arthrobacter, Bacillus Chromobacterium, corynebacterium, Flavobacterium, Paracocus, Psedomonas, Spirillum, Vibrio*, although some autotrophic bacteria such as *Nitrosomonas europaea and Planctomycetales* are also capable of denitrification (Tchobanoglous et al., 2003). Denitrification can be shown by the following equation:

$$NO_3^- + \text{organic carbon} \rightarrow NO_2^- + \text{organic carbon} \rightarrow N_2 + \text{carbon dioxide} + \text{water}$$

$$\text{nitrate} \qquad\qquad \text{nitrite} \qquad\qquad \text{nitrogen gas}$$
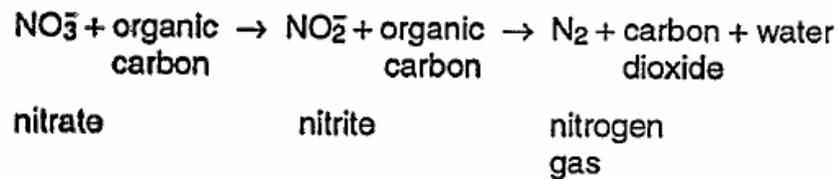
(Source: U. S. EPA 1993)

Denitrifying bacteria grow in anoxic environments, free from oxygen, with organic carbon as the carbon source and electron donor. Even small amounts of dissolved oxygen (0.2 mg/l) can inhibit the denitrifying activity of the bacteria (Tchobanoglous et al., 2003). Denitrification is less sensitive to pH changes, but even small temperature changes have a significant effect on the rate of denitrification (Katers, 1996).

2.4 Effects of Unionized Ammonia and Nitrate on Environment and Health

Groundwater is the main source of water for drinking and domestic use in many places. Nitrate contamination of ground water can cause serious effects on human health and the environment. Most of the nitrogen in septic tank wastewater is ammonia in the form of ammonium ion and unionized ammonia. Unionized ammonia is toxic at concentrations as low as 0.5 mg-N/l in aquatic environments and can be lethal to sensitive species. Toxic effects include impairement of physiological factors such as growth rate, oxygen consumption and disease resistance in most species of fish (Chan, 2003; Aiyuk et al., 2004). Ammonia is converted to nitrate by the nitrification process. There are serious health effects associated with high levels of nitrate in drinking water such as Methaemoglobinaemia (World Health Organization, 2003). Nitrate and ammonia nitrogen can have harmful effects on the environment, the most common being eutrophication. Natural eutrophication is a phenomenon of aging of lakes through which they become more productive. Anthropogenic eutrophication is caused by addition of high levels of nutrients such as nitrogen and phosphorus (World Health Organization, 1999). Eutrophication is recognized as a pollution problem which "leads to algal mats and plentiful macrophyte growth, hampered light penetration, undesirable species and low species diversity" (Aiyuk et al., 2004). "A survey showed that in the Asia Pacific Region, 54 per cent of lakes are eutrophic; the proportions for Europe, Africa, North America and South America are 53 per cent, 28 per cent, 48 per cent and 41 per cent respectively" (World Health Organization, 1999).

2.5 Clinoptilolite

Clinoptilolite is both the most abundant and most commonly used (95 %) natural zeolite in the United States (Knoff, 1999). Clinoptilolite has a large pore space, and has many applications such as gas absorption, odor control and water filtration for municipal and residential drinking water and aquariums. "Notable occurrences include the widespread tuffaceous volcanic rocks of Arizona; the type locality of Hoodoo Mountains and the Yucca Mountains of Nevada; Altoona, Washington; Agate Beach and Madres, Oregon and several sites in California, USA. Also found at Austria; Bulgaria; British Columbia, Canada; Ortenberg Quarry, Germany; Alpe di Siusi, Italy; Kuruma Pass, Japan; McQueens Valley and Moeraki, New Zealand and Chinchwad, India" (Amethyst Galleries Inc., 1999). "The largest producers of clinoptilolite in the United States are St. Cloud Mining (Winston, NM), Zeotech (Tilden,TX) and Teagure Mineral Products (Adrain, OR)." (Knoff, 1999)

2.5.1 Properties of Clinoptilolite

The physical structure of clinoptilolite consists of three dimensional arrangements of SiO^{4-} and Al^{-2} tetrahedra consisting of a network of interconnected tunnels and cages as shown in Figure 2.2. The tetravalent silica ions are replaced by trivalent aluminium ions and the resulting negative charge balanced by cations. The greater the aluminium ion replacement the greater is the capacity for cation absorption. Thus the Si/Al ratio, which varies from 2.7 to 5.2 (for clinoptilolite), and cation content determine the properties of most zeolites.

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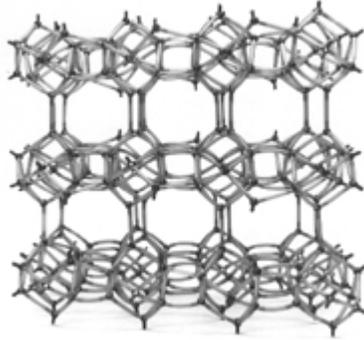
Figure 2.2 Structure of clinoptilolite

(Source: http://mineral.galleries.com/minerals/silicate/clinopti/clinopti.htm)

The free volume of clinoptilolite is closely related to the volume of the cations. This blocking effect can be reduced by acid treatment, which leaches the aluminium ion and increases the adsorption capacity (Mumpton et al., 1981; Rhodes, 1997).

2.5.2 Ion Selectivity of Clinoptilolite

The ability of clinoptilolite to selectively adsorb cations is called as "molecular sieving." "The order of preference for various cation in decreasing order is $Cs^+ > Rb^+ > K^+ > NH_4^+ > Ba^{+2} > Sr^{+2} > Na^+ > Ca^{+2} > Fe^{+3} > Al^{+3} > Mg^{+2} > Li^+$ (Ames), $Pb > NH_4^+ > Cu$, $Cd > Zn$, $Co > Ni > Hg$ (Blanchard) and $K^+ > NH_4^+ > H^+ > Na^+ > Sr^{+2} > Ca^{+2} > Mg^{+2} > Li^+$ (Goto and Ninaki)" (Knoff, 1999). Clinopotilolite has a cation exchange capacity of at least 1.25meq/g and has been exploited in many ways for use as an ion exchange material for water purification. Ion exchange capacity of 1.55 meq/g was reported in a study in mexico on the zeolite-TAM2 (Leyva-Ramos et al., 2004). Research carried out on Chilean zeolite composed of clinoptilolite and modernite showed an ion exchange capacity of 1.02 meq NH_4^+ g^{-1} (Englert et al., 2005). An ion exchange capacity of 8.149 to 15.169 mg N/g has been reported by Bernal and Lopez

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(1993). Clinoptilolite has a high affinity for NH_4^+ over other cations and is used as an ion exchange resin for ammonia removal (Knoff, 1999). The ion selectivity of clinoptilolite depends upon the capacity of the hydrated cation to move in the pores and channels, which depends on the nature of cages, a function of the Si/Al ratio in clinoptilolite.

2.5.3 Use of Clinoptilolite for Wastewater Treatment

Clinoptilolite is widely used for various kinds of waste treatment applications. Jorgensen and Weatherley (2003) studied the removal of ammonia by ion exchange in a simulated wastewater. They concluded that presence of organic compounds enhances the uptake of ammonium ion and also stated the various advantages of using clinoptilolite, including the ability to handle shock loading. In the study they compared steady state equilibrium results for ammonium ion exchange on clinoptilolite with two synthetic ion-exchange resins: Dowex 50w-x8 and Purolite MN500. Enhancement in ammonia exchange in presence of organics such as whey protein and lipase affirmed that clinoptilolite could be used for treating wastewater, which has readily available organic matter (Jorgenson et al., 2003).

Klieve and Semmens (1980) studied ion exchange qualities of four different zeolites: clinoptilolite, erionite, mordenite and phillipsite. They concluded that phillipsite has the highest affinity; clinoptilolite and mordenite, with approximately the same affinity, were the second highest and erionite has the least affinity. Phillipsite is not feasible to use in wastewater treatment naturally because it is friable and would need a binder, making it very expensive. Thus, clinoptilolite is the only natural zeolite

which is not very expensive to use and has high affinity for ammonia. There are many pretreatment methods and chemicals for altering the exchange characteristics of zeolites. Inglezakis et al., (2000) described how pretreatment can remove certain ions from the structure of zeolite and replace them with an increased number of a single ion by washing the material in an ion exchange packed bed with solution with salts, acid or alkali under specific operating conditions to form the homoionic form of zeolite, which has a increased overall exchange capacity. Researchers experimented on four homoionic forms of clinoptilolite: potassic zeolite (KZeo), magnesic zeolite (Mg-Zeo), sodic zeolite (Na-Zeo) and calcic zeolite (Ca-Zeo) and reported the ammonia exchange capacity of modified clinoptilolite in diminished order as : Na-Zeo > Ca-Zeo > K-Zeo > Mg-Zeo (Milan et al., 1996). Klieve and Semmens (1980) investigated and reported that heat treatment at 600 ^{o}C for 1 hour improved the ammonia selectivity of zeolite significantly.

Research has been done on use of clinoptilolite for industrial, municipal, domestic wastewater purification and some researchers have also investigated treatment of municipal landfill leachate. Use of NH_4-form zeolite in the flocculation/zeolite adsorption process for printing ink removal from simulated wastewater showed 95 percent organic removal (Metes et al., 2004). To accelerate the nitrification in an activated sludge system, a comparison batch test study of activated sludge with and without clinoptilolite was conducted by researchers by varying the C/N ratio from 3.25 to 7.5 and hydraulic detention time of 3 hours (Park et al., 2002). In this study, enhanced nitrification was observed in activated sludge with clinoptilolite as the C/N

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ratio was increased (Park et al., 2002). One study investigated using columns of chabazite for ammonia adsorption (Lahav et al., 1998). A single reactor was operated in two sequential modes: ion exchange and regeneration. In the regeneration mode the same column is operated as fluidized bed for the bioregeneration of the saturated zeolite, which acts as a carrier for a nitrifying biofilm. A nitrifying population of 9-10 g VSS/L was maintained on the zeolite (Lahav et al., 1998).

Pilot scale studies in a 250 mm diameter batch column were carried out with an Australian natural zeolite by researchers using a wastewater feed with a suspended solid value of 20 mg/l in a 250 mm diameter batch column. The adsorption experiments were successful in reducing the ammonium ion concentration from influents ranging from 25 to 50 mg/l to 1 mg/l of NH_4-N/L in the effluent. The zeolite had an adsorption capacity of about 4.5 mg NH_4-N/g (Booker et al., 1996). Other research was conducted on the same Australian clinoptilolite in a fixed bed ion exchanger system operated in downflow mode. Chemical regeneration using 0.6 NaCl at pH 10 with flow-rates of 1 and 2 BV/h resulted in high ammonia removal efficiency (Cooney et al., 1999). A study conducted on ammonia removal from pretreated domestic sewage was done using a downflow packed column of clinoptilolite with media ranging in size from of 1-2.5 mm. A removal efficiency of 95 percent was observed and the ion exchange capacity of the clinoptilolite was found to be 14 g Ammonium-N/kg zeolite. The clinoptilolite was regenerated using 0.21 g NaCl l^{-1}. Researchers investigated the removal efficiency for various flow ranging from 1-6 min HRT and concluded that efficiency decreased with increase in flow (Aiyuk et al., 2004). Research was conducted at the University of

Texas at Arlington using clinoptilolite for ammonia removal from a simulated dairy lagoon wastewater (Rhodes, 1997). Reactors filled with clinoptilolite were used for both COD and total nitrogen removal. The clinoptilolite was regenerated using nitrifying bacteria, in naturally aerated beds. Ammonia removal efficiency was 50 % (Rhodes, 1997).

<div align="center">2.6 Septic Tank Systems</div>

A conventional septic system consists of two parts: a septic tank and a leachfield, also referred to as a drainfield or soil absorption system (see Figure 2.3). Raw sewage is piped from a home to a buried septic tank which is a large, watertight sedimentation tank usually made of concrete or fiberglass designed with a 1000–2000 gallon liquid capacity, with an ability to retain wastewater for 24 to 36 hours. The septic tank functions as a anaerobic bioreactor that removes seattleable solids and floatable material and partially digests the organic matter (Minnesota Pollution Control Agency, 2001; USEPA 1993, 1999). The inlet and outlet have designated devices to prevent sludge from being released in the effluent. Due to anaerobic activity, gases such as methane, hydrogen sulfide and carbon dioxide are generated; thus proper venting also has to be provided to septic tank systems (Katers, 1996). The effluent from the septic tank typically disposed of in a leachfield, which also provides some additional treatment (Community Environmental Services Inc., 2001).

