

Monitoring of a pilot GFO filter for removal of low-concentration arsenic in water

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Abstract

Many water treatment plants (WTPs) were designed to remove ammonia, iron, and manganese simultaneously using biofilters. In some cases (especially in the Pianura Padana area, in Italy) such plants were designed without a specific treatment stage for arsenic removal because its concentration in the groundwater (i.e. 10 to 20 µg/L) was lower than the previous maximum contaminant level (MCL) of 50 µg-As/L; therefore, specific treatments for arsenic removal must be introduced or upgraded in WTPs. In this work, the results of a 19-month monitoring campaign are reported for a pilot granular ferric oxide (GFO) filter installed in an Italian WTP as a polishing stage. The aim was to investigate the performance of GFO with low arsenic concentrations. The results show that, if the groundwater arsenic concentration is close to the MCL, GFO treatment can be cost effective (approximately 80,000 bed volumes have been treated). It was confirmed that GFO can be effective for the removal of both As(III) and As(V) species.

Key words: arsenic removal, drinking water, GFO, iron-based sorbents, WTPs

INTRODUCTION

Arsenic (As) is a metalloid element. It can be converted into species with different valencies or react to form insoluble compounds – e.g. with iron. Its presence in natural waters is caused by leaching from source rocks and sediments (Hering & Elimelech 1995), or uncontrolled human activity (e.g. in agriculture, arsenic is used as a fungicide and insecticide; in the wood industry, for preservation; in the mining industry, for smelting metal ores, etc.). Details of the environmental origins, occurrence, events and impacts on human health of arsenic can be found in Mandal & Suzuki (2002).

Generally, arsenic reaches the human body through the water (Mondal *et al.* 2013). Arsenic contamination in natural waters can range upward from a few µg/L and is a worldwide problem that affects at least 100 million people (Bhattacharya *et al.* 2007). The countries affected include Argentina, Bangladesh, Cambodia, Canada, Chile, China, Italy, the UK, and the USA.

Arsenic occurs in several oxidation states (valencies) in the environment (e.g. -III; 0; +III; V) although, depending on the environmental pH and redox potential (Eh), the predominant species are As(III) and As(V). Arsenic (V) is generally predominant in oxygen-rich surface waters while As (III) usually predominates in groundwater (Choong *et al.* 2007; Giles *et al.* 2011).

Effects on human health related to the ingestion of water containing high levels of arsenic – the most serious is ‘black foot disease’ – are well documented (e.g. Mandal & Suzuki 2002; Choong

et al. 2007). Both the concentration and valencies of the arsenic in any water are critical for human health. In fact, as reported in many studies (e.g. *Arienzo et al.* 2002; *Qu* 2008; *Zhang et al.* 2010), As (III) is more toxic than As(V). The very high toxicity of trivalent arsenic arises from its strong affinity for thiol groups, which inactivates enzymes (*Ahuja* 2008).

Arsenic can be removed from water using various techniques based on different chemical, physico-chemical or biological principles; details can be found in, e.g., *USEPA* (2003); *Mondal et al.* (2006); *Choong et al.* (2007); *Ahuja* (2008) and *Singh et al.* (2015).

Some arsenic removal techniques (e.g. coagulation and flocculation, lime softening, membrane filtration, adsorption on activated carbon) are well known and widely applied around the world, and are known as *consolidated techniques*. Others, e.g., activated alumina, titanium dioxide, sulfur modified iron – known as *emerging techniques* – have not yet been applied in water treatment plants (WTPs), even though their effectiveness has been established in some cases.

Since about 2005, the processes involving iron-based sorbents (IBs) have become some of the most extensively applied for arsenic removal. Two IBs are commercially available and widely used: granular ferric hydroxide (GFH, available since 1997) and granular ferric oxide (GFO) such as Bayoxide[®] E33 (developed in 2000). *Streat et al.* (2008a) report both the preparation and physical characteristics of GFH and GFO.

