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(54) **Title**: REMOVAL OF ARSENIC FROM WATER

(57) **Abstract**: There is disclosed a water purification process comprising, adding Fe(II) ions to water, b) subjecting the water to oxidizing conditions,c) leaving any formed complex in contact with the water for at least one hour, d) filtering away formed complexes, wherein complexes are in contact with the water which passes the filter, and e) retaining complexes in the filter for at least 24 hours, wherein the pH of the water is adjusted to be in the interval 5-7.5, wherein Fe(II) ions first are added in a ratio of Fe to As from 50 to 5000, and wherein Fe(II) ions subsequently are added in a ratio of Fe to As from 10 to 50. Advantages include a low consumption of Fe(II) ions, a possibility to reach very low concentrations of arsenic, no harmful or toxic additives are used, and arsenic is bound in stable complexes after removal from the water.

Removal of arsenic from water

Technical field

The present invention relates to removal of arsenic from water.

Background

It is desirable to remove arsenic from water because of its well known toxic properties. Arsenic has been linked for instance to cancers of the lung, bladder, and skin in humans

Arsenic is present in many minerals. One example of mineral comprising arsenic is arsenopyrite (FeAsS). Under oxygen rich conditions arsenic may be present in groundwater in compounds such as but not limited to arsenate (AsO_4^{3-}) compounds, where arsenic is present as As(V). Under conditions with low oxygen arsenic may be present in groundwater in compounds such as but not limited to arsenite (AsO_3^{3-}), where arsenic is present as As(III).

Many countries have limits for arsenic in drinking water. Examples of limits for arsenic in drinking water include but are not limited to 10 µg/l.

Removal of arsenic from water has been studied before.

US 5,252,003 discloses a method for treatment of particulate materials containing arsenic compounds. The method comprises contacting the material with a source of iron(III) ions and magnesium(II) ions to stabilise the material against leaching of arsenic there from.

US 5,358,643 describes a method to remove arsenic from water comprising adding an iron salt such as $FeSO_4$.

DE 10 2006 028 172 A1 discloses a process to remove arsenic from water, in particular *in situ* from ground water by precipitation after contacting with aqueous iron hydroxide.

US 2006/0243668 A1 teaches increased dissolved oxygen level in the ground water in order to lower undesirable constituents such as iron and arsenic.

Summary

It is an object of the present invention to provide an improved process for the removal arsenic from water.

In a first aspect there is provided a process for the removal of arsenic from water comprising the steps:
a) adding an aqueous solution comprising Fe(II) ions to the water, said aqueous solution comprising Fe(II) ions having a pH from 1 to 6,
b) subjecting the water to oxidizing conditions to at least partially oxidize Fe(II) to Fe(III), whereby complexes are formed,
c) leaving any formed complex in contact with the water for at least one hour,
d) filtering away essentially all of any formed complexes, wherein complexes which are retained in the filter are in contact with the water which passes the filter, and
e) retaining complexes in the filter for at least 24 hours, wherein the pH of the water is adjusted during the process to be in the interval 5-7.5, wherein Fe(II) ions first are added in a ratio of Fe to As from 50 to 5000 in order to create large complexes, and wherein Fe(II) ions

subsequently are added in a ratio of Fe to As from 10 to 50.

Further aspects and embodiments are defined in the appended claims, which are specifically incorporated herein by reference.

One advantage is that the consumption of Fe(II) ions is reduced.

Another advantage is that it is possible to reach very low concentrations of arsenic.

A further advantage is that no harmful or toxic additives are used.

Yet another advantage is that arsenic is bound in stable complexes after removal from the water.

Definitions

Before the invention is disclosed and described in detail, it is to be understood that this invention is not limited to particular compounds, configurations, method steps, substrates, and materials disclosed herein as such compounds, configurations, method steps, substrates, and materials may vary somewhat. It is also to be understood that the terminology employed herein is used for the purpose of describing particular embodiments only and is not intended to be limiting since the scope of the present invention is limited only by the appended claims and equivalents thereof.