2.6.1 Soil Absorption Systems

Common soil absorption systems are trenches and beds called leachfields, mounds, seepage pits, leaching chambers and evaporation-infiltration lagoons. Since

simplicity and reliability are desirable in onsite system operation, conventional onsite systems use leachfields following the septic tank (see Figure 2.3). The size and the type of the soil absorption system is based upon size of the house and soil characteristics (Katers, 1996; U.S. EPA, 1999). Soil absorption systems used for disposal of septic tank effluent convert ammonia to nitrate by nitrification. In a case study on three site samples collected from well 23m from leachfields in Little St. George Island, Florida, it was observed that nitrogen and phosphorus concentrations were elevated above the natural levels (1.1 ± 0.1 mg N l^{-1}, 0.20 ± 0.02 mg P l^{-1}) by as much as 2 and 7 times, respectively (Corbett et al., 2002). Alternative septic tanks effluent treatment systems are also used currently with raising concern about contamination of ground waters by soil absorption systems. These systems are described in the following sections.
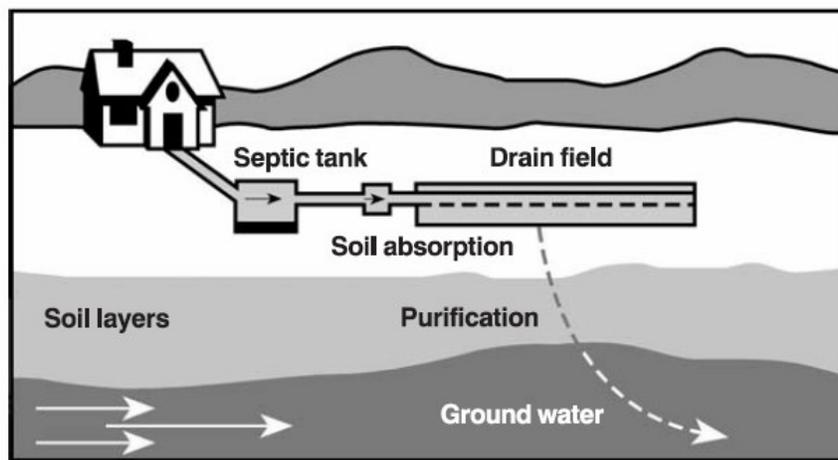
Figure 2.3 Conventional septic tank systems (Source: U. S. EPA 1993)

2.6.2 Peat Filters

In Maine and Ontario province, peat beds have been used for residential sewage treatment since 1970. In a peat system the absorption beds are replaced with 2 to 3 feet of peat, and effluent is treated in the system before being discharged. Peat systems

require less maintenance then the other onsite systems and are better suited for colder climates where there are fewer problems with the decomposition of the peat (Heufelder et al., 1997; Community Environmental Services Inc., 2001). Wastewater from the house flows to a septic tank and then through a distribution box to the peat bed (see Figure 2.4). The water can be either pumped or it flows due to gravity. Peat filter systems require fairly large areas but are reliable and inexpensive. The total nitrogen removal efficiency can vary from 30% to 65%, and BOD removal efficiency ranges from 80% to 99% depending upon the type of peat used (Heufelder et al., 1997). Properly functioning peat filters have effluent discharge characteristics of 1-30 mg/L BOD_5, 5-15 mg/L TSS and 10-30 mg/L of total nitrogen (Community Environmental Services Inc., 2001). A study conducted in West Virginia on a peat filter treating effluent discharged from residential septic tanks and aquaculture wastewater supernatant found ammonia-N removal of 86 % for 150 gpd influent flow-rate (50 % of design flow) and 54 % removal for 300 gpd influent flow-rate (100 % of design flow) (Ebeling et al., 2003). Table 2.1 gives a cost estimation of the capital cost for peat filter system installation and cost of maintenance and energy if a pump is required.

Table 2.1 Estimated capital and maintenance cost of a peat filter
(Source: Community Environmental Services Inc. 2001)

Estimated initial lined peat bed costs, installed, and including septic tank for pretreatment, pump, pump tank, control and alarm	$ 7000
Equipment (pump repair/replacement costs, estimated at $60/year)	$ 5/month
Replacement of peat bed after 7 and 14 years intervals (NPW for Future costs of $4,000 each time bed replaced)	$ 3600

Table 2.1 *Continued*

Septage and sludge pumping once every 3-1/2 years	$ 4.17/month
Energy costs (using 0.051 KWH/day energy use)	$ 0.124/month
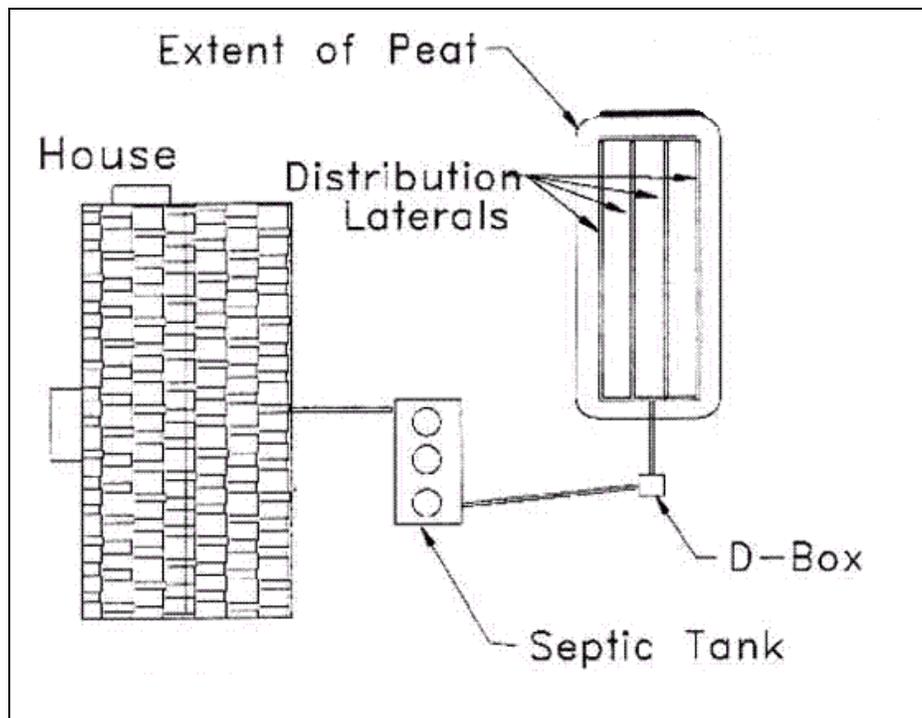
Figure 2.4 Typical Layout of a peat system (Source: Heufelder et al., 1997)

2.6.3 RUCK Systems

RUCK systems use two separate septic tanks for treatment of household greywater (wash water) and blackwater (toilet and kitchen sink waste); with the effluent is disposed into a conventional leaching facility. The blackwater flows through first septic tank and the effluent is nitrfied in a RUCK filter. The RUCK filter is an aerobic filter with in-drains provided for air transfer. The in-drains are overlain by layers of

sand and filter cloth. The wastewater from the RUCK filter flows to a second septic tank where it is denitrified under anaerobic conditions with greywater, which is a rich source of carbon (Heufelder et al., 1997). The denitrified effluent from the second septic tank is disposed to a leachfield (see Figure 2.5). Effluent data collected for a system with a RUCK filter followed by an upflow rock filter show 75% to 90 % nitrification (ammonia removal), 60 to 80 % total nitrogen removal, BOD_5 range of 20-30 mg/L, and TSS 20-30 mg/L, respectively (Community Environmental Services Inc., 2001). Researchers in Rhode Island studied removal performance of buried sand filter/grey water systems similar in design to a RUCK filter in a field laboratory and two full scale systems and found 50-80 % nitrification and close to 100 % denitrification (Lamb et al., 1991). Table 2.2 is a cost estimation of the capital cost for a RUCK filter followed by a upflow rock filter system, along with cost of maintenance and energy requirement if a pump is required.
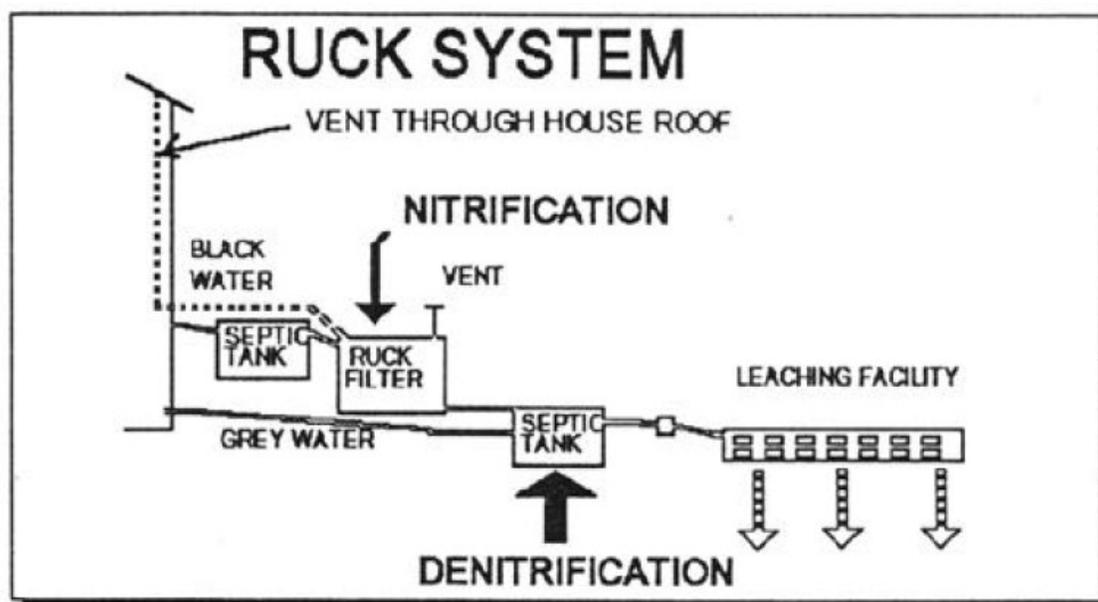
Figure 2.5 Typical layout of a ruck system (Source: Heufelder et al., 1997)

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Table 2.2 Estimated capital and maintenance cost of a ruck filter
(Source: Community Environmental Services Inc., 2001)

Blackwater septic tank (1,000 gal.), and intermittent sand filter unit costs, including pump, pump tank, control and alarm, and valves	$6,500
Upflow rock filter unit, installed	$2,000
Greywater septic tank, installed	$2000
Equipment repair/replacement costs, estimated at $60/year	$ 5/month
Energy costs (using 0.051 KWH/day energy use)	$ 0.124/month
O&M, with a maintenance contract of $720/year (est. four 90-day filter runs, with sand removal/replacement twice annually, and raking performed twice annually) 24 hrs. @ $15/hour * 2.0, including overhead andpump/controls servicing,	$ 60/month
Septage and sludge pumping once every 2 to 5 years, and sand/biosolids mixture transported and disposed of twice annually (@ $100 per haul, incl. landfilling fee)	$20.83/month

2.6.3 Intermittent Sand Filters

Intermittent sand filters are sand beds, either buried or open, which are dosed with wastewater intermittently during the day by gravity flow or pumping devices. Depending upon the media size used in a intermittent sand filter, the effluent quality for an open sand filter ranges from 70-98 % nitrification (NH_3 removal), 3-30 mg/L BOD_5 and a TSS value of 10-40 mg/L in the effluent (Community Environmental Services

Inc., 2001). In a laboratory study on organic carbon removal and nitrification in a two stratified sand filter, with a layer of sand gravel, four feed doses were applied each day. Synthetic wastewater with characteristics similar to wastewater from a dairy farm was used for the study. The ammonia removal results varied with varied hydraulic loading rate, i.e. a loading of 6.67 $L/m^2/d$ resulted in 100 percent removal and a loading of 20 $L/m^2/d$ resulted in 88 percent removal (Rodger et al., 2005). Though the intermittent sand filter has a high ammonia removal efficiency, a very acute problem in the filter is clogging occurring between the pore spaces of the media by accumulation of biomass and deposition of inert and suspended solids; thus, a high maintenance cost associated with operation (Rodger et al., 2005).