The main advantages of using GFO and GFH are: (i) high potential removal efficiency (up to 98%), (ii) few maintenance, operating or manpower requirements, (iii) absence of waste sludge, (iv) simplicity of spent material disposal (*Streat et al.* 2008b; *Singh et al.* 2015), and (v) low treatment cost (*Mohan & Pittman* 2007). The treatment cost is strongly related to the arsenic concentration in the raw water, because higher arsenic concentrations reduce the life of the material, and the WTP's configuration – e.g., whether the GFO/GFH filter is used for polishing or as a single-stage for arsenic removal. *Sorlini et al.* (2014) who surveyed Italian WTPs for arsenic removal, indicate that, if the influent arsenic concentration is between 16 and 100 µg/L, the operating cost of IBs is comparable to that of membrane filtration and ion exchange.

High removal rates (>90%) can be obtained with GFO. These are frequently achieved in batch adsorption experiments on solutions spiked to multiple mg-As/L concentrations (*Mohan & Pittman* 2007). Few if any studies were aimed at investigating GFO performance in removing low arsenic concentrations – i.e., 10 to 20 µg/L. In many areas of Pianura Padana, arsenic concentrations in groundwater are close to the maximum contaminant level (MCL) of 10 µg/L established under Italian Drinking Water Legislation (Legislative Decree 2 February 2001, n. 31), so a long term experiment aimed at investigating GFO performance under those conditions could be useful for many WTP managers.

On this basis, a pilot GFO filter was installed at a WTP treating an average flow of 5 m³/h, for 700 inhabitants. The WTP, in the Province of Pavia, northern Italy, has a treatment configuration typical of many small plants that have been operating since about 1980s and were designed for the simultaneous removal of ammonia, iron, and manganese by biofiltration, without a specific arsenic removal stage. The pilot plant, fed continuously with water from the biofilters (mean arsenic concentration 9.6 µg/L), was monitored (with arsenic speciation) for 19 months.

MATERIALS AND METHODS

Case study

The GFO pilot filter was installed in a WTP in the north of Italy (Lombardy, Italy). A schematic diagram of the plant is shown in *Figure 1*. Contaminants in the groundwater exceeding the MCLs

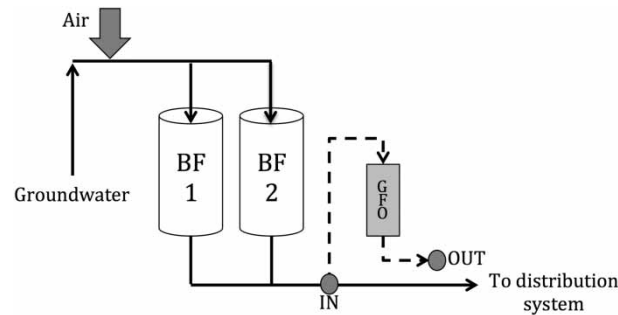


Figure 1 | Schematic diagram of the WTP (continuous lines), highlighting the pilot GFO filter location (dotted lines). Legend: grey dots: sampling points; BF: biological filter filled with sand (80%) and pyrolusite (20%); GFO: pilot plant.

established under Italian Drinking Water Legislation (*op cit*) include: ammonia (mean: 0.70 mg-NH₄⁺/L), manganese (mean: 94 µg/L) and arsenic (mean: 11 µg-As_{tot}/L).

In the full-scale plant, contaminants are removed by a pre-aeration stage (in-line injected air) and two biofilters (BFs) filled with sand (80%) and pyrolusite (20%). Pyrolusite consists essentially of manganese dioxide (MnO₂) and its use was introduced in BFs in 2014 for arsenic control (Mohan & Pittman 2007; USEPA 2011). Following the methodological approach reported in Sorlini *et al.* (2015a), a monitoring campaign was conducted and two problems arose: (i) arsenic removal in the BFs was below 10% because the pyrolusite worked mainly as an oxidizer, and (ii) the mass of pyrolusite in the BFs fell due to backwashing. In agreement with the WTP managers, it was decided to investigate the suitability of an additional arsenic removal (polishing) stage, to keep its concentration as low as possible – following the WHO guidelines (2011). Of the arsenic removal technologies available, GFO was chosen because the process is simple (low maintenance and manpower requirements) and reliable. It is also sludge-free so there are no waste disposal costs.

