IN SITU NITRATE REMOVAL FROM GROUNDWATER USING FREELY AVAILABLE CARBON MATERIAL AT AN INDUSTRIALLY POLLUTED SITE

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Abstract

Groundwater pollution by nitrate is a known problem which occurs all around the world. In its nitrate (NO_3^-) and other forms, nitrogen can move through soil into groundwater. Nitrogen can also contribute to surface water quality problems. In elevated concentrations, nitrate in drinking water can be toxic to infants and young animals. Groundwater use is a cost effective option in rural areas. Rural areas have small scale distribution systems and larger expensive treatment options that are practised in cities are not suited to these areas. Treatment of nitrates with minimal costs and safe methods is thus a required technological endeavour in the more rural parts of Africa. In situ treatment could be a robust and effective technique for removal of nitrate, iron, and manganese. *In situ* denitrification is essentially the removing of nitrate-nitrogen from groundwater while it is still within the aquifer. The basic principle is that a carbon source is placed perpendicular to the groundwater flow such that flow occurs through the carbon source layer. Required layer thickness and depth vary from site to site. A chemical reaction mediated by naturally available bacteria then takes over and accelerates the natural process of denitrification. This paper discusses the procedures and calculations used to design laboratory experiments as well as to upscale the method and its field application. Laboratory denitrification experiments showed successful denitrification in the short term using sawdust or woodchip as carbon source. A field site was then equipped for testing the technology. A 5 m diameter x 1.37 m height tank filled with woodchips was buried to 3 m at a site with industrially high nitrate concentrations (above 1000 mg/L as NO_3). This represents an extreme case of groundwater pollution from nitrate in South Africa. Seven boreholes in the vicinity of the carbon source were monitored for 6 months. Measurements of redox potential, electrical conductivity, total dissolved solids and temperature were routinely done bi-weekly. Laboratory analyses of nitrate, ammonia, sulphate, iron and manganese were also done bi-weekly. The results showed that denitrification from above 300 mg/L to < 1 mg/L within the treatment zone was complete within 2 days, while the closest points, approximately 2.5 m away from the centre of the carbon source tank, showed total denitrification within one month. The presence of trace metals on soil surfaces (e.g. manganese, iron, and copper) and within the saturated zone may have enhanced the denitrification and nitrification at certain boreholes. Boreholes installed 10 and 30 m away from the carbon source showed some effects of denitrification approximately 2 and 4 months after the treatment started. It was successfully demonstrated that in situ denitrification can effect total removal of nitrate of industrially high nitrate concentrations using sawdust as a cheaply available slowly degradable carbon source

1. INTRODUCTION

Groundwater pollution by nitrate is a known problem which occurs all around the world. In its nitrate (NO_3^-) and other forms, nitrogen can move through soil into groundwater. Nitrogen can also contribute to surface water quality problems. Nitrate concentration in groundwater is of concern due to potential effects on human health as well as effects on livestock, crops, and industrial processes at high concentrations. A condition called methaemoglobinaemia also known as "blue baby syndrome" results from the ingestion of high concentrations of nitrate in its inorganic form (ITRCWG, 2000). Infants as well as children and adults suffering from maladies or treatments that lower the levels of stomach acid, are vulnerable to methaemoglobinaemia (ITRCWG, 2000). Above 300mg/L as N, nitrate poisoning may result in the death of livestock consuming water. At lower concentrations, other adverse effects occur in animals, these include increased incidence of still born calves, abortions, retained placenta, cystic ovaries, lower milk production, reduced weight gains and vitamin A deficiency. Recommended levels of nitrate for stock watering (livestock and poultry) in the US is below 100mg/L as N (ITRCWG, 2000 and Innovative Technology, 2000).