It must be noted that, as used in this specification and the appended claims, the singular forms “a”, “an” and

"the" include plural referents unless the context clearly dictates otherwise.

If nothing else is defined, any terms and scientific terminology used herein are intended to have the meanings commonly understood by those of skill in the art to which this invention pertains.

The term "about" as used in connection with a numerical value throughout the description and the claims denotes an interval of accuracy, familiar and acceptable to a person skilled in the art. Said interval is ± 10 %.

A roman number used in connection with the symbol of an element denotes the oxidation number of that element. For instance As(III) denotes arsenic with an oxidation number of +3. As(V) denotes arsenic with an oxidation number of +5.

"Aerate" as used throughout the description and the claims denotes the process of supplying air. Water can be aerated by mixing air with the water. Examples of mixing air and water include stirring and bubbling of air through the water.

"Complex" as used throughout the description and the claims denotes an assembly of atoms and/or ions such as for instance an assembly comprising Fe(III) ions.

"Drinking water" as used throughout the description and the claims denotes water which is intended to be potable or purified to be potable. The term drinking water includes contaminated water which has not yet been

purified, but which is intended to be purified to be potable.

"Ratio of Fe to As" as used throughout the description and the claims denotes the ratio between the content of Fe as calculated by weight and the content of As as calculated by weight. The weight of Fe and As is calculated as the corresponding amount of elemental Fe and As respectively.

Detailed description

In a first aspect there is provided a process for the removal of arsenic from water comprising the steps:
a) adding an aqueous solution comprising Fe(II) ions to the water, said aqueous solution comprising Fe(II) ions having a pH from 1 to 6,
b) subjecting the water to oxidizing conditions to at least partially oxidize Fe(II) to Fe(III), whereby complexes are formed,
c) leaving any formed complex in contact with the water for at least one hour,
d) filtering away essentially all of any formed complexes, wherein complexes which are retained in the filter are in contact with the water which passes the filter, and
e) retaining complexes in the filter for at least 24 hours, wherein the pH of the water is adjusted during the process to be in the interval 5-7.5, wherein Fe(II) ions first are added in a ratio of Fe to As from 50 to 5000 in order to create large complexes, and wherein Fe(II) ions subsequently are added in a ratio of Fe to As from 10 to 50.

In one embodiment the water is drinking water.

The steps of the process are described in greater detail in the following:

Step a):

In one embodiment the Fe(II) ions are added in an acid solution. The pH is between 1 and 6. In one embodiment the pH is from 2 to 3. The low pH prevents oxidation of the iron in the aqueous solution. If the pH of the drinking water has to be lowered the acid will act to adjust the pH.

Step b):

Examples of oxidizing conditions include but is not limited to aerating and stirring the water. Air can be bubbled through the water. In one embodiment oxidation agents are added. Examples of oxidation agents include but are not limited to $KMnO_4$ and H_2O_2.

Fe(II) is at least partially oxidised to Fe(III). During oxidation of Fe(II) to Fe(III) the molecular structure changes. Without wishing to be bound by any particular scientific theory the inventor believes that the reason is that the ion Fe(III) does not exist in drinking water solutions and that $Fe(OH)_6^{3+}$ are formed which are deprotonized and forms large complexes with a positive charge. If arsenic is present in the solution, being it as arsenite or arsenate, these negative ions will be built into the complexes formed.

However, in drinking water there are other ions present which can compete with the arsenite/arsenate ions to join the complexes. Such ions are PO_4^{2-} and SO_4^{2-}. To get a good precipitation of arsenic a surplus of Fe(II) is needed due to the coprecipitation of other ions which can compete as

they are frequently present at higher concentrations than arsenic. A so called coprecipitation takes place forming amorphous structures of nano size. During this stage As(III) is at least partly oxidized to As(V).

In one embodiment the ratio of Fe to As is from 10 to 5000.

