Research Article

Iron Nanoparticle Filter for Remediation of Groundwater Contaminated by Geogenic Arsenic

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Abstract

Naturally occurring arsenic in geological formations is a major source of groundwater contamination worldwide, including Bolivia. Physical-chemical studies have identified high concentrations of arsenic in water from deep and shallow wells in various Bolivian regions. In the Lake Poopó basin, arsenic levels in shallow wells exceed Bolivian and WHO standards by up to 25 times. In Cochabamba, groundwater arsenic content reaches up to 113 μg-As/l. Chronic arsenic exposure poses health risks such as neurological, dermatological, and carcinogenic effects. To date, no measures have been taken to mitigate this issue, posing a serious threat to the health and livelihoods of the rural population. In this study, iron and iron oxide nanoparticles obtained from recycling were used to filter arsenic, successfully reducing concentrations below Bolivian drinking water standards.

Keywords: Water; Treatment; Adsorption; Nanotechnology

Introduction

The geogenic presence of arsenic in groundwater represents a global health threat and currently affects all 5 continents. Its permanent consumption causes the appearance of various types of cancer, such as cancer of the skin, lungs, kidneys, liver, and potentially the prostate, as well as non-cancerous disorders including skin lesions, cardiovascular diseases, effects on the nervous system, and diabetes [1,2]. It is estimated that about 220 million people worldwide are potentially exposed to high concentrations of arsenic in groundwater with the prospect of further increases as new contaminated areas are constantly being discovered [3,4]. In Bolivia, so far, some studies have documented the presence of geogenic arsenic in the Bolivian Altiplano, in quite high concentrations in the southern part of Lake Poopó, especially in the town of Quillacas with approximately 250 μg/l and the lower valley of Cochabamba [5-7]. Recent research shows the distribution of arsenic in different regions of Bolivia, including some wells in the lowlands, which have As concentrations higher than the maximum permissible value of 10 μg/l established by the Bolivian regulation NB 512 for drinking water [8].

There are several technologies available to achieve the demanding arsenic removal required for human consumption of water, however, the economic variable is a determining factor [9].

According to Hao, et al. (2018) among many currently available techniques for arsenic removal from water, the adsorption process is considered one of the most promising techniques due to its low cost, high efficiency, and ease of operation [10]. Iron-based adsorbents have been developed extensively and showed good removal efficiency of arsenic species from water. Removal of arsenic from water by adsorption on nanomaterials is being studied as a promising alternative method to conventional methods (reverse osmosis, nanofiltration, ion exchange, etc.) because of its large specific surface area, catalytic potential, low energy consumption, high efficiency, and reversibility [11].

A literature search was performed in the Scopus database, focusing on articles related to the remediation of groundwater contaminated with arsenic by adsorption with zerovalent iron nanoparticles (ZVI) and iron sesquioxide (magnetite). 51 records were found. It can be concluded that the application of nanotechnology in the treatment of arsenic-contaminated water, particularly with ZVI nanoparticles and magnetite, is a topic of growing importance in the last decade.

Casentini, et al. (2019) analyzed the use of Fe hydroxide nanoparticles encapsulated in an Exopolysaccharide Gel (FeEPS) for arsenic removal in aqueous solutions [12].

The pH of the hydrogel was found to affect its adsorption capacity, being less favorable than magnetite nanoparticles. The higher removal efficiency was observed in more concentrated FeEPS solutions due to a higher amount of iron nanoparticles present. Immobilization of FeEPS on carbonaceous shells was shown to be effective in arsenic adsorption.

Shahmohammadi, et al. (2019) investigated the adsorption of arsenic in well water using a filter with an iron matrix (ZVI) [13]. The effectiveness of all layers of the filter was examined and variables such as pH, flow rate, arsenic concentration, turbidity, and chloride concentration were considered to optimize the process. The filter was applied for 10 hours. It was found that the turbidity and flow rate had the highest correlation with adsorption efficiency. Optimal adsorption conditions included: Initial arsenic concentration 500 µg/L, flow rate 2 L/h, pH 7, turbidity 0 NTU, and absence of calcium hypochlorite. The filter had a removal efficiency of 98.7%. In conclusion, the proposed filter demonstrated high efficiency in the adsorption of arsenic in water.