In situ biological denitrification refers to processes of enhancing the natural system's ability to denitrify water. The method is capable of denitrifying groundwater, waste water, treated effluent from waste water works and other polluted water. It requires the addition of a suitable substrate, normally a carbon source if heterotrophic denitrifcation is desired. Autotrophic denitrifcation takes place when reduced sulphur compounds, ferrous iron or hydrogen is added to the subsurface (Mateju *et al.* 1992; Mercado *et al.*, 1988). Permeable reactive barrier walls are constructed by digging a trench or hole of suitable size and configuration perpendicular to the groundwater flow direction, and mixing aquifer material with organic matter, e.g. sawdust, woodchips, which acts as a carbon source to stimulate denitrification (Schipper *et al.* 2004, Schipper and Vojvodic-Vukovic 1998, 2000, Robertson and Cherry 1995, 1997, Robertson *et al.* 2000).

The efficient functioning of a reactive barrier can often depend on the proper emplacement of the actual barrier and its effectiveness at allowing flow of source water, be it groundwater or waste water through it. Many studies have shown successful denitrification all over the world. However, some of the sites were not always as successful, and often scientists who have successfully denitrified their source water through a permeable reactive barrier in one location have had failures in other areas using similar techniques. Successful denitrification of groundwater has been practiced for more than a decade in New Zealand (Schipper and Vojdovic-Vukovic,1998, 2000, 2001, and 2004). The latest attempted denitrification of non point sources of nitrate from shallow groundwater failed due to hydraulic constraints on the performance of the denitrification wall (Schipper *et al.*, 2004). Denitrification walls are most successful and effective at protecting downstream water quality when as much groundwater as possible is intercepted by them (Schipper *et al.*, 2004, Barkle *et al.,* 2008 and Robertson *et al.*, 2003). Hence the hydraulic properties of both the wall and aquifer are integral properties to be monitored and assessed throughout the life of an operation (Barkle *et al.,* 2008).

Using the aquifer's natural flow to drive the reaction was also part of the controls of the project. Field and pilot scale studies show very promising results in other countries where they have been implemented and testing of denitrification in the field in a South African field scale site is a required progression. The main objectives of the experiment were to test the feasibility of field denitrification at industrially high nitrate levels in groundwater. Testing whether denitrification will take place successfully where clay is present was another objective as this presented some uncertainty. Brief discussions of laboratory denitrification results are included. Field characterisation procedures for in situ denitrification are recorded for the site. The actual field implementation exercise and available results of the field study are shown. Key implications of field implementations and limitations that may arise and lessons learnt during the field implementation are discussed.

2. SITE CHARACTERISATION AND PRE-FIELD IMPLEMENTATION PROCEDURES

Laboratory tests preceded the field characterization and implementation phases. These took the form of treatability tests done on bench scale using various carbon sources (Israel et al., 2007) and laboratory flow through testing with a selected carbon source from treatability tests. The laboratory bench scale treatability experiment was used to decide on a most suitable carbon source for the desired use. A long term low maintenance and slowly degradable carbon source with effective denitrification was deemed ideal. Carbon sources used during treatability tests included glucose, methanol, maize meal and sawdust. Sawdust was selected as a carbon source for field and laboratory flow through experiments due to its slow degradability and effective denitrification and minimal by-products.

The laboratory flow tank experiment was used to consider design parameters for field scale testing e.g. amount of sawdust required based on requirements for denitrification. Barrier thickness or barrier concentration could be determined from calculations using fundamental stoichiometric relationships and incorporating flow and area. Nitrite was produced during the experiment which indicates incomplete denitrification. The sawdust requirement for this experiment was not met hence the maximum period of denitrification occurred during the initial 10 days as well as between days 20 and 40 of the experiment.

