

A  
Dissertation Report  
On  
**ASSESSMENT OF OPERATIONAL PARAMETERS AND EFFICACY OF SAND  
FILTRATION FOR THE REMOVAL OF TKN, AMMONICAL NITROGEN,  
NITRATES AND PHOSPHATES FROM GREYWATER**



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## List of Abbreviations

BOD = Biochemical Oxygen demand

CFU = Colony Forming Unit

COD = Chemical Oxygen demand

CPHEEO = Central Public Health and Environmental Engineering Organization

FC = Fecal coliform

GW = Greywater

L/cap/day = liter per capita per day

L/hr/m<sup>2</sup> = liter per hour per square meter

m<sup>3</sup>/hr/m<sup>2</sup> = cubic meter per hour per square meter

Mg/l = milligram per liter

ml/min = milliliter per minute

NH<sub>4</sub>-N = Ammonical nitrogen

NO<sub>3</sub>-N = Nitrate Nitrogen

NTU = Nephelometric Turbidity Unit

OECD = Organization for economic co-operation and development

P = Phosphorus

PO<sub>4</sub><sup>3-</sup> = Phosphates

TDS = Total dissolved solids

TKN = Total Kjeldahl Nitrogen

TSS = Total suspended solids

μm = Micrometer

μS/cm = Micro Siemens per centimeter

## **ABSTRACT**

*In the wake of water scarcity it becomes important to save water. In arid and semi-arid areas of Rajasthan particularly this becomes very essential to counter the problem of water scarcity. An important initiative in this matter is recycling of wastewater. In this study the recycling of greywater was sought because it is low in organics and nutrients as compared to black water and hence does not command very high level of treatment. Also a majority portion of the domestic waste water generated is greywater (about 75%). To further make the process low cost it was thought to use the locally available sand for the design of filter bed. If greywater is recycled on domestic level by much of the population it could reduce load on the centralized water supply systems and further reduce load on the waste water treatment facility.*

*In this study 4 column studies were performed: two studies with constant loading rates of 10 ml/min and 20 ml/min, other two studies with constant head of 3 cm and 5 cm. The greywater was collected from MNIT, Jaipur girls' hostel. It was characterized for pH, TKN, NH<sub>4</sub>-N, NO<sub>3</sub>-N and phosphates. The effluent concentration of these parameters after filtration were compared with the reuse guidelines as provided by the CPHEEO and hence the efficiency, effect of various process parameters on efficiency and suitability of the process was ascertained.*

## **1. INTRODUCTION**

This chapter discusses the need for this study and specific need for the treatment of greywater, followed by aim and objectives of this study.

### **1.1. NEED OF THE STUDY**

Lately, most of the developing countries around the world are facing water scarcity. It is estimated that within the next 50 years, more than 40% of the world's population will face the problem of water scarcity (Sandeep Thakur, 2013). Therefore, there arises a need of waste water recycling. This is particularly important in arid zones, where water is scarce and recycling GW (Greywater) for private and public landscape irrigation could reduce potable water use by up to 50% (A. Gross, 2007). The population growth, climate change and the enhancement in the standard of living, especially for personal hygiene; are the main factors for the increased domestic water consumption (Zhe Li, 2010). While water withdrawals worldwide are 4,000 to 5,000 km<sup>3</sup> per year, the annual water consumption in the world accounts for 2,500–3,000 km<sup>3</sup>. In the OECD countries the average of the domestic consumption is approximately 180 L/d/cap. But there are some disparities around the world. Countries like Canada and Japan consumes more than 250 L/cap/day (326 and 278 L/cap/day respectively), for domestic purposes whereas in several African countries the daily intake remains lower than 30 L/cap/day (Katell Chaillou, 2011). Currently the large scale centralized water supply system has led to the over-exploitation of water. This approach was supported by economy and better professionals available. But today as the urban population is continuously sprawling there is lack of funds for urban infrastructure, leakages and water theft, so it has become harder to maintain and operate the centralized system. Therefore there arises a need for the decentralized treatment which would offer more opportunities for recycling. Such systems are in vogue in countries like Japan, U.S., EU, Australia, Israel, etc. (Eran Friedler, 2008). In this study it is aimed to recycle waste water, by using greywater particularly as the waste and hence recycle and reuse it for various purposes, primarily toilet flushing.

### **1.2. NEED FOR THE TREATMENT OF GREYWATER**

Greywater (GW) has attracted global attention as an alternative water source over the last few decades. GW treatment and reliable reuse require the overall qualitative characterization of samples from different sources. Household greywater, comprising wastewater from bathing (light GW) as well as that from laundry, excluding wastewater from toilets, is not waste but an alternative water source. It is

comparatively low in organics and nutrients than black water and so requires relatively cheap treatment. In a number of known domestic activities (toilet flushing, soaking, window cleaning or car washing, irrigation etc.), the use of potable water is not necessary. An average person typically produces 150-250 liters of domestic wastewater per day, and GW accounts for up to 75% of household wastewater. About 50–80% of domestic wastewater which originates from dishes, showers, sinks, laundries and toilet flushing, accounts for around 20–30% of the total household water usage (Nurul Widiastutia, 2008). A study reported that gardens account for 34% of the water budget and toilets 20% (Diana Christova-Boal, 1996). Thus if greywater is recycled then a lot amount of water can be saved on a daily basis. This is particularly important in arid zones, where water is scarce and recycling GW for private and public landscape irrigation could reduce potable water use by up to 50% (A. Gross, 2007). The first case of greywater treatment was mentioned in 1975 by the NASA (Katell Chaillou, 2011). Domestic in-house water demand in industrialized countries ranges between 100 to 150 L/cap/day, of which 60–70% is transformed into greywater, while most of the rest is consumed for toilet flushing. Greywater reuse for toilet flushing (if implemented) can reduce the in-house net water consumption by 40–60 L/cap/day, and can reduce urban water demand by up to 10–25%, which is a significant reduction of the urban water demand (E. Friedler, 2005). In Arizona it is documented that an average household generate about 30,000 to 40,000 gallons of GW per year (Al-Jayyousi, 2003).

The quality of GW depends on the type of the source, the quality of the water supply, the type of distribution system, household occupancy and the occupants' genders, age distribution and activities. The analysis of the quality of GW streams originating from different sources is essential before reuse. The concentrations of organic compounds, solids, salts, pathogens, phosphorus and nitrogen forms in GW vary widely by source and depend significantly on the volume of water used (Ildiko Bodnar, 2014). Treated greywater is most appropriate for water applications that are frequently used and require low-quality water. Toilet flushing is the best application. The 27% of domestic water used for toilet flushing could be entirely replaced by treated GW. The 12% of water that is used for other applications such as garden watering and car washing could also be replaced by treated GW (Li et al, 2010). The total water for toilet flushing (about 15 to 55 L/cap/day) can be substituted with service water without a hygienic risk or comfort loss (Nolde, 1999).



### **1.3. AIM OF THE STUDY**

With this study it is aimed to reduce the water shortage by recycling greywater by cost effective methods. The aim of this study is to characterize greywater and subsequently use the locally available sand for the removal of various parameters to make the method cost effective. It is aimed to reduce the consumption of water by MNIT hostel. As the hostel already have plumbing fixtures for using the recycled water for flushing purposes so it was thought to recycle greywater because of its large volume generated daily. It was thought to assess the capability of the sand filters for recycling greywater and study greywater characteristics and feasibility of this process.

### **1.4. OBJECTIVES OF THE STUDY**

- To characterize greywater for TKN,  $\text{NO}_3\text{-N}$ ,  $\text{NH}_4\text{-N}$ , pH and  $\text{PO}_4^{3-}$
- To assess the capacity of locally available sand for the removal of above mentioned parameters
- To study the removal efficiency under constant overloading rate conditions for 2 different rates: 10 ml/min and 20 ml/min
- To study the removal efficiency under constant head apparatus for 2 different heads: 3 cm and 5 cm
- To compare the efficiencies of removal of all the above mentioned parameters with time (at the end of each day)
- To study the effect of various parameters like pH, initial concentration, contact time and loading rate on the removal efficiencies and explore the physico-chemical aspects of filtration
- To compare the effluent characteristics with the reuse guidelines and hence determine the usefulness of the process for greywater recycling

## **2. REVIEW OF LITERATURE**

This chapter discusses the characteristics of grey water, challenges involved in its use, reuse guidelines for its reuse, various treatment technologies in vogue, the theory of filtration as a whole, removal mechanism of different forms of nitrogen and phosphates.

### **2.1. GREYWATER CHARACTERISTICS**

The published literatures indicate that the typical volume of grey water varies from 90 to 120 L/cap/d depending on lifestyles, living standards, population structures (age, gender), customs and habits, water installations and the degree of water abundance. The quality of GW varies from source to source. The main characteristic of greywater is its high variability. It depends on the quality of the water supply, the type of distribution system and the activities of occupants in the house (Zhe Li, 2010). Not only this the quality of greywater also change with time during a day; showering and shaving may take place in the morning, hand washing during the day and make-up removal and oral care in the evening. The water quality is therefore expected to vary accordingly in the greywater produced (Eva Eriksson, 2009).

Bathroom grey water is deficient in both nitrogen and phosphors due to the exclusion of urine and faces (Fangyue Li, 2009). Furthermore, there could be chemical and biological degradation of the chemical compounds, within the transportation network and during storage. The micro-organisms can be introduced into greywater by hand washing after toilet use, washing of babies and small children connected with diaper changes and diaper washing. In a study the total nitrogen concentration of the grey wastewater was lower than in domestic wastewater, 0.6–74 and 20–80 mg/l, respectively. The main source for nitrogen in domestic wastewater, urine, should not be present in grey wastewater. The total phosphorus concentrations were generally higher in laundry grey wastewater compared to bathroom grey wastewater, 0.1–57 mg/l and 0.1–2 mg/l, respectively. BOD of the greywater ranged from 40-250 mg/l and COD ranged from 100-600 mg/l. The average fecal coliform count was  $5.6 \times 10^5$  CFU/100 ml (Friedler et al, 2005). In another study average COD, BOD and TSS values were 2568 mg/l, 1056 mg/l and 845 mg/l, respectively (M. Halalsheh, 2008). The greywater is relatively low in turbidity which indicates that it has large amount of dissolved impurities.

The separation of the toilet stream from domestic wastewater generates effluents which have reduced levels of nitrogen, solids, and organic matter (especially the barely degradable fraction),

but often contain elevated levels of surfactants, oils, boron and salt. The components in GW may alter soil properties, damage plants and contaminate groundwater (A. Gross, 2007).

All types of grey waters show good biodegradability in terms of the COD: BOD<sub>5</sub> ratios. Compared to the suggested COD: N: P ratio of 100:20:1 for sewage wastewater, bathroom grey water is deficient in both nitrogen and phosphorus due to the exclusion of urine and feces. Similar to the bathroom greywater, the laundry grey water and the mixed grey water are also deficient in nitrogen. In some cases, the laundry grey water and the mixed grey water are low in phosphorus due to the use of phosphorus free detergent.

Kitchen greywater contributes the highest levels of organic substance, suspended solids, turbidity and nitrogen. Differing from other grey waters, the kitchen grey water doesn't lack nitrogen and phosphorus. Some authors exclude kitchen wastewater from the other streams. However, if grey water is intended to be treated through a biological process, it is suggested that the small amount of kitchen grey water should be collected together with other streams to maintain an optimal COD: N: P ratio. This is because grey water from kitchen sinks and dishwashers contributes most of the biodegradable organic substances and particulate nitrogen. The analysis of the grey water characteristics by different categories also shows that the bathroom and laundry grey water are less contaminated by the microorganisms compared to the other grey water streams. Due to the presence of the large amount of easily biodegradable organic substances, kitchen grey water is more contaminated by the thermal tolerant coliforms than other grey water streams.

Greywater along with microorganisms also contains various chemical contaminants. These chemical contaminants are majorly surfactants which are present due to the detergents used in the laundry. Surfactants are combined to another chemical compounds known as builders. The builders are alkali substances which makes the greywater alkaline in nature.

The major characteristics of greywater are a high COD/BOD ratio, nutrient deficiency, high variability associated with organic concentration, low suspended solids to turbidity ratio (majority particles in 10–100 µm range). These characteristics suggest that advanced biological processes combined with efficient solid separation process are likely to be the most suitable technology for grey water recycling (B. Jefferson, 1999). The characteristics of greywater from various literatures are compiled in the following table (Table 2.1).

**Table 2.1. Characteristics of greywater originating from bathrooms and showers**

<b>BOD (mg/l)</b>	<b>COD (mg/l)</b>	<b>TN (mg/l)</b>	<b>Turbidity (NTU)</b>	<b>Total P (mg/l)</b>	<b>pH</b>	<b>Reference</b>
108	586	-	330	-	7.6	(Sandeep Thakur, 2013)
200	421	15.9	125	0.20	7.34	(Katell Chaillou, 2011)
50-300	100-663	3.6- 19.4	44-375	0.11-48.8	6.4- 8.1	(Fangyue Li, 2009)
50-100 (BOD <sub>7</sub> )	100-200	5-10		0.2-0.6		(Nolde, 1999)
280-688	702-984	25-45.2		17.2-27	6.3- 7.0	(A. Gross, 2007)
76-200		4.6-20 (TKN)	60-240	0.11-1.8	6.4- 8.1	(Diana Christova-Boal, 1996)
121	371	1(NH <sub>3</sub> -N)	69	0.36		(Al-Jayyousi, 2003)
72.7		4.1	38.8		6.8	(M. Gual, 2008)
104		4.6	34	0.7		(Eran Friedler, 2008)
95	148		33			(Yael Gilboa, 2008)
39	144	7.6	35	0.5 (PO <sub>4</sub> <sup>3-</sup> )	6.6- 7.6	(Marc Pidou, 2008)
167			96	16 (PO <sub>4</sub> <sup>3-</sup> )		(Stewart Dallas, 2004)
47 (BOD <sub>7</sub> )		3.72		3.73		(Gunther, 2000)
	244	41.2		6.6		(Luc'ia Hernandez Leal, 2010)
597	1489	105 (TKN)	26		7.6	(M. Halalsheh, 2008)
33.3	143		44.5			(B. Jefferson, 1999)
76-200		4.6-20 (TKN)	60-240	0.11-1.8	6.4- 8.1	(Eva Eriksson, 2002)

216	424	1.56 (NH <sub>3</sub> -N)	92	1.63(PO <sub>4</sub> <sup>3-</sup> )	7.6	(Eva Eriksson, 2002)
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## **2.2. CHALLENGES OF GREYWATER TREATMENT**

The characterization of grey water reveals that the grey water shall be treated to a higher standard before reusing to avoid the health risk and negative aesthetic and environmental effects. This happens especially in the warm climate where temperature favors the pathogen growth. As such highly efficient and reliable conveyance, storage and treatment systems should be developed.

The major target of grey water reclamation and reuse is to reduce the suspended solids, the organic strength and the micro-organisms due to its relationship with the aesthetic and health characteristics of the product water and directly through legislative requirements. It is also important to reduce the micro-nutrients to avoid the regrowth and fouling potential in the reuse system. The risk of contamination may arise due to direct contact with greywater through hands or mouth. The risk of pollution of the soil and receiving waters is another problem due to the contamination by phosphates and Xenobiotic compounds. (Nurul Widiastutia, 2008). Storage of untreated or treated greywater before reuse promotes the growth of microorganisms like fecal coliforms, as well as there is evolution of offensive sulphide-containing odors. (Bongumusa M. Zuma, 2009). The different exposure scenarios — direct contact, irrigation of sport fields and groundwater recharge can give unacceptably high rotavirus risks despite a low fecal load. The poor reduction of somatic coli phages, could be the one main reason which suggests additional treatment of the greywater (Jakob Ottosona, 2003). A number of problems are encountered while installing greywater systems like insufficient head, long collection, distribution and overflow pipelines, anchoring of collection tanks to the ground, etc. It is advisable that the greywater system should be constructed simultaneously when is house is being constructed. This could be followed for the new houses and buildings being constructed. In countries such as the USA and Australia, where regulations for the use of GW have been established, they concentrate on issues associated with public health but do not consider potential harmful environmental impacts like that of dioxins. (A. Gross, 2007)

There are a number of problems related to the reuse of untreated grey wastewater. The risk of spreading of diseases, due to exposure to micro-organisms in the water, will be a crucial point if the water is to be reused for e.g. toilet flushing or irrigation. There is a risk that micro-organisms in the water will be spread in the form of aerosols that are generated as the toilets are flushed (Eva Eriksson, 2002). Also, NOM is a precursor for biological growth in water and waste water

treatment and distribution systems (Kerry E. Black, 2014). The major difficulty presented for treatment of greywater is the large variation in its composition. Reported mean COD values vary from 40 to 371 mg/l between sites, with similar variations arising at an individual site. This variation has been attributed to changes arising in the quantity and type of detergent products employed during washing. Grey water quality is also subjected to dynamic variation: significant chemical changes may take place over time periods of only a few hours, though little appears to have been reported on this phenomenon (B. Jefferson, 1999)+.