According to Sista, et al. (2021), Iron Nanopowder (INP) is a prominent option due to its low cost, high reactivity, and commercial availability [14]. It uses various mechanisms to remove arsenic, such as oxidation, reduction, adsorption, precipitation, and co-precipitation. The properties of the (INP) and the aqueous system, such as pH and the presence of other contaminants, influence its effectiveness. (INP) obtained by electrolytic and reduction routes are preferred. This review highlights the potential of (INP) at micrometric and nanometric levels in arsenic removal in water remediation. The different synthesis routes, removal mechanisms, research evolution, and commercial occurrence of (INP) are also discussed.

The objective of our research is the design, construction, and performance evaluation of a simple and functional filter applied to arsenic removal from groundwaters contaminated with arsenic of geogenic origin. The filter contains Zero-valent Iron (ZVI) and iron oxide nanoparticles obtained from recycling metal chips by top-down and bottom-up technology, combined with other easily accessible artisanal filtering materials such as charcoal, ground brick, and river sand, in the form of layers for arsenic adsorption. The filter is designed to be used as a household device for water purification.

Materials and Methods

Water characterization

Sampling followed the standard protocol [4,15]. The pH, electrical conductivity, temperature, and redox potential were determined *in situ* using a Hanna Instruments model HI 9828 meter. Alkalinity was measured *in situ* using a Hach model 16900 titrator. Samples were microfiltered (0.45 µm) in polyethylene bottles, acidified with 1% HNO_3, for analysis of the major cations and trace metals in well water, For anions the sample was not acidified. All samples were refrigerated at 4°C before analysis. Cl^-, NO_3^-, NO_2^-, SO_4^{2-}, F^- and PO_4^{3-} ions were determined by ion chromatography (Dionex ICS-1100). Ca, Mg, Na, and K were quantified using a Perkin Elmer Analyst 200 flame atomic absorption spectrophotometer at the Instituto de Investigaciones Químicas, Universidad Mayor de San Andrés. Heavy metals and arsenic were determined by inductively coupled plasma mass spectrometry, ICP-MS, (Agilent 7500) at the laboratory of the Instituto Geológico y Minero de España, IGME, Spain. For adsorption tests and filter performance, arsenic concentrations were measured using a PG Instruments Model AA500 atomic absorption spectrometer with a hydride generator in the laboratories of the Universidad Privada Boliviana in Cochabamba.

Obtaining nanoparticles

The iron nanopowder was obtained with top-down technology in a high-energy ball mill, selection, and magnetic separation as reported by Torres (2021) [16]. The collection was optionally performed by:

1. the wet method: In a liquid solution of iron particles plus isopropyl alcohol with a magnetic field and

2. the dry method: In a high-energy ball mill with a filter.

To obtain the iron oxide nanopowder the bottom-up technology was used with the aid of electrochemical synthesis, from a solution of iron sulfate in distilled water. DC electric current was applied between the cathode (stainless steel plate) and the anode (conductive bar of compacted iron microparticles of 45 µm), leading to the deposition and accumulation of iron oxide nanoparticles on the cathode plates and the bottom of the tank [17].

Characterization of the nanoparticles

The chemical composition of both nanoparticles was quantified by Scanning Electron Microscopy with Energy Dispersive X-Ray Spectroscopy (SEM-EDX).

The size distribution of the nanoparticles was performed on a TESCAN VEGA3 LMU scanning electron microscope. Both analyses were performed at the physics laboratory of the University of San Simon in Cochabamba Bolivia.

Design and construction of the pilot prototype filter

The filter was constructed with a 2" (50.8 mm) diameter High-density Polyethylene (HDPE) SCH 40 pipe, one meter long, made of recycled material, with threads at the ends to install couplings for reduction to ½" (12.7 mm) as a commercial measure. The tube was filled as shown in Figure 1 and Table 1.

