Removal of Heavy Metals in Drinking Water by Iron-based Sorption Materials

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Abstract. Adsorption on a suitable adsorption material is the most frequently used methods in water treatment. In terms of plant operation, filtration (sorption) process represents a simple, effective and economical friendly method of heavy metals removal namely for the possibility of using a large scale of substances with a sorption ability – sorbents. Oxides, oxyhydroxides and hydroxide containing iron are among the most frequently used sorbents of the heavy metal removal. The quality of the treated water (pH, silica, phosphorus, fluorides, sulphates, total mineralization, iron and manganese, organic matter, etc.), redox conditions, the valency of metal and filtration conditions has the impacts on heavy metal removing efficiency. The objective of this work was to verify the sorption properties of granular iron-based sorption materials (CFH12, CFH18, Bayoxide E33, GEH) in removal of arsenic, antimony and nickel from drinking water. Under the given operational conditions (average concentration of arsenic in raw water 52.96 μg/L, average velocity 5.6 m/h, concentration As 10 μg/L at the outlet of media) adsorption capacity of filtration material CFH12 and Bayoxide E33 for arsenic was 1.203 mg/g and 1.463 mg/g, respectively. The limit of 10 μg/L of As was exceeded after 24048 BV and 28623 BV (Bed volume), respectively. According to the model tests (concentration of nickel in raw water of about 50 µg/L, filtration rate 5.8 m/h, concentration Ni 20 µg/L at the outlet of media, pH 7.0) the adsorption capacity of nickel for Bayoxide E33 was set to 198 μg/g, for CFH18 107.5 μg/g, GEH 97.5 μg/g and CFH12 38.1 μg/g. The limit of 20 μg/L of Ni was exceeded after 4808 BV for Bayoxide E33, 1409 BV for CFH12, 3007 BV for CFH18 and 3218 BV for GEH. For the average antimony concentration of 58.35 μg/L in raw water and filtration rate between 4.27-4.51 m/h, the limit concentration of 5 μg/l at the outlet of the sorption column was reached at the bed volume 3470 for GEH, 2225 for CFH 12 and 1274 for Bayoxide E33. The adsorption capacities were as follows: 167.8 μg/g for GEH, 105.7 μg/g for CFH12 and 90.9 μg/g for Bayoxide E33. The experiments proved that iron-based sorption materials GEH, CFH12, CFH18 and Bayoxide E33 can help reduce the concentration of heavy metals in water below the limit of the Slovak Republic Decree No. 247/2017 on Drinking Water.

1. Introduction

Heavy metals are a naturally occurring compound that is found in the environment. The sources of heavy metals in natural water may originate from geochemical reactions, uncontrolled industrial waste discharges, agricultural use of pesticides containing heavy metals, or chemical warfare. The presence of the increased concentration of heavy metals in water has been reported to be extremely harmful to human health. The risk of the heavy metals presence rests mainly in their tendencies of being accumulated in the tissue of plants and animals, some of them are a carcinogen. The
knowledge about the health aspects of heavy metals presence in drinking water are included in the paper Water Quality and Treatment, A Handbook of Community Water Suppliers [1] and literature [2-8].

For the supply of drinking water in Slovakia are used mainly groundwater resources (86% of inhabitants are supplied with drinking water from underground resources). In terms of quality of ground water the main indicators are the amount of iron, manganese, heavy metals (e.g. arsenic, antimony), etc. 23% of groundwater sources require treatment for drinking purposes.

Since 1998, an intensive attention has been paid to the presence of heavy metals in the water, when technical standard STN 75 7111 Drinking Water was introduced into the Slovak legislation. By transposition of European Direction 98/83/EC and WHO recommendation [9,10] into our legislation, the limit concentrations of some of the heavy metals (e.g. As, Sb) were decreased, resp. determined for the first time which caused that some of the Slovak water sources has become nonconforming and they need to be adjusted properly for their next use.

Now, on Slovakia is an acceptable concentration of heavy metals in drinking water defined under the Decree of the Ministry of Health of the Slovak Republic No. 247/2017 Col. The limit concentration for arsenic is 10 µg/L, for antimony 5 µg/L and nickel 20 µg/L.

There are several technological methods for removing heavy metal in the water treatment process: precipitation, ion exchange, membrane processes, adsorption, electrochemical and biological methods.