As can be observed from the Figure, the disinfection stage is not in operation in order to minimize the negative effects that can arise from the use of disinfectants such as chlorine dioxide (ClO₂) and, consequently, to keep the WTP's process scheme as simple as possible. In fact, if ClO₂ is used for final disinfection, specific removal stages for chlorine disinfection by-products removal (e.g. activated carbon filtration) could be provided (Sorlini *et al.* 2015b, 2015c).

Adsorbent material

Bayoxide[®] E33 was chosen from the commercially available iron-based materials. Its main characteristics are listed in Table 1, while those of the water treated are reported in Table 2. Bayoxide[®] E33 (E33, below) was chosen because: (i) it is less sensitive to high pH values (>7) than GFH (the water's pH is close to 7.6, the pH_{ZPC} of the GFH); (ii) it shows better As(V) removal than GFH

Table 1 | Properties of the E33 used in the test (data from the manufacturer)

Parameter	Value
Chemical class	Synthetic iron hydroxide (Goethite)
Content of Fe ₂ O ₃	>70%
Physical appearance	Amber granules
Brunauer-Emmett-Teller surface (m ² /g)	120–200
Bulk density (g/cm ³)	0.40–0.60
Particle size (mm)	0.25–2.00
Sieve sizes (U.S. Standard)	10 × 35

Table 2 | Influent parameters to the GFO pilot filter monitored during the test

Parameter	Units	Average concentration
pH	–	7.6 ± 0.3
Ammonia	mg-NH ₄ ⁺ /L	0.28 ± 0.2
Antimony	µg/L	<0.5*
Arsenic (total)	µg/L	9.6 ± 1
Arsenic (III)	µg/L	0.65 ± 0.5
Arsenic (V)	µg/L	8.95 ± 1
Calcium	mg/L	16.3 ± 0.2
Chloride	mg/L	1.0 ± 0.1
Iron	µg/L	40 ± 30
Magnesium	µg/L	4 ± 1
Manganese	µg/L	11 ± 10
Orthophosphate (as PO ₄ ³⁻)	mg/L	0.80 ± 0.3
Silica (as SiO ₂)	mg/L	24 ± 2
Sodium	mg/L	11.7 ± 0.3
Sulfate	mg/L	0.30 ± 0.1
Vanadium	µg/L	<0.5*

Note: antimony (Sb) and vanadium (V) – marked with ‘*’ – were always below the limit of detection. Variation from the mean is indicated by ‘±’.

(AWWA 2005), and, the concentration of competing ions like phosphate, vanadium, and silicate is low or close to the interference threshold reported in literature (>0.5 mg-PO₄³⁻/L; >50 µg-V/L; >13 mg-SiO₂/L at pH = 7.5) (AWWA 2005; Jeong *et al.* 2007; Nguyen *et al.* 2007; Zeng *et al.* 2008).

GFO pilot plant characteristics

The GFO pilot filter consisted of a Plexiglas column (diameter: 3 cm; height: 80 cm) containing 42 g of E33 operating under continuous down-flow conditions. Water leaving the BFs was fed into the pilot plant at 1 L per hour using a metering pump. The empty bed contact time in the filter was 5 minutes and no backwash was implemented during testing.

Analytical methods

Water quality monitoring was conducted by taking an average of one sample per week throughout the 19-month test from both the influent (IN) and effluent (OUT) streams – see Figure 1. A total of 152 samples were analyzed. Almost all analyses were carried out according to Italian standards (APAT-IRSA/CNR 2003), but Standard Method n. 3125 (APHA AWWA & WEF 2012) was used for total arsenic, as well as As(III) and As(V). This was done because, due to its neutral charge at neutral pH levels (6–9), As(III) is hard to remove from water by chemical speciation or adsorption (USEPA 2003; Giles *et al.* 2011). Arsenate (As(V)) can be removed readily due to its positive charge. Because of this consideration, the As(III) concentration in the samples was measured by determining the total arsenic (As_{tot}) remaining in water filtered through SERDOLIT[®] resin, which is selective for As(V) (McNeill & Edwards 1995; Sharma *et al.* 2016).