Table 1: Detail of each filter layer, component, size, weight, density, and volume of each one of the filter media

Component	Size (µm)* (nm)**	Weight (g)	Density (g/cm³)	Volume (cm³)
Sand	2000*	219	1.6	136.88
Iron microparticles	250*	300	7.86	38.17
Sand	2000*	208	1.6	136.25
Ground brick	4000*	144	1.4	102.86
Sand	2000*	219	1.6	136.88
Iron oxide nanoparticles	20-200**	25	5.2	4.81
Polyurethane sponge	-	0.1	0.024	4.17
Sand	4000*	219	1.6	136.88
Iron nanoparticles	20-500**	64	5.2	12.31
Sand	4000*	219	1.6	136.88
Charcoal	2000*	56	0.3	186.67
Sand	4000*	219	1.6	136.88
Coarse ground brick	4000*	357	1.4	255
(µm)* micrometer (nm)** nanometer				

Figure 1: ZVI and magnetite nanoparticle filter with layers detailed in Table 1

Adsorption tests

To determine the adsorption capacity, different doses (0.01 g-10 g) of both nanoparticles were placed in contact with synthetic solutions of As(V), As(III), and groundwater. The solutions were stirred for varying periods (20 min to 1 d). The initial and final As concentrations were measured to determine the removal efficiency and adsorption capacity.

Groundwater filtration test

Filtration was performed by gravity in continuous downflow mode, and arsenic removal performance was monitored as a function of the volume treated and the number of volumes of nanoparticle beds in the filter. Arsenic concentrations were measured at the inlet and outlet at different times, collecting samples for subsequent analysis.

Results

Untreated water quality

Table 2 presents the results of the physical and chemical parameters of the well located in District 4 of Cochabamba Bolivia (Cbba. Bo.). The water is slightly alkaline and the oxidation-reduction potential, Eh, showed a relatively oxidizing system, +198 mV. Its salinity is low to moderate (252 µS/cm). The majority of cations and anions indicate that the water is of the Na-Ca-Mg-HCO$_3$ type, as reported by Escalera and Ormachea (2017), for a set of wells located in the western peri-urban zone of (Cbba. Bo.) [18]. The quality satisfies the drinking water standard NB 512, except for arsenic and manganese contents. The arsenic content is 113 µg-As/l, much higher than the limit of 10 µg-As/l established by Bolivian standard NB 512. The contents of competitive anions; i.e. sulfates, nitrates, nitrites, and chlorides are relatively low except for phosphates. Arsenic is present in water in the form of arsenites, As(III). This species is more difficult to adsorb than arsenate, As(V).

Characterization of nanoparticles

Iron oxide nanoparticles (Magnetite): The percentage elemental composition is: Fe (57.7%), O (35.8%), Si (4.9%), S (1.3%), and Ti (0.3%). The size distribution is from 60 to 200 nm.
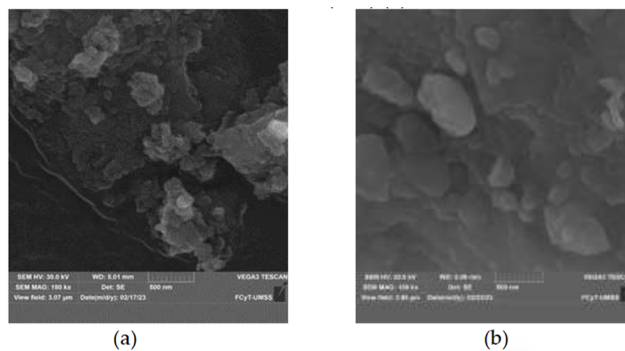
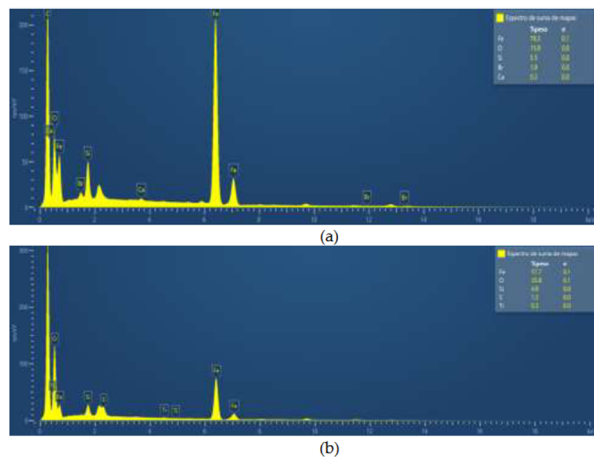Iron nanoparticles (ZVI). The chemical composition is Fe (76.3%), O (15.9%), Si (5.5%), Br (1.9%), and Ca (0.3%). The nanoparticles have a regular morphology, with sizes ranging from approximately 60 nm to 200 nm, (Figures 2 and 3) and (Table 3).