2.6.4 Recirculating Sand Filters

Recirculating sand filters are similar to intermittent sand filters with a recirculation or dosing tank used for collecting the sand filter effluent for recirculation. Figure 2.6 below shows a typical recirculating sand filter. The effluent from the septic tank is pumped to the top of the sand filter. The sand filter has conditions suitable for nitrification and thus ammonia is converted to nitrite in the sand filter by biological activity. A portion of the effluent, usually one-fifth, is passed through the leaching facility and the remaining effluent is pumped back to either the septic tank or the recirculation tank. Better denitrification can be achieved if the effluent is recirculated to the septic tank (see Figure 2.6). Typical recycle ratios of 3: 1 to 5:1 are common for these systems (Heufelder et al., 1997; Community Environmental Services Inc., 2001). Properly functioning   recirculating sand   filters   have   effluent   quality   range

values of 70-98 % nitrification, 30-80% total nitrogen removal, effluent BOD_5 5-10 mg/L, TSS 5-10 mg/L (Community Environmental Services Inc., 2001). Table 2.3 gives a cost estimation of the capital cost for recirculating sand filter system installation and cost of maintenance and energy if a pump is required.
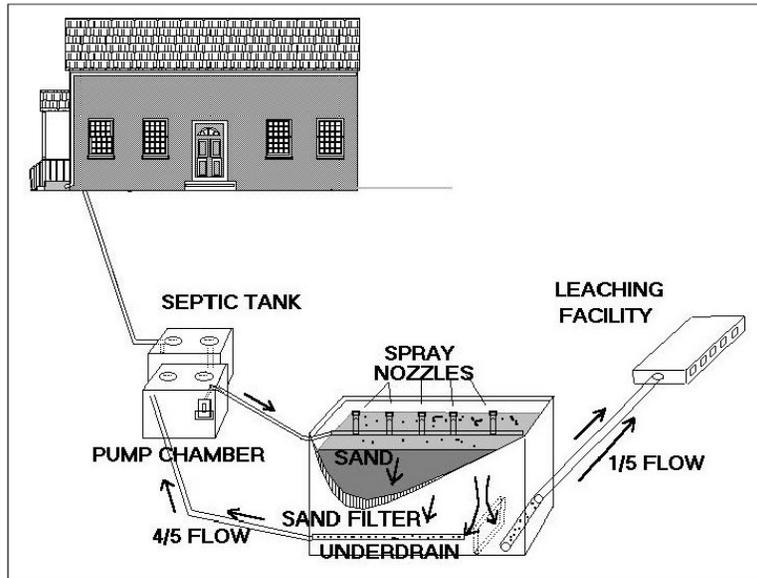
Figure 2.6 Recirculating sand filter (Source: Heufelder et al., 1997)

Table 2.3 Estimated capital and maintenance cost of a recirculating sand filter
(Source: Community Environmental Services Inc., 2001)

Estimated initial Intermittent sand filter unit costs, installed, and including septic tank for pretreatment, pump, pump tank, control and alarm, and valves,	$7,000
Equipment repair/replacement costs, estimated at $60/year (pump repair/replacement)	$ 5/month
Septage and sludge pumping once every 2 to 5 years	$4.17/month
O&M (does not include periodic sand removal/replacement and	

Table 2.3 *Continued*

disposal if/as needed), with a maintenance contract of $240/year (est. two 180-day filter runs with system checks and maintenance performed twice annually) 8 hrs. @ $15/hour * 2.0, including taxes, overhead, profit, and including pump/controls servicing	$20/month
Energy costs (using 0.051 KWH/day energy use)	$ 0.124/month

2.7 Recirculating Course Media Biofilters

The term biofilter is used in air pollution control to describe a unit for treating gaseous pollutants consisting of microbes growing on natural or synthetic media such as compost. However, biofilter in this report refers to a filter used for treatment of wastewater in a biological attached growth system similar to a trickling filter as opposed to biofilter used for gaseous pollutant treatment. In this research nitrogen removal by nitrification and denitrification was to be achieved in a single recirculating biofilter using course media. Recirulating course media biofilters have the advantage of larger pore size, which helps in oxygen transfer. As corroborated by literature review clinoptilolite was selected as a course media in this research for the upper portion of one of the laboratory recirculating biofilters because of its affinity for ammonia adsorption and potential to achieve better nitrogen removal. The initial goal of this research was to investigate ways to optimize nitrogen removal in a recirculating biofilter using gravel and clinoptilolite. The main objective of the research was to compare the ammonia and COD removal efficiencies of a recirculating clinoptilolite biofilter to a gravel biofilter.

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CHAPTER 3

FACILITIES AND METHODOLOGY

3.1 Description of Equipment

Experiments were performed in the Civil and Environmental Engineering Laboratory at University of Texas, Arlington using two pilot scale biofilters. The objective was to compare the performance of a clinoptilolite biofilter with a gravel biofilter operated at the same loading rates throughout the duration of the study using a simulated septic tank effluent feed prepared in the laboratory.

The two pilot scale biofilters used for laboratory experiments are shown in the Figure 3.1. One biofilter was filled with 1 ft of clinoptilolite over 1 ft of gravel and the other biofilter was filled with 2 ft of gravel. The two biofilters are each 4 ft (1.22m) long, 1 ft (0.305 m) wide and 3 ft (0.915 m) deep (see Figure 3.1). The material used for the biofilter floor and walls was plywood supported by 2" x 4" and 2" x 6" wood. Since the biofilters were to remain in constant contact with water, 4 to 5 coats of water proofing material were applied to the floor and walls to prevent water damage. For further protection, a polyethylene liner was provided to protect the water proofing coating from damage (Vemulapalli et al., 2003). A side view of the biofilters is shown in figure 3.2.

Simulated septic tank effluent feed was prepared in two 33 gallon plastic tanks. The inlet and the outlet to the biofilters are perforated pipes two inches above the base

of the reactor. Perforated pipes are used at the inlet and outlet for better distribution of the feed and better effluent collection. The pipes are covered with medium sized gravel to avoid clogging and intrusion of gravel media at the inlet and the outlet. Feed was pumped using half inch flexible tubing from feed tanks to both the biofilters with a dual channel peristaltic pump P-1, which maintains the same flow into both biofilters. The inlet pipe was raised to the height of the reactor to take care of the head buildup in the systems.

Figure 3.1 Two pilot scale biofilters for laboratory experiments

For the simultaneous nitrification and denitrification to occur in both biofilters, aerobic conditions followed by anaerobic conditions are required. The upper layer consists of clinoptilolite in the one biofilter and gravel in the second biofilter.

Nitrification occurs in the upper 1 ft layer of the biofilters by maintaining aerobic condition in this zone (see Figure 3.2). This can be achieved by air transfer in the upper zone and minimal air presence in the lower zone. Air transfer is achieved by recirculation of the effluent using a distribution manifold on the top of the media. The distribution manifold was made of two, half inch diameter plastic pipes with holes drilled at equal intervals along their lengths. A junction box was placed before the outlet to produce the required water level for pumping the effluent back to the reactor using half inch flexible plastic tubing and a dual channel peristaltic recycle pump, P-2 (see Figure 3.2). Both the biofilters are filled with gravel in the lower zone where denitrification occurs in anaerobic conditions. The outlets of the biofilters were kept 1 ft above the base of biofilters to maintain the water level in the biofilters at 1 ft for maintaining anaerobic conditions in the denitrification zone (see Figure 3.2).
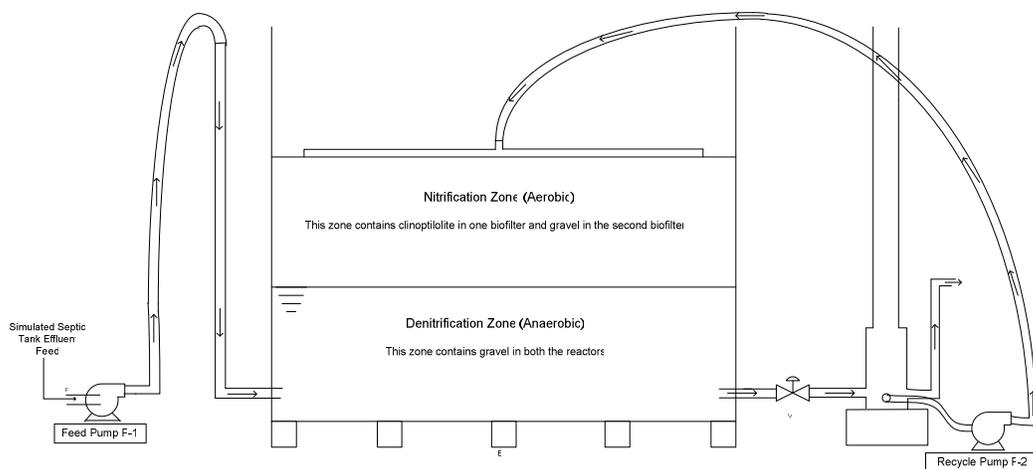
Figure 3.2 Side view of the biofilters

3.2 Description of Materials Used

3.2.1 Clinoptilolite and Gravel

Clinoptilolite obtained from Ash Meadows LLC, a subsidiary of Badger Mining Corporation, was used in the biofilter. It is dry, pale greenish-yellow in color, and has an earth-like odor. Composition of the clinoptilolite used for the study is 95 % clinoptilolite and 5 % Silica, quartz and silicon dioxide. Pea gravels used for the study were a combination of brown, black and white course media pebbles. Clinoptilolite used for study was assumed to be harder than the earlier pilot-scale study conducted at University of Texas at Arlington to overcome the issue of breakage of clinoptilolite due exposure to water.

3.2.2 Simulated Septic Tank Effluent

The simulated septic tank effluent was formulated based on typical values found in the literature (see Table 3.1). The biofilters were operated for a period of six months. The desired characteristics as given in the table 3.1 were maintained but different

Table 3.1 Typical range of constituent of septic tank effluent

Constituent	Range
pH	7 – 7.5
COD	350-450 mg/l
Ammonia as N	30-35 mg/l
Phosphate as P	8-10 mg/l

chemicals were used depending upon availability and cost to achieve the objective and consistency was maintained for result comparison and achieving steady state in the biofilters. A brief description of the chemicals used is given below.

1.) <u>Vinegar</u>

Household vinegar with chemical composition of approximately 5% CH_3COOH and 95 % water was used throughout the course of the lab experiments. Vinegar was purchased from grocery stores as it is easily available and cost effective.

2.) <u>Tryptone and peptone</u>

Tryptone manufactured by Fisher Biotech Inc. and peptone manufactured by Alpha Biosciences Inc. were part of the simulated wastewater. The above chemicals were used in preparing microbiological culture media. These chemicals contain organic nitrogen, which is readily available for bacteria growth and typical of the organic nitrogen found in septic tank effluents. Tryptone is pancreatic digest of casein and is especially rich in tryptophane and peptone. The typical properties of tryptone are pH range (in a 2% solution) of 6.5-7.5, total nitrogen of 10 % and amino nitrogen of 4 %. The typical properties of peptone are pH (in a 1% solution) of 7.0, total nitrogen of 15.5 % and amino nitrogen of 3.1 %. 54.7 mg/l of tryptone or peptone was determined by calculation to be added for feed preparation.

3.) <u>Ammonium Chloride</u>

Ammonium chloride (NH_4Cl, M. W. = 53.49) manufactured by Fisher Scientific Inc. was used as the main source of ammonia to achieve a desired range of 30-35 mg-N/l.

4.) <u>Ammonium Sulfate</u>

Ammonium sulfate (NH_4Cl, M. W. = 53.5) manufactured by AFP was used in the latter stages of the lab experiments for maintaining the desired value of ammonia as N in the range of 30-35 mg-N/l. 166 mg/l of ammonium sulfate was calculated (by the percent of nitrogen present) to be added in tap water for simulated septic tank effluent feed preparation in the laboratory.

5.) <u>Potassium Phosphate Dibasic</u>

Potassium phosphate dibasic (K_2HPO_4, M. W. = 174.18) manufactured by EM Industries Inc. was used as the main source of phosphate as P to achieve a desired range of 8-10 mg-P/l.

6.) <u>Super Phosphate</u>

Super phosphate, a fertilizer manufactured by Lily Miller was used as the source of phosphorous in the later course of the laboratory experiments. The desired amount required to achieve 8-10 mg-P/l of phosphate as P was measured and added to water to achieve the target simulated wastewater septic tank effluent.