### 2.3. GREYWATER APPLICATIONS AND REUSE GUIDELINES FOR TOILET FLUSHING

The most commonly described application for grey water reuse is toilet/urinal flushing which can reduce water demand within dwelling by up to 30%. However, grey water has been considered for many other applications including irrigation of lawns at cemeteries, golf courses and college campuses, vehicle washing, fire protection, boiler feed water, concrete production and preservation of wetlands (B. Jefferson, 1999). Other than domestic recycling greywater is also being recycled in a Tourist Hotels world over. A social survey in Melbourne showed that people were interested in using greywater treatment and its reuse provided there was a short payback period of about 2-4 years (Diana Christova-Boal, 1996). The guidelines for reuse of various countries is given in (Table 2.2)

**Table 2.2. Greywater reuse guidelines in various countries for toilet flushing**

Country	pH	TDS (mg/l)	Turbidity (NTU)	BOD (mg/l)	NH <sub>3</sub> -N (mg/l)	Fecal coliform (CFU)	References
Germany				5 (BOD <sub>7</sub> )		< 10 / ml	(Nolde, 1999)
Japan	5.8-8.6			<20		≤1000/ml (total)	(Fangyue Li, 2009)
China	6-9	<1500	<5	<20	<10	<3/100 ml	(Fangyue Li, 2009)
U.S.A.	6-9		<2	10		ND /100 ml	(Fangyue Li, 2009)
Australia		<30(TSS)		<20		<100/100 ml	(Fangyue Li, 2009)
Canada		<10 (TSS)	<2	<10			(Katell Chaillou, 2011)

The following are the reuse guidelines as prescribed by the Government of India (Table 2.3)

**Table 2.3. The Wastewater reuse guidelines for toilet flushing in India (CPHEEO)**

<b>Parameter</b>	<b>Values for Reuse (mg/l)</b>
Turbidity(NTU)	<2
SS	NIL
TDS	2100
pH	6.5-8.3
Temperature	Ambient
Oil and Grease	10
Minimum residual chlorine	1
TKN	10
BOD	10
COD	AA
Dissolved Phosphorous as P	1
Nitrate Nitrogen as N	10
Fecal Coliform in 100 ml	NIL
Odor	No foul odor

AA: As arising when other parameters are satisfied

All units are in mg/l unless specified



## 2.4. GREYWATER TREATMENT TECHNOLOGIES

Greywater and its characteristics are highly variable as it is generated as a result of the living habits of the people, the products used and house plumbing installation. The treated grey water should fulfill four criteria for reuse for reuse:

1. Hygienic safety
2. Aesthetics
3. Environmental tolerance
4. Economic feasibility

One should also make a note that different reuse applications require different water quality parameters and thus in every case different combination of treatment system varying from simple processes to advanced will be required. There has been no uniformly enforceable international water reuse guideline to control the quality of the reclaimed wastewater. In many cases, the national water reuse guidelines vary from one state to the other. There is considerable variation among these guidelines, particularly regarding identifiable values and the limited parameters. Technologies applied for grey water treatments include physical, chemical, and biological systems or a combination of these three systems. Most of these technologies are preceded by a separation step as pre-treatment in which heavier suspended particles are separated by sedimentation of grit chamber to avoid clogging of the subsequent treatment and followed by a disinfection step as post treatment to achieve considerable removal of pathogens.

The most commonly used greywater treatment technologies are:

**a) Physical processes-** These process encompasses treatment by depth filtration by using sand or other media and membranes. Coarse filtration followed by disinfection is a commonly used method. Coarse filtration is obtained by using metal strainers and disinfection is by using chlorine and bromine. Obviously, coarse filtration and soil filtration alone are not able to reduce the physical, chemical and microbiological parameters to the values required by the non-potable reuse guideline. The micro filtration and the ultrafiltration membrane provide a limited removal of the dissolved organics but an excellent removal of the suspended solids, turbidity and pathogens. The key constraint for economic viability of membrane systems is the fouling of the membrane surface by pollutants species. This increases the hydraulic resistance of the membrane, thereby increasing the energy (Al-Jayyousi, 2003). The application of membrane technology requires greater energy consumption. A low strength bath grey water was treated

using a nylon sock type filter, followed by a sedimentation step and a disinfection step. The COD, the turbidity, the SS and TN were reduced from 171 mg/l, 20 NTU, 44 mg/l and 11.4 mg/l in the influent to 78 mg/l, 16.5 NTU, 18.6 mg/l and 7.1 mg/l respectively in the effluent (Fangyue Li, 2009). In another study the COD, the BOD, the SS, the TN and the TP in the kitchen sink grey water were reduced from 271 mg/l, 477 mg/l, 105 mg/l, 20.7 mg/l and 3.8 mg/l in the influent to 40.6 mg/l, 81 mg/l, 23 mg/l, 4.4 mg/l and 0.6 mg/l respectively in the effluent by using a slanted soil filter (Fangyue Li, 2009). The soil filtration system is found to remove pollutants by an amalgamation of various processes in a single system, like simultaneous nitrification-denitrification, adsorption, straining, etc. The pre-treatments such as septic tank, filter bags, screen and filters are applied to reduce the amount of particles and oil and grease (Fangyue Li, 2009). In a study by (M. Halalsheh, 2008) intermittent sand filter was used for greywater filtration along with septic tanks, wetlands and UASB- hybrid reactor. In the sand filter solids were reported to be removed by both physical and biological processes. When properly designed, the filter can remove as high as 90% of the BOD<sub>5</sub> and 80% of the COD. In a study by (George Nakhla, 2003) the slow sand filtration was assessed for simultaneous nitrification and denitrification. Obviously, coarse filtration and soil filtration alone are not able to reduce the physical, chemical and microbiological parameters to the values required by the non-potable reuse guideline. The micro filtration and the ultrafiltration membrane provide a limited removal of the dissolved organics but an excellent removal of the suspended solids, turbidity and pathogens. Greywater mixed with RO reject was treated using sand filtration in 2 steps. The first filter was of nylon (1mm mesh) and the second was of sand (0.7 to 1 mm) (M. Gual, 2008). Ultrafiltration of greywater was done and it was found that permeate was low in nutrients and organics (Eran Friedler, 2008).

**b) Chemical processes** – Chemical processes like photo-catalytic oxidation, coagulation, ion-exchange, and granular activated carbon, magnetic ion-exchange (Marc Pidou, 2008) are generally used methods for chemical treatment. The direct treatment of greywater without any pretreatment may result into early fouling of the membrane and clogging of the filters. It was reported by (Nurul Widiastutia, 2008) that natural zeolites are good potential material for water and wastewater treatment because of their low cost and abundance. Also it is reported that natural zeolite removes more than 50 wt. % of ammonical nitrogen (Rozic et al, 2009). Due to the fact that zeolite has negative charge within the pores balanced by positively charged ions

such as  $\text{Na}^+$ ,  $\text{K}^+$ ,  $\text{Ca}^{2+}$ , and  $\text{Mg}^{2+}$  with weaker electrostatic bonds. Those cations, therefore, can be easily exchanged with cations such as  $\text{NH}_4^+$  in waters and wastewaters. Greywater was pretreated using hypochlorite solution before sand filtration. It was reported that a residual chlorine concentration of 1 mg/l showed negative results for coliform test (M. Gual, 2008). In a study chlorine demand of the greywater was reported to be 10–20 mg/l (Eran Friedler, 2008). In the same study ferric chloride was tested as a coagulant. A dose of 50 mg/l of ferric chloride reduced the UF flux decline rate by 43%, mainly due to a 38% reduction of the organic load. A combination of chlorination and coagulation resulted in a TN concentration as low as 1.1 mg/l and P was 0.1 mg/l which was better than using these two processes separately.

**c) Biological processes** – Many processes like vertical flow constructed wetland (VFCW) (A. Gross, 2007), sand filtration, RBC (E. Friedler, 2005), RBC followed by sedimentation (Yael Gilboa, 2008), UASB (Luc'ia Hern'andez Leal, 2010) have been used. (E. Friedler, 2005) developed a greywater treatment system for an eight storied building. The treatment system comprised of several units: fine screens, equalization basin, Rotating biological contactor, sedimentation basin, pre-filtration storage tank, sand filtration and disinfection. The pilot plant successfully removed 58%, 87%, 96% and 72% of the TP, TKN, ammonia and organic nitrogen, respectively. 100% of the FC was removed by the pilot plant. The COD removal was 64% and turbidity removal was 98%. The final BOD and turbidity was 2.3 mg/l and 0.6 NTU respectively. Mulch tower treatment has been used for greywater filtration. The tower comprised of a mulch layer of 20 cm at the top followed by 10 cm of coarse sand, 1mm mesh, 15 cm fine gravel layer and 15 cm coarse gravel layer (Bongumusa M. Zuma, 2009). Ecological sanitation system of horizontal sub-surface flow constructed wetland was modelled by (Darrien Yau Seng Mah, 2009). *Triglochin huegelii*, a Western Australian species for the greywater treatment was studied and it was reported that *Triglochin* has consistently removed nitrogen and phosphorus (R. Mars, 2003). Reed beds have been used for the treatment of greywater in Costa Rica, Central America (Stewart Dallas, 2004).

## **2.5. THE THEORY OF FILTRATION**

The method of purification of water using sand is in vogue since the 19<sup>th</sup> century. The illustrious and continuous use of this method is attributed to its simplicity, low cost and reliability. The use of locally available sand makes it even more economical. Generally a water treatment method is judged on the basis of water quality and quantity, raw water sources, efficiency, economy and safety. For the selection of an optimum combination of treatment units it is important to ascertain the required water quality after treatment. Then comes the role of economy which encompasses running and maintenance costs. So it is important to set goals in this direction.

### **History**

The first instance of filtration dates back to 1804 when John Gibbs designed a slow sand filter for bleachery in Paisley, Scotland. It was in 1829 that a slow sand filter was first used for a public supply. It was James Simpson who created the sand filter to be used by Chelsea Water Company in London. At that time filtration was considered as a method to remove turbidity and suspended solids from water and the presence of pathogenic organisms was unknown. There was lack of insight in to the complex removal mechanisms taking place. In 1885 and after discoveries by Pasteur, Escherich and others contributed to the science of pathogens. In 1885 the first mechanical filters were installed in USA. In 1899 England patented pressure filters. (L. Huisman)

### **Types of filters**

On the basis of driving force there are two types of filters – Gravity and Pressure Filter. In a pressure filter the media is enclosed in a vessel and the water is forced in under pressure. In a gravity filter the media is kept in a vessel which is open from the top and the water is allowed to flow down under gravity. The pressure filters require frequent attention and are costly. The gravity filters are cheap comparatively and does not require much attention. Further gravity filters are of two types – Slow and Rapid. Both these filters are down flow filters with single media.

### **Elements of Filtration**

#### **Filter Sand**

For slow sand filtration locally available sand and builder grade sand can be used. For support round gravels can be used, if not available hard broken stones can be used. In rapid sand filtration the effective size ( $D_{10}$ ) of the sand shall be to 0.45 to 0.75 mm. Uniformity coefficient

shall be 1.3 to 1.7. Specific gravity shall be in the range of 2.55-2.65. In a slow sand filter the effective size should be 0.2-0.3 mm and uniformity coefficient should be 5 (CPHEEO, 1999).

#### Rate of filtration

For a slow sand filter the rate of filtration should be 0.1 m/hr under normal operation and 0.2 m/hr for maximum overload. For a rapid sand filter the rate should be 4.8-6 m/hr.

#### Depth of the sand

The various elements for slow sand filter – free board (0.2 m), supernatant water (1 m), filter sand (1m), supporting gravel (0.3 m), under drainage system (0.2 m) summing to a total depth of 2.7 m shall be used. For a rapid filter the depth of the sand bed should be 0.60 to 0.75 m with a minimum free board of 0.50 m. The depth of the sanding water should be 1-2 m.

#### **Hydraulics of filter**

The resistance (H) offered by the filter bed to the flow of water can be given by Darcy's law:

$$H = v_f \cdot h/k$$

Where h is the bed thickness, k is the coefficient of permeability (m/sec) and  $v_f$  is the filtration rate (volume/time/area).

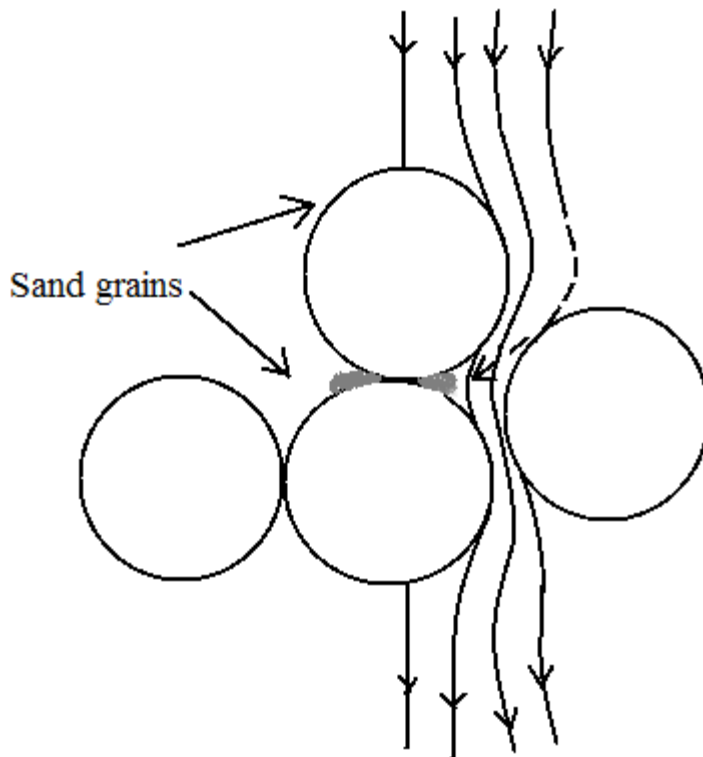
The CPHEEO manual has recommended Kozeny's equation for the theoretical calculation of head loss in a filter.

During filtration most of the impurities are strained on the top layer of the sand. As the time increases more and more impurities get deposited which leads to a decrease in the pore size and hence the resistance increases. The resistance increases in the initial stages slowly but in the later phase it increases very sharply. The resistance is developed entirely on the schmutzdecke and the top layer as the impurities and large amount of turbidity is deposited on this top layer. In a clean filter there is a gradual decrease in the hydrostatic pressure from top to bottom which is equal to initial head loss  $H_0$ . Further operation of filter leads to more and more clogging resulting in further lowering of pressure. This lowering is felt immediately below the filter skin which could result in the development of negative pressure.

#### **Purification Mechanisms**

The principle removal mechanisms that occur in a slow sand filter as described as under:

- 1) Straining or Screening – The most obvious removal mechanism is screening in which the suspended solids having diameter greater than the pore size of the sand are retained on the uppermost layer of the sand. The pores of a tightly packed sand grains are able to retain particles of size greater than  $1/7^{\text{th}}$  the size of sand grains. Also some smaller particles which can otherwise pass through the sand, combine with other smaller particles and undergoes agglomeration during the twisting motion of water across the grains and becomes large enough to be deposited. The removal efficiency is also improved by the formation of schmutzdecke layer which further decreases the pore size.
- 2) Sedimentation – In a filter all the upward surface area of the sand is available. The sedimentation efficiency is a function of the ratio of surface loading rate and settling velocity. When the settling velocity is greater than surface loading rate complete removal may be achieved. Smaller and lighter particles are also removed due to flocculation.