Table 2: Analysis of the quality of groundwater

Parameter	Analytical method	Limit of quant.	Result	Bolivian standard 512
Temperature (°C)	SMWW 2550 B. Temperature	-	19.3	-
Specific conductivity, (mS/cm)	SMWW 2510 B. Conductivity	-	252	1500
pH (-)	SMWW 4500 H+ B.	-	6.9	6.5-9.0
Eh, mV	Electrometric SMWW 2580 B. Oxidation-Reduction Potential	-	198	-
Total alkalinity, mg-CaCO$_3$/L	SMWW 2320 B. Titration	-	157	370
Fluoride, mg-F$^-$/L	SMWW 4110 F- B. Ion Chromatography	0.5	<LD	1.5
Chloride, mg-Cl$^-$/L 0.5	SMWW 4110 NO3- B. Ion Chromatography	0.5	0.3	250
Nitrate, mg-NO$_3$$^-$/L 0.5	SMWW 4110 NO2- B. Ion Chromatography	0.5	<LD	45
Nitrite, mg-NO$_2$$^-$/L 0.5	SMWW 4110 PO43- B. Ion Chromatography	0.5	<LD	0.1
Phosphate, mg-PO$_4$$^{3-}$/L 0.2	SMWW 4110 SO42- B. Ion Chromatography	0.2	9	-
Sulphate, mg-SO$_4$$^{2-}$/L 0.5	SMWW 3111 B. Direct Air-acetylene flame	0.5	0.3	400
Calcium, mg-Ca^{++}/L 0.5	SMWW 3111 B. Direct Air-acetylene flame	0.5	31.2	200
Magnesium, mg-Mg^{++}/L 0.05	SMWW 3111 B. Direct Air-acetylene flame	0.05	9	150
Sodium,, mg-Na$^+$/L 0.1	SMWW 3111 B. Direct Air-acetylene flame	0.1	29.9	200
Potassium, mg-K$^+$/L 0.2	SMWW 3111 B. Direct Air-acetylene flame	0.2	0.97	-
Aluminum, µg-Al/L	SMWW 3125 B. ICP/MS	1	1.9	100
Antimony, µg-Sb/L	SMWW 3125 B. ICP/MS	0.05	0.49	5
Arsenic, µg-As/L	SMWW 3125 B. ICP/MS	0.06	113	10
Barium, µg-Ba/L	SMWW 3125 B. ICP/MS	-	70.9	700
Cadmium, µg-Cd/L	SMWW 3125 B. ICP/MS	0.2	<LD	5
Copper, µg-Cu/L	SMWW 3125 B. ICP/MS	0.2	0.28	1000
Chrome, µg-Cr/L	SMWW 3125 B. ICP/MS	0.1	0.51	700
Iron, µg-Fe/L	SMWW 3125 B. ICP/MS	0.1	7.5	300
Manganese, µg-Mn/L	SMWW 3125 B. ICP/MS	0.1	442	100
Nickel, µg-Ni/L	SMWW 3125 B. ICP/MS	0.1	0.25	-
Lead, µg-Pb/L	SMWW 3125 B. ICP/MS	0.1	4.09	10
Zinc, µg-Zn/L	SMWW 3125 B. ICP/MS	0.1	10.9	5000