**Adsorption** processes are based on the adsorption of contaminants on the surface of an adsorption material. The molecules of the contaminant pass from the water environment to the solid adsorbent. It is possible to use iron oxides, oxyhydroxides or ferric hydroxide (GEH, CFH12, CFH18, Bayoxide E33, Everzit As), activated alumina, iron-activated alumina (Fe-AA), activated carbon (AC), iron-activated carbon (Fe-AC), zirconium-activated carbon (Zr-AC), zero-valent iron (Fe(0)), media containing TiO₂, CeO₂, ZrO₂ or MnO₂ layers on their surface, sand covered by iron hydroxide, low cost materials (zeolites, carbonates, clay, peat, moss, ash, chitosan, sawdust, coconut husk, living or non-living biomass etc.), for removal of heavy metals.

Efficiency of heavy metal removal by adsorption material depends on the pH of water, oxidation-reduction potential of a given metal in water, initial heavy metal ion concentrations in water, the solid/liquid ratio, concentration of substances in water that have a potential to affect (interfere with) adsorption or modify adsorbent surface loading, concentration of substances and colloid particles that can physically block the entry into the particle and the access to grains of adsorption media, respectively (concentration of iron, manganese, phosphorus, silicon, fluorides, sulphates, organic matter, etc.) [11,12], the particle size of a sorption material, specific surface area and distribution of pores of adsorption material, hydraulic properties of filtration media in treatment (filtration rate, the Empty Bed Contact Time - EBCT, the filter medium height).

Adsorption using an appropriate sorption material is preferred for water treatment when considering small water resources. Sorption is a simple (regarding its operation) and effective method of heavy metal removal, mainly because of the wide range of sorption materials that can be used in this process [13,14].

The literature mostly describes the use of iron oxides and oxyhydroxides for removal of arsenic and other heavy metals from water. A number of experiments and model studies on the adsorption of heavy metals are described in various publications [15-22].
2. Material and Methods

Experimental part of this work is divided into three parts while aim of the experiments was to verify the sorption properties of used iron-based sorption materials (CFH12, CFH18, Bayoxide E33, GEH) in removal of the arsenic, antimony and nickel from the drinking water.

2.1. Iron-based sorption materials

Bayoxide E33 is a granular iron oxide-based medium. It was developed by Severn Trent in cooperation with Bayer AG for the removal of arsenic and other contaminants from water. The advantage of this material is its ability to remove both As$^{3+}$ and As$^{5+}$ from drinking water together with iron and manganese. Under high pH conditions, high levels of vanadium, phosphate (>1.0 mg/L) and silica (>40 mg/L) can present interference and reduce the media’s adsorption capacity for arsenic [23,24].

CFH12 and CFH18 are granular sorption materials based on iron oxyhydroxides. It was developed by Kemira Finland as effective product for the removal of arsenic and other contaminants from water by adsorption. The advantage is their high adsorption capacity and higher efficiency at a lower cost, provided that the adsorption capacity is fully used (optimum filtration, backwash and pH). CFH12 and CFH18 differ from each other by their grain size [25-27].

The GEH was obtained from the supplier (GEH Wassermchemie, Germany). GEH is a sorption material developed by the Department of Water Quality Monitoring of the University of Berlin for the purpose of arsenic removal from water [28-30]. GEH is highly selective towards arsenate; therefore, it requires an initial oxidation step in the presence of arsenite [31] (Bissen and Frimmel, 2003). Its adsorption capacity is dependent on the characteristics and composition of the water treated as well as the operating conditions. Increasing phosphate and sulfate concentrations in influent water greatly reduces arsenic removal.

The basic physical and chemical properties and composition of these commercial sorption materials are listed in the Table 1 and Table 2. Chemical composition was determined by using the methods of X-ray microanalysis and SEM.

Table 1. Physical and chemical properties of the selected sorption materials

| Parameter                  | Bayoxide E33 | CFH12, CFH18 | GEH          |
|----------------------------|--------------|--------------|--------------|
| Matrix/ Active agent       | Fe$_2$O$_3$>70% and FeOOH | FeOOH Fe$^{3+}$ >40% | Fe(OH)$_3$ and 52-57% β-FeOOH |
| Bulk density [g/cm$^3$]     | 0.45         | 1.12-1.2     | 1.22-1.28    |
| Specific surface area [m$^2$/g] | 120-200     | 120          | 250-300      |
| Grain size [mm]            | 0.5-2.0      | 0.5-2.0      | 0.32-2.0     |
| Grain porosity [%]         | 85           | 75-80        | 72-80        |