The site used for field testing was in the Somerset West area. Historical reports for the site and available data were consulted to obtain information about the site. The area to be remediated is situated where an ammonium nitrate warehouse was previously operating. The area is approximately 3.6 ha. The surface elevation is approximately 6.5 m amsl. A water body (Paardevlei, figure 3) is situated adjacent to the site and covers an area of approximately 50 ha (SRK, 2006). Natural drainage channels to the lake have been

canalised away from it and over the years the walls have been raised. The site is situated on coastal plain underlain by Malmesbury Shale. Malmesbury rocks, comprised of dark green-grey shale, hornfels and quartzite, are generally well fractured. Fractures, particularly in the vadose zone, are generally filled with very stiff, dark green-grey clay. The site occurs within a zone of interface between the Coastal Plain and Coastal Dune Belt. Quaternary sediments of the Langebaan geology occur at the site.

Somerset West normally receives about 568 mm of rain per year and because it receives most of its rainfall during winter it has a Mediterranean climate. It receives the lowest rainfall (10 mm) in February and the highest (96 mm) in June. The average midday temperatures for Somerset West range from 16.2°C in July to 26.1°C in February. The region is the coldest during July when the mercury drops to 7.2°C on average during the night. The majority of the lowland area in Somerset West is heavily transformed by agriculture and urbanisation and there is minimal natural vegetation remaining but rather predominantly alien herbaceous vegetation (Holmes, 2002). The AECI in particular have mainly bluegum trees as well as grasses across the site. A general soil profile (SRK, 2006) for the study site is presented in the table below.

Table 1. Generalized soil profile for the AECI site, Somerset West, after SRK (2006).

Strata	Depth (m bgl)	Description
Surface	0 – 0.4	Grassed surface with red gravel and ash fill
Transported/Alluvium	0.4 - 1.5	Slightly moist, brown, loose, fine-medium SAND
Transported/Alluvium	1.5 – 2	Moist, light brown sandy CLAY.
Transported/Alluvium	2 – 3	Moist – wet, light brown to grey, clayey SAND with fine- calcrete gravel concretion and medium rounded gravel. Groundwater seepage encountered at this depth.

Sampling of groundwater, soil, and water level depth measurements were taken prior to installation of the treatment zone. Sampling and analyses of nitrate was done to establish the distribution of nitrate concentrations across the area where the nitrate removal would be tested. Distances between points were measured and water level data were used to construct contours of groundwater level data and determine groundwater flow vectors. Groundwater nitrate concentrations as N and winter groundwater levels together with the distances between boreholes and position of carbon source tank are shown in figure 1.
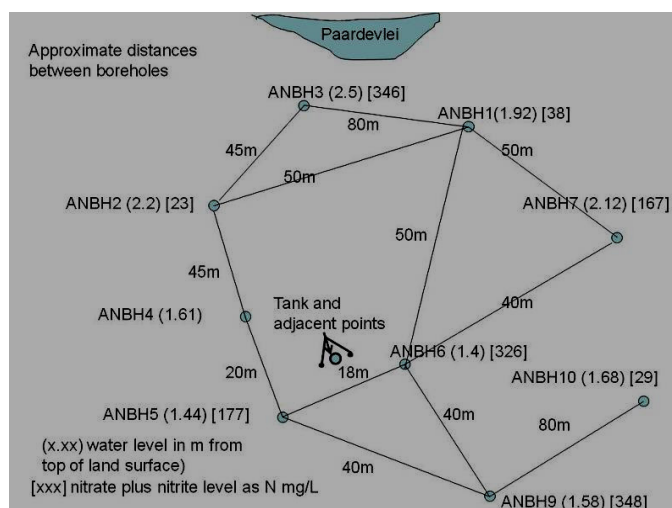

Figure 1. Borehole positions and proximity from each other and the Paardevlei water body. Water table depth and nitrate as N indicated for boreholes were measured.