Table 3: Elemental composition of ZVI and iron oxide nanoparticles in weight percentage, (%)

Component	Composition of iron oxide nanoparticles (%)	Composition of ZVI nanoparticles (%)
Fe	57.7	76
O	35.8	15.9
Si	4.9	5.5
S	1.3	-
Ti	0.3	-
Br	-	1.9
Ca	-	0.3

(a)   (b)

Figure 3: Size distribution, (a) Image of iron oxide nanoparticles, (b) ZVI nanoparticles Observed at 200,000X magnification

Adsorption capacity

Table 4 and Figure 4 present the results of the treatment of groundwater characterized in Table 1. The percentage removal of total As (mostly As(III)) and the adsorption capacity of zero-valent iron and magnetite nanoparticles are presented. Starting from an As(III) concentration of 83 ppb, removal efficiencies of up to 43% could be achieved with ZVI and 41% with magnetite. After 20 min, a saturation of the active sites of the nanoparticles is reached, which allows the establishment of the equilibrium condition to calculate the adsorption capacity, reaching values of approximately 4 mg-As(III)/g for both nanoparticles.

Figure 2: Average chemical composition by EDS method of (a) iron nanoparticles (ZVI), (b) iron oxide

Table 4: Results of adsorption test on metallic iron and iron oxide nanoparticles. Nanoparticle concentration=10 mg/l

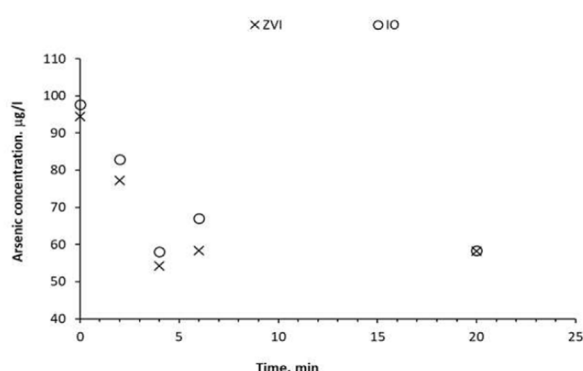| Time, min	Arsenic concentration, g-As(III)/L, ZVI mg/l	Arsenic concentration, g-As(III)/L, IO mg/l	Adsorption capacity, mg-As(III)/g. ZVI	Adsorption capacity, mg-As(III)/g. IO	As(III) removal efficiency, %, ZVI	As(III) removal efficiency, %, IO
0	94.44	97.73	0	0	0%	0%
2	77.34	82.85	1.71	1.49	18%	15%
4	54.29	58	4.02	3.97	43%	41%
6	58.37	66.99	3.61	3.07	38%	31%
20	58.27	58.4	3.62	3.93	38%	40%

Figure 4: Adsorption test results on metallic iron and magnetite nanoparticles. The concentration of nanoparticles=10 mg/l

Figure 5 presents the results of the adsorption tests in synthetic waters with As(V) with ZVI iron nanoparticles. Solutions of 500 ppb were subjected in each of the 13 runs and residual concentrations were determined after 1 h or 2 h, times at which equilibrium is achieved. Adsorption capacities ranged from 0.86 mg-As(V)/g to 2.99 mg-As(V)/g with an average of 1.54 mg-As(V)/g.
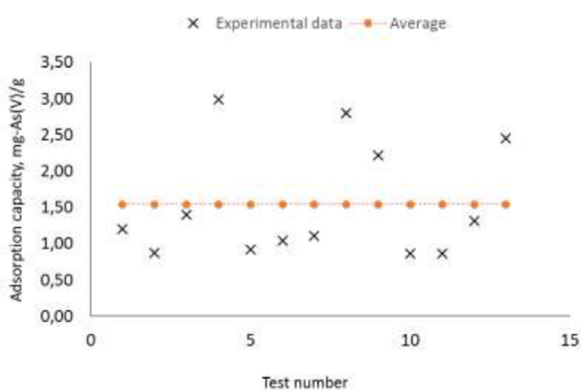
**Figure 5:** Adsorption capacity of As(V) on iron nanoparticles, ZVI