Table 2. Chemical composition of selected sorption materials [14]

| Material | MgO [mass %] | Al$_2$O$_3$ | SiO$_2$ | P$_2$O$_5$ | SO$_2$ | K$_2$O | CaO | TiO$_2$ | Fe$_2$O$_3$ |
|----------|--------------|-------------|---------|------------|--------|--------|-----|---------|-------------|
| E33      | 0.97         | 6.59        | 12.75   | 0.34       | 0.31   | 0.37   | 2.01| 0.91    | 75.28       |
| CFH12    | 3.75         | 0.45        | 1.18    | -          | 8.49   | 0.27   | 2.72| 0.50    | 82.65       |
| CFH18    | 5.19         | 0.48        | 1.47    | 0.28       | 4.58   | -      | 1.41| 0.30    | 86.29       |
| GEH      | -            | 1.74        | 3.05    | 0.21       | 0.54   | 0.08   | 0.18| -       | 91.92       |
2.2. Model filtration system
Filter columns was originally developed for testing the arsenic and nickel removal with granular sorption materials. It was made of glass and consisted of two parts. Internal column (diameter of 2.8 cm) was filled with adsorption material. Adsorption column was cooled by water from the outside in order to ensure stable temperature of the column (external column was used as a cooler). The height of glass column was 76 cm while the height of filling was 58 cm which represented 6.157 cm² of the column surface and 357.1 cm³ of its volume.

Two commercial media (E33 and CFH12 or GEH and CFH18) were tested in parallel on the same influent water source. Sorption materials were added to columns using drinking water to flush the material down into the columns. Columns were backwashed to remove fine particles by operating the column in upflow mode with drinking water until the effluents ran clear. The experiments were conducted under laboratory conditions.

The effectiveness of antimony removal was verified using adsorption columns containing selected sorption material. Adsorption column was made of glass with a diameter of 5.0 cm. A height of media was 58 cm, and the total height of glass column was 80 cm, which represented 19.635 cm² of the column surface and 1138.8 cm³ of its volume. The system of several valves was used for feeding the water for filtration system (from top to bottom) and for filter backwash (from bottom to top) as well as for regulating filtration rates.

2.3. Water source and chemical spiking
Drinking water from water supply system was used for the experiment. Certified reference material containing As and Ni was added to the water in order to obtain the concentration of about 50 µg/L. Water used in the model was prepared in 100 litre storage tank and pumped with a dosing pump.

Groundwater in locality Dúbrava (Brdáre well) passed through the filtration system, and the concentration of antimony ranged from 51.2 to 64.9 µg/L was monitored in raw and treated water at the outlet of filtration columns. Simultaneously, the flow rates were measured at the outlet of each column.

2.4. Analytical methods
Heavy metals (arsenic, antimony, nickel) samples after passing through columns were collected into plastic bottles and immediately acidified with highly pure nitric acid (Merck). All bottles were submerged in 10% nitric acid solution over 3 days and triple rinsed with de-ionized water. Agilent 7500CE ICP-MS (ORS technology) was used to determine heavy metals concentration in solution. The detection limit for these metals by ICP-MS was 2 µg/L.

3. Results and discussions
Treatment technology includes contaminant sorption process using granular iron-based sorption materials placed in the reactor through which treated water flows by scheme:

Raw water → filtration and adsorption (without regeneration and washing the filters)

Raw water without any pre-treatment flowed through the filtration equipment while the heavy metals concentrations were monitored in a raw water and in a treated water outflowing from the individual filtration columns. The flow of the water at the discharge from each column and the amount of filtered water were monitored also.

3.1. Experiment 1 – Arsenic removal
Arsenic concentration in a model water were about 46.5 - 63.1 µg/L (average 52.96 µg/L) during the experiments, filtration velocity had been reaching approx. 5.6 m/h, detention period of the water in column (EBCT) 6.2 min. Filtration conditions (average values) are listed in the Table 3.
Table 3. Filtration conditions

| Parameter                             | Bayoxide E33 | CFH12 |
|---------------------------------------|--------------|-------|
| Grain size [mm]                       | 0.5 – 2.0    | 1 – 2 |
| Medium height [cm]                    | 58           | 58    |
| Average flow through column [ml/min]  | 57.66        | 57.36 |
| Average filtration rate [m/h]        | 5.62         | 5.59  |
| Empty Bed Contact Time (EBCT) [min]  | 6.19         | 6.22  |

Figure 1 shows arsenic concentration in the raw water and values of arsenic determined behind the examined sorption materials depending on the filtration time and the ratio V/V₀ (V - treated water volume, V₀ – filtration filling volume). This ratio is called "Bed volume". In the Figures is also shown the limit value for arsenic in the drinking water (10 μg/L) that is set by the Decree of Ministry of Health of the Slovak Republic No. 247/2017 Col. that documents the specifications for drinking water quality, control of drinking water quality, monitoring program and risk management of drinking water supply.