The complexes are formed instantaneously and arsenic is linked strongly to the complexes. The structure of these complexes varies and is dependent on the actual concentrations in the solutions and each of them has a different solubility product.

When the relationship of Fe to As is low, such as below 50, small complexes with a size of about 10 microns are formed. If a high relationship Fe/As is used, such as above 50, big complexes are formed which easily can be separated by for instance sedimentation or filtration. The presence of ions in the water influences the ratio of Fe to As that is necessary.

Step c):
In step c) when the complex is in contact with the water different processes take place.

The time during which any formed complex is in contact with the water is calculated from the formation of the complex to the point where the water leaves the filter.

In one embodiment any formed complex is in contact with the water for at least 1 hour.

In one embodiment any formed complex is in contact with the water for at least 10 hours.

In one embodiment any formed complex is in contact with the water for at least 50 hours.

In one embodiment any formed complex is in contact with the water for at least 70 hours.

In one embodiment any formed complex is in contact with the water for at least 100 hours.

In one embodiment any formed complex is in contact with the water for at least 120 hours.

The arsenic ions $H_2AsO_4^-$ and $HAsO_4^{2-}$ are the main ions present after oxidation at pH levels which are the normal for water.

In order to further remove arsenic a different mechanism will be used, called arsenic adsorption. The adsorption mechanism involves adsorption of arsenic to the surface of the complexes. The mechanism is dependent on pH and the ionic strength. In one embodiment the pH is between 6 and 7. In an alternative embodiment the pH is between 5 and 7.5. The process is most efficient at around neutral pH or slightly acid conditions, whereas arsenic can be desorbed at basic conditions. Therefore highly basic conditions are less preferred. If the pH is too high the pH should be lowered by addition of an acid. In one embodiment an acidic solution comprising Fe(II) ions is used. One advantage of using an acidic solution comprising Fe(II) ions is that oxidation of Fe(II) is avoided during storage.

The time required for the adsorption mechanism is longer than for the precipitation of complexes. Also in this stage there is a competition between ions.

Adsorption of arsenic on the surface of Fe(III) oxide complexes is also efficient at low concentrations of arsenic, providing an efficient way to further decrease the concentration of arsenic. Large Fe(III) oxide complexes are also efficient to adsorb other small Fe(III) oxide complexes to prevent them from passing a filter.

Step d):
In step d) essentially all of the formed complexes are removed. Essentially all means more than 98 wt% of all complexes, preferably more than 99.5 wt%. The filter is designed so that the complexes which are retained in the filter are in contact with the water which passes the filter.

Step e):
During step e) there is a transfer of arsenic from the surface of the Fe(III) oxide complexes to the inner parts. Through this process strong links are formed and the material gets more or less crystallized.

The transfer which takes place is slow but increases the use of the material as the surface gets reactivated and can be used to capture more arsenic. The complexes that are retained in the filter are retained for at least 24 hours. In one embodiment at least a part of the complexes are retained in the filter for 1-30 days. In one embodiment complexes are retained in the filter at least 1 day. The filter may be cleaned or replaced at suitable

intervals such as but not limited to one week. In one embodiment the complexes are retained in the filter until the filter is cleaned or replaced.

The material gets stabilized as arsenic will be more strongly connected inside the particles. From a stability point of view it is an advantage that the residual product comprising arsenic is stable.