Finally, in line with the procedure reported in the citations as well as in Henry *et al.* (1979) and Henry & Thorpe (1980), the water's As(V) was calculated as the difference between the concentrations of As_{tot} in the unfiltered sample and that of the filtered sample containing only As(III); (As(V) = As_{tot} – As(III)).

RESULTS AND DISCUSSION

The main characteristics of the GFO pilot filter effluent are reported in [Table 3](#). During the test, the pH of both influent and effluent remained essentially constant at 7.6. As this is between 6 and 9, it is within the recommended range for GFO treatment ([USEPA 2003](#)), so removal occurred in the optimal treatment range.

Table 3 | Effluent parameters from the GFO pilot filter monitored during the test

Parameter	Units	Average concentration
pH	–	7.6 ± 0.3
Ammonia	mg-NH ₄ ⁺ /L	0.26 ± 0.2
Antimony	µg/L	<0.5*
Arsenic (total)	µg/L	See Figure 4
Arsenic (III)	µg/L	See Figure 4
Arsenic (V)	µg/L	See Figure 4
Calcium	mg/L	16.3 ± 0.2
Chloride	mg/L	1.0 ± 0.1
Iron	µg/L	45.1 ± 40
Magnesium	µg/L	4 ± 1
Manganese	µg/L	8 ± 8
Orthophosphate (as PO ₄ ³⁻)	mg/L	0.64 ± 0.3
Silica (as SiO ₂)	mg/L	23 ± 2
Sodium	mg/L	11.7 ± 0.2
Sulfate	mg/L	0.28 ± 0.1
Vanadium	µg/L	<0.5*

Note: antimony (Sb) and vanadium (V) – marked with ‘*’ – were always below the limit of detection. Variation from the mean is indicated by ‘±’.

Comparison of the concentrations of ammonia, magnesium, calcium, chloride, sodium, iron, manganese, sulfate, and antimony reported in [Tables 2](#) and [3](#), shows that no significant variations were detected during the test.

Among the species that can potentially affect arsenic adsorption, only silica (average feed concentration = 24 mg-SiO₂/L) and orthophosphate (average = 0.80 mg-PO₄³⁻/L) exhibited concentrations close to the maxima permitted. The concentration of silica was substantially the same in the influent and effluent, confirming that it was not adsorbed. The concentration of vanadium was always below its detection limit and so no further consideration was given to it.

The influent and effluent concentration trends of the orthophosphate (PO₄³⁻) are reported in [Figure 2](#). As first comment, it is noted that the phosphorus concentration in the influent was very variable during the study and two annual macro-cycles can be expected: the first one, coinciding with the autumn-winter period, with an orthophosphate concentration in the influent exceeding 0.8 mg-PO₄³⁻/L and the second with a phosphorus concentration of between 0.6 and 0.8 mg-PO₄³⁻/L, coinciding with the spring-summer period. This variability depends substantially on the groundwater and the fact that there is no specific treatment stage in the WTP for this parameter. In order to test the E33 under the same working conditions as the WTP, no phosphorus pre-treatment controls were done during the experiments.

The arsenic breakthrough curve is presented in [Figure 3](#), which shows that the arsenic-adsorption capacity of the E33 was substantially exhausted in the first 20,000 bed volumes (Bv) ($C/C_0 = 0.75$). Comparison of [Figures 2](#) and [3](#) shows that competitive adsorption occurs between arsenic and