Table 4. Effectiveness of arsenic removal from water during filtration – adsorption

| Parameter                                      | Bayoxide E33 | CFH12 |
|------------------------------------------------|--------------|-------|
| Total filtration time [hrs]                   | 3578         | 3578  |
| Filtration time [hod] after exceeding the limit 10 μg/L | 2989         | 2505  |
| Total volume of overflowing water [m³]        | 12.213       | 12.201|
| Volume of overflowing water [m³] up to the limit 10 μg/L | 10.222       | 8.588 |
| Bed volume (the ratio V/V₀) up to the limit 10 μg/L | 28623        | 24048 |

Limit value 10 μg/L As was exceeded after 2989 hours of filtration device operation while the Bayoxide E33 was used as the sorption material (Table 4). The amount of water that overflowed through this filtration device for this period of time represents 10.222 m³, i.e. 28623 fold filling, while the capacity of adsorption filling has been not depleted completely.
In case of sorption material CFH12 (Kemira) the limit value 10 μg/L for As was exceeded after 2505 hours of operation of the filtration device (Table 4). Water volume that overflown this filtration device during this period of time was 8.588 m$^3$, i.e. 24 048 fold of column volume while the adsorption capacity of filling has not been completely depleted.

On the basis obtained the results and using the material balance we were calculated the amount of arsenic on the inflow to the columns, on the outflow from the filters, the amount of arsenic absorbed by the filter filling for the particular time intervals of sampling and the amount of arsenic adsorbed after exceeding the limit 10 μg/L (Decree No. 247/2017 Col.).

Under the given operational conditions (average concentration of arsenic in raw water 52.96 μg/L, average velocity 5.59 m/h, total filtered volume 8.588 m$^3$) the arsenic at the amount 454.9 mg was adsorbed by the filling CFH12 with the weight 378 g. It accrues from these results that the adsorption capacity of filtration material CFH12 is 1.203 mg/g.

Under the given operational conditions (average concentration of arsenic in raw water 52.96 μg/L, average velocity 5.62 m/h, total filtered volume 10.222 m$^3$) the arsenic at the amount 541.4 mg was adsorbed by the filling Bayoxide E33 with the weight 370 g. It accrues from these results that the adsorption capacity of filtration material Bayoxide E33 is 1.463 mg/g (Figure 2).

![Figure 2](image)

**Figure 2.** The amount of adsorbed arsenic in relation to the filtration time (left) and value of adsorption capacity for used sorption materials up to the limit 10 μg/L of As (right)

### 3.2. Experiment 2 – Nickel removal

In the following stage of experiments, the efficiency of nickel removal from water using the sorption materials GFH, CFH12, CFH18 and Bayoxide E33 was monitored for filtration rates in the range 5.84-5.89 m/hour and the height of filter media in the column of 58 cm. The concentration of nickel in raw water was in the range from 48.5 to 51.1 μg/L, the temperature of raw water was in the interval from 9 to 14 °C and the water pH was adjusted to 7.0. The filtration conditions are shown in Table 5.

The Figure 3 shows the nickel concentrations at the outlets from the adsorption column in dependence on the operational time and ratio V/V$_0$ (Bed volume). The Figure 4 shows the values of the adsorption capacity and the V/V$_0$ ratio for used adsorption materials when the limit concentration of nickel (20 μg/L) was achieved at the outlets of media.
Table 5. Filtration conditions

| Parameter                                           | E33   | CFH12  | CFH18  | GEH     |
|-----------------------------------------------------|-------|--------|--------|---------|
| Grain size [mm]                                     | 0.5 – 2.0 | 0.5 – 2.0 | 0.8 – 1.8 | 0.32 – 2.0 |
| Medium height [cm]                                  | 58    | 58     | 58     | 58      |
| Average flow through column [ml/min]                | 60.39 | 60.25  | 60.12  | 60.08   |
| Average filtration rate [m/hour]                    | 5.885 | 5.871  | 5.858  | 5.854   |
| Empty Bed Contact Time (EBCT) [min]                 | 5.914 | 5.928  | 5.940  | 5.944   |
| Total filtration time [hour]                        | 497   | 480    | 480    | 497     |
| Total amount of water passed through filtration column [m³] | 1.798 | 1.715  | 1.724  | 1.789   |