**Figure 2.1. Transportation and sedimentation of heavier particles**

- 3) Inertial and centrifugal forces – The swirling twisting motion of water through the sand grains cause the particles heavier than water to leave the flow lines and settle in to the spaces between two sand grains shown in (Figure 2.1).
- 4) Diffusion (Brownian motion) – This phenomenon acts independently of the filtration rate. The suspended particles come into contact with the surface of the media. It occurs through the entire depth of the filter.
- 5) Mass attraction (Van der Waals force) – These forces are much weak forces than the chemical bonds. But at moderate distances these forces play a significant role in bringing particles close to each other. In a filtration system these forces are responsible for transport and attachment of suspended particles in the water to the sand grains. This force plays considerable role in holding the particles to the surface once they are attached.
- 6) Electrostatic and electro-kinetic attractive forces (Coulomb forces) – The force occurs between the oppositely charged particles in the water and surface of the sand media. This force may also contribute to the transport mechanism. However, when the charges are alike the particle will not be attached to the surface and will continue on its course. Owing to its origin clean quartz sand has a net negative charge on its surface. The particles of organic origin like bacteria are negatively charged and so there is very low removal in the initial stages. However after the operation of filter for some days there is accumulation of positive charge, after sometime the sand surface becomes over saturated with positive charge which leads to charge reversal and hence leads to the attraction of the negatively charged organics.
- 7) Biological Purification – During the ripening of the filter media the organics get arrested on the top surface of the filter and this surface becomes breeding ground for bacteria and other microorganisms. This causes development of a sticky gelatinous layer known as schmutzdecke layer. This layer arrests organic and also inert material until it is removed by sand bed cleaning operation. This deposited organic matter is used as food which is oxidized by bacteria for their metabolism (dissimilation) and a part of it is converted to living cells (assimilation). The biological activity is accompanied by equivalent dying off. The availability of organic matter is limited by flow rate. The organic matter is transported to the lower depths gradually. Below 30-40 cm there is very low biological

activity. Gradually all of the biodegradable matter is converted into carbon dioxide, water, inorganics like sulphates, nitrates and phosphates which is then discharged into the effluent. For biochemical oxidation to occur there should be sufficient oxygen and contact time which is obtained by keeping the filtration rate low. Anaerobic decomposition occurs when whole of the oxygen is depleted. This leads to the formation of ammonia, hydrogen sulphide and other odor producing substances.

## **2.6. NITROGEN REMOVAL BY NITRIFICATION, DENITRIFICATION AND ADSORPTION IN A SAND FILTER**

These days world over high concentration of nitrogen are found in ground water and surface water. Man-made activities like excessive use of fertilizers have contributed to the increased concentration of nitrogen in ground water majorly in the rural areas. Further discarding of waste water into rivers and lakes have resulted into increased nitrogen concentration in surface water bodies leading to eutrophication.

The inorganic forms of nitrogen are ammonium ( $\text{NH}_4^+$ ), nitrite ( $\text{NO}_2^-$ ), nitrate ( $\text{NO}_3^-$ ), nitrous oxide ( $\text{N}_2\text{O}$ ), and dissolved elemental nitrogen or nitrogen gas ( $\text{N}_2$ ). Gaseous nitrogen includes nitrogen gas ( $\text{N}_2$ ), nitrous oxide ( $\text{N}_2\text{O}$ ), nitric oxide ( $\text{NO}_2$ ), and free ammonia ( $\text{NH}_3$ ) (Tanveer Saeed, 2012).

Traditional methods for nitrogen removal from wastewater are biological processes (denitrification, nitrification), chemical processes (breakpoint chlorination, selective ion exchange) and physical operation (ammonia stripping). Other operations or processes are conventional treatment (primary, secondary), biological processes (bacterial assimilation, harvesting of algae, oxidation ponds), chemical processes (chemical coagulation, adsorption), physical operations (ammonia stripping, electro-dialysis, filtration, reverse osmosis), land application (irrigation rapid infiltration, overland flow) (Ne,se Öztürk, 2004). Nitrate removal has been achieved by adsorbents like natural zeolites and clays (M. Rozica, 2000), carbon based adsorbents, chitosan adsorbents, agricultural waste, industrial waste, etc. (Amit Bhatnagar, 2011).

The sand filters use the biological mechanisms for the removal of nitrogen present in the influent in the form of organic nitrogen, nitrates, nitrites and ammonical nitrogen. As the water percolates through the sand filter the particulate nitrogen is filtered by surface filtration, while the dissolved



nitrogen in the form of organics is oxidized by the bacteria cultures to nitrates. Ammonia removal takes place by volatilization in the form of  $\text{NH}_3$ . The oxygenation of the sand filter is ensured by gaseous exchange between the atmosphere and the interstices of sand when the surface of the filter is dry during the periods of rest (M. Achak, 2009).

The major elimination of nitrogen takes place by denitrification. But there can be intervention by the retention by the sand and/or volatilization of ammonium ions. Ammonia volatilizes by conversion of ammonium ions  $\text{NH}_4^+$  into volatile ammonia  $\text{NH}_3$ , particularly when pH of the influent varies between 7.8 and 8.4 and if the inorganic carbon ( $\text{CO}_2$ ) is low in the wastewater. A significant amount of  $\text{NH}_4^+$  removal takes place by adsorption on organic matter physically and chemically. As all the living organisms utilize nitrogen in the form of ammonia for cellular metabolism, therefore it is very much possible that the microbial assimilation had a very significant role to play in this removal, but this removal is limited by the available carbon. Nitrification consists indeed of the  $\text{NH}_4^+$  transformation into  $\text{NO}_2^-$  and finally into  $\text{NO}_3^-$  which causes to decrease the  $\text{N-NH}_4^+$  concentration.

The process of denitrification takes place in the anoxic zones and consists of transformation of  $\text{NO}_3^-$  into  $\text{NO}_2^-$  then  $\text{N}_2\text{O}$  and finally the nitrogen molecular  $\text{N}_2$ . The nitrogen molecular, thus obtained from nitrates by the breathing of denitrifying bacteria, evaporates then in the atmosphere.

The majority of bio-denitrification relies on heterotrophic bacteria that require an organic C-source (Sukru Aslan, 2007).

The carbon availability is very significant for the biological processes. (M. Achak, 2009). Also pretreatment can cause the removal of organics which could result in a decrease in the carbon availability (Hong-Duck Ryu, 2008). In a study by (Sukru Aslan, 2007) a low C/N ratio resulted in low nitrogen removal efficiency and high  $\text{NO}_3\text{-N}$  level at the end of the study. The optimum C/N ratio was found to be 1.8 when using acetic acid in batch tests. At C/N ratios below the optimum ratio the  $\text{NO}_3\text{-N}$  removal was dependent upon the C concentrations. Although no significant improvement in  $\text{NO}_3\text{-N}$  removal was observed when the C/N ratio was greater than the optimum value. Most of the  $\text{NO}_3\text{-N}$  removal was observed at the upper layer of the 10 cm filter bed. Lower  $\text{NO}_3\text{-N}$  concentration was observed for the filtration rates up to 0.03 m/h than the standard limit of 10 mg  $\text{NO}_3\text{-N/l}$ . Increasing the filtration rates to 0.04, 0.05, and 0.06 m/h, decreased the  $\text{NO}_3\text{-N}$  removal to 55%, 51%, and 47% at 10 cm and 100%, 99%, and 94% at 80

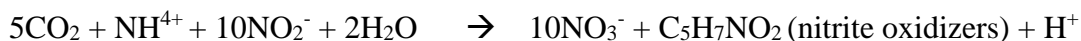
cm filter depth, respectively.  $\text{NO}_3\text{-N}$  concentration was still below the acceptable level for the drinking water at the filtration rates between 0.015 and 0.05 m/h at 10 cm filter depth. Alkalinity is produced during the conversion of nitrate to nitrogen gas resulting in an increase in effluent pH. The final pH at the effluent was slightly higher than initial pH and in the range of 7.6–8.5. Most of the  $\text{NO}_3\text{-N}$  was removed in the top layer of the sand filter. In another study at influent TKN and TN concentrations of 3–3.9 and 3.9–4.9 mg/l, respectively, the slow sand filters affected 42.4–78.4% nitrification, and 45–67.5% total nitrogen removal efficiencies, producing effluent TKN and TN concentrations in the range 0.6–1.75 and 1.5–2.7 mg/l respectively, at the low bed depth of 0.5m and high filtration rate of 0.38 m/h clearly demonstrates that the slow sand filter can be effectively used for tertiary wastewater treatment for simultaneous removal of organics and nitrogen (George Nakhla, 2003).

Denitrification can be brought about by both heterotrophic and autotrophic bacteria. Heterotrophic bacteria under anoxic conditions utilize organic substrates such as methanol, ethanol, and acetic acid for the conversion of nitrate to nitrogen. Gaseous organic substrates such as methane and carbon monoxide can also serve as substrates for the denitrification of water (Anoop Kapoor, 1997).

Adsorption technology has been found successful in removing different types of inorganic anions from waste waters by using various materials as adsorbents. It should be noted here that for achievement of optimum removal rates require selection of appropriate material for the removal of specific types of anions. Various conventional and non-conventional materials from different origins have been assessed for the removal of  $\text{NO}_3^-$  from water.

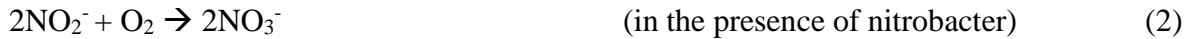
### **The process of nitrification and denitrification**

Nitrifying and denitrifying bacteria are equally prevalent in most of the aerobic biological systems. The oxidation of ammonia and nitrites is carried out by autotrophs known as nitrosomonas and nitrobacter respectively. If the gross composition of biomass is considered to be  $\text{C}_5\text{H}_7\text{NO}_2$  then the following equations represent the growth of the biomass:



### **Stoichiometry of biological nitrification**

The energy yielding process of nitrification occurs in two steps:

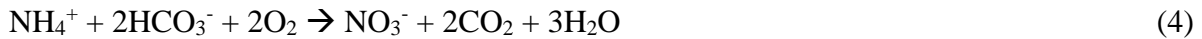


Total oxidation reaction:



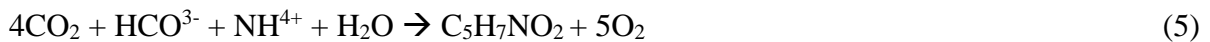
The stoichiometric quantities of oxygen required to oxidize 1 g of  $\text{NH}_3\text{-N}$  and 1 g of  $\text{NO}_2\text{-N}$  is 3.43 g and 1.14 g respectively.

The alkalinity required to carry out the process of nitrification can be calculated by the equation:



For each gram of ammonium (as nitrogen) converted, 7.14 g of alkalinity as  $\text{CaCO}_3$  will be required.

Along with obtaining energy a portion of ammonium is assimilated into cell mass. The synthesis reaction is as follows:



The half equations discussed above can be used to create a complete equation for nitrification:



From the above equation it can be deduced that for 1g of ammonia (as N) converted, 4.25 g of  $\text{O}_2$  is utilized, 0.16 g of new cells are formed, 7.07 g alkalinity as  $\text{CaCO}_3$  is consumed and 0.08 g inorganic carbon is utilized in the formation of new cells. However, the oxygen required is less than the theoretical value of 4.57 g calculated from the equation number (3) because the ammonia for cell synthesis is not considered in this equation. For the similar reason the alkalinity consumed is 7.07 g which is less than the theoretical value of 7.14 g (Bhavender Sharma, 1977).

### Parameters affecting nitrification

- 1) Dissolved oxygen: The stoichiometric quantities of oxygen required to oxidize 1 mg of  $\text{NH}_3\text{-N}$  and 1 mg of  $\text{NO}_2\text{-N}$  is 3.43 mg and 1.14 mg respectively (Bhavender Sharma, 1977).

- 2) Temperature: Nitrification is better in warm climate. The optimum temperature for the growth of nitrifying bacteria lies in the range 28-36<sup>0</sup>C. Temperature of 42<sup>0</sup>C has been reported for nitrobacter. Studies have revealed that a 4<sup>0</sup>C increase in the temperature lead to a 50 % increase in the rate of nitrification while a 1.5<sup>0</sup>C decrease lowered the rate by 8 % (Bhavender Sharma, 1977).
- 3) Effect of pH: The process of nitrification is sensitive to pH. The rate of nitrification is reduced significantly below the pH of 6.8. The rate at pH=6 is 10-20% of the rate at pH=7. (Metcalf and eddy). The pH of 7.5-8 corresponds to optimum rate of nitrification. For the wastewaters having low alkalinity, alkalinity is added.
- 4) Effect of ammonia and nitrite ion concentration-Substrate and product inhibition: Both the groups nitrobacter and nitrosomonas are sensitive to their substrate and each other's substrate. Ammonia-ammonium and nitrite-nitrous acid equilibria influences the degree of inhibition. Ammonia and un-dissociated nitrous acid have significant effect on the inhibition of nitrification. However, normal nitrite and ammonia ion concentration is not in the inhibiting range in domestic wastewaters.
- 5) Concentration of nitrifies: The rate of nitrification is greatly influenced by the concentration of nitrifying bacteria. The initial rate of ammonia oxidation increases with increase in initial seed concentration in VSS. The rate of nitrification also depends upon the ammonia to nitrifier ratio.
- 6) Sludge age, organic loading and detention time: Other parameters being constant the degree of nitrification depends greatly on organic loading rate. Nitrification is found to decrease with increase in loading rate. 3-4 days of sludge age is required for achieving high degree of nitrification. The detention time usually employed for suspended growth systems is greater than attached growth systems. The detention time and organic loading rate are the tools to achieve required rate of nitrification.
- 7) Effect of Surfaces and turbulence: De Marco concluded that high turbulence causes an increase in the rate of ammonia oxidation. It was found that mud interface contained 450 times as many oxidizers and 256 times as many nitrite oxidizers as did surface water on a volumetric basis. In the seas generally the nitrifiers are found in the sand bed. It was also found that surface muds of stratified lakes are good source of nitrifiers.
- 8) Effect of light: Light is found to inhibit the activity of nitrifying bacteria.

- 9) Micronutrients: There are a number of other compounds that stimulate or inhibit the rate of nitrification. The inhibitors may owe their action to the products formed after the attack of microbes.
- 10) Organic matter as inhibitor: Organic matter is found to depress the activity of nitrifiers. An increase in carbon: nitrogen ratio is found to decrease the efficiency of nitrification. This effect may be due to dissolved oxygen concentration limits due to competition between nitrifiers and common heterotrophs. In a study the optimum C/N ratio was found to be 1.8 for NO<sub>3</sub>-N removal. At lower ratios than optimum NO<sub>3</sub>-N removal was found to depend upon carbon present but at higher ratios no significant removal was observed (Sukru Aslan, 2007).
- 11) Adaptability and microbial interactions: The adaptability can be known from the culture history and symbiotic effects in mixed cultures.

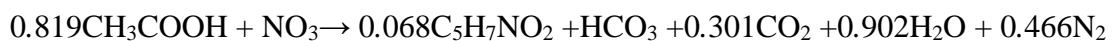
### **Stoichiometry of the biological denitrification**

Biological denitrification (BD) occurs naturally when certain bacteria (nitrobacter) use nitrate as a terminal electron acceptor in their respiration process under anoxic conditions i.e. absence of oxygen. Denitrification is composed of a sequence reactions conducted by different enzymes.

During denitrification nitrate is transformed into nitrogen gas, with formation of a number of nitrogen intermediates like  $\text{NO}_3^- \rightarrow \text{NO}_2^- \rightarrow \text{NO} \rightarrow \text{N}_2\text{O} \rightarrow \text{N}_2$ .