The process comprises the following steps of adding Fe(II) in different ratios Fe to As which are further described:

i) adding Fe(II) ions in a ratio of Fe to As from 50 to 5000 a relatively short period of time such as but not limited to a few minutes to a few hours, examples include but are not limited to 5 minutes, 10 minutes, 15 minutes, and 20 minutes. In one embodiment this period of time is from 1 minute to 10 hours. In another embodiment this period of time is from 5 minutes to 60 minutes. In an alternative embodiment this period of time is from 1 hour to 10 hours. This is done in order to create large complexes. In one embodiment the Fe(II) ions are added in a ratio Fe to As from 50 to 5000 during a period of time from 1 minute to 10 hours. In one embodiment the high ratio of Fe to As is from 50 to 500. In one embodiment the high ratio of Fe to As is from 50 to 1000.

ii) adding Fe(II) ions in a lower ratio of Fe to As from 10 to 50 for a longer period of time, examples include but are not limited to half a day, one day, one week, and one month or more. In one embodiment this period of time is from 6 to 120 hours. In an alternative embodiment this period of time is from 1 day to 1 week. In one embodiment the Fe(II) ions are added in a lower ratio of Fe to As

during an extended period of time that lasts until the filter is replaced or cleaned. In one embodiment the Fe(II) ions are added in a ratio Fe to As from 10 to 50 during a period of time from 6 hours to one month. In one embodiment the Fe(II) ions are added in a ratio Fe to As from 10 to 50 during a period of time from 12 hours to 10 days. In another embodiment the lower ratio of Fe to As is from 5 to 50.

The high ratio of Fe to As creates large complexes with a large surface which can adsorb arsenic and also smaller complexes. The large complexes are efficient to capture both arsenic and also small complexes.

When the concentration of Fe ions is lowered, i.e. a lower ratio of Fe to As is used, smaller complexes will be formed, but the smaller complexes will be captured by the large complexes which are still in the system. In one embodiment the small complexes have a size of around 10 µm, which would pass many filters, but due to the initial use of a high ratio of Fe to As large complexes with a large surface have been created and the small complexes will be adsorbed to the large complexes. The larger complexes initially formed will also take up arsenic from the water by adsorption to the surface of the complexes. In one embodiment the low ratio of Fe to As is from 10 to 50. One advantage is that a lower amount of Fe ions can be used.

In one embodiment the steps of i) adding Fe(II) ions in a ratio of Fe to As from 50 to 5000 and b) adding Fe(II) ions in a ratio of Fe to As from 10 to 50 are repeated.

Examples of duration times between the repetitions of the steps a) and b) include but are not limited to, 10 hours, 24 hours, 36 hours, 48, hours, 5 days, 10 days, 1 month, and three month.

In one embodiment $FeSO_4$ is used as a source of Fe(II) ions. This is an advantage since $FeSO_4$ is cheap and does not give any harmful effects in the environment.

In one embodiment at least steps c), d) and e) are performed in the soil in the boulder.

The porosity of the soil has to be such that a good flow is achieved. A compact clay soil is not suitable.

When the process is carried out in the boulder, the oxidation is done by air, which in one embodiment is added into the ground water through satellite wells placed in a ring around the production well, from which the cleaned potable water is pumped up. The ground water on its way to the production well passes the aerated area. Air is in one embodiment added through an injector.

In one embodiment a solution comprising Fe(II) ions is added to the satellite wells and oxidation will take place by injected air in the zone between the satellite and production wells forming complexes which will behave as described above and form stable crystalline complexes comprising iron and arsenic. In one embodiment the porous volume in the boulder is enough to act as a filter for a life span over thousands of years. Advantages of this embodiment include that it is simple and does not require maintenance of a filter. The arsenic will be bound in the soil.

Other features and uses of the invention and their associated advantages will be evident to a person skilled in the art upon reading the description and the examples.

It is to be understood that this invention is not limited to the particular embodiments shown here. The following examples are provided for illustrative purposes and are not intended to limit the scope of the invention since the scope of the present invention is limited only by the appended claims and equivalents thereof.

Examples

Example 1

In a laboratory test drinking water with an arsenic content of 53 micrograms of As per litre was added a solution of FeSO4 in distilled water in an amount which corresponded to 530 microgram of Fe per litre, which means a relationship of Fe/As of 10. The sample was aerated for one hour and then stirred for another hour. The sample was filtered through a filter with a pore size of 20 microns. The filtrate thus obtained was analyzed with AAS which demonstrated that it contained less than 5.2 micrograms As per litre, which is the detection limit of the laboratory used. The pH of the water was 6.