7.) <u>Sodium Carbonate</u>

Sodium carbonate (Na_2CO_3, M. W. = 106) or Soda ash or pH balancer of various brands such as Leslie's Soda Ash, All Clear Soda Ash, Aqua chem. pH Add and Hth pH Plus Balancer were used to neutralize the acid (vinegar) and maintain the pH of the simulated feed in the desired range of 7-7.5.

The above described chemicals were used for preparation of the simulated septic tank effluent feed depending upon availability (see Table 3.2).

Table 3.2 Amount of chemicals used per **30 gallon** tap water
for preparation of the simulated feed for one biofilter

Chemical	Used between 3/8 and 6/30	Used between 7/1 and 9/14
Vinegar	710 ml	710 ml
Tryptone	6.21 g	-
Peptone	-	6.21 g
NH_4Cl	15.2 g	-
$(NH_4)_2SO_4$	-	18.9 g
K_2HPO_4	2.99 g	-
Super Phosphate	-	4.54 g
Na_2CO_3	72.2 g	72.2 g

3.3 Description of Operation and Maintenance

Laboratory scale biological systems typically need a startup time of three to four weeks. The biofilters were started on 2/20/05 with seed activated sludge from the City of Fort Worth's Village Creek Wastewater Treatment Plant. To allow the biological growth to develop in the biofilters, they were run on continuous recycle for a period of approximately three weeks. After this initial startup operation and observation of growth in the transparent plastic tubing, the simulated feed flow input was started on 3/8/05. An influent flow of 30 gallons/day was applied to both the biofilters throughout the duration of the laboratory experiments. Influent feed flow with pumping rate of 945 ml/min was applied to both biofilters twice a day for a period of one hour. The recycle pumping rate was also setup at 945 ml/min with the recycle flow being applied once

every two hours, i.e. 12 times a day. Two dual layer peristaltic pumps and a Chrontrol Corporation table top programmable timer were used to set the above operating conditions. In research conducted at the University of Texas at Arlington by Vemulapalli Gopala, better nitrogen removal was achieved with an operating recycle ratio of 6. In this study the same recycle ratio was used with a surface hydraulic loading rate of 7.5 gal/ft^2/day. Biological systems are slow in reaching steady state and true data comparison is done with steady state values. Both biofilters were operated for two months before the data collection was started. Effluents from the biofilters were periodically checked for ammonia-N values and COD values to check for steady state conditions. Consistency in the feed was maintained throughout the experiments for accurate comparison of the results. The feed chemicals were changed after approximately three months of operation due to cost and availability considerations. To ensure true steady state and consistency in operating conditions, the biofilters were operation for a month using the new chemicals before any further data collection.

Nitrification and denitrification produces sludge and growth in the biofilters. Slime layer formation was observed in the plastic tubing used for recycle. The soft plastic tubes were cleaned after every two weeks of operation with chlorine (bleach). Drilled holes in the recycle distribution manifold used for better distribution were to be cleaned to prevent clogging occurring due to excessive growth. Chlorine was used for cleaning tubes and piping. All equipment was thoroughly cleaned with tap water before resuming operation to ensure no residue of bleach in the cleaned equipment.

3.3.1 Sampling Procedure

The influent feed to the biofilters was pumped for a period of one hour. To compare performance, the most representative sample should be collected and analyzed. Samples were collected at 10 minutes, 30 minutes, 50 minutes and 68 minutes from the starting of feed application to the biofilters to see if the concentration changed over time.

Table 3.3 Test analysis results of samples taken at different time intervals

Biofilter	Time of Sample	COD (mg/l)	Ammonia as N (mg/l)
Clinoptilolite	Influent	378	31
Gravel		384	34
Clinoptilolite	10 minutes	-	2.8
Gravel		-	0.56
Clinoptilolite	30 minutes	58.4	0.65
Gravel		60.9	6.16
Clinoptilolite	50 minutes	101	3.9
Gravel		98.6	11.2
Clinoptilolite	68 minutes	96.6	3.98
Gravel		103	12.3

The concentrations do change significantly making composite sampling necessary (see Table 3.3). Equal volume samples were taken at 15 minutes intervals and mixed together to obtain composite samples.

<u>3.4 Sample Analysis and Equipment</u>

The composite samples from the biofilters were tested for COD, ammonia as N and Nitrate as N values. Laboratory tests were done on influent feed samples and composite effluent samples from both biofilters to determine the removal efficiencies. COD removal rates are important to determine the overall efficiency of the system, nitrification efficiency can be known by sample analysis for ammonia as N and denitrification efficiency can be determined by sample analysis of nitrate as N for the biofilters. A brief description of the equipment and test procedure is given below. A battery operated Orion AQUAfast Colorimeter, a preprogrammed colorimetric system manufactured by Thermo Electron Corporation was used for testing of the above three characteristics of the wastewater using the tablet chemistry reagents for analysis (see Figure3.3). Following is a brief description of the procedures for analysis of COD, ammonia-N and nitrate-N.

3.4.1 *COD Analysis*

Low range (0-150 mg/l) COD vials manufactured by Thermo Electron Corporation were used for COD analysis. 2 ml of the sample was taken in the vial. The vials were stirred for mixing of the contents and heated in a preheated digester at 150^0 C for two hours. A blank was prepared using the same procedure with deionized water. The vials were cooled for 45 minutes and COD values were read from the calorimeter.

3.4.2 *Ammonia-N Analysis*

Tablet chemistry reagents AC 2012 were used for ammonia-N analysis. 1 ml of sample and 9 ml of deionized water were taken in sample cuvettes and the reagents

tablets were crushed and mixed in the cuvettes. This instrument displays the analysis value in terms of ammonia (mg/l) (range 0-10 mg/l), which was then converted to ammonia-N by a conversion factor of 0.227.

Figure 3.3 Orion AQ 4000 used for sample analysis

3.4.3 Nitrate-N Analysis

Tablet chemistry reagents AC 2046 were used for nitrate-N analysis. 5 ml of sample and 5 ml of deionized water was taken in sample cuvettes and the reagents tablets were crushed and mixed in the cuvettes. This instrument displays the analysis value in terms of nitrate-N (mg/l).

CHAPTER 4

RESULTS AND DISCUSSION

The data collected from testing of the composite samples is presented in section 4.1 of this chapter. The performance of clinoptilolite and gravel biofilters is compared using the removal efficiency of the biofilters. Section 4.3 describes the mechanisms of oxygen transfer in the biofilter and provides suggestions for scale-up design and operation based on laboratory results and performance.

4.1 Analysis of Results

The influent ammonia-N concentration values ranged from 27 mg/L to 34 mg/L for the clinoptilolite biofilter and from 25 to 36 mg/L for the gravel biofilter with an average value of 32.5 mg/L for both biofilters (see Table 4.1). Nitrate-N was not added to the simulated wastewater but influent values ranged from zero to 2 mg/L in both biofilters. The influent samples for clinoptilolite and gravel biofilters had average COD values of 357 and 351 mg/L, respectively. Variation in the influent concentration of ammonia-N and COD can be accounted for by manual error in chemical measurement, changes in tap water concentrations or sample analysis error.

During the initial weeks of sample collection (three months after start-up), the clinoptilolite biofilter performed better in ammonia-N and COD removal than the gravel

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Table 4.1 Test results for clinoptilolite and gravel biofilter

| Date | Biofilter | Ammonia (as N) | | Nitrate (as N) | | COD | |
		Influent (mg/L)	Effluent (mg/L)	Influent (mg/L)	Effluent (mg/L)	Influent (mg/L)	Effluent (mg/L)
6/15	Clinoptilolite	27.0	2.8	1.53	1.22	340	69.7
	Gravel	24.5	6.8	1.36	1.19	348	97.1
6/17	Clinoptilolite	35.9	2.53	1.70	0.32	408	59.5
	Gravel	35.7	8.95	1.65	0.20	401	97.9
6/19	Clinoptilolite	29.8	3.5	1.62	0.601	-	-
	Gravel	35.7	8.18	1.85	0.31	-	-
6/21	Clinoptilolite	27.4	2.51	1.57	0.10	348	65.0
	Gravel	31.2	7.2	1.51	0.079	342	84.1
6/23	Clinoptilolite	33.5	4.28	2.12	0.27	-	-
	Gravel	31.2	8.98	1.93	0.023	-	-
6/25	Clinoptilolite	32.9	6.4	1.90	0.34	334	73.6
	Gravel	30.4	9.57	1.85	0.508	335	74.7
6/27	Clinoptilolite	33.1	5.6	1.51	N A	348	84.3
	Gravel	32.4	9.43	1.29	N A	336	79.3
8/10	Clinoptilolite	31.8	4.13	0.95	0.350	388	66.0
	Gravel	32	4.95	0.91	0.350	392	56.0
8/12	Clinoptilolite	33.0	8.4	1.45	2.04	-	-
	Gravel	32.4	8.98	1.50	1.100	-	-

Table 4.1 *Continued*

Date	Biofilter	Ammonia (as N)		Nitrate (as N)		COD	
		Influent (mg/L)	Effluent (mg/L)	Influent (mg/L)	Effluent (mg/L)	Influent (mg/L)	Effluent (mg/L)
8/14	Clinoptilolite	32.8	7.16	1.04	Neg	352	105
	Gravel	33	8.63	1.18	Neg	352	77.0
8/16	Clinoptilolite	34.8	8.68	0.79	0.320	-	-
	Gravel	35.2	8.3	0.84	1.100	-	-
8/18	Clinoptilolite	36.0	6.8	0.03	Neg	351	92
	Gravel	35.6	7.3	0.030	1.100	350	74.0
8/20	Clinoptilolite	31.6	8.32	1.389	Neg	-	-
	Gravel	31.4	9.18	1.469	Neg	-	-
8/22	Clinoptilolite	33.8	7.84	Neg	Neg	340	88.0
	Gravel	34.0	10.2	Neg	1.30	332	62.0
8/24	Clinoptilolite	32.0	8.05	0.400	0.200	-	-
	Gravel	32.2	9.3	0.250	Neg	-	-
8/26	Clinoptilolite	32.8	8.95	0.681	0.800	358	95.0
	Gravel	32.2	9.8	0.636	1.05	320	71.0
9/8	Clinoptilolite	34.7	10.2	0.795	0.073	-	-
	Gravel	33.2	10.2	0.840	0.329	-	-

biofilter. Towards the end of sample collection, the performance of clinoptilolite biofilter in inorganic nitrogen and COD removal decreased, probably due to clogging at the interface of the clinoptilolite and gravel layer in the clinoptilolite biofilter (see Table 4.1). Figures 4.1 and 4.2 are graphical representations of the influent and effluent values of ammonia-N and nitrate-N for the clinoptilolite biofilter versus the sampling date. It can be inferred from Figure 4.1 that better ammonia-N removal was observed during the first two weeks of experimental data collection, when the range of ammonia-N removal was between 81 % and 93 %. The subsequent gradual clogging of the biofilter over the 2 months resulted in a decrease in the removal efficiency to a range of 80 to 70 % removal. The lowest value of effluent ammonia-N in the clinoptilolite biofilter was 2.5 mg/L in the beginning of sample collection and the highest value of effluent ammonia-N was 10.2 mg/L during the last 2 weeks of sample collection. The overall average ammonia-N removal efficiency for the 3 month period was 81 %. The nitrate-N effluent values ranged from negligible to 2.04 mg/L, with an average of 0.55 mg/L.