Water table elevation data were then interpolated and used to determine groundwater flow at the site based on groundwater elevation gradients. Figure 2 shows the groundwater interpolated elevation map. Groundwater flow is from high elevation toward lower elevation. Figure 2 shows that flow occurs towards the Paardevlei area adjacent to BH3 as well as toward the coastal dune area just East of BH9, although measures were put in place to divert or channel flow away from the Paardevlei. Local flow toward the boreholes may be occurring based on figure 2. The carbon source tank was placed between BH5 and BH6. Soil samples were collected during excavation. Figure 3 shows the results of soil tests.
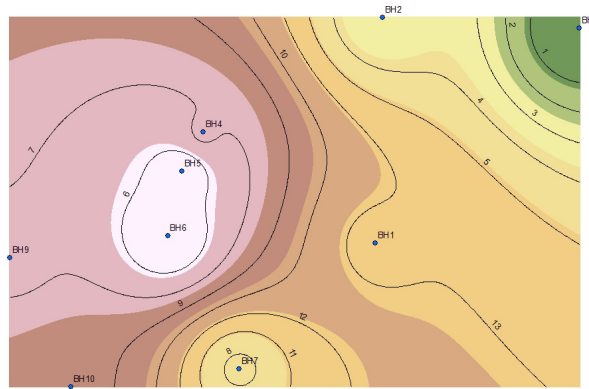
Figure 2. Water level elevation at the experimental site
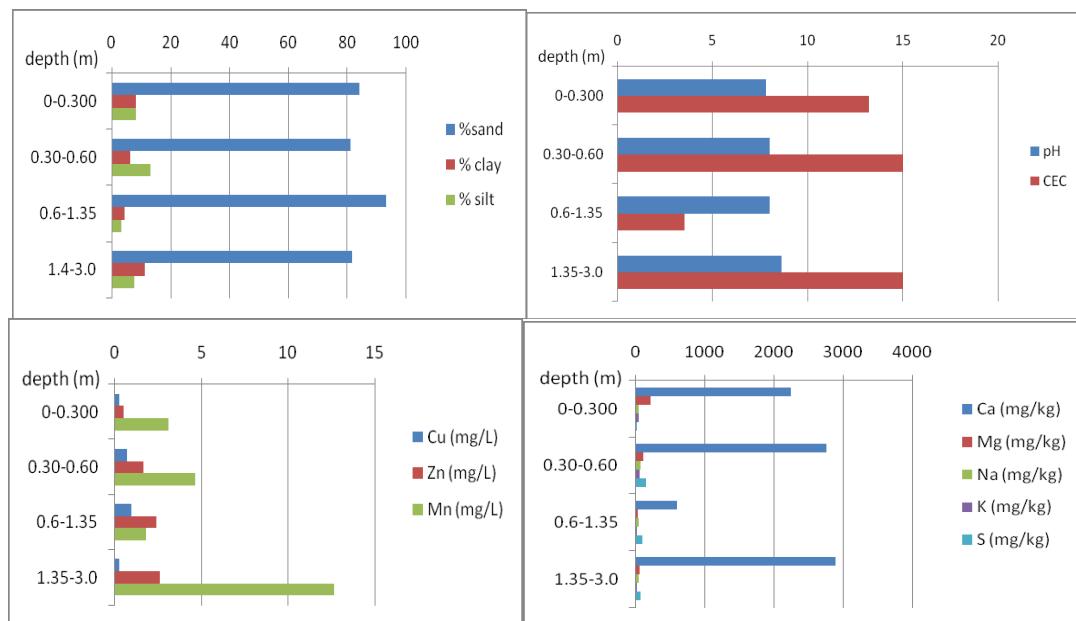(contours represent groundwater elevation in m).

Figure 3. Results of soil analyses. These include upper left: particle size analyses; top right:
pH and cation exchange capacity; bottom left: metals; and bottom right: exchangeable cations.

Most of the soil profile is composed of predominantly sand sized particles, with the soil depth of 1.4 to 3 m having the highest clay content of 10%. This relates to a higher cation exchange capacity for this depth as more charged surfaces are available in clay particles. The texture of the aquifer was estimated to be about that of sand 20-35% based on the particle sizes present.