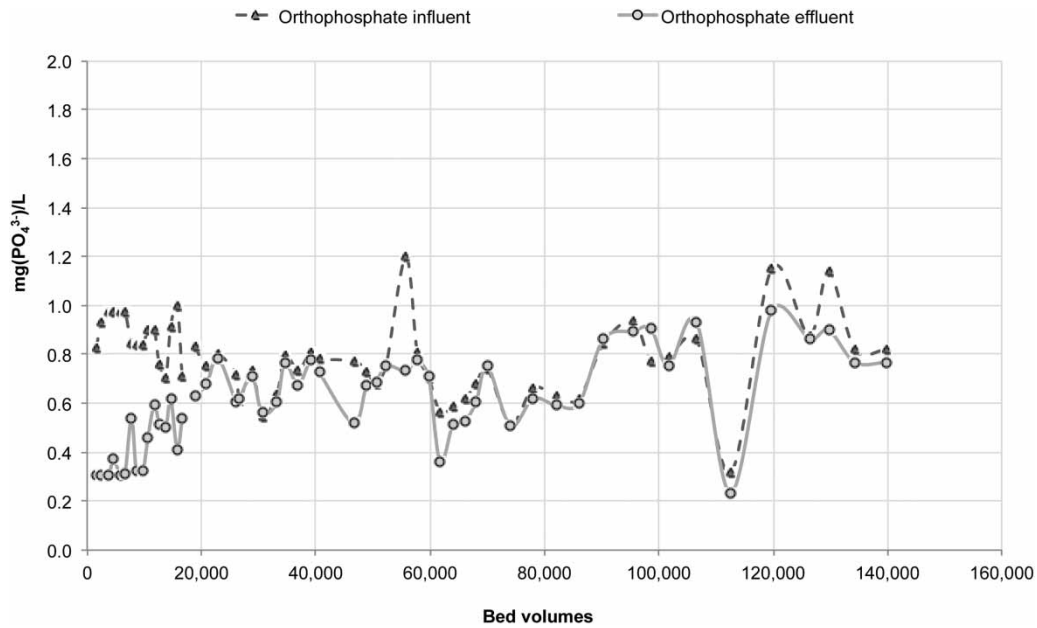


Figure 2 | Orthophosphate concentration trends in the influent (triangles) and effluent (bullets).

phosphorus. In fact, [Figure 2](#) shows average PO_4^{3-} removal efficiency of 40% in the first 20,000 Bv, with maximum removal efficiency of about 65% in the first 5,000 Bv. After 20,000 Bv, some phosphorus removal capacity remains although the difference between the influent and effluent concentrations tends towards zero. These data confirm that phosphorus was removed at the same time as arsenic throughout the tests.

Although the phosphorus compromised the E33's removal efficiency, the media did remove a useful amount of the arsenic up to 40,000 Bv. After this, the E33 was essentially exhausted, technically ($C/C_0 = 0.85$), and media replacement was necessary. In order to investigate the complete exhaustion of the E33, the experiment was extended to 140,000 Bv. From [Figure 3](#) it can be seen that E33 exhaustion was reached at about 80,000 Bv, when $C/C_0 = 0.90$.