Denitrification involves conversion of nitrate to nitrogen gas; hence it allows closing of the natural nitrogen cycle. Denitrification can be achieved in autotrophic and heterotrophic ways, and both can be developed under anaerobic conditions. Heterotrophic denitrification (HD) requires an organic carbon rich environment, which can be supplied by soluble or insoluble carbon source. As soluble carbon sources sucrose, methanol, ethanol, etc., are the most widely cited ones, while as insoluble carbon sources vegetable oil, natural organic substrates such as liquorice, giant reed, wheat straw and cotton, bio-polymer and methane can be used. Autotrophic denitrification (AD), based on an inorganic carbon source (CO<sub>2</sub>), involves sulfur or hydrogen gas as the electron donor for the bacterial metabolic chain (Claudio Della Rocca, 2007).

The biological denitrification using acetic acid as a carbon source including assimilation can be explained as:



The reduction of 1g of  $\text{NO}_3$  produces 0.55 g of new cells (Sukru Aslan, 2007).

### **Parameters affecting denitrification**

- 1) pH: During nitrification alkalinity is consumed which could result in a substantial drop in pH which could hinder denitrification. Denitrification can be hindered at pH<6 and pH>8. There is maximum denitrification in the range of 7.5-8 pH. Methane forming bacteria operate at the range of 6.5-7.5 and a deviation from this range could result in the inhibition of denitrification.
- 2) Availability of oxygen: Denitrification requires essentially anoxic conditions i.e. complete absence of oxygen.
- 3) Availability of carbon source: In the sand filter, significant nitrate elimination can be obtained when the effluent from the biological unit include sufficient organic carbon.
- 4) Temperature: The process of denitrification is found to increase until an optimal temperature of 40 °C is reached.
- 5) Hydraulic loading rate and retention time: The increase in the hydraulic loading rate leads to a lower retention time of the waste water and there is quick passage of water from the bed. This lowers the contact time and a reduction in the rate of denitrification.

### **2.7. PHOSPHATE REMOVAL**

Phosphorus is an essential nutrient for the growth of algae and other biological organisms. The amount of phosphorus compounds in domestic and industrial discharges must be controlled using either chemical or biological techniques otherwise the rampant discharge of such waste water would cause algal blooms resulting in the eutrophication of the surface water bodies. Municipal wastewater may contain from 4 to 15 mg/l phosphorus as P but industrial wastewaters may contain phosphate levels well in excess of 10 mg/l (Yildiz, 2004). The usual forms of phosphorus found in aqueous solutions include orthophosphate, polyphosphate and organic phosphate. (Mevra Yalvac Cana, 2006). In temperate fresh waters, dissolved phosphorus is the limiting nutrient (Andrew J. Erickson, 2007).

Phosphorus can be treated by biological treatment, chemical treatment, chemical precipitation, crystallization technologies, sludge based process and many other processes in vogue (G.K. Morse, 1998). Mechanisms such as exchange, adsorption and precipitation have been proposed to explain the removal of phosphate from aqueous solutions. Adsorption and chemical precipitation among the above methods have been widely used for phosphate removal. The removal of phosphate from aqueous streams consists of the conversion of soluble phosphate to

an insoluble solid phase. This solid phase can be separated from water by means of sedimentation or filtration. In all cases, phosphorus is removed by converting the phosphorus ions in wastewater into a solid fraction. This fraction can be an insoluble salt precipitate, a microbial mass in an activated sludge, or a plant biomass in constructed wetlands (Luz E. de-Bashan, 2004).

### *1. Chemical precipitation*

In wastewater applications, the most common and successful methods to precipitate phosphate involve the dissolved cations  $\text{Al}^{3+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Fe}^{3+}$  and to a lesser extent of  $\text{Fe}^{2+}$  (Yildiz, 2004). Chemical precipitation is a physico-chemical process, comprising the addition of a divalent or trivalent metal salt to wastewater or passing the water through such filter media having divalent or trivalent metal ions, causing precipitation of an insoluble metal phosphate that is separated by sedimentation. Chemical precipitation typically produces phosphorus bound as a metal salt within the wasted sludge. It therefore has potential value to be used in agriculture (G.K. Morse, 1998).

### *2. Biological removal*

The conventional biological treatment processes are disadvantageous because they have limited phosphate removal capability because microbes utilize phosphorus during cell synthesis and energy transport. As a result 10–30% of the influent phosphorus only can be removed during biological treatment (Mevra Yalvac Cana, 2006). It is known that activated sludge takes up phosphorous for biomass growth. An anoxic zone ahead of an aerobic zone is required in the activated sludge process.

### *3. Miscellaneous removal technologies*

The removal of phosphate has also been done by adsorption on fly ash (S.G. Lu, 2009), fly ash using crossflow microfiltration (Yildiz, 2004), adsorption on polonite (Agnieszka Renman, 2010), adsorption using iron tailings (Le Zenga, 2004). Investigations on phosphate removal using different adsorbent such as fly ash slag and bagasse shows that phosphate precipitates with calcium as apatites and especially hydroxapatites (HAP). It was found that the extent of HAP formation is critically dependent on factors such as degree of super saturation, pH and initial phosphate concentration (Mevra Yalvac Cana, 2006). It was reported by (S.G. Lu, 2009) that the removal of phosphorus by using fly ash was primarily due to the adsorption and precipitation of phosphorus as calcium phosphate on fly ash.



The removal of phosphate depends on many environmental factors like pH, nitrates, BOD, aeration, retention time, contact time, etc. The removal of phosphate is accompanied by a decrease in the  $\text{Ca}^{2+}$  ion concentration and pH value (S.G. Lu, 2009). It was also reported that significant difference in the efficiency of phosphate removal among different fly ashes was attributed to the different Ca content and pH of fly ashes. The pH of the solution is found to play a critical role in the rate of removal and mechanism of the removal of phosphate (S.G. Lu, 2009). In another study when the initial pH increased the removal efficiency increased but after reaching pH=6 it reduced. (Mevra Yalvac Cana, 2006)

### **3. METHODOLOGY**

This chapter discusses the procedure of model setup involving its location, sample collection, design of filter bed, operation of setup and laboratory analysis of the raw and treated water.

#### **3.1. LOCATION AND TIMING**

The greywater sample was collected from Gargi girls' Hostel, MNIT Campus, Jaipur. It was collected in the morning hours between 9-10 a.m. The sample was collected in plastic cans of 5 L capacity and was brought to the Public Health and Environment Lab in the Civil Engineering department, MNIT, 5-10 minutes after collection. The treated water after 6 hours of filtration was collected in glass beakers and was tested for various parameters. The filtered sample was stored under a temperature of 4<sup>0</sup>C in case of delay.

#### **3.2. REACTOR DESIGN**

The column was created using plastic measuring cylinder. The dimensions of the column is shown in (Figure 3.3).

Total capacity of cylinder = 1000 ml

Total height of the cylinder = 42 cm

Diameter = 6.5 cm

Area of the column = 33.16 cm<sup>2</sup>

Diameter of the inlet and outlet holes = 1 cm

Height of the sand bed = 23 cm

The greywater was poured into the filter column using peristaltic pump maintained at constant RPM (Figure 3.2). The schematic representation of the setup is shown in (Figure 3.1). The treated water was collected into a glass beaker. Experiments were performed on the constant loading rates of 10 ml/min (181 L/hr/m<sup>2</sup>) and 20 ml/min (362 L/hr/m<sup>2</sup>), the corresponding speeds in the pump were 3.1 and 6.2 RPM respectively. The setup was run for 6 hours every day. Another setup was run for constant head studies of 3 cm and 5 cm. The head was maintained by creating two outlets each at an elevation of 3 cm and 5 cm above the top sand layer. The same setup was used in a thesis titled "Assessing the efficacy of sand filtration for greywater treatment" by Ms. Swati Singh.

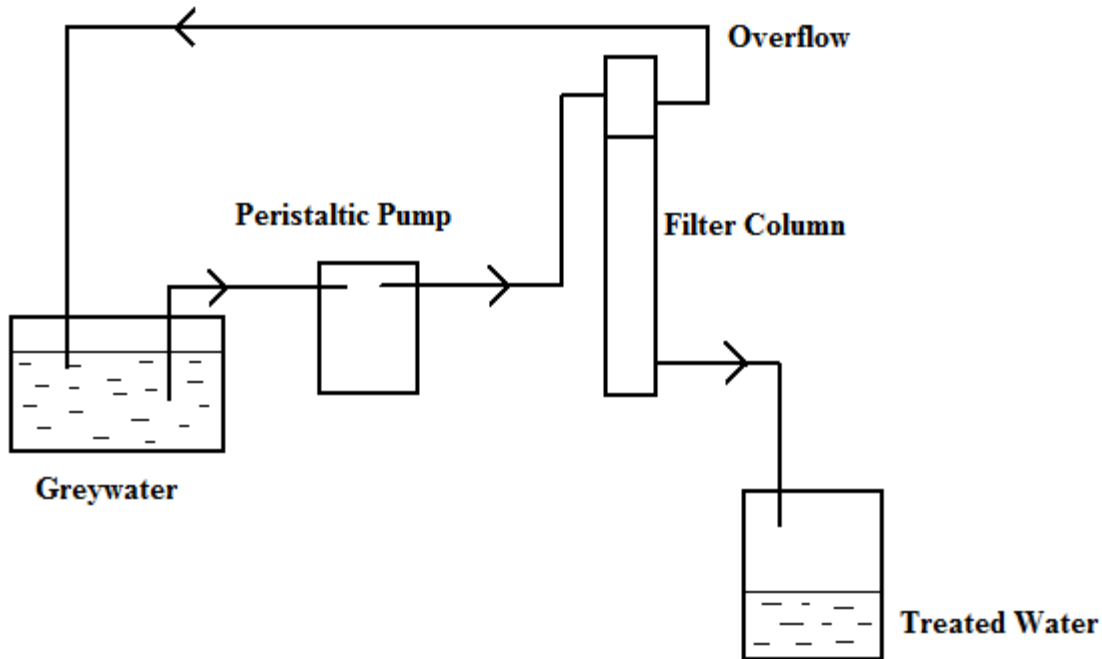


Figure 3.1. Schematic diagram of the setup

### Mode of application

Proper choice of feed mode is extremely important, as it can maximize wastewater mixing through the packed media. Till now different types of influent feed modes have been used such as: continuous, intermittent, batch, tidal, and step feed mode.

Intermittent loading can enhance the removal of organics and nitrogen. Alternative dry and wet periods can enhance the diffusion of oxygen into the media. In this study intermittent mode of feed was used.

In a study it has been noted that denitrification improved in the system (i.e. 89%), when the load was intermittently fed (e.g. 4 times a day), coupled with external carbon addition. However, when the same wetland was operated under continuous loading, with the previous carbon addition mode (i.e. 4 times a day), denitrification was significantly reduced. The authors hypothesized that, the uneven distribution of organic carbon during continuous mode might have limited  $\text{NO}_3^-$  N reduction in their system. The increased removal rates could be due to the

applied flushes (during intermittent feeding) induced greater turbulence inside the media, allowing passage of wastewater through aerobic and anaerobic zones (Tanveer Saeed, 2012).



**Figure 3.2. Actual laboratory setup**

### **Filter Media**

The locally available sand from the MNIT campus was taken for making the setup an economical one. Freshly excavated sand was used. It was then washed and sieved. The fraction of sand of size 150-300  $\mu\text{m}$  was used in the filter column. The sand was supported on gravel bed (Figure 3.4). The size gradation and depth of sand and gravels is given in (Table 3.1).

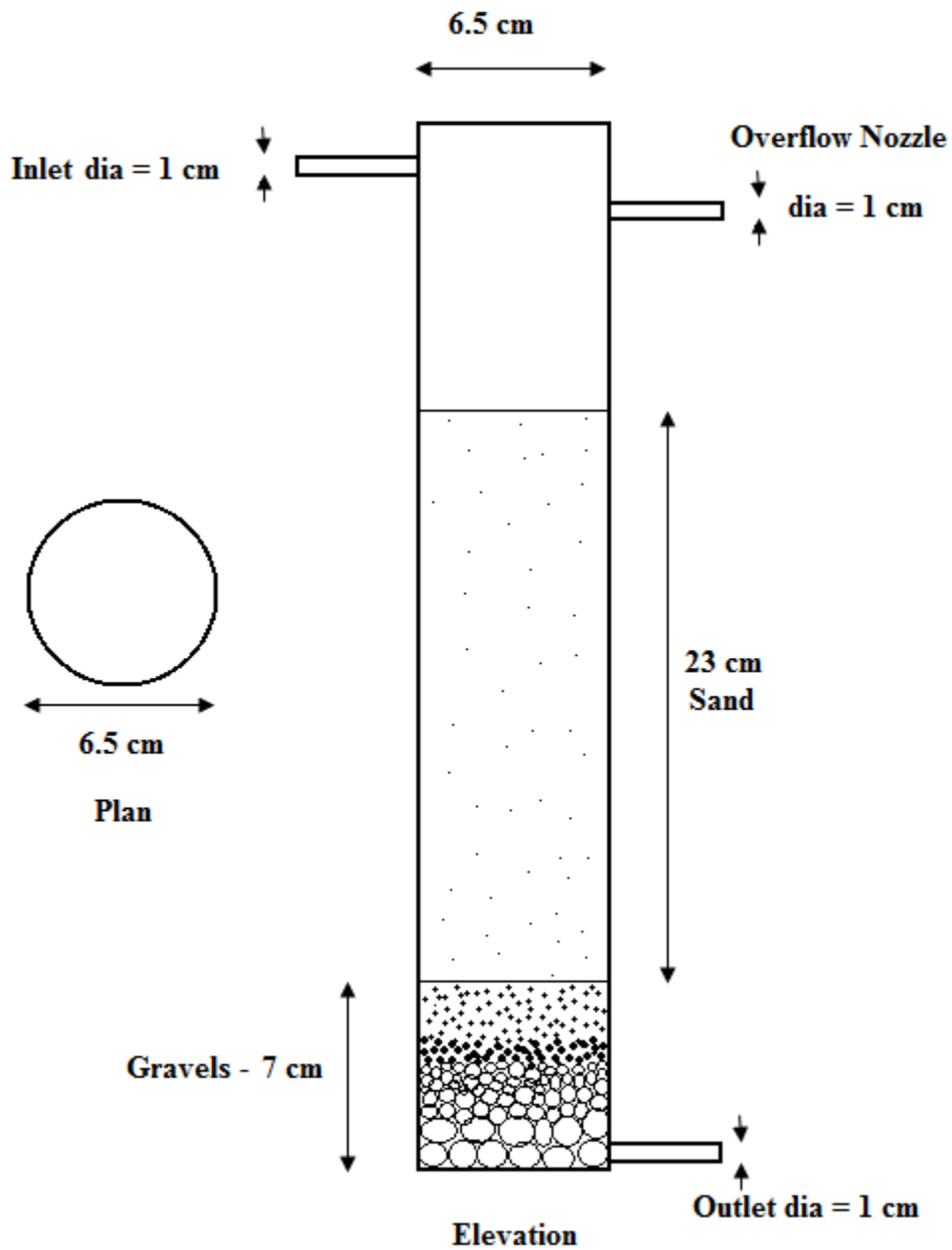


Figure 3.3. Plan and elevation of the filter bed

**Table 3.1. Gradation of media (Top to bottom)**

<b>S.No.</b>	<b>Media</b>	<b>Depth (cm)</b>	<b>Size</b>
1	Sand	23	150 – 300 $\mu\text{m}$
2	Gravel	0.5	300 – 600 $\mu\text{m}$
3	Gravel	1	0.6 – 1.18 mm
4	Gravel	1.5	1.18 – 2.36 mm
5	Gravel	2	10 – 15 mm
6	Gravel	2	3 – 2 cm



**Figure 3.4. The sand and gravel layers in the filter bed**

### 3.3. LABORATORY ANALYSIS

The treated and raw greywater was analyzed for TKN, ammonical nitrogen, phosphorous, nitrates, pH and alkalinity.