Trace metals copper (Cu), zinc (Zn) and manganese (Mn) were analysed for as it is documented that the presence of metals can enhance denitrification rates at various concentrations (Labbe *et. al*., 2003). The groundwater level was at 1.35 m below surface at the area where excavating took place. It varied across the site. Results show that manganese is the dominant trace metal at most soil depths and it is particularly concentrated within the saturated zone below 1.35 m depth, while zinc is more concentrated just above the water table. Manganese oxides are among the strongest naturally occurring oxidizing agents in the environment, having high sorptive capacities and participating in various redox reactions with both organic and inorganic compounds.

Exchangeable nutrients were analysed as an indication of availability in the soil environment. The results of the cations or exchangeable nutrients show that calcium is the dominant cation in the soil at all depths. The presence of calcareous boulders and calcrete nodules at some depths as well as the fact that the position of the excavation is at a contact between more clayey type geology and coastal calcareous sands support the fact that calcium is the dominant cation at all soil depths. Sulphur as a plant nutrient was plotted to show the reserve of sulphur within the soil. Sulphur is a redox sensitive species which may be affected by oxidation reduction processes or condition of the subsurface. Figure 4 shows the % carbon along the soil profile. This was tested to evaluate the possible occurrence of natural denitrification.
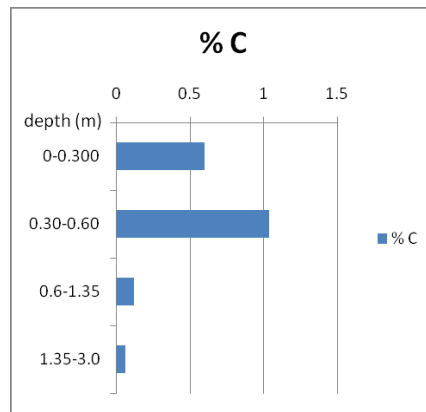
Figure 4. % carbon in the soil profile.

The % carbon results show that the maximum carbon is just over 1%. This would result in a low likelihood for natural denitrification at the site, hence it was deemed necessary to amend the subsurface with a slowly degradable carbon source.

3. FIELD IMPLEMENTATION AND RESULTS OF MONITORING AT THE SITE

Results from previous laboratory experiments (Israel, 2007 and Israel et al., in press) were used as an indication of treatability and rates of reactions for the selected carbon source. Barrier or wall thickness as well as concentration or mass of carbon source required for a particular nitrate concentration was determined using stoichiometric relationships for denitrification as well as results from a laboratory flow experiment. Calculations took into consideration an average or potential flow rate based on soil type, size of the area to be treated as well as desired resultant nitrate concentration. The selected carbon source was chosen based on availability, treatability, costs, effectiveness and minimal nuisance products or side reactions during laboratory experiments as well as longevity of the carbon source which would in turn reduce the need or frequency for maintenance at the site. Untreated woodchips were freely available for the experiment on site. 5000 kg of woodchips were used; this was limited by the size of the treatment vessel (tank) used. The determination of the mass of a litre of sawdust was calculated via a mini experiment. It was determined that the mass to volume ratio for sawdust was roughly 1:1. The calculated requirement for sawdust was between 4980 kg and 6350 kg which compared favourably with the amount that could be accommodated for in the tank. Photos were taken throughout the implementation phase. Figure 5 shows the installation of the treatment vessel.

Figure 5. Digging and emplacement of tank used for carbon source during the experiment.