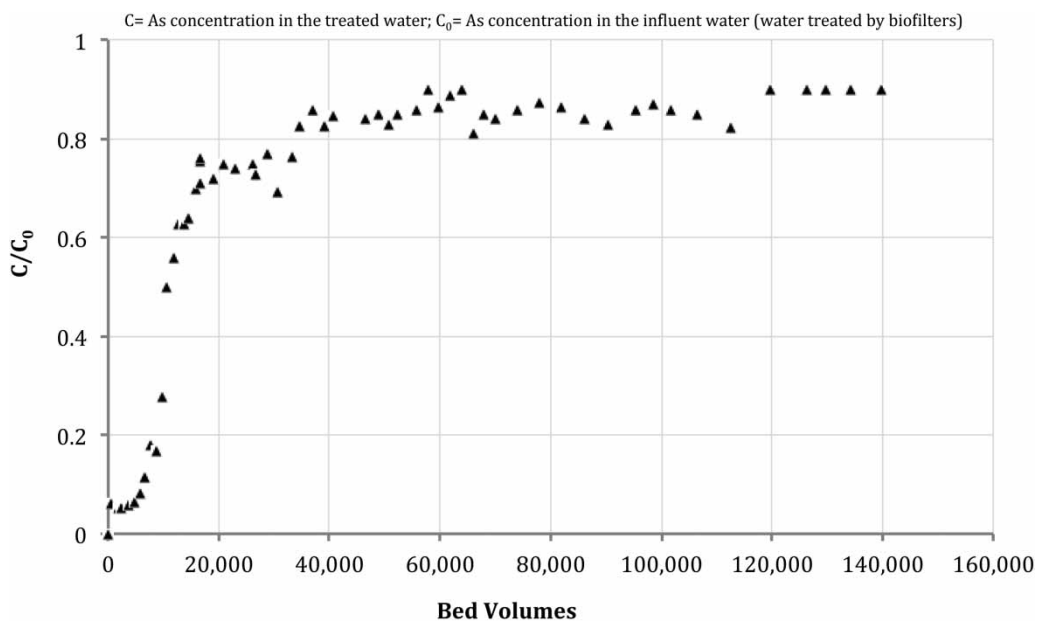


Figure 3 | Total arsenic breakthrough curve for the GFO pilot filter.

The Bv value achieved in the tests suggests that long operating cycles and low operating costs can be achieved in plants operating with groundwater arsenic concentration close to the MCL and low concentrations of other compounds that can affect the adsorption, i.e. interferents (e.g. silica, phosphorus, vanadium, etc.).

Figure 4 shows the concentrations of different chemical species of arsenic in the influent and effluent waters. The high content of As(V) in the influent (78% of the water's total arsenic content) is consonant with the WTP's configuration. In the biological filters the As(III) naturally present in the groundwater (where As(III) is the prevalent species, accounting for 92% of the total arsenic) is oxidized by the pyrolusite and biological processes (Singh *et al.* 2015).

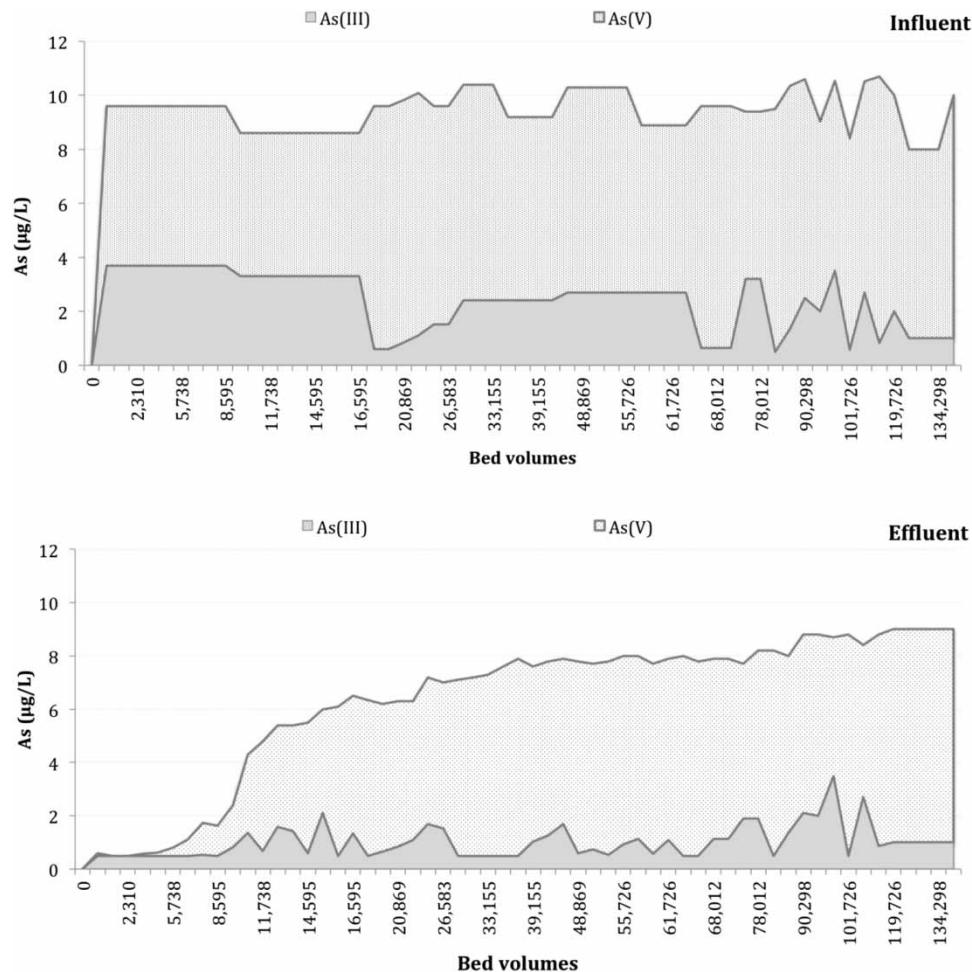


Figure 4 | Comparison of the concentrations of the chemical species As(III) and As(V) in the influent and effluent waters.