#### a) TOTAL KJELDAHL NITROGEN (TKN)

**TKN = Organic nitrogen + Ammonical nitrogen**

##### Principle

The sample was digested in the presence of  $H_2SO_4$ , potassium sulfate ( $K_2SO_4$ ), and cupric sulfate ( $CuSO_4$ ) catalyst to convert amino nitrogen of many organic materials to ammonium. The free ammonia also converts to ammonium. Distillation was performed after addition of base (NaOH), and absorbed in boric acid. Then, ammonia was determined by titration with a diluted acid.

##### *Digestion:*

- To 20 ml treated wastewater in a distillation flask, 10 mL conc.  $H_2SO_4$  was added with 5g mixture of  $K_2SO_4$  and  $CuSO_4$  (in 4: 1) (3.33g  $K_2SO_4$  + 0.67g  $CuSO_4$ ).
- The sample was digested in a distillation assembly (Figure 3.5) which raised the temperature of the digestion mixture maximum up to  $420\ ^\circ C$ .
- After digestion the mixture was cooled to room temperature and diluted to 100 mL with distilled water.

##### *Distillation:*

- 20 mL 40% NaOH was added to 10 ml of the digested sample and the volume was made to 150 ml. This mixture was poured into a distillation flask. Similarly a blank sample was prepared by taking 20 ml distilled water instead of sample.
- In another conical flask 25 ml boric acid with indicator was poured.
- Flask was connected to a steamed-out distillation apparatus (Figure 3.6) and 150 ml condensate was collected into the conical flask.
- As the ammonia gets absorbed into boric acid, the dark purple color changed to green.

##### *Titration:*

- Titration was performed on the green colored solution obtained in the conical flask with 0.02 N  $H_2SO_4$  till the color changed to pink.

A= ml of H<sub>2</sub>SO<sub>4</sub> used to titrate sample B= ml of H<sub>2</sub>SO<sub>4</sub> used to titrate blank.

$$\text{mg of NH}_3 - \text{N} = \frac{(A - B) \times 280}{\text{ml of sample}(20 \text{ ml})}$$



Figure 3.5. TKN Digestion assembly



Figure 3.6. TKN distillation unit



**b) AMMONIACAL NITROGEN:** Phenate method was used for ammonia measurement as per (APHA, **Standard Methods for the Examination of Water and Wastewater 1999**) (4500-NH<sub>3</sub> F)

*Principle:* An intensely blue compound, indophenol, is formed by the reaction of ammonia, hypochlorite, and phenol catalyzed by sodium nitroprusside.

#### **Procedure**

- To a 25-mL treated wastewater sample in a 100mL beaker was added, with thorough mixing after each addition, 1 mL phenol solution, 1 mL sodium nitroprusside solution, and 2.5 mL oxidizing solution. Similarly blank sample was prepared by taking 25 ml distilled water instead of sample.
- The sample was covered with paraffin wrapper film.
- Color was allowed to develop at room temperature (22 to 27°C) in dark for at least 1 h.
- Absorbance was measured at 640 nm using spectro photometer (Figure 3.9).

**c) PHOSPHORUS:** Stannous Chloride Method was used as per (APHA, **Standard Methods for the Examination of Water and Wastewater 1999**) (4500-P D. Stannous Chloride Method)

*Principle:* Phosphates that respond to colorimetric tests without preliminary hydrolysis or oxidative digestion of the sample are termed “reactive phosphorus.” While reactive phosphorus is largely a measure of orthophosphate, a small fraction of any condensed phosphate present usually is hydrolyzed unavoidably in the procedure. Reactive phosphorus occurs in both dissolved and suspended forms.

#### **Procedure**

- To 50 mL treated wastewater sample, 0.5 g K<sub>2</sub>S<sub>2</sub>O<sub>8</sub>, 1 ml strong acid (H<sub>2</sub>SO<sub>4</sub> + HNO<sub>3</sub>), 5-7 drops phenolphthalein indicator were added.
- The sample was digested (Figure 3.7) till its volume reduced to 30 ml.
- To the digested sample was added 5-7 drops phenolphthalein and was titrated against 1 N NaOH till the color changed to pink.
- 5 ml of the titrated sample was taken and the volume was made to 100 ml. Similarly a blank sample was prepared by taking 100 ml distilled water.
- To each solution 4.0 mL molybdate reagent and 0.5 mL (10 drops) stannous chloride reagent were added.

- After 10 min, but before 12 min, color was measured photometrically at 690 nm.



Figure 3.7. Phosphate digestion assembly

#### d) NITRATE

The nitrates were determined using electrode method (Figure 3.8).

- The standard nitrate solutions were prepared: 1.3709 g of  $\text{NaNO}_3$  was dissolved in 1000 ml to make 1000 ppm solution. Using successive dilutions were prepared 100 ppm, 10 ppm, 1 ppm and 0.1 ppm solutions. 50 ml measured volume of standard solutions was taken in 100 ml beaker. 50 ml of the sample was taken in a 100 ml beaker.
- 1 ml ISA was added to the standard solutions and samples.
- The electrode was calibrated using standard solutions and then reading was taken.



Figure 3.8. Nitrate electrode



Figure 3.9. Shimadzu Spectrophotometer

e) **pH:** pH of the treated wastewater sample was measured using pH meter available in the PHE laboratory, MNIT, Jaipur.

**Procedure**

The pH meter was calibrated by immersing the electrode in the buffer solution of known pH, normally 4.0 and 7.0.

- The electrode was rinsed with distilled water.
- The pH of the treated water sample was read.

f) **ALKALINITY:** The titration method was used as per (APHA, Standard Methods for the Examination of Water and Wastewater 1999) (2320 B. Titration Method).

**Procedure**

- 25 ml of treated wastewater sample was used for testing.
- 2- 3 drops of Phenolphthalein solution, alcoholic, pH 8.3 indicator was added. If pink color is observed phenolphthalein alkalinity is present.
- Titration was performed with 0.02 N H<sub>2</sub>SO<sub>4</sub> till the color disappeared. The reading was noted and phenolphthalein alkalinity (P) was calculated. Where , A = ml of acid used N = normality of the acid
- Then 2-3 drops of methyl orange solution were added, pH 4.5 indicator. Pale yellow color was observed.
- Again titration was performed with 0.02 N H<sub>2</sub>SO<sub>4</sub> till orange color appeared. Note the reading and calculate total alkalinity (T).

$$\text{Alkalinity , mg CaCO}_3/\text{L} = \frac{\text{B} \times \text{N} \times 50 \times 1000}{\text{ml of sample}}$$

Where, B = ml of acid used      N = normality of the acid

Calculation of alkalinity relationships: The results obtained from the phenolphthalein and total alkalinity determinations offer a means for stoichiometric classification of the three principal forms of alkalinity present in many waters. The classification ascribes the entire alkalinity to bicarbonate, carbonate, and hydroxide. According to this scheme:

**1. Carbonate (CO<sub>3</sub><sup>2-</sup>) alkalinity** is present when phenolphthalein alkalinity is not zero but is less than total alkalinity (P<T).

**2. Hydroxide (OH<sup>-</sup>) alkalinity** is present if phenolphthalein alkalinity is more than half the total alkalinity ( $P \geq 1/2 T$ ).

**3. Bicarbonate (HCO<sub>3</sub><sup>-</sup>) alkalinity** is present if phenolphthalein alkalinity is less than half the total alkalinity ( $P < 1/2$ ).

### 3.4. INSTRUMENTS USED

The various instruments used for the laboratory analysis are given in (Table 3.2).

**Table 3.2. Instruments used in the laboratory**

S.N.	Instrument	Company	Model
1.	Digital pH Meter	Labtronics	LT-11
2.	UV Spectrophotometer	Shimadzu	UV-1800
3.	Microwave	Samsung	
4.	TKN Unit	Kelplus	KES04LE
5.	Weighing balance	CAS	CAUW220D
6.	Soxhlet unit	Khera Instruments pvt. ltd.	
7.	Distilled water unit		
8.	Peristaltic Pump	Entertech	ENPD 1000 Victor
9.	Nitrate electrode	Thermo scientific Orion 5 star	

## **4. RESULTS AND DISCUSSION**

### **4.1. GREYWATER CHARACTERIZATION**

**Table 4.1. Greywater characterization**

<b>Parameter</b>	<b>Mean</b>	<b>Standard Deviation</b>	<b>N (Sample size)</b>	<b>Maximum values reported</b>	<b>Reuse Guidelines (CPHEEO)</b>
pH	7.35	0.32	21	7.83	6.5-8.3
Phosphates (mg/L)	0.107 as PO <sub>4</sub> <sup>-</sup>	0.058	20	0.193	1 as P, 3.07 as PO <sub>4</sub> <sup>-</sup>
Nitrates (mg/L)	8.11	4.68	21	17.7	10
TKN (mg/L)	4.97	6.16	17	23	10
Ammonical Nitrogen(mg/L)	2.05	0.81	13	3.38	--
Alkalinity (mg/L)	660	55.86	5	752	--

The coliform count of the greywater was  $>9.2 \times 10^5$  CFU/100ml. Further tests of coliforms could not be conducted due to unavailability of resources. The pH, phosphates, TKN, ammonical nitrogen and nitrates showed compatibility with the data reported in the literature (Table 4.1). The pH of the greywater was alkaline. The maximum pH was reported to be 7.83. The raw pH was already in the limits prescribed for reuse. The TKN is also very low indicating a deficiency of nutrients. The raw water TKN concentration was maximum 23 mg/l which is greater than reuse guideline of 10 mg/l. The concentration of phosphates was also very low, maximum being 0.193 mg/l as PO<sub>4</sub><sup>3-</sup>, which is way below the reuse standards. The major contributor of phosphates are detergents where the phosphate containing detergents are still being used. A low concentration of phosphates indicates high per capita use of water. Concentrations between 6 and 23 mg Tot-P/l can be found in traditional wastewaters in areas where phosphorus detergents are used. However, in regions where non-phosphorus detergents are used the concentrations range between 4 and 14 mg/l. In bathroom grey wastewater the total phosphorus and phosphate concentrations were, 0.1–57 and 0.1–2 mg/l, respectively (Eva Eriksson, 2002). The (Washington state department of health, 2009) reported mean of total P to be 2.8 mg/l in the greywater including kitchen waste water and laundry waste water. The total P concentration of 1.37 mg/l in greywater were reported (B. Jefferson, 2004). Although the mean concentration of

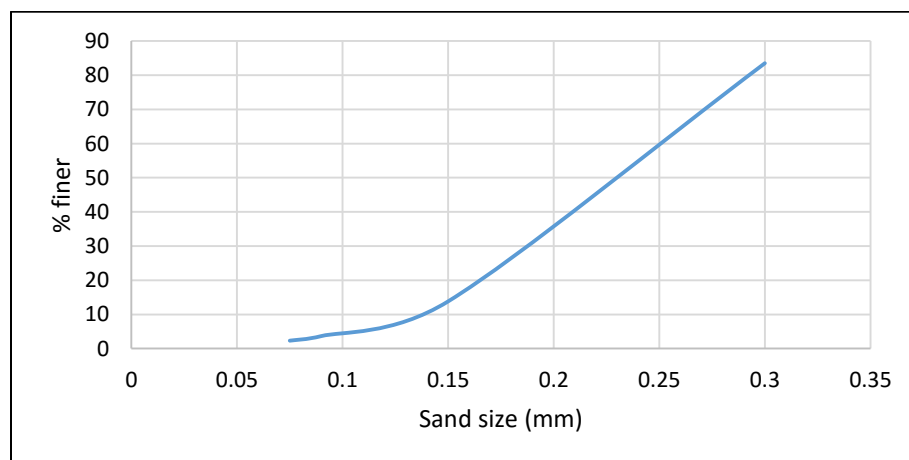
nitrate was 8.11 mg/l which is just below the reuse guidelines, the maximum concentration of nitrate went as high as 17.7 mg/l, which is above the reuse guidelines. It appears that TKN and nitrate are major concerns for removal. Further ammonical nitrate removal becomes important so that a reduction can be obtained in TKN concentration as TKN includes organic and ammonical nitrogen. Phosphates being very low does not pose a threat for reuse.

#### 4.2. SAND CHARACTERIZATION

The sand was collected from the MNIT campus. The pH of sand was found out to be 8.05 (sand and distilled water in 1:1 ratio). The conductivity was 0.211  $\mu\text{S}/\text{cm}$  (sand: water = 1:2.5). The sand was washed and sieved. The fraction of sand of size 150-300  $\mu\text{m}$  was used for filter bed. The bulk density of sand was 1.43  $\text{g}/\text{cm}^3$ . A total of 2 kg sand was used for sieve analysis (Table 4.2) (Figure 4.1).

**Table 4.2. Sieve Analysis**

Sieve size ( $\mu\text{m}$ )	Weight (g)	% weight retained	Cumulative weight retained %	% Finer
300	330	16.5	16.5	83.5
150	1393.63	69.68	86.18	13.82
90	202.77	10.14	96.32	3.68
75	27	1.35	97.67	2.33
<75	36.88	1.84	99.5	0.49



**Figure 4.1. Sieve Analysis**

The majority fraction of sand was in the range of 150-300  $\mu\text{m}$  (69.68 %). The  $D_{10}$  and  $D_{60}$  size were 0.127 mm and 0.2 mm. The uniformity coefficient ( $C_u$ ) was 1.57. Soils with a value of  $C_u$

less than 2 are uniform soils. The MIT system of classification of soils classify the particle size of 0.06 mm to 2 mm as sand. In our study the particle size is 0.15 mm to 0.3 mm. So it is a sandy soil. The Indian Standard classification system classifies soils into three types:

1. Coarse grained, when 50% or more of the total material by weight is retained on 75 $\mu$  (0.075 mm) IS sieve.
2. Fine grained, when more than 50% passes 75 $\mu$  IS sieve.
3. Organic soils

In this study more than 50% was retained on 150 $\mu$  IS sieve. So the soil in this study is sandy soil.

### **4.3. RETENTION TIME**

Assuming that the greywater while passing through the filter used entire volume of sand, the initial theoretical retention time was 71.7 minutes and 35.8 minutes for 10 ml/min and 20 ml/min loading rates respectively.

### **4.4. pH**

The mean pH of the untreated greywater was 7.48 which indicates that the greywater was alkaline which conforms well to the literature sought. The pH of the treated greywater conformed well to the pH recommended by reuse guidelines. A number of reactions responsible for the removal of various nutrients and organics depend on the pH of the waste water. During nitrification alkalinity is consumed. The optimum pH for the nitrification to occur lies in the range 7.5-8. Hence, if after the treatment of waste water there is a reduction in the pH of the waste water it can be assumed that a significant nitrification is taking place. During nitrification alkalinity is consumed which could result in a substantial drop in pH which could further hinder denitrification. Denitrification can be hindered at pH<6 and pH>8. During denitrification alkalinity is generated. But here, there is very low possibility of denitrification due to absence of anoxic conditions. In case of constant flow rate studies the pH of the effluent was observed to be more than the influent. This surge of pH could be due to the alkalinity of soil due to presence of carbonates and bicarbonates, as the pH of soil was 8.05.

In case of constant head study the pH of the treated and the untreated greywater is nearly the same (Figure 4.2). Alkalinity is consumed in the process of nitrification, when ammonia is oxidized while it is produced in the case of denitrification. So it can be deduced that nitrification is taking place but denitrification is not taking at an appreciable rate. The reason for the

denitrification not taking place could be due to absence of anoxic conditions. Comparing the constant head studies and constant flow rate studies the driving force is more in the case of constant flow rate studies. This driving force is responsible for turbulence and makes it possible for the waste water to reach all the aerobic and anaerobic zones resulting in an efficient nitrification and during its course the water was able to dissolve carbonates and bicarbonates resulting in an increase in the pH. The optimum range of pH for nitrification to occur is 7.5-8. The pH of the greywater varied from 7 to 8 which shows that conditions were favorable for nitrification to take place in the sand filter. pH also plays a role in adsorption. Acidic pH promotes the adsorption of anions and basic pH promotes the adsorption of cations. This basic pH could have played a role in the adsorption of ammonium ions. Also the removal of phosphate is accompanied by a decrease in the  $\text{Ca}^{2+}$  ion concentration and pH value (S.G. Lu, 2009) The dependence of removal of various parameters on pH are discussed separately in the following sections.