The tank fully was slotted to allow flow through the treatment material and into the aquifer. The upper left picture in figure 5 shows the start of the digging process, while the lower left picture represents the tank used during the experiment. The upper right picture shows the tank in place within the groundwater table, while the lower right picture shows the fully slotted PVC pipes used as monitoring points to track trends of groundwater quality within the tank and adjacent to the tank. The tank used was a 5000 L tank

with height 1.37 m, diameter 2.15 m. Monitoring of the central point in the tank, the two adjacent points as well as already existing boreholes continued two days after the tank was in place. Figure 6 shows some results of nitrate concentrations for the tank and the two points adjacent to the tank.
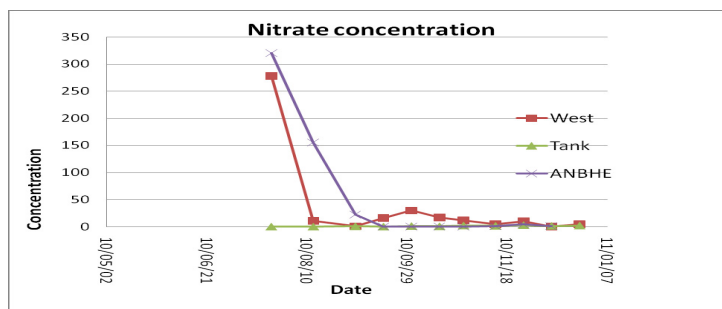

Figure 6. Nitrate concentration mg/L as NO3 in the tank and two adjacent boreholes from July 2010 up to December 2010.

The boreholes adjacent to the tank had initial concentrations of 320 mg/L (East- right of tank) and 280 mg/L (West-left of tank) on the first day of sampling (Figure 6). On the second sampling trip (2 weeks after), both boreholes showed a considerable decrease in their nitrate concentration, with the East showing a 51.6% decrease in concentration to 155 mg/L and the West a 96% removal of nitrate to 11 mg/L. Within 2 months, which equates to 4 sampling events, all the nitrate was reduced in these boreholes. Figure 6 shows that the nitrate concentration was below 1 mg/L in the tank from day two of installation. The nitrate levels remained low for the rest of the experiment in all these boreholes.

Summary plots of parameters per borehole were done for boreholes ANBH4, ANBH5 and the two boreholes adjacent to the tank in figure 7.
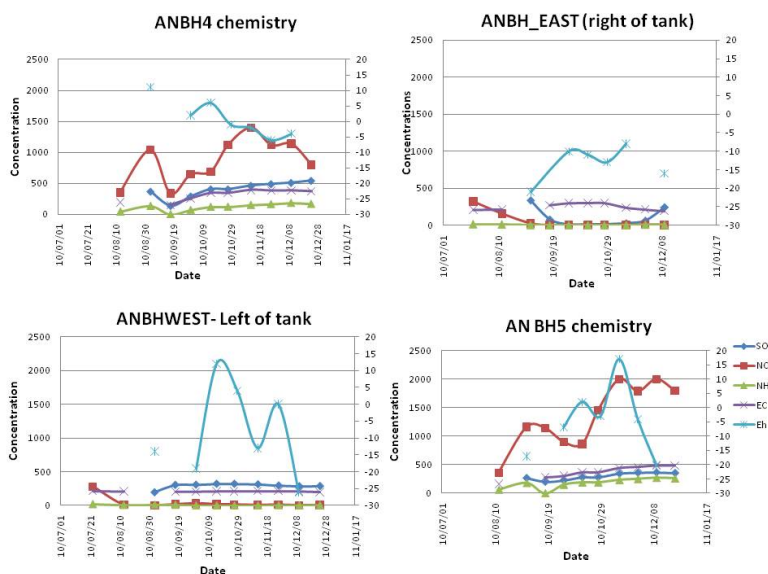

Figure 7. Summary plots per borehole for all measured parameters at the experimental site.

Boreholes further away from the tank showed a decline in nitrate concentration approximately 2 and 4 months after the tank was put in place, then showed an increase. The boreholes are approximately 10 and 30 m away from the tank. The percentage nitrate removal was 40% in ANBH5 and 45% for ANBH4 prior to a subsequent increase in concentration. It is envisaged that a diluted and partially consumed dissolved organic carbon concentration compared to the concentration in the tank may have reached these points. It appears that preferential flow took place even though the two closest monitoring boreholes were more or less equidistant from the tank. The borehole logs showed that the boreholes in the preferred direction of greater flow velocity are drilled through the main water bearing lithology on the site composed of sand and clay, while other boreholes are drilled into clay which has slower flow velocities.