Example 2

Example 1 was repeated with the same water at different relations of Fe/As, 50, 100, 300, 600, 900 and 1200 at a pH of 6. The filtrate thus obtained was analyzed with AAS which demonstrated that it contained less than 5.2 micrograms As per litre, which is the detection limit of the laboratory used.

Example 3

Tests of the same water with Fe/As = 30 and 80 and with pH 7.79 and 7.84 respectively, the same time for aerating and mixing but in which the sample was allowed to stand for sedimentation for 120 hours prior to the filtration had after filtration concentrations in the filtrate of 11.4, 16.0 and 17.3 micrograms As per litre. In the samples poorly yellow particles could be seen floating in the bottom of the beaker.

Example 4

Example 3 was repeated with Fe/As ratio = 100, with pH 6 and where the sample was allowed to stand for sedimentation for 72 hours prior to the filtration. The sample had after filtration a concentration of As less than 5.2 micrograms per litre. Very small cream color particles could be observed.

Example 5

Example 1 was repeated with the same water but with the difference that the water had not been passing a de-ionization stage, meaning that the concentrations of calcium and other ions were much higher, treatment at a Fe/As-relation of 10 with 1 hour aeration and 1 hour mixing followed by filtration gave a concentration if As in the filtrate of 7.9 microgram per litre.

Example 6

In a laboratory test with water having a concentration of 13 micrograms of As per litre and a pH of 7.7 it was treated with acid $FeSO_4$ to get a pH of 7.4 and a relation Fe/As of 100. The water was standing for 2 hours in contact with the precipitate and then filtrated. The

filtrate contained 0.3 micrograms As per litre. The detection limit was 0.1 micrograms As per litre drinking water.

Example 7

Water with a pH of 8.6 and a concentration of As of 84 micrograms per litre was treated with acid $FeSO_4$ to obtain a pH of 6.9 and a Fe/As-relation of about 50. After aeration for 60 minutes the water was filtrated and a concentration of 46 micrograms As was obtained.

Example 8

Example 7 was repeated but letting the sample stand in contact with the precipitate for 90 minutes and the As concentration was analyzed to be 2.5 micrograms per litre after filtration. The detection limit was 0.1 micrograms As per litre drinking water.

Example 9

This test was made in an installation designed for 10 consumers of a drinking water containing 56 micrograms of As per liter.

The installation was made up of a pump from the well, a pressure tank, a storage tank for an acid solution of $FeSO_4$, a storage tank for a solution of $KMnO_4$ and a sand filter with automatic cleaning.

The $FeSO_4$ solution was fed into the water pipe immediately after the pump at times when the pump was working. The $KMnO_4$ solution was fed into the water pipe immediately after the feeding point for $FeSO_4$ and controlled by the water flow from the pressure tank.

The amount of $KMnO_4$ that was fed was determined by the amount of $FeSO_4$ in such a way that a stochiometric surplus of the permanganate was assured.

In the test the $FeSO_4$ solution was fed until the Fe/As ratio was calculated to be 95. After some 46 hours a water sample was taken from the pressure tank and the pH was measured to 5.5. The feeding rate was then reduced to get a calculated Fe/As ratio of 18 and after further 5 days a sample was taken from the clean water which was analyzed to contain about 1 microgram of As per liter.

Example 10

In a similar test as in example 9 was made at another occasion in the same installation, a clean water sample was taken at somewhat different conditions: The calculated Fe/As ratio was 90, the pH in the pressure tank was measured to be 5.4 and after two hours of operation the clean water was analyzed to contain 28 micrograms of As per liter.