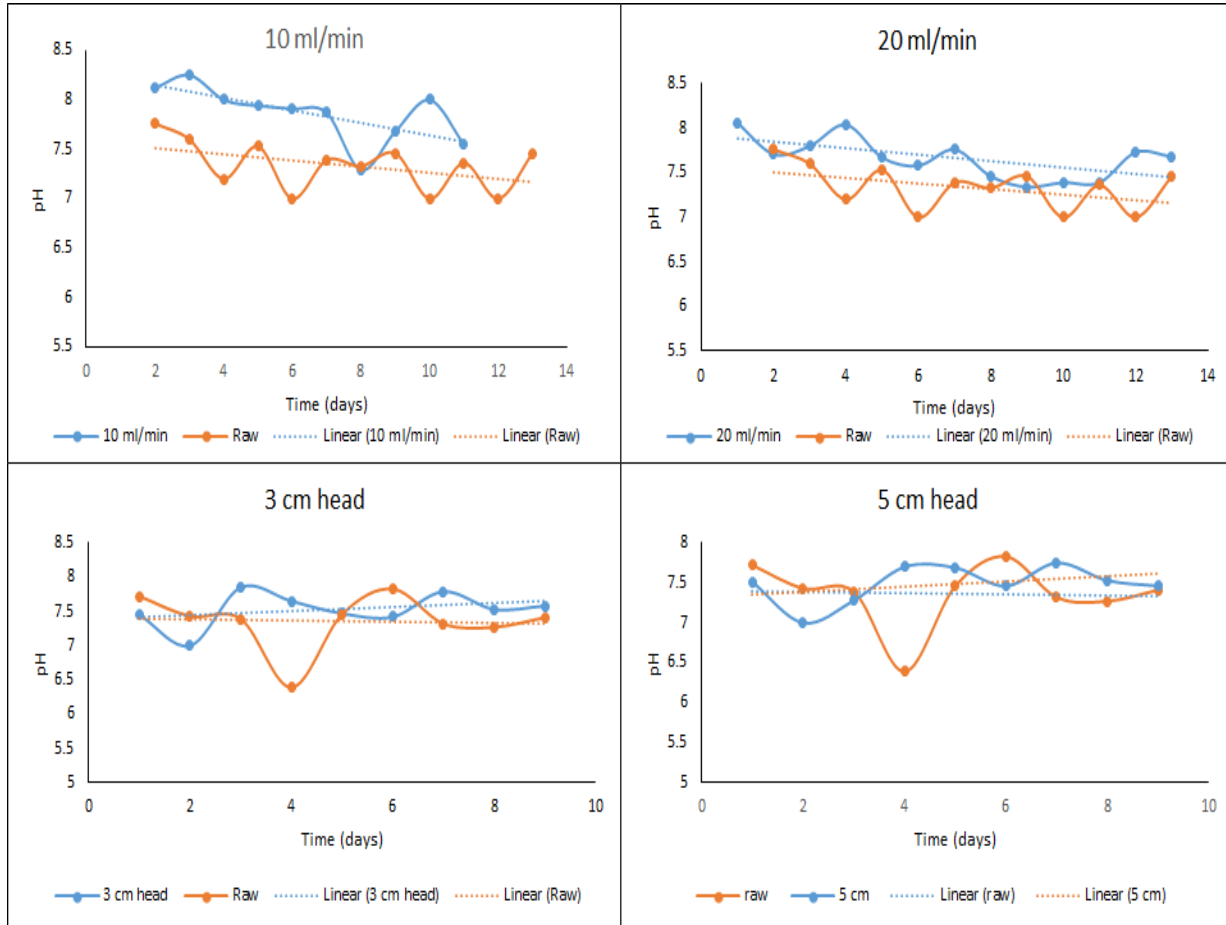
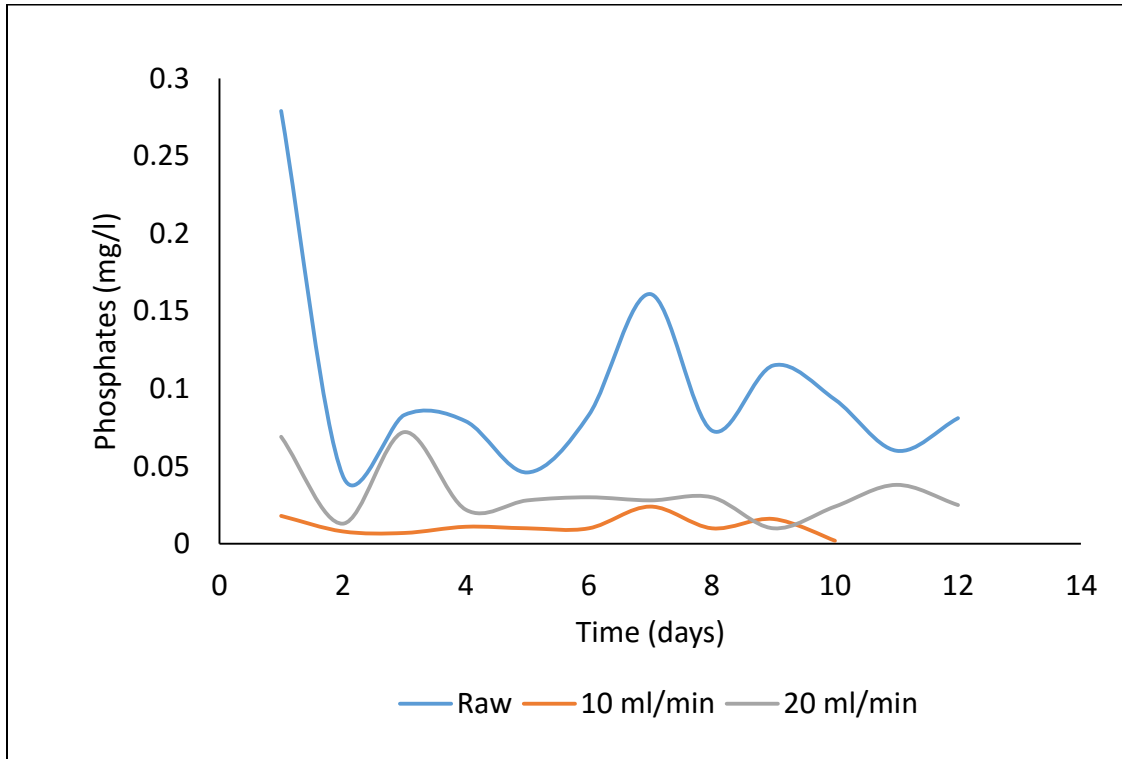


Figure 4.2. Evolution of pH with time

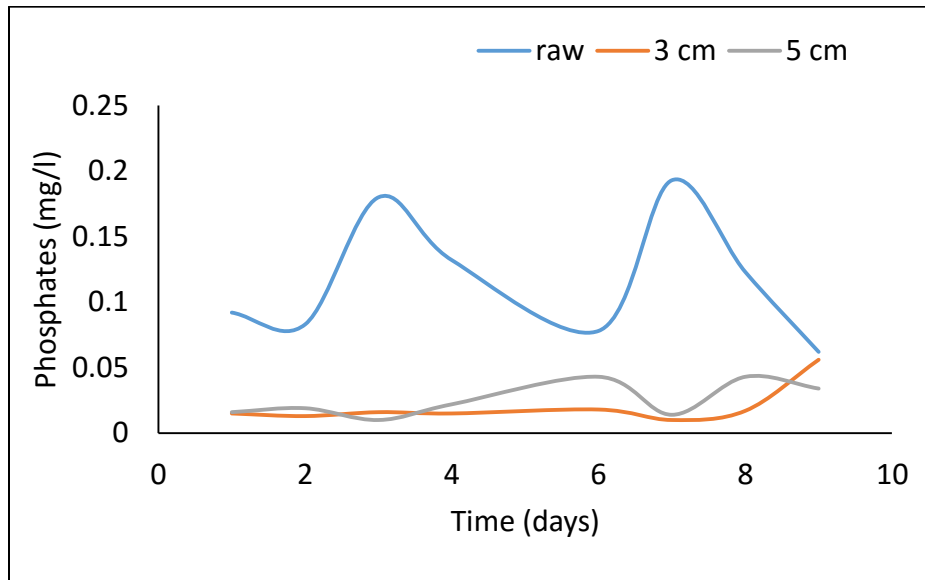
#### 4.5. PHOSPHATE REMOVAL



**Fig.4.3. Phosphate concentration of effluent with time (constant flow rate)**

The CPHEEO manual recommends a value of 1 mg/l as phosphorous and 3.07 mg/l as phosphates for reusing the greywater for flushing purposes. The mean concentration of phosphates in the raw greywater in this study was 0.107 mg/l. This concentration was already way below the prescribed standards and also after sand filtration the total phosphates went as low as 0.002 mg/l.

The removal of phosphates can be attributed to the adsorption on the positively charged ions present in the sand. The maximum removal that occurred was 97.84 % and 91.30 % for the loading rate of 10 ml/min and 20 ml/min respectively. The high removal efficiency of the former filter can be attributed to the fact that it offers more contact time for adsorption to occur. The results indicate that better removal is obtained by the lower loading rate of 10 ml/min. In the (Figure. 4.3) it can be seen that there is a slight increase in the concentration of phosphates after 8 days which could be due to the progressive bed exhaustion.



**Figure 4.4. Phosphate concentration of treated and untreated greywater with time (constant head)**

In the constant head studies the maximum removal efficiency for the two heads i.e. 3 cm and 5 cm was 94.82 % and 94.44 % respectively. After some 8 days both the filters showed a lowering trend in removal efficiency indicating exhaustion of the filter bed (Figure 4.4). It was reported by (S.G. Lu, 2009) that, the removal of phosphorous by using fly ash was primarily due to the adsorption and precipitation of phosphorous as calcium phosphate on fly ash. Here in this study it would be important to know if there is possibility of phosphates to combine with calcium ions present in the sand. The pH of the waste water is found to play a critical role in the rate of removal and mechanism of the removal of phosphate. The removal of phosphate is accompanied by a decrease in the  $\text{Ca}^{2+}$  ion concentration and pH value (S.G. Lu, 2009). But due to simultaneous nitrification and denitrification (which also leads to pH changes) reactions happening in the bed it would not be advisable to deduce or compare the extent of removal of phosphates by looking at effluent pH. Due to adsorption onto Fe, Al or Ca containing minerals and precipitation with each of these elements from the dissolved phase could be the major removal mechanism (M. Achak, 2009). In this study the phosphate removal can be attributed to the presence of ions like  $\text{Ca}^{+2}$ ,  $\text{Mg}^{+2}$ ,  $\text{Fe}^{+3}$  and other cations in the soil.

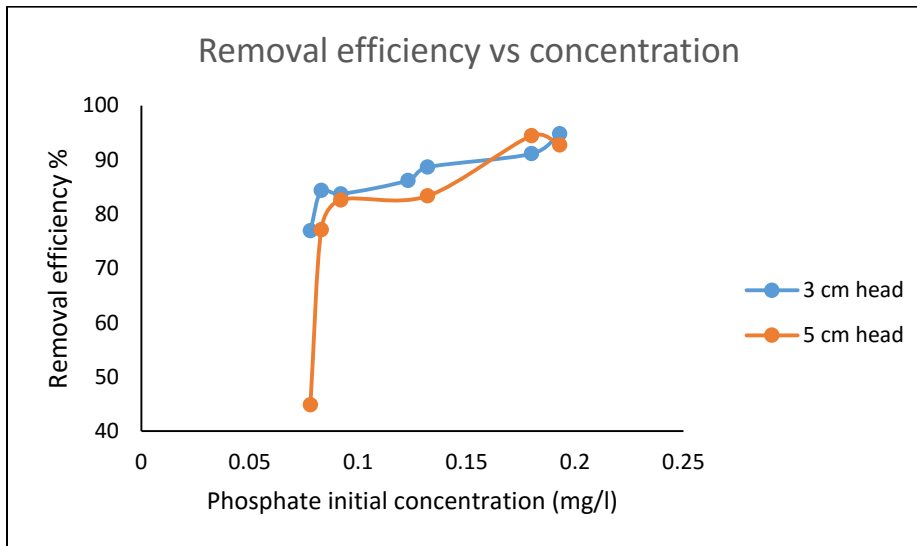
#### ***Effect of various process parameters***

Various parameters like pH, flow rate, head, contact time, initial concentration effects the removal efficiency of phosphates. pH and initial concentration cannot be controlled. It will

depend on the greywater quality. The only parameter which can be controlled is flow rate and initial concentration, optimizing this can result in the optimal removal of the phosphates or any other impurity.

#### *Effect of initial concentration*

It was found that removal efficiency depended greatly on the initial phosphate concentration.



**Figure 4.5. Dependence of removal efficiency on initial phosphate concentration**

It can be seen in the (Figure 4.5) that, as the phosphate concentration of the influent greywater increased the removal efficiency also increased. This could be attributed to the concentration gradient created which caused more adsorption of ions on the soil surface. As the concentration of incoming phosphate was more the concentration gradient was more. This driving force in the form of concentration gradient led to the adsorption of phosphates.

#### *Effect of contact time and loading rate*

As previously discussed the lesser the flow rate or head, more was the contact time and more was the removal efficiency. In case of constant loading rate studies for 10 ml/min and 20 ml/min the better removal was for 10 ml/min. In case of constant head the better removal was for 3 cm head. So it can be said that contact time plays an important role in the removal of phosphates.

**Table 4.3. Efficiency under various loading rates and heads**

Operating head/flow rate	Maximum efficiency (%)	Range of efficiency (%)
10 ml/min	97.85	78.26 – 97.85
20 ml/min	91.3	13.25 – 91.30
3 cm	94.82	76.9 – 94.82
5 cm	94.44	44.87 – 94.44

It is evident here that the filter with a flow rate of 10 ml/min gives the maximum efficiency of 97.85 % with the variation of 78 to 97 % (Table 4.3). So for optimum removal of phosphates one can choose the loading rate of 10 ml/min.

#### *Effect of pH*

The pH of the solution affects the surface charge of the adsorbents as well as the degree of ionization of different pollutants. The hydrogen ion and hydroxyl ions are adsorbed quite strongly on the sand surface and therefore the adsorption of other ions is affected by the pH of the solution. At lower pH more H<sup>+</sup> ions are hydrolyzed on the surface of the adsorbent and consequently adsorb negatively charged ions. Change of pH affects the adsorptive process through dissociation of functional groups on the adsorbent surface active sites. This subsequently leads to a shift in reaction kinetics and equilibrium characteristics of adsorption process (Mall et al, 2006). In this study the pH of influent varied from 7 to 7.8. The effect of pH on the removal efficiency is shown (figure 4.6.). This is a small range, so it would be difficult to comment on the dependability of removal efficiency on pH. There was no strict dependence of removal efficiency on the raw water pH in this range. At the same pH the removal efficiency was both high and low, e.g. at pH=7.4 for 5 cm head, the removal efficiency varied from 50-80% (fig 4.6). As the range of pH is not very wide so it would be better to check the effect of pH in other pH ranges and also pH is not the sole criteria for removal efficiency, the initial concentration and flow rate have also a major role to play.