For As (V) in synthetic waters, a significant variability of the initial adsorption capacities is noted which is related to the variability of their size 20 nm-200 nm and their specific surface area (436 m^2/g-131 m^2/g). The adsorption capacities of nanoparticles are higher than other larger adsorbent materials such as ferric oxide-coated sands with 0.64 mg-As/g [18]. In contrast, smaller magnetic nanocomposites (11 nm-15 nm) have much higher capacities (74.9 mg-As/g), due to their huge specific surface area (thousands of m^2/g) [19].

For As(III) in well water, despite the presence of phosphates (9 mg/l), the adsorption capacity was 4 mg-As/g for ZVI and magnetite, a result comparable with that reported by Kanel, et al. (2005) for well water in Bangladesh (maximum capacities of 3 mg/g-5 mg/g of ZVI). The authors used a size between 10 nm-100 nm with 60% smaller than 50 nm and indicated phosphates reduce adsorption capacities by up to 66% [20,21].

Nanofilter performance

Figures 6 and 7 illustrate the good performance of the Nanofilter in treating groundwater. The concentrations of the treated water are presented as a function of the volume treated and the number of bed volumes, achieving reductions to values below the maximum permissible limit of 10 ppb established by Bolivian standard 512.
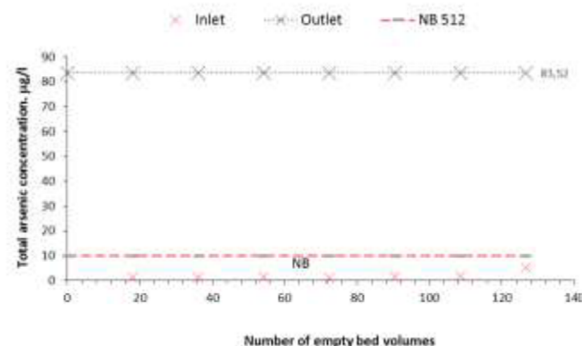
Figure 6: Total arsenic concentration vs number of volumes per bed
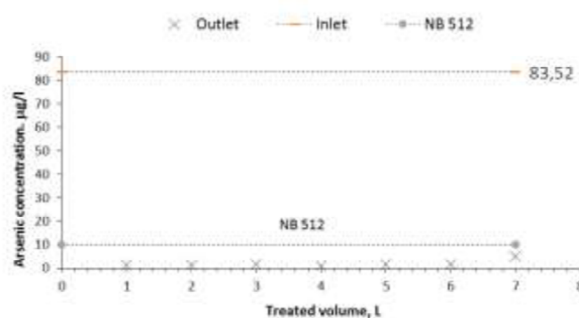
Figure 7: Total arsenic concentration vs volume treated

Discussion

Synthesis of ZVI and magnetite nanoparticles

The original material for obtaining both nanoparticles is iron shavings from machining process. This study takes advantage of this low-cost waste due to its widely known

potential to remediate As(V) and As(III) contaminated waters. Top-down and bottom-up technologies are also simple and widely used for the fabrication of micro and nanoparticles. In contrast, the synthesis of uniform-sized and smaller nanoparticles is more complex and diverse. These aspects make the fabrication cost quite lower in the case of this study.

Nanofilter performance

Although the masses of magnetite and zerovalent iron nanoparticles in the prototype were quite small, the As removal efficiency is over 95%. In treated well water, arsenic is present in the water in the form of arsenites, As(III) which is more difficult to adsorb than arsenate, As(V). However, in the nanofilter it was possible to reduce its content to 50% below that established by Bolivian Standard 512, at a low cost since nanoparticles obtained by the recycling process of iron chips by top-down and bottom-up methods were used, a technology developed and tested to obtain iron and iron oxide nanoparticles with size and chemical composition characteristics suitable for the remediation of water contaminated with arsenic.