As can be seen in Figure 4, the ratio of As(III)/As(V) in the influent remained broadly constant (0.30) during the test but dropped to 0.18 in the effluent. Comparison of the As(III) concentration trends in the influent and effluent waters shows that the average concentration in the latter is about half that in the former (1.0 vs 2.3 µg/L). This indicates that As(III) is first converted in As(V) and then adsorbed by the media mainly in this form, confirming the findings of Mondal *et al.* (2013). Moreover, it can be also seen that the oxidation capacity of the E33 does not change during the experiment, in fact, the amount of As(III) oxidized is substantially constant throughout the test.

Comparing the trends of As reported in Figure 4, it seems that the arsenic is mainly adsorbed by the media as arsenic (V); in fact, in the first 5,000 Bv all As(V) contained in the feeding water has been removed while As(III) is found in the effluent.

In order to investigate the prevalent species removed by the E33, four different media response periods (periods that can be assumed homogenous with respect to operating conditions) were analyzed: 0–5,000; 5,001–20,000; 20,001–82,000; 82,001–140,000 Bv. In these the removal efficiencies have been calculated for the two most important arsenic species and the results are reported in Table 4.

Table 4 | Arsenic removal efficiencies in the different response periods

Response period (Bv)	Average removal efficiencies (%)		
	As _{tot}	As(III)	As(V)
0–5,000	94.3	96.4	99.1
5,001–20,000	51.6	67.8	44.8
20,001–82,000	20.2	52.9	10.6
82,001–140,000	9.2	9.8	7.3

For both species – As(III) and As(V) – the highest removal efficiencies were obtained in the first 5,000 Bv, after which removal decreased progressively. In the last removal period (82,001–140,000 Bv), As_{tot} removal efficiency was very low (9%) confirming that the E33 was effectively exhausted ($C/C_0 > 0.9$) and needed to be replaced.

With respect to the high removal efficiencies found for As(III), it must be underlined that it was not possible to distinguish the contributions of the adsorption and oxidation processes, so, removal must be considered as the sum of those contributions and some adsorption of As(III) cannot be excluded. This confirms reports by Mohan & Pittman (2007).

When the experiment ended, the adsorption capacity of the GFO was estimated as 0.57 $\mu\text{g-As}_{\text{tot}}/\text{L}$.

CONCLUSIONS

Among IBs, GFO is available for arsenic removal in water treatment. In many parts of Pianura Padana arsenic occurs in the groundwater at concentrations of 10 to 20 $\mu\text{g/L}$, slightly above the MCL of 10 $\mu\text{g/L}$.

A GFO pilot filter was installed and monitored for 19 months as a polishing stage in a WTP. The mean influent arsenic concentration in the pilot plant was reasonably constant during the tests at about 9.6 $\mu\text{g-As}_{\text{tot}}/\text{L}$.

Other chemical species present in the groundwater that could potentially have affected arsenic adsorption were orthophosphate (0.80 mg/L) and silica (24 mg/L). The results show that only orthophosphate affected arsenic removal, especially in the first 20,000 Bv, when competitive adsorption occurred with arsenic. Nevertheless, the GFO was able to remove some arsenic up to 40,000 Bv, showing a total adsorption capacity of 0.57 $\mu\text{g-As}_{\text{tot}}/\text{mg}_{\text{GFO}}$. Beyond this point, the GFO can be considered exhausted technically ($C/C_0 = 0.9$) and GFO replacement must be considered. The results also show that As(III) was removed at a low rate and its average concentration in the effluent was negligible.

Economically, GFO filter installation could be attractive because the operating costs – related to the As concentration in the influent – are comparable to those of other physico-chemical processes.

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