Claims

1. A process for the removal of arsenic from water comprising the steps:

a) adding an aqueous solution comprising Fe(II) ions to the water, said aqueous solution comprising Fe(II) ions having a pH from 1 to 6,

b) subjecting the water to oxidizing conditions to at least partially oxidize Fe(II) to Fe(III), whereby complexes are formed,

c) leaving any formed complex in contact with the water for at least one hour,

d) filtering away essentially all of any formed complexes, wherein complexes which are retained in the filter are in contact with the water which passes the filter, and

e) retaining complexes in the filter for at least 24 hours,

wherein the pH of the water is adjusted during the process to be in the interval 5-7.5,

wherein Fe(II) ions first are added in a ratio of Fe to As from 50 to 5000 in order to create large complexes, and wherein Fe(II) ions subsequently are added in a ratio of Fe to As from 10 to 50.

2. The process according to claim 1, wherein the water is drinking water.

3. The process according to any one of claims 1-2, wherein any formed complex is in contact with the water for at least 10 hours.

4. The process according to any one of claims 1-2, wherein any formed complex is in contact with the water for at least 50 hours.

5. The process according to any one of claims 1-4, wherein said aqueous solution comprising Fe(II) ions has a pH from 2 to 3.

6. The process according to any one of claims 1-5, wherein the steps of adding Fe(II) ions in a ratio Fe to As from 50 to 5000 and subsequently in a ratio of Fe to As from 10 to 50 are repeated.

7. The process according to claim 6, wherein the duration time between the repetitions are at least 10 hours.

8. The process according to any one of claims 1-7, wherein Fe(II) ions are added in a ratio Fe to As from 50 to 5000 during a period of time from 1 minute to 10 hours.

9. The process according to any one of claims 1-8, wherein Fe(II) ions are added in a ratio Fe to As from 10 to 50 during a period of time from 6 hours to one month.

10. The process according to any one of claims 1-9, wherein $FeSO_4$ is used as a source of Fe(II) ions.

11. The process according to any one of claims 1-10, wherein at least step c), d) and e) of claim 1 are performed in the soil in the boulder.

INTERNATIONAL SEARCH REPORT

International application No.
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A. CLASSIFICATION OF SUBJECT MATTER

IPC: see extra sheet
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC: C02F, B09C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-INTERNAL, WPI DATA, PAJ, COMPENDEX

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	DE 102006028172 A1 (UNIVERSITÄT HANNOVER), 20 December 2007 (20.12.2007), paragraphs [0012]-[0019],[0039]	1-11
A	US 20060243668 A1 (MILLER G.P. ET AL), 2 November 2006 (02.11.2006), paragraphs [0032]-[0034],[0051]-[0052],[0065]	1-11
A	CN 101264965 A, UNIV HARBIN POLYTECHNIC, 2008-09-17: (abstract) Retrieved from: WPI database, WEEK 200867, AN 2008-L34348	1-11

[X] Further documents are listed in the continuation of Box C. [X] See patent family annex.

* Special categories of cited documents:
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Date of the actual completion of the international search	Date of mailing of the international search report
22 Sept 2009	23 -09- 2009
Name and mailing address of the ISA/ Swedish Patent Office Box 5055, S-102 42 STOCKHOLM Facsimile No. +46 8 666 02 86	Authorized officer Jens Waltin/ELY Telephone No. +46 8 782 25 00

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International application No.
PCT/SE2009/050799

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	DE 4313425 A1 (HÖLZEL, GERD), 30 June 1994 (30.06.1994), abstract --	1-11
A	US 20020003116 A1 (GOLDEN J.H.), 10 January 2002 (10.01.2002), paragraphs [0012]-[0016] -- --------	1-11

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C02F 1/62 (2006.01)
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Paper copies can be ordered at a cost of 50 SEK per copy from PRV InterPat (telephone number 08-782 28 85).

Cited literature, if any, will be enclosed in paper form.

DE	102006028172	A1	20/12/2007	NONE
US	20060243668	A1	02/11/2006	NONE
DE	4313425	A1	30/06/1994	NONE
US	20020003116	A1	10/01/2002	NONE