Figure 3. Progress of nickel concentration at the outlets of the adsorption media CFH12 and CFH18 (A) or GFH and Bayoxide E33 (B) depending on Bed volume (V/V₀)

Figure 4. Adsorption capacity of nickel and the values of V/V₀ ratios for limit concentration (20 µg/L of Ni) at the outlet of adsorption media
According to the obtained results presented in the Fig. 3 it can be stated that Bayoxide E33 is the most suitable for nickel removal as compared to other tested sorption materials. For Bayoxide E33 the nickel concentration 20 μg/L at the outlet of the media with the height of 58 cm was reached for the ratio V/V₀ = 4808. The value for CFH12 was V/V₀ = 1409, for CFH18 V/V₀ = 3007 and for GFH V/V₀ = 3218. In these conditions the adsorption capacity for Bayoxide E33 was 198.4 μg/g, CFH18 107.5 μg/g, GEH 97.5 μg/g and CFH12 38.1 μg/g.

The Figure 5 shows the amount of adsorbed nickel depending on the V/V₀ ratio, as well as the amount of adsorbed nickel in adsorption media when the limit concentration Ni (20 μg/L) was achieved at the outlets from the adsorption column.

Figure 5. Amount of adsorbed Ni in adsorption media in dependence on the filtration time (left) and amount of adsorbed nickel in sorption materials when was reached the limit concentration 20 μg/L of Ni at the outlet from the columns (right)

3.3. Experiment 3 – Antimony removal

The third part of this work was aimed at monitoring of the efficiency of sorption materials GEH, CFH12 and Bayoxide E33 for the removal of antimony from water. The concentrations of antimony in raw water ranged from 51.2 to 64.9 μg/L (average value 55.64 μg/L) and the filtration rates were 4.27-4.51 m/h. The filtration conditions are shown in Table 6.

Table 6. Filtration conditions

| Parameter                           | E33   | CFH12 | GEH   |
|-------------------------------------|-------|-------|-------|
| Grain size [mm]                     | 0.5−2.0 | 0.5−2.0 | 0.32−2.0 |
| Medium height [cm]                  | 58    | 58    | 58    |
| Volume of adsorption column [cm³]   | 1138.8 | 1138.8 | 1138.8 |
| Average flow through column [ml/min]| 140.0 | 147.8 | 147.3 |
| Average filtration rate [m/hour]    | 4.27  | 4.51  | 4.50  |
| Empty Bed Contact Time (EBCT) [min] | 8.41  | 7.98  | 8.0   |
| Total filtration time [hour]        | 1174.5| 1174.5| 1174.5|
| Total amount of water passed through filtration column [m³] | 9.74 | 10.08 | 10.11 |
The Figure 6 shows the concentration curve of antimony at the outlets of adsorption media in relation with operational time and the concentration curve of antimony in relation with \( V/V_0 \) ratio (where \( V \) is a volume of treated water that flowed through the column in a given time and \( V_0 \) is a volume of adsorbent media), i.e. bed volume. In the Figures 3 is also shown the concentration of antimony in raw water on the inflow to the filtration columns and the limit value for antimony in the drinking water (5 µg/L) that is set by the Decree of Ministry of Health of the Slovak Republic No. 247/2017 Coll.

![Figure 6](image1)

**Figure 6.** The concentration of antimony (µg/L) at the outlets of adsorption media (h=58 cm) in relation with operational time (left) and \( V/V_0 \) ratio (right) at average concentration of Sb in raw water 55.64 µg/l of Sb, filtration rate 4.27-4.51 m/h.

![Figure 7](image2)

**Figure 7.** The values of \( V/V_0 \) ratio and adsorption capacity (µg/g) for each sorption material (h=58 cm) when reaching the limit concentration of antimony (5 µg/L)

Considering the minimum differences in filtration rates and based on the results presented in the Figure 7, it can be concluded that GEH is the most suitable material for antimony removal compared to other sorbents used in the test. The following \( V/V_0 \) ratios were measured for the antimony concentration (5 µg/L) at the outlet of 58 cm high adsorbent media: \( V/V_0 = 3470 \) for GEH, \( V/V_0 = 2225 \) for CFH 12.
and \(V/V_0 = 1274\) for Bayoxide E33. The