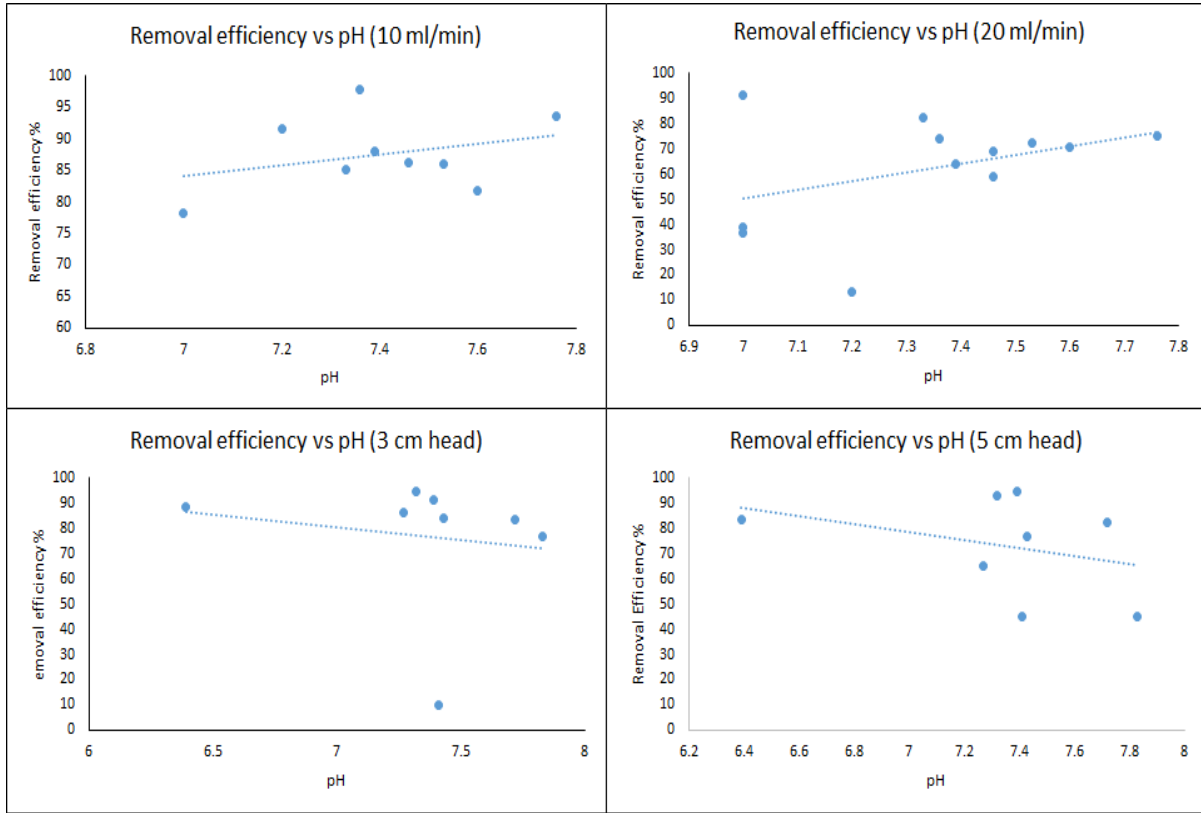


Figure 4.6. Removal efficiency vs pH

#### 4.6. TKN AND AMMONICAL NITROGEN REMOVAL

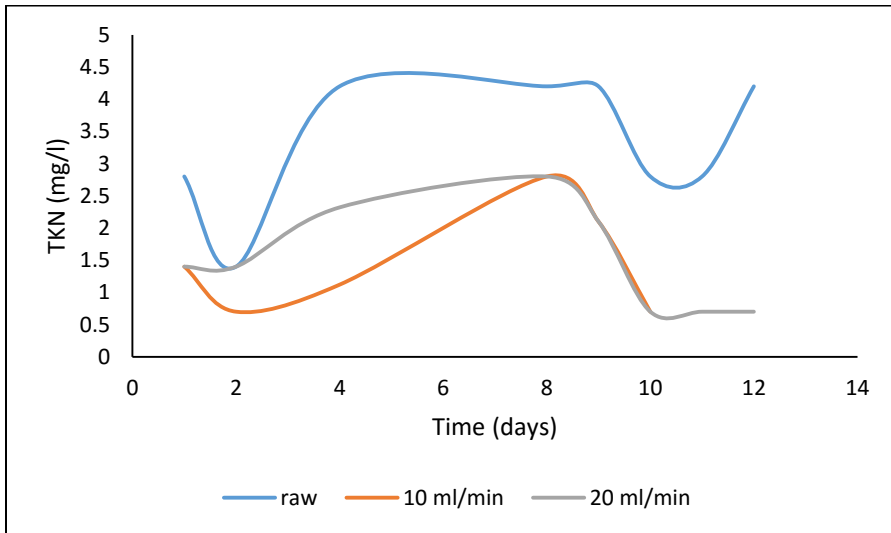
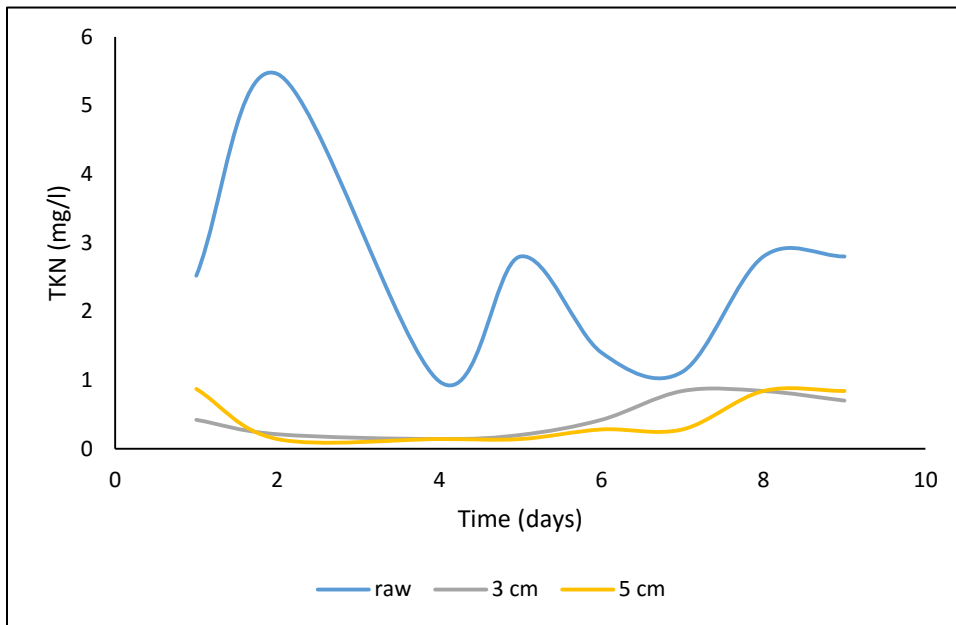


Figure 4.7. TKN concentration with time for constant flow rate studies

The removal of TKN is essentially due to straining and subsequent nitrification. In this study the initial plunge in the TKN concentration in the effluent could be due to the straining of suspended

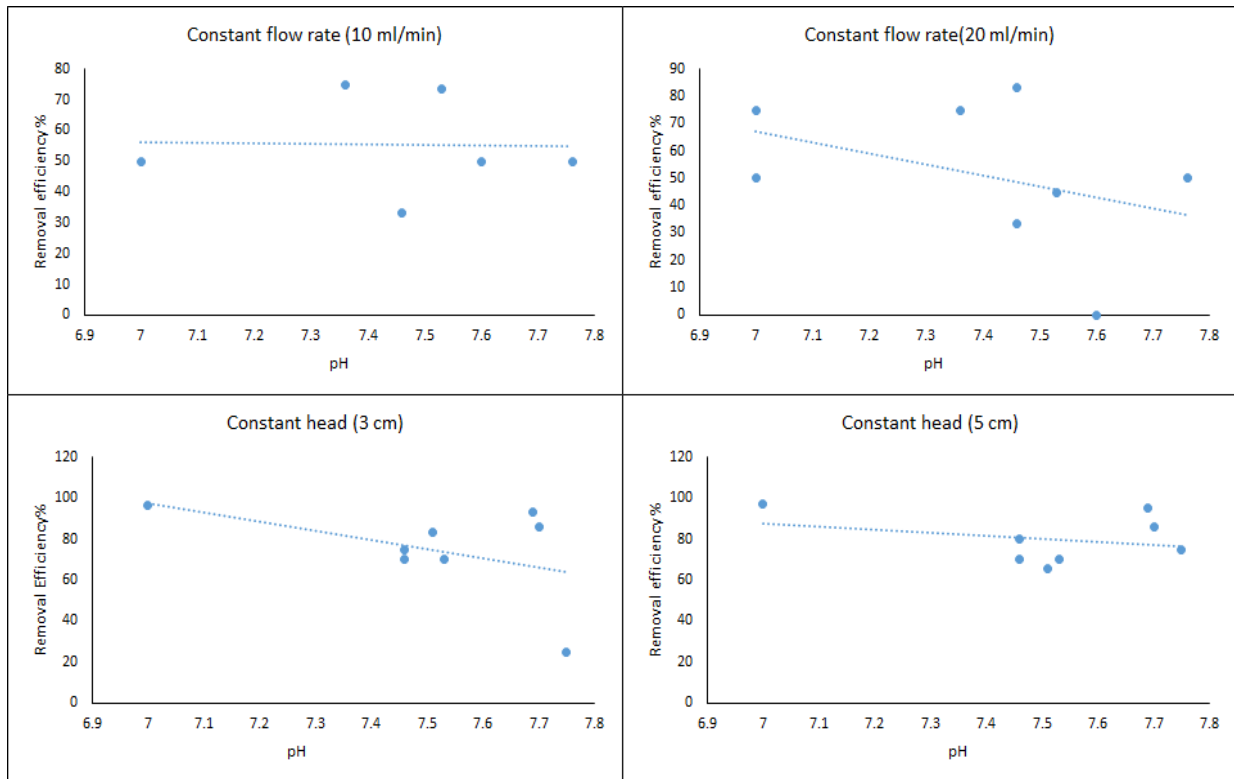
filterable organics in the top layers and then continuation of nitrification during the subsequent days. The reaction of nitrification is pH dependent it reduces below pH of 6.8. The pH of the influent remained well above 7.0 so the pH favored the reaction. Organic matter is found to depress the activity of nitrifies. An increase in carbon: nitrogen ratio is found to decrease the efficiency of nitrification. The low efficiency of removal for 8<sup>th</sup> and 9<sup>th</sup> day could be due to high carbon content. Also 10 ml/min is showing a better removal because of the low flow rate and high contact time. The highest removal efficiency for loading rate of 10 ml/min and 20 ml/min was 73.33 % and 44.77 % respectively (Figure 4.7).



**Figure 4.8. TKN concentration with time for constant head studies**

The removal of TKN in the constant head study is better than in constant flow rate. The head of 3 cm and 5 cm provided more contact time than constant head apparatus. The maximum removal for 3 cm and 5 cm head was 96.15 % and 97.44 % respectively (Figure 4.8). This could be due to the fact that the in the constant head studies the driving force is lower which enhanced contact time.

*Effect of pH*



**Figure 4.9. Effect of pH on TKN removal efficiency**

The removal efficiency of TKN was plotted (Figure 4.9.) against pH. The optimum pH for nitrification is 7.5-8. But here, a fair removal was seen at pH=7. Most of the times the pH of the influent greywater was between 7.4 and 7.8. So the pH conditions were near to optimum for nitrification to occur. There was low removal efficiency at some places even in this pH range. This could be due to deficiency of dissolved oxygen. The optimum pH is not the sole criteria for nitrification to occur. A number of other parameters define the efficiency of this reaction like temperature, dissolved oxygen, effect of ammonia and nitrite ion concentration, concentration of nitrifies, sludge age, organic loading and detention time, effect of surfaces and turbulence, effect of light, micronutrients, presence of organic matter, adaptability and microbial interaction.

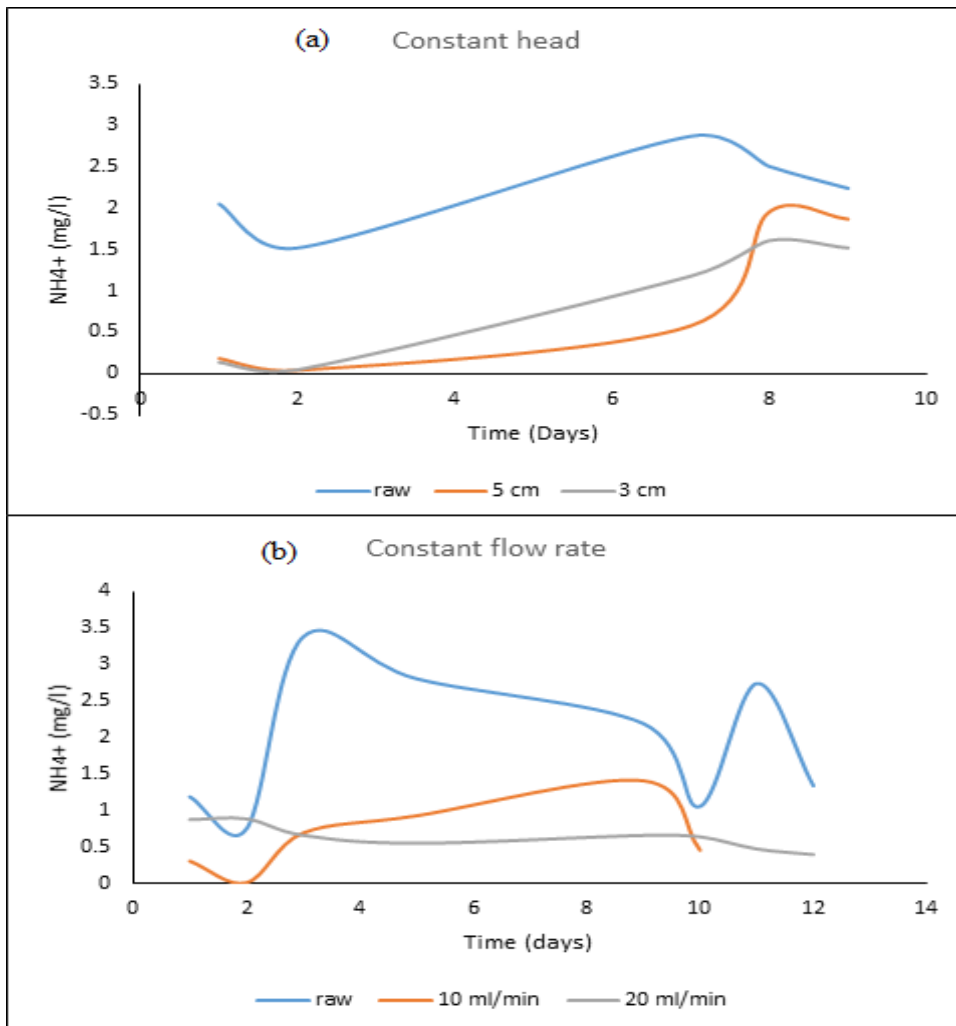


*Effect of flow rate and operating head*

**Table 4.4. Efficiency under various loading rates and heads**

Operating parameter	Range of efficiency (%)
10 ml/min	73.33 – 33.33
20 ml/min	83.33 – 33.33
3 cm	96.15 – 25
5 cm	97.44 – 65.48

It can be seen here (Figure 4.4) that the filter with 5 cm head offers maximum efficiency and the overall range of efficiency (97.44-65.48 %) is also higher than other filters.



**Figure 4.10. Ammonical nitrogen concentration with time for constant flow rate studies and constant head studies**

The maximum removal efficiency of ammonical nitrogen for flow rate of 10 ml/min and 20 ml/min was 97.88 % and 82.57 % respectively and that of 3 cm and 5 cm head was 96.77 % and 97.30 % respectively (Figure 4.10). Only in the case of 20 ml/min flow rate a low removal efficiency was observed. The plausible reason could be the low contact time and increased turbulence. The removal efficiency of ammonia increased, reached a peak value and after that decreased in all the filters. This can be due to the decrease in the oxygen content with time which reduces the rate of nitrification. A significant amount of  $\text{NH}_4^+$  removal takes place by adsorption on organic matter physically and chemically. As all the living organisms utilize nitrogen in the form of ammonia for cellular metabolism, therefore it is very much possible that the microbial assimilation had a very significant role to play in this removal, but this removal is limited by the available carbon. The effectiveness of removal of  $\text{N-NH}_4^+$  seems to support the assumption of existence of a significant nitrification. It is also possible that the ammonia ion is exchanged with the weakly bonded cations like  $\text{Ca}^{+2}$ ,  $\text{Mg}^{+2}$ ,  $\text{Na}^+$  and other positively charged ions.