Higher initial manganese levels in the ANBHE borehole may have contributed to the rapid reduction of both nitrate and sulphate, together with the highly reduced conditions produced by the strong oxidizing agent.

4. CONCLUSIONS

It was concluded that *in situ* field denitrification successfully removed nitrate within a short period of time, in this case about one month for an area of approximately up to 2.5 m away from the centre of the tank. Proximity to the main denitrification zone, as well as ease of flow path toward other boreholes and effects of infiltration, dilution, dissolution or replacement on soil particles as well as competing reactions of nitrification and denitrification all affect the results seen at a specific point in time. It was concluded that the tank's carbon source reached boreholes ANBH4, ANBH5 and ANBH6 during the course of the experiment, however it may have been in insufficient concentration to effect total denitrification at these points. The presence of trace metals on soil surfaces and within the saturated zone may have enhanced the denitrification and nitrification at certain boreholes. Sulphate reduction occurred due to nitrate already being used up in ANBHE. This is explained by the redox sequence in waterlogged soils and the saturated zone. When sulphate is available in the sub surface while carbon is still available as a reducing agent, and reducing conditions prevail, sulphate reduction will occur. An important issue that came across from reviewing the methods used is that budgets play a big role in the construction and implementation time and sophistication. It was successfully demonstrated that in situ denitrification can effect total removal of nitrate of industrially high nitrate concentrations using sawdust as a cheaply available slowly degradable carbon source. Despite the presence of clay, the reaction proceeded successfully. Continual monitoring will enable actual rate calculation for points further from the tank. The technology is cheaply implementable, and proved feasible for removing nitrate for the area around the tank within 2 months. Continual monitoring of the site is ongoing.

5. LIMITATIONS OF THE SYSTEM

The tank used was only 1.37 m in height while the winter water table was at about 1.4 m depth below ground level and the summer water table depth ranged from 1.8 (minimum depth) to over 3 m (maximum water table depth) in some boreholes. This means that the tank's location was limited to between 2.8 m bgl and 1.48 m. Uncertainty with respect to the ability to sample all points during summer, as well as summer reaction rates or occurrence exist. Certain boreholes have water levels below 2.8 m during summer. The bedrock was not encountered during the excavation process, hence there is uncertainty about underflow in the treatment zone as well as flow by-passing the treatment zone. The presence of disconnected clay lenses as well as calcareous sands in certain parts of the aquifer presented potential for areas or zones of variable flow. Monitoring was done bi-weekly using indicator parameters to accommodate the available budget; this limited the amount of data obtainable for the study area. Uncertainty with respect to distribution of clay lenses and calcrete boulders presented some limitation with respect to the local flow direction and velocity at the site. The area immediately around the tank had a rapid rate at which denitrification occurred. The presence of clay is not ideal, but not limiting either, it simply means that flow is much slower than desired in some areas of the site, and hence the denitrification will take place over a longer period in those areas.

6. RECOMMENDATIONS

To test such or similar technologies in the field, site characterization is essential as every site has its own unique prevailing conditions. This includes a minimum of testing soil and groundwater physical and chemical properties, flow direction and flow rate if possible. Ideally, monitoring the site prior to implementation for flow directions and rates; especially taking changes in flow direction and rate into account during seasonal changes.It is also important to test for nitrate and or other indicator parameters for the specific site to understand the distribution of pollutants at the site prior to selecting the most suitable area to place the treatment barrier. Historical data for any site can give insight regarding the pollution or spill type events, water level trends, chemistry, pollution or clean-up initiatives that have been done at the site. Treatability studies using material from the site may prove invaluable during field implementation.

7. ACKNOWLEDGEMENTS

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