Figures 4.3 and 4.4 are graphical representations of the influent and effluent values of ammonia-N and nitrate-N for the gravel biofilter. The gravel biofilter ammonia-N removal efficiency ranges from 68% to a high of 85 % removal. The lowest value of ammonia-N in the effluent was 4.95 mg/L. The gravel biofilter showed a consistent removal of greater than 70 percent but significantly less than that achieved in the clinoptilolite biofilter. The gravel biofilter also gradually clogged over a 6 month period and the removal percentage decreased. The average rate of ammonia–N removal

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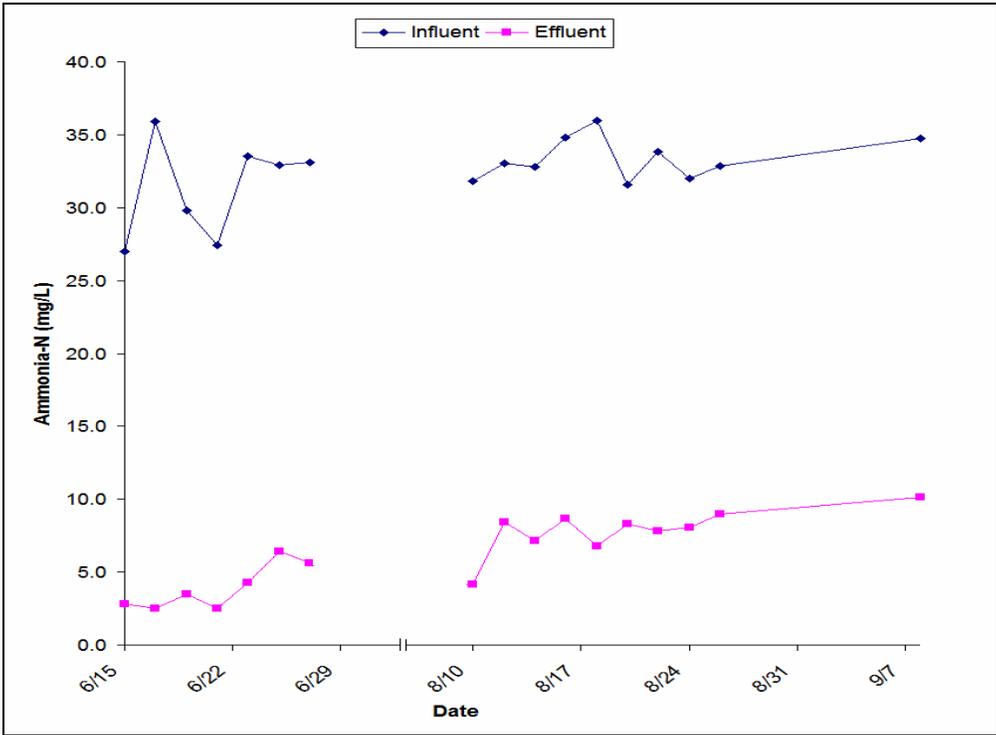
Figure 4.1 Ammonia-N influent and effluent values for clinoptilolite biofilter
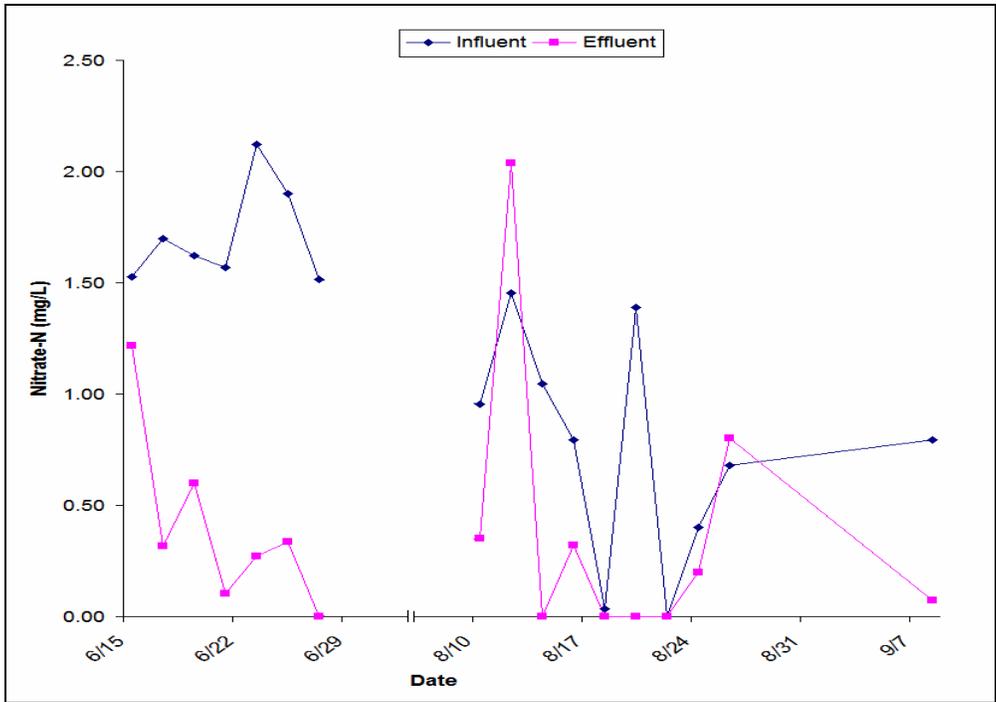
Figure 4.2 Nitrate-N influent and effluent values for clinoptilolite biofilter

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was 73.5 % over the period of sample collection. The effluent values of nitrate-N ranged from zero to 1.3 mg/L with an average value of 0.66 mg-N/L. The gravel biofilter had high values of nitrate-N in the effluent in the last four weeks of testing as compared to the clinoptilolite biofilter effluent during the same period. A possible explanation for higher nitrate-N values could be short circuiting occurring in the denitrification zone of the gravel biofilter due to biological clogging.

The COD values in mg/L for the influent and the effluent for the clinoptilolite and gravel biofilter are represented in Figures 4.5 and 4.6, respectively. Better COD removal was observed in the clinoptilolite biofilter in the initial two weeks of sample collection, but during the latter four weeks of sample collection the gravel biofilter performed better in COD removal.
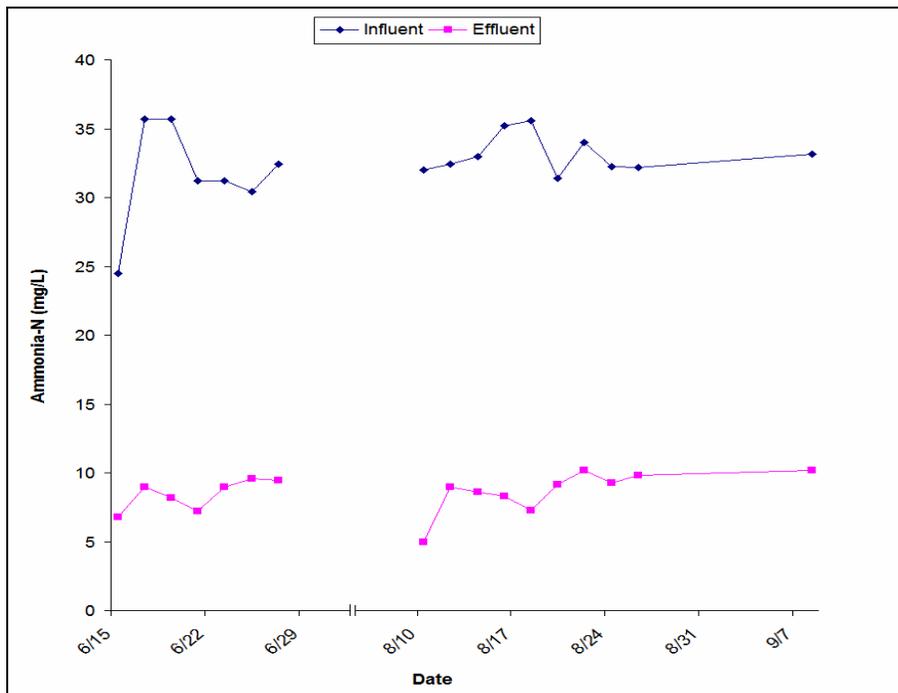

Figure 4.3 Ammonia-N influent and effluent values for gravel biofilter
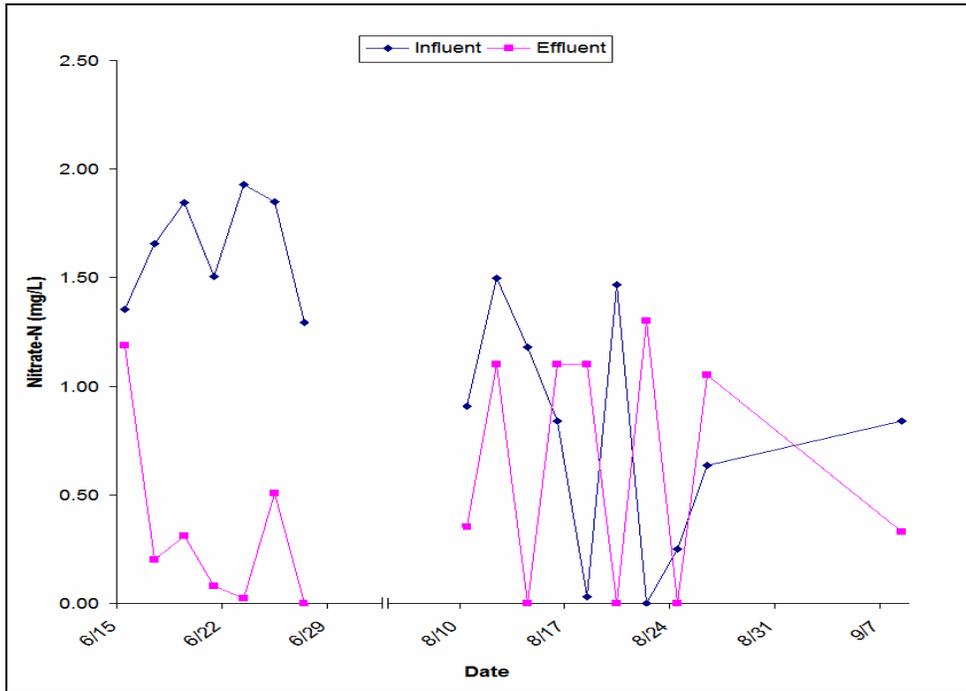
Figure 4.4 Nitrate-N influent and effluent values for gravel biofilter
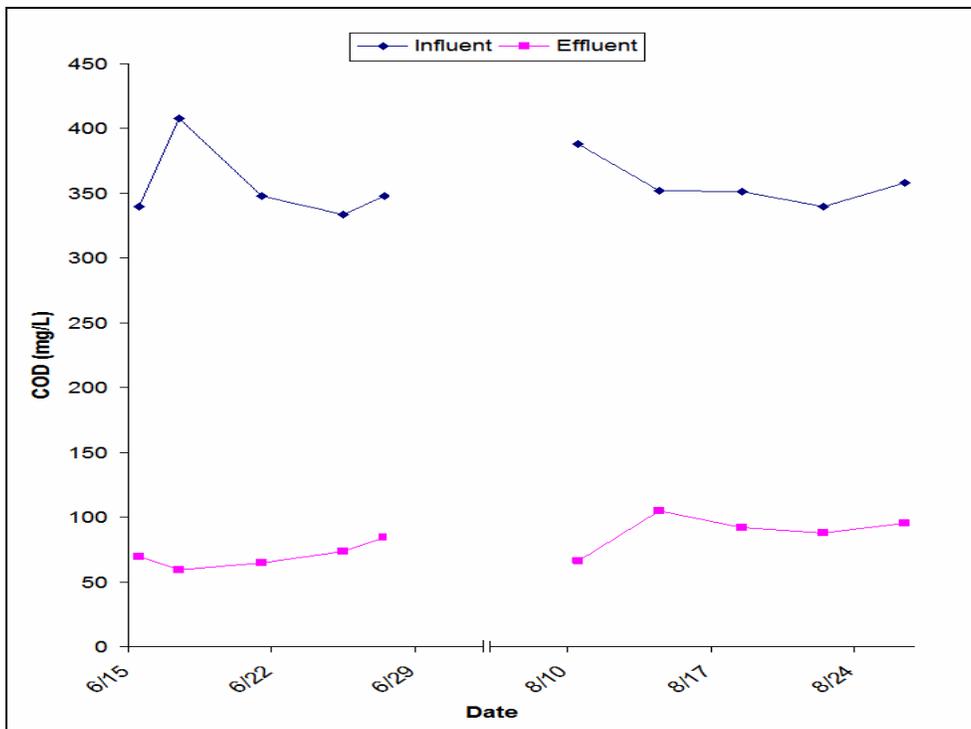
Figure 4.5 COD influent and effluent values for clinoptilolite biofilter
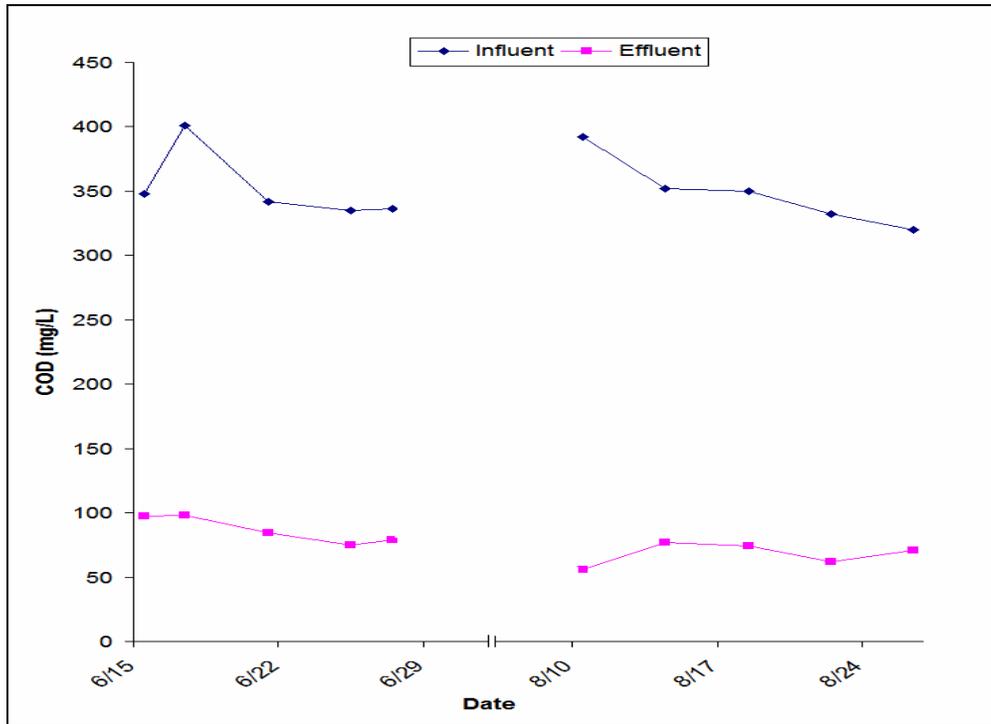
Figure 4.6 COD influent and effluent values for gravel biofilter

The data collected over a period of five weeks of sampling is analyzed by taking an average of the influent and effluent values of both biofilters. The average value of ammonia-N and nitrate-N is added to get the value of total inorganic nitrogen in the influent and effluent. Table 4.2 is a weekly average of total inorganic nitrogen influent and effluent values for both the reactors. Table 4.3 is a weekly average of influent and effluent COD values for both biofilters. Figure 4.7 is a comparison of performance of clinoptilolite and gravel biofilter in terms of removal percentage of COD and total inorganic nitrogen. For comparison the weekly average of total inorganic nitrogen and COD were calculated and the corresponding removal percentage was calculated.

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Table 4.2 Total inorganic nitrogen influent and effluent values for both biofilters

	Clinoptilolite		Gravel	
	Influent (mg/L)	Effluent (mg/L)	Influent (mg/L)	Effluent (mg/L)
Week 1	32.5	3.66	33.6	8.54
Week 2	33.5	4.88	32.9	8.95
Week 3	33.7	7.36	33.7	8.00
Week 4	34.9	8.04	34.8	8.99
Week 5	33.3	8.61	33.1	10.55

Table 4.3 COD influent and effluent values for both biofilters

	Clinoptilolite		Gravel	
	Influent (mg/L)	Effluent (mg/L)	Influent (mg/L)	Effluent (mg/L)
Week 1	374	64.6	375	97.5
Week 2	343	74.3	338	79.4
Week 3	370	85.5	372	66.5
Week 4	346	90.0	341	68.0
Week 5	358	95.0	320	71.0

From Figure 4.7 it can be inferred that total inorganic nitrogen removal of clinoptilolite and gravel biofilter decreased during the five weeks of sample collection over a period of 3 months. The total inorganic nitrogen removal tracked the COD

removal for both the reactors. As the COD removal increased or decreased in both the biofilters, the total nitrogen removal likewise increased or decreased. A possible explanation for this correlation between COD removal and total inorganic nitrogen removal is associated with oxygen transfer, i.e. when the oxygen transfer decreased due to clogging, the removal percent of COD and total organic nitrogen decreased.
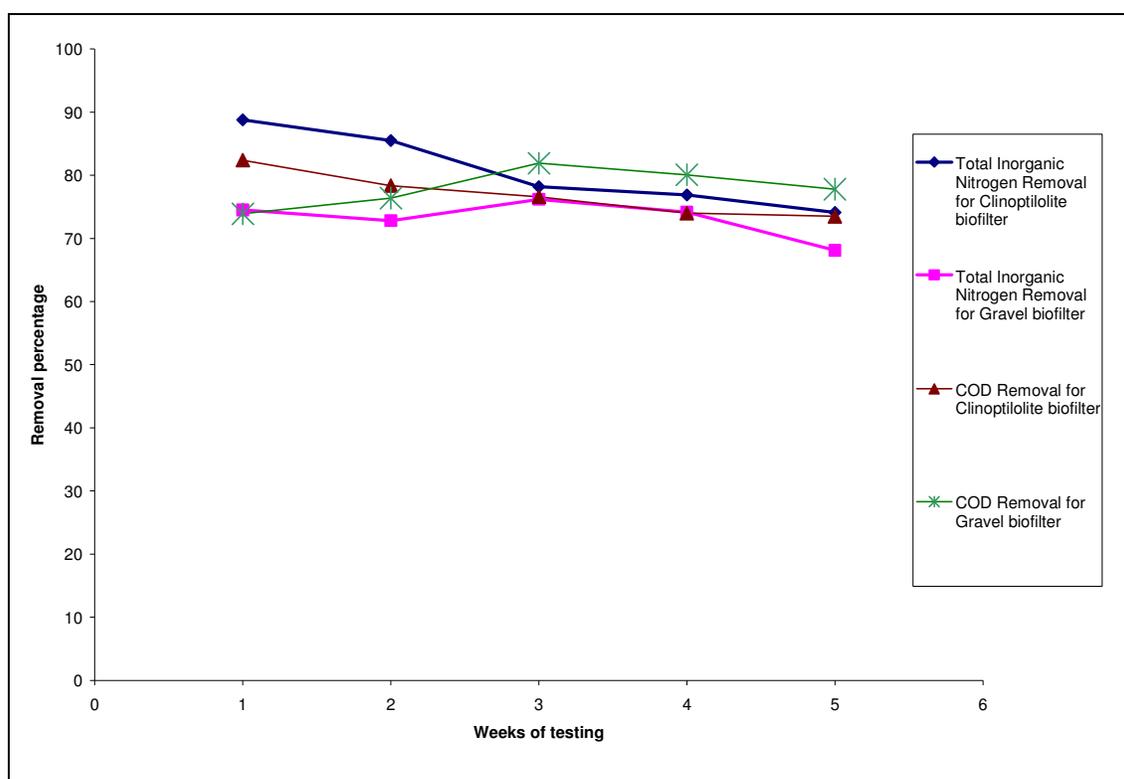

Figure 4.7 Comparison of COD and total inorganic nitrogen removal for both biofilters

The gravel and clinoptilolite biofilters can be assumed to be divided into two zones, the upper unsaturated zone where nitrification occurs and the lower zone where denitrification occurs. Although nitrification occurs in both biofilters, the clinoptilolite

biofilter has the advantage of ammonia adsorption. It is assumed that during the recycle application, nitrification and adsorption of ammonia occurs on the clinoptilolite surface. It is further assumed that the adsorbed ammonia is nitrified during periods of no recycle, which helps to restore the adsorption capacity of clinoptilolite. These assumed mechanisms can explain why the clinoptilolite biofilter performs better in COD and total inorganic nitrogen removal.

4.2 Observations

- The clinoptilolite layer dropped by about 1.25 inches during the 6 months of operation. This observation was similar to that experience in an earlier UTA investigation but was not as severe.

- After approximately 6 months of operation, water accumulation was observed on the clinoptilolite biofilter during the feed application, which led to the conclusion that there was clogging in the biofilter.

- To investigate the causes of clogging of the clinoptilolite biofilter, samples of clinoptilolite and gravel were taken from the clogged biofilter and analyzed for volatile and fixed solids. The test results suggested that clogging in the clinoptilolite biofilter was probably due to two factors: breakage of clinoptilolite into fine particles and accumulation of these fines on the gravel clinoptilolite interface in the denitrification zone with a combination of biological clogging due to excessive biomass growth.

- Two weeks after the clinoptilolite biofilter showed clogging, the gravel biofilter also showed clogging but in a different way. During influent pumping, some of

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the influent was lost from the inlet pipe, probably due to biomass accumulation in the anaerobic zone of the biofilter.

- During the third and fourth months of operation, odors were detected at the top of biofilter during feed application. Also, small insects were observed on both biofilters after about five months of operation.

- Biological growth in the recycle piping to the clinoptilolite biofilter was observed to be greater than that in the gravel biofilter recycle piping. This result was unexpected because the biofilters performed similarly and no reason is evident.

- Hydrographs were measured during the course of study to check for clogging of the biofilters. Figure 4.8 contains the hydrographs of both biofilters when clogging was suspected in the biofilters. As can be seen in Figure 4.8, there was a large variation in the outflow of the biofilters as compared to the inflow.

One possible solution to biological clogging is to rest the system without any feed application, which then leads to endogenous decay of biomass. Both biofilters were shutdown and no feed application or recirculation operation was done for a period of three weeks. Figure 4.9 contains the hydrographs of both biofilters after resting for three weeks. As can be seen in Figure 4.9, the effluent hydrographs of both biofilters are similar to the influent hydrographs, indicating that resting can be effective in preventing clogging due to bioaccumulation.
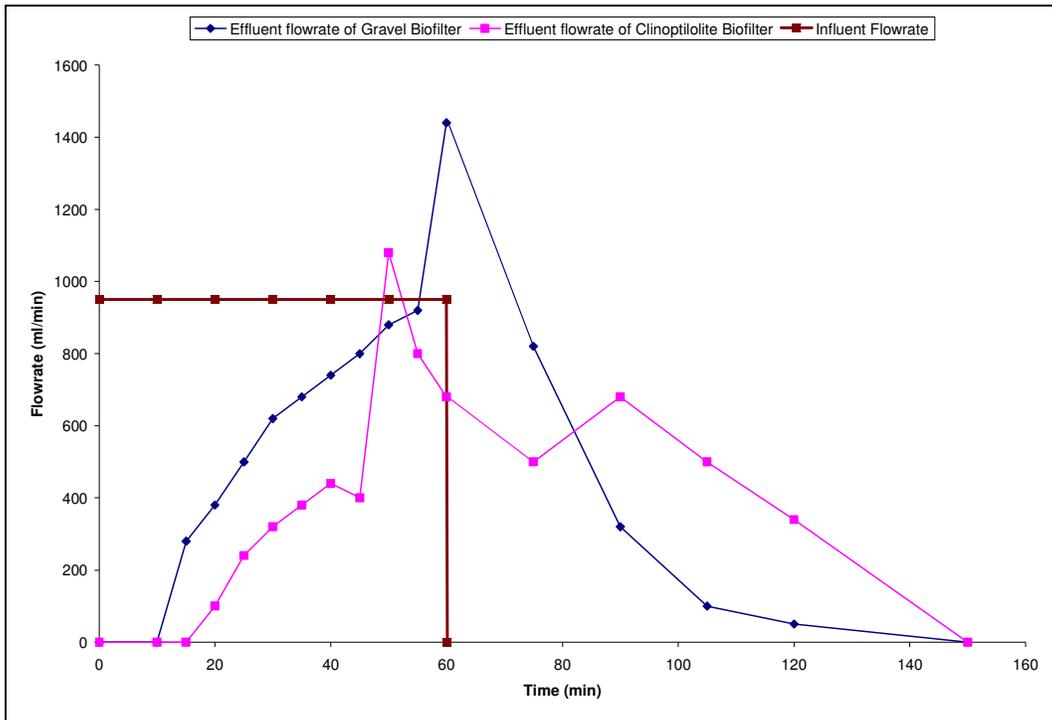
Figure 4.8 Hydrograph for the biofilters when clogging was observed in the clinoptilolite biofilter
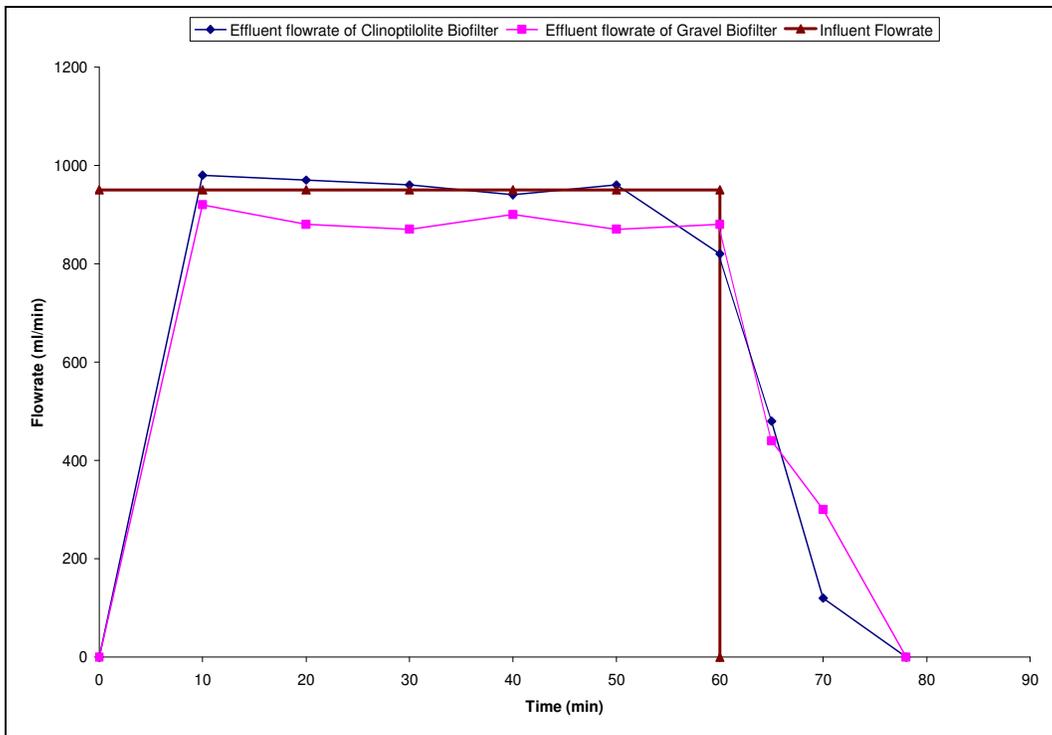

Figure 4.9 Hydrograph for the biofilters after resting the biofilters for 3 weeks

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4.3 Discussion

It is assumed that COD removal and nitrification is primarily the result of aerobic biological activity in the upper unsaturated zone of the biofilter. Although some COD is removed in the lower anaerobic zone by denitrification, the nitrate for that reaction was formed in the upper aerobic zone. The following sections discuss factors which affect the oxygen transfer into the aerobic zone of the biofilters.

4.3.1 Factors Affecting the Percentage of the Void Volume Occupied by Gases

As can be seen in figure 4.10, clinoptilolite or gravel account for sixty percent of the volume in the upper portions of the biofilters. The remaining forty percent, the void volume, is assumed to be occupied by some combination of biomass, water and air. During recirculation the percentage of air decreases as the percentage of water increases. It is also possible that biological growth over extended periods may accumulate in the void volume leading to clogging.

Clinoptilolite or Gravel	Bio-Mass	Water	Air
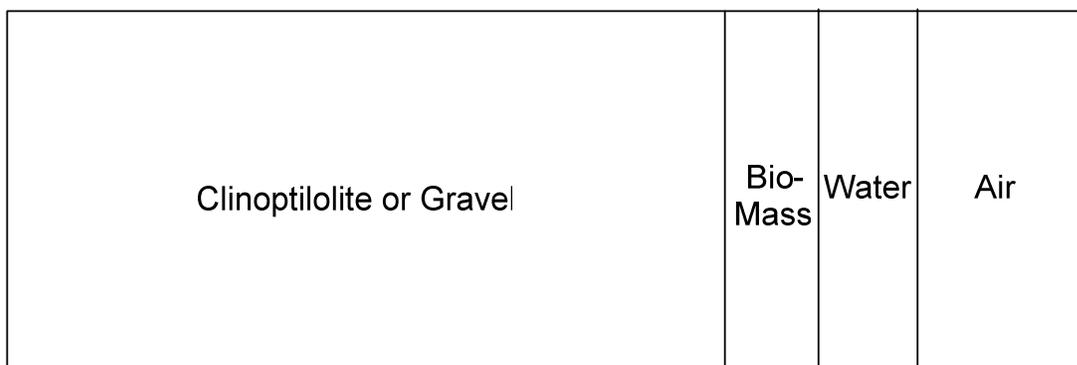
Figure 4.10 Schematic diagram of upper portion of the biofilters

4.3.2 Mechanisms of Oxygen Transfer

Oxygen is required for nitrification in the nitrifying zone. The oxygen enters the biofilter by three assumed mechanisms: gas phase convection, water phase absorption and convective transport and gas phase diffusion. Movement of air from the atmosphere into the biofilters can be compared to soil aeration. Convection can be significant in soils with large pores. The clinoptilolite and gravel are similar to a soil with very large pores. Convection occurs due to a gas pressure gradient where gas moves from the high pressure zone to the low pressure zone. Convection can be caused pressure differences between air present in pores of soil and air above the soil caused by changes in barometric pressure, temperature gradients, wind gusts over soil surfaces, and penetration of water during infiltration (Hillel, 1982). Penetration of water during infiltration into soil is similar to the recirculation of effluent over the gravel and clinoptilolite media; however, this type of transport is limited when the soil pores are large. The laboratory biofilters were operated at room temperature and it can be assumed that the temperature of water and air were same. Thus convective transport due to gas phase temperature variations can also be largely neglected for the laboratory biofilters. While convective transport was assumed to be negligible in the laboratory biofilters, convective transport in full-scale systems may be more important and is discussed later in this section.

A second mechanism assumed to be responsible for oxygen transfer is water phase absorption and convective transport. The effluent from the biofilter outlet is recirculated and applied to the top of the biofilter. The recirculated effluent has low

dissolved oxygen and when it comes in contact with the atmospheric air at the point of release, it can absorb oxygen before entering the biofilter. The oxygen added to the recirculated effluent will be used for biological uptake; however, the contact time for this type of gas transfer is very short in the biofilters and therefore the amount of oxygen transferred is likely to be very small.

This leaves diffusion as the final mechanism responsible for oxygen transport. Diffusive transport of oxygen occurs in both the liquid and gas phases in the biofilters. In the clinoptilolite and gravel biofilters, microorganisms in the biofilm use oxygen for respiration and growth. Concentration differences between both phases due to the biological uptake of oxygen in the biofilm creates a driving force for oxygen transfer due to diffusion (see Figure 4.11).
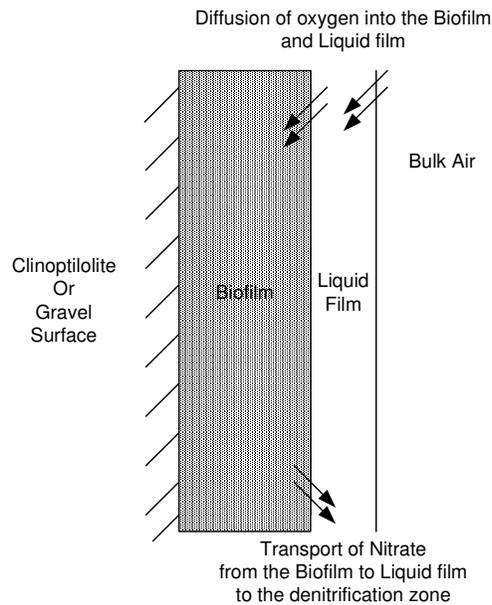
Figure 4.11 Biofilm present in the biofilter

4.4 Experimental Ways of Measuring Oxygen Transfer

The amount of oxygen transferred by convection and diffusion can be measured experimentally using the reactor depicted in figure 4.12. The reactor can be constructed of PVC pipe with plates attached by flanges used for support of media. The aim of the experiment is to determine the amount of oxygen transferred during recirculation of effluent in the biofilters. Above the water level in the lower part of the reactor, an arrangement with holes for air transfer can be built to study the effects on the performance by induced air transfer. Clean media such as clinoptilolite, gravel or plastic packing can be used to study the amount of oxygen transferred.
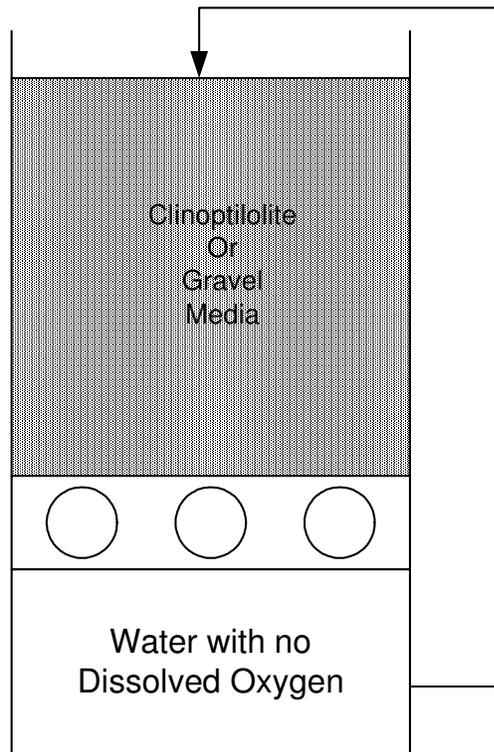

Figure 4.12 Schematic diagram of experimental setup

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At the start of the experiment water is depleted of dissolved oxygen by bubbling nitrogen gas through it. The water with no dissolved oxygen can be recycled and allowed to trickle over the media. As the water trickles down the media, the oxygen concentration of the water rises due to gas transfer. Water samples can be collected from the lower portion of the reactor and analyzed for dissolved oxygen to compute the amount of oxygen transferred in the system by convection, absorption and diffusion. The height of the media can be changed to study its effects on oxygen transfer. Forced draft can be created by inducing the transfer of air into the reactor through the holes above the water level to determine change in oxygen transfer efficiency.

4.5 Mass Transfer Approach for Computing the Amount of Oxygen Transfer

A theoretical mass balance can be performed on the biofilter to investigate whether diffusion alone can provide the amount of oxygen transfer required for COD removal and nitrification. Taking an oxygen mass balance on the biofilter:

$$\frac{dO_2}{dt}V = QO_{2\,Influent} - QO_{2\,Effluent} - OUR + OIR$$

Where O_2 $\qquad$ = Dissolved oxygen concentration in the biofilter

$\qquad$ $O_{2\,Influent}$ $\qquad$ = Dissolved oxygen concentration in the Influent

$\qquad$ $O_{2\,Effluent}$ $\qquad$ = Dissolved oxygen concentration in the Effluent

$\qquad$ V $\qquad$ = Volume of the biofilter

$\qquad$ Q $\qquad$ = Flow-rate into the biofilter

$\qquad$ OUR $\qquad$ = Oxygen Uptake Rate by microorganism

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OIR = Oxygen Input Rate to the biofilter

Assuming steady state and $O_{2\,influent}$ and $O_{2\,effluent}$ to be negligible, we get

OUR = OIR

We know that Q = 30 gallons/day, COD $_{influent}$ = 400mg/L, COD $_{effluent}$ = 80mg/L (Assuming 80% COD removal), $TKN_{influent}$= 39mg/L (Vemulapalli, 2003), NO_3 -$N_{effluent}$ = Zero, NH_3-$N_{effluent}$ = 2.5 mg/L (lowest value in sample analysis).

Solving for OUR

OUR (grams/day) = Q [(COD $_{influent}$ – COD $_{effluent}$)

+ 4.57 mg O_2/mg NH_3-N ($TKN_{influent}$ –NH_3-$N_{effluent}$)

- 2.8 mg O_2/mgNO_3-N ($TKN_{influent}$ – NO_3 -$N_{effluent}$ – NH_3-$N_{effluent}$)]

OUR = 30 gallons/day x 3.78 L/gallon [(400 mg/L – 80 mg/L)

+ 4.57 mg O_2/mg NH_3-N (39 mg/L –2.5 mg/L)

- 2.8 mg O_2/mgNO_3-N (39 mg/L – 0-2.5mg/L)]

= 43,614 mg/day

= 43.6 g/day

From the discussion earlier we know,

$$OIR = \begin{bmatrix} gO_2 \text{ / } day \\ transfered \\ by \\ Absorbtion \end{bmatrix} + \begin{bmatrix} gO_2 \text{ / } day \\ transfered \\ by \\ Convection \end{bmatrix} + \begin{bmatrix} gO_2 \text{ / } day \\ transfered \\ by \\ Diffusion \end{bmatrix}$$

Assuming the main mechanism for oxygen transfer is diffusion we get,

$$OIR = \begin{bmatrix} gO_2 \ / \ day \\ transfered \\ by \\ Diffusion \end{bmatrix}$$

OIR (gm/d) = Flux (mass/area-time) x Area

By Fick's law of diffusion:

$$flux = D_m \frac{dC}{dz}$$

Where, D_m = Diffusion coefficient of oxygen for the media

(cm^2/sec)(gravel or clinoptilolite)

dC = concentration gradient

dz = difference in height

Since the gravel and clinoptilolite media can be compared to soil with very large pores, we can assume that the diffusivity coefficient for soil is equal to diffusivity coefficient of the media $D_s = D_m$

$$D_m = D_s = 0.66 f_a D_o$$

Where, D_s = Diffusivity coefficient of oxygen in soil (cm^2/sec)

0.66 = Tortuosity coefficient

f_a = Air filled porosity

D_o = Bulk-air diffusivity coefficient (cm^2/sec)

= 0.189 cm^2/sec (Hillel, 1982)

The value of tortuosity coefficient ranges from 0.6 to 0.7, depending on the air filled porosity in media, and is assumed to be 0.66. The value of air filled porosity f_a is assumed to be 0.4. Substituting the value in above equation we get:

D_m = 49.9 x 10^{-3} cm^2/sec

Saturated concentration of oxygen in air at STP
= 0.23 x 32 gm / 22.4 liters
Csat = 3.28 x 10^{-4} gm/cm^3

Z = 1 ft = 30.5 cm

C=0
(Assuming oxygen entirely utilized by nitrifiers)
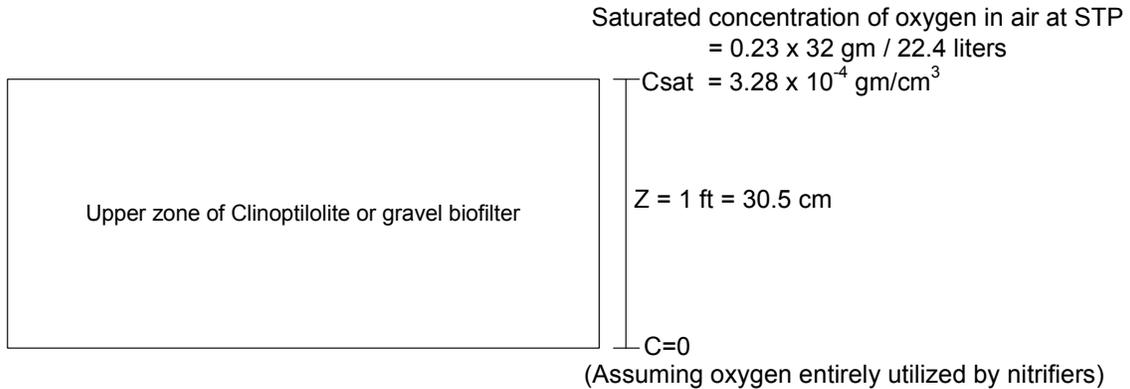
Figure 4.13 Schematic diagram of upper zone of biofilter

From Figure 4.13 and substituting values in equation of Fick's law of diffusion:

$$Flux = 49.9 \times 10^{-3} cm^2 / \sec(3.28 \times 10^{-4} gm / cm^3 - 0) \times 60 \sec \times 60 \min \times 24 hours / 30.5 cm$$

$Flux = 46.4 \times 10^{-3}$ $gm/cm^2/day$

OIR = Flux x area

Area = 3716 cm^2

OIR = 172 gm O_2/day

Thus OIR ≈ 4OUR

It can be concluded that the maximum oxygen input rate by diffusion can be adequate for oxygen uptake required by the biofilters for COD removal and nitrification.

4.6 Recommended Design Modifications for a Full Scale System

In this section modification for a full scale system are proposed to increase pollutant removal performance and minimize clogging. Oxygen transfer is the main limiting factor for successful operation. The laboratory setup had no arrangement for natural or forced draft to occur. The denitrification zone in a full scale system can be constructed below the ground level. It can be filled with gravel with a plastic lining surrounding it to prevent the seepage of nitrified wastewater into the ground. The base can be built with concrete blocks for solid foundation and support. The advantage of concrete blocks is that holes can be easily drilled in them to allow for movement of air into and out of the biofilter (see Figure 4.14).
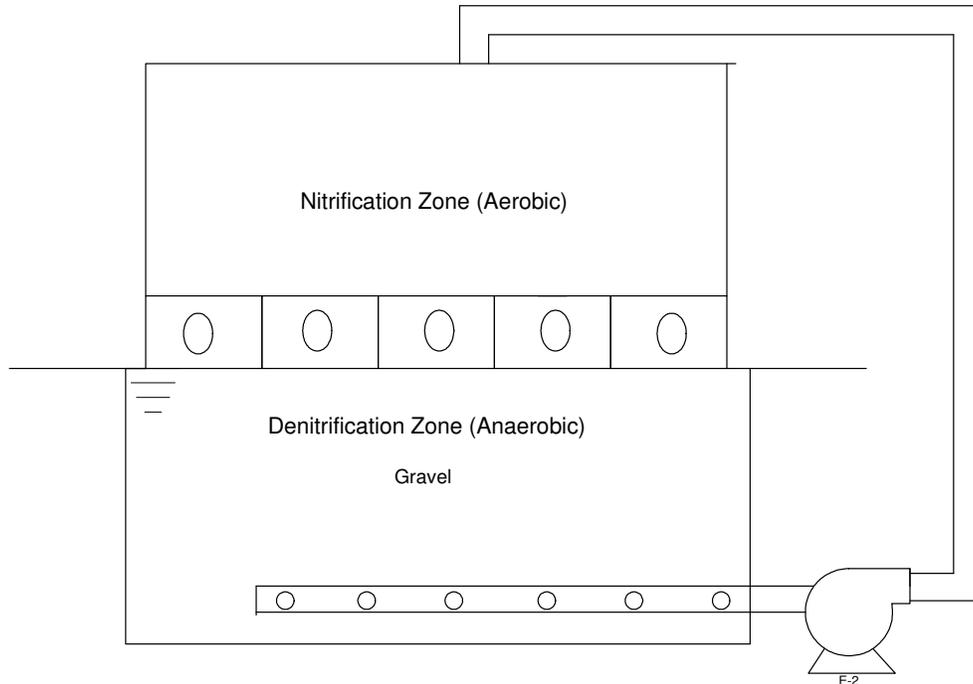
Figure 4.14 Schematic diagram of recommended full scale system

Natural air draft could be created in the system as a result of the above modification is ancillary to the recirculation system and is analogous to air transfer in a windrow composting system. Windrow composting is the most common method for on-farm composting. A raw mixture of organic materials is placed in a trapezoidal pile. Air transfer through the pile occurs due to the chimney effect (see figure 4.15). The primary mechanism of oxygen transfer in windrow composting is convection. Another type of windrow composting is the passively aerated windrow composting. With the purpose of enhancing aeration through the compost media perforated pipes are inserted at specific locations along the porous base such as gravel or straw of the compost pile (Fogiel et at 1998). A similar modification is possible for the bioreactors by adding a blower in the nitrification zone and triggering the blower on during the application of feed to the system. However, adding mechanical equipment such as a blower requires maintenance with least maintenance is desirable.
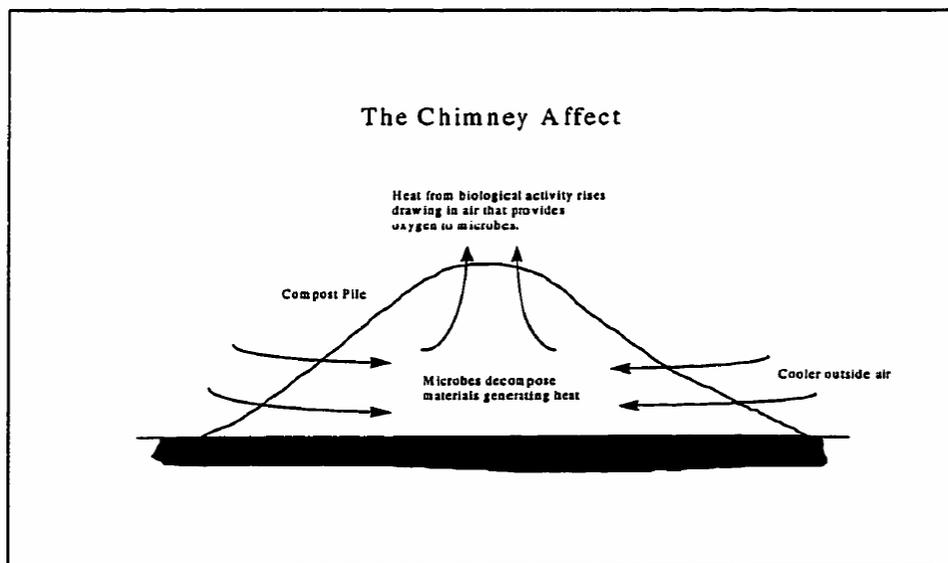

Figure 4.15 Schematic of chimney effect (Source : Fogiel 1998)

Natural draft systems are preferred over forced draft systems. The main driving force in a natural draft system is the resulting pressure head created as a result of temperature difference between the air. In the recommended full scale biofilter density difference between air inside the biofilter can causes the air to either rise or sink. The magnitude of density difference depends upon the temperature and humidity of ambient air. Thus in summer season when $T_{ambient}$ (temperature of ambient air) $> T_{pore}$ (temperature of air inside the pores of the media), air flows downwards, and in the winter when $T_{ambient} < T_{pore}$, air flows upwards. A potential problem occurring in natural draft systems is stagnation, especially in spring when the neutral density conditions occurs, i.e. $T_{ambient}$ and T_{pore} are so close that no air flows occurs. Forced draft system could be an alternative option and fans can be installed for active air flow, but mechanical means require additional maintenance. Instead, an arrangement of pipes could be setup in wind direction to direct the air inside the biofilter through the holes in the concrete base for achieving induced air flow without moving parts during the spring season (Rittmann et al., 2001).

Another modification recommended for scale-up operation is use of V-Notch weirs for recirculation. The laboratory flow distribution manifold has a disadvantage of holes clogging due to excessive biological growth. During the laboratory experimentation the pipes were dismantled and cleaned every two weeks with chlorine, but for a home owner cleaning the piping would add a lot of maintenance costs. A submersible pump can be used for recycle, and the effluent can be pumped to V-notch weirs for better absorption and to avoid maintenance required for cleaning the pipes

(see Figure 4.14). To avoid clogging observed in the biofilter due to excessive biological growth, an additional biofilter should be operated thus keeping the one biofilter at rest to improve hydraulic conductivity of the biofilter by endogenous decay of biomass during the resting period. Depending upon the cost and availability, alternative media such as gravel, plastic packing can also be used instead of clinoptilolite.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

After reviewing the results and observations from the laboratory study the following conclusions can be drawn:

- Simulated feed with a surface hydraulic loading of 7.5 gal/ft^2/day without recycle and 52.5 gal/ft^2/day with recycle and recycle ratio of 6 was applied to compare the performance of clinoptilolite and gravel biofilters. The clinoptilolite biofilter due to ion selectivity of ammonia displayed better nitrification, with total average inorganic nitrogen removal of 80.4 % as compared to 73 % removal efficiency for the gravel biofilter.

- The decrease in height of the clinoptilolite in the biofilter, hydrographs of biofilters and fixed solids tests on the clinoptilolite and gravel interface validate the assumption that the biofilter clogged due to combination of excessive biomass formation and breakdown of clinoptilolite due to disintegration.

- From observations of the laboratory biofilter, in order to solve the biological clogging associated with excessive biomass growth, a standby biofilter should be incorporated into a full scale system to allow resting of a filter and restoration of hydraulic capacity.

5.2 Recommendations for Future Research

- Nitrification efficiency of alternative media such as plastic packing can be studied in the laboratory.

- Nitrification and denitrification efficiency can be studied using clinoptilolite in a fill and draw laboratory operation using siphon for flow regulation, which would save recycle pumping costs.

- Optimum operating conditions, such as different combinations of influent feed application duration, frequency of feed applications with varying recycle ratio, or effects of removal efficiency with continuous feed application, can be researched further.

- Research can be done to study the effects of recycle by analyzing samples for COD and ammonia during recirculation cycles to find optimum recycle ratio.

- Forced draft conditions can be simulated in the laboratory to research the effect of induced air transfer on removal efficiency.

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BIOGRAPHICAL INFORMATION

Born in the city of Mumbai, India on July 29, 1978, Adnan Sadikot completed his initial schooling in Ahmedabad and started pursuing his Bachelor of Chemical Engineering from University of Pune, India in 1995. His senior year project titled 'Upgradation of Heavy Water using Highly Efficient Structured Packing by Distillation' was conducted at Bhabha Atomic Research Center, Mumbai, India.

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