#### **4.7. NITRATE REMOVAL**

##### *Constant flow rate studies*

The mean nitrate concentration of the raw greywater was found to be 8.11 mg/l. The Jaipur soil is rich in nitrates. The medium of filter bed being made of Jaipur soil showed leaching of nitrates in the initial hours of operation. There was heavy leaching in the first 4 hours. The concentration of nitrates for 10 ml/min and 20 ml/min loading rate went as high as 112 mg/l and 81.1 mg/l respectively after one hour of filter operation (Figure 4.11). It gradually reduced and came down to 3.25 and 4.82 mg/l after 3 hours. After that the concentration of nitrates in the effluent was monitored at the end of each day. The nitrates reduced to a concentration of less than 1 mg/l after 4 days. This could be due to the fact that after initial leaching of the nitrates the sites left vacant were occupied by the new incoming nitrate from the greywater.

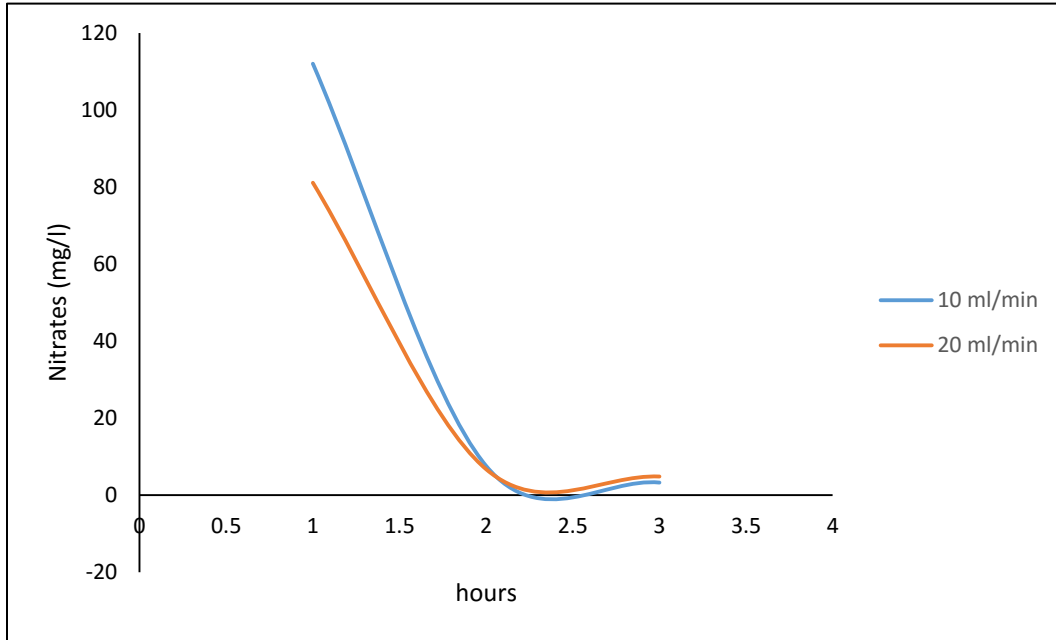


Figure 4.11. Nitrate leaching in the initial hours of filter operation

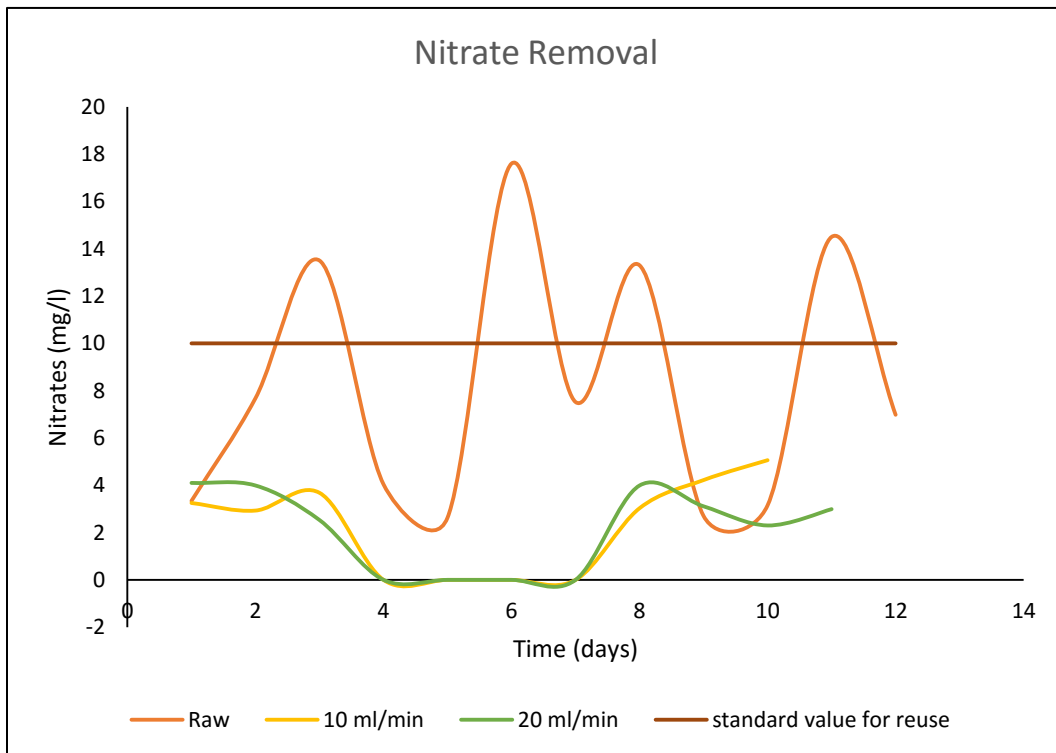


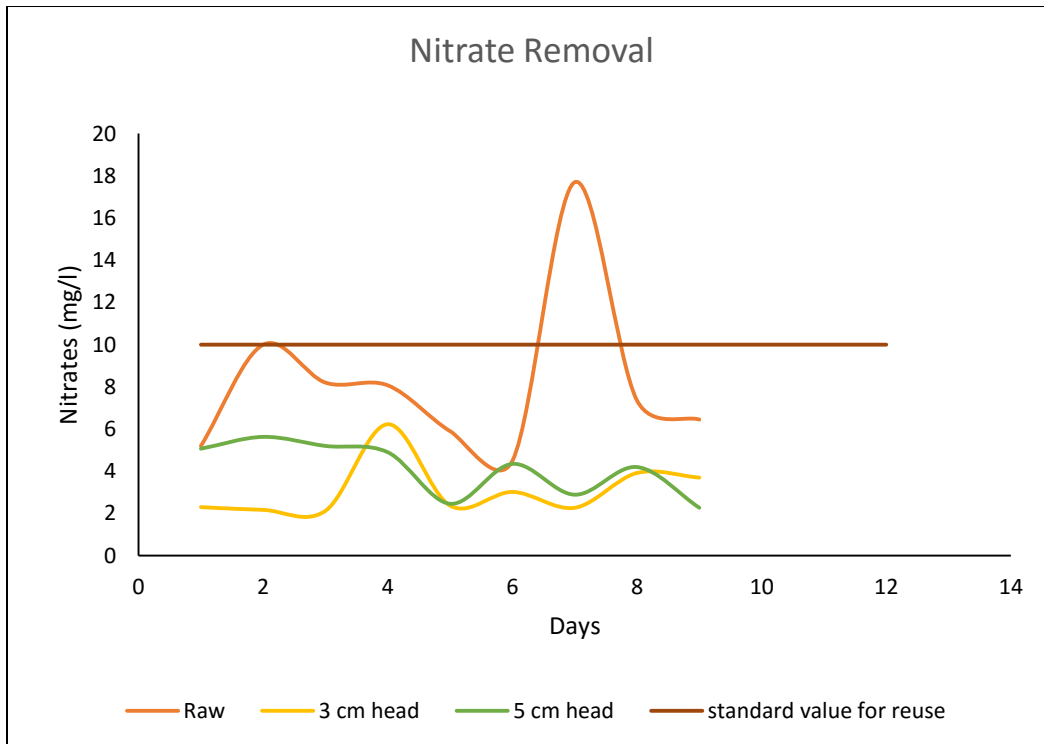
Figure 4.12. Nitrate concentration with time (constant flow rate)

The nitrate removal efficiency was 100 % at the end of 4<sup>th</sup> day and afterwards up to 7<sup>th</sup> day (Figure 4.12). After the 7<sup>th</sup> day the concentration of nitrates in the effluent started to increase but

still remained lower than the influent. The traditional method for nitrate removal is denitrification. For denitrification to occur anoxic conditions are required. In this study the DO of the effluent was 5-8 mg/l which shows that there was no anoxic condition in the filter. Also the filter was intermittently fed so there was ample exchange of air in the media.

The effect of loading rate was not much pronounced as both the setups showed similar trend irrespective of loading rate.

#### *Constant head studies*



**Figure 4.13. Nitrate concentration with time (constant head)**

In the constant head studies the maximum removal efficiency was 87.18 % and 83.67 % for 3cm and 5 cm head respectively (Figure 4.13). The nitrate concentration remained fairly low in the effluent, about 2-5 mg/l. In this study the operating head was not as much as it was in the constant flow rate.

#### 4.8. CLOGGING OF THE FILTER BED

The columns were operated until they got clogged. The filter was clogged after 12-14 days of operation. The reason for this clogging was straining of suspended particles in the top layer and development of schmutzdecke layer on the top. The effluent showed a visible reduction of turbidity (Fig. 4.14). The raw grey water had turbidity in the range 10-15 NTU. The turbidity of the treated greywater was well below the prescribed reuse standard of 2 NTU. This plunge in the turbidity suggest that there was straining of the suspended particles in the top layer. The decrease in the effluent flow rate suggest the progression of clogging.

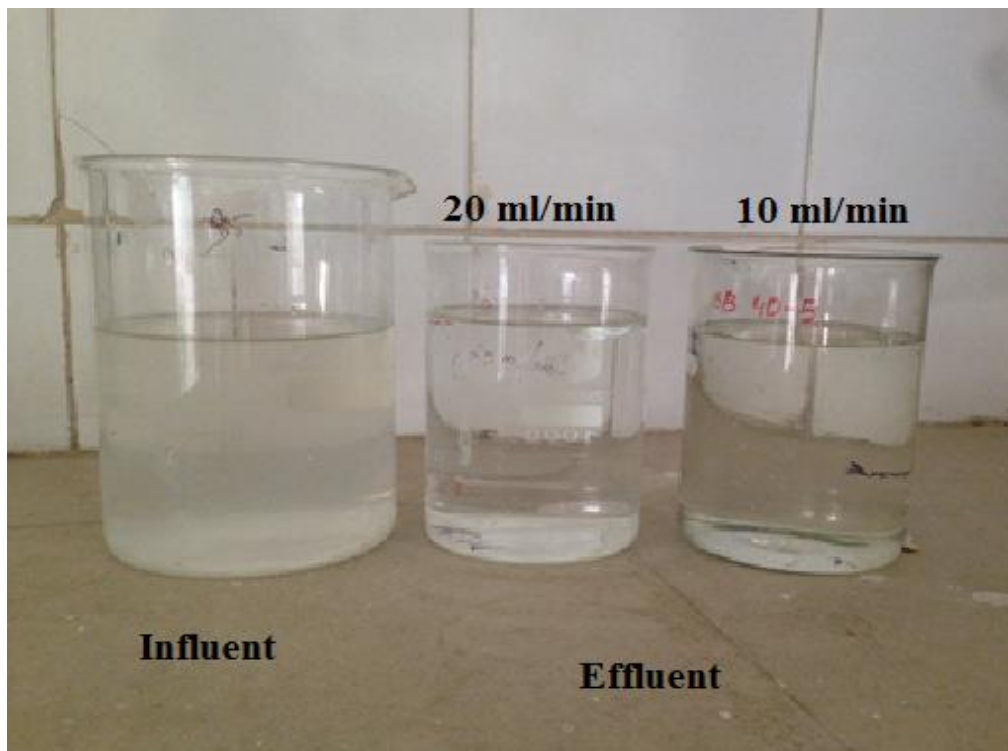


Figure 4.14. Influent and effluent water

The rate of filtration for the loading rate of 10 ml/min was initially about 5-6 ml/min and gradually reduced to 1-2 ml/min after 10-12 days before clogging. Similarly the other filter with the loading rate of 20 ml/min showed an initial filtration rate of about 11 to 8 ml/min and it was reduced to 4 to 6 ml/min after 10-12 days. In case of the filters with constant head of 3 cm and 5 cm the initial rate of filtration was 3-4 ml/min and 5-7 ml/min respectively which then gradually reduced to 1-2 ml/min and 3-4 ml/min respectively, after 7 to 8 days. There is early clogging in

the constant head studies due to their low loading rate which increases the removal of suspended solids and hence results in very efficient removal of turbidity.

## **5. CONCLUSIONS AND FUTURE RESEARCH SCOPE**

The locally available Jaipur soil has potential to be used as a filter media. There was removal of Phosphates, TKN, ammonical nitrogen and  $\text{NO}_3\text{-N}$  up to 97%, 97%, 97% and 83.33% respectively. The concentration of TKN, nitrates and phosphates were well below the reuse standards after filtration. The removal was primarily due to straining of suspended matter, nitrification of TKN, adsorption of nitrates and phosphates. It could be said that the sand has good anion adsorption capacity. In general the removal of phosphates, TKN, ammonical nitrogen and  $\text{NO}_3\text{-N}$  was found to be dependent on contact time. Each parameter showed a better removal in the case of flow rate of 10 ml/min and 3 cm head. The head causing flow was more in the case of 10 ml/min filter than 3 cm head filter. This obviously shows that the filtered water collected would be more in the case of rate with 10 ml/min. Also in case of both these filters the efficiency is nearly same. So one can adopt a flow rate of 10 ml/min for achievement of more efficiency and high rate of treatment. The dependence of removal efficiency on the initial concentration was found in case of phosphates indicating that concentration gradient has a role to play in the adsorption of phosphates. The removal of TKN was achieved fairly well as the pH of the greywater was well within 7 and 8. Various process parameters influence the rate of removal but practically one can control only the loading rate. So it becomes essential that for an optimal removal of various impurities one should ascertain optimal working parameters.

The future research can be carried out for this system by developing pilot scale plants to monitor the real time removal efficiencies. One of the major operating problem of sand filter is clogging. Another problem could be the disposal of sand in an environment friendly manner. A pre-treatment with mesh can be provided so that much of the larger suspended matter can be removed and consequently filter could be operated for more days continuously. Further column studies can be conducted by changing the bed depth and the effluent characteristics can be monitored at different depths. This would provide better insights into the treatment mechanisms happening in the filter. A post-treatment step of disinfection could be provided to achieve better removal efficiency throughout the filter operation.

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## 7. APPENDIX

### The Wastewater reuse guidelines in India (Source: CPHEEO)

Parameter	Toilet Flushing	Fire Protection	Vehicle exterior Washing	Non-contact impoundments	Landscaping, Horticulture and agriculture			
					Horticulture, Golf course	Crops		
						Non-edible crops	Crops which are eaten	
							raw	cooked
Turbidity(NTU)	<2	<2	<2	<2	<2	AA	<2	AA
SS	NIL	NIL	NIL	NIL	NIL	30	NIL	30
TDS	2100							
pH	6.5-8.3							
Temperature	Ambient							
Oil and Grease	10	Nil	Nil	Nil	10	10	Nil	Nil
Minimum residual chlorine	1	1	1	0.5	1	Nil	Nil	Nil
TKN	10	10	10	10	10	10	10	10
BOD	10	10	10	10	10	20	10	20
COD	AA	AA	AA	AA	AA	30	AA	30
Dissolved Phosphorous as P	1	1	1	1	2	5	2	5
Nitrate Nitrogen as N	10	10	10	5	10	10	10	10
Faecal Coliform in 100 ml	NIL	NIL	NIL	NIL	NIL	230	NIL	230
Color						AA		
Odor	No foul odor							

AA: As arising when other parameters are satisfied

All units are in mg/l unless specified