The novelty of this work lies in the development of a new low-cost filtration system using known materials that are easily accessible, which allows predicting that this remediation method can be widely applied in all the sites identified with this problem.

In a work similar to this study [19], a gravity filter was developed using polyurethane (PU) foam support loaded with iron oxide nanoparticles. The author comments that it is an innovative filter that is low cost, easy to manufacture, fast, practical, and efficient for arsenic removal, demonstrating between 50% and 70% effectiveness. However, it should be noted that the iron oxide nanoparticles used were purchased at an average cost of hydrated iron oxide is $ 586/lb, and nanoscale zero-valent iron of $ 4,850/lb, respectively is over 50% of the cost that we produced.

From the results obtained in this research, it can be concluded that the filter constructed with iron and iron oxide nanoparticles has a higher efficiency because it was able to reduce the arsenic in contaminated water with 83.52 mg/l to 5 mg/l, representing a 94% reduction. On the other hand, the cost and availability of the materials used make the nanofilter very accessible in technical and economic terms.

Proprietary and innovative technological development

The filter built with iron and iron oxide nanoparticles and other filler elements as easily accessible filtering elements, the object of the present research, was able to reduce the arsenic content in groundwater to 50% below that established by Bolivian Standard NB 512, at a low cost since nanoparticles obtained by the recycling process of iron chips by top-down and bottom-up method were used. Technology was developed and tested to obtain iron and iron oxide nanoparticles with size and chemical composition characteristics suitable for the remediation of water contaminated with arsenic. Its construction at industrial

and commercial scale is feasible because its technology is within reach and available in the environment.

Quality of life and socioeconomic aspects

The potential of the nanofilter as a system applied at the point of use lies in the possibility of directly benefiting people who do not have alternative access to safe water sources, in the 3 ecological floors of Bolivia that have the problem of arsenic-contaminated water especially in rural areas. So it could improve the quality of life with the improvement of water quality.

Alternative to the current and future situation

It is a fact that one of the current and future problems in our country is the scarcity of drinking water sources for human consumption and irrigation. Hence, the optimization of its use is essential. The filter also has the potential to remediate water contaminated with heavy metals in industrial and mining processes for its reuse, once it's scaling up becomes possible.

Conclusion

From the results obtained in the present investigation, it can be concluded that the filter constructed with iron and iron oxide nanoparticles has a higher efficiency than that achieved in other similar studies, managing to reduce the arsenic in well water naturally contaminated with 83.52 mg/l to 5 mg/l, which represents a 94% reduction. On the other hand, the cost and availability of the materials used make this filter very accessible in technical and economic terms.

The technology developed and tested is suitable for obtaining iron and iron oxide nanoparticles with size and chemical composition characteristics suitable for the remediation of arsenic-contaminated water. This is confirmed by similar work using the adsorption remediation process.

The industrial and commercial scale construction of the nanofilter is feasible because its technology is within reach and available in the environment.

One of the crucial problems identified in Bolivia is the presence of arsenic in groundwater in very high concentrations, particularly in rural areas in the 3 ecological levels: Altiplano, valleys, and the Amazon, with greater incidence in rural areas. This fact endangers the health of the inhabitants of these areas and their biodiversity since this liquid element coming from wells is the only source of supply for human use and irrigation. The potential of the nanofilter as a system applied at the point of use lies in the possibility of directly benefiting the population that does not have alternative access to safe water sources. With the application of this filtration system, the quality of life could be improved by improving water quality.

graduate of the Production Engineering Department at Universidad Privada Boliviana, who carried out the experimental adsorption runs.

To Dr. Victor Achá from La Salle University in France for the preliminary results of nanoparticles characterization.

Data Availability Statement

The data used in this research are available in this paper and are included in the author's references so other researchers can use them.

Conflict of Interest

Authors declare no conflict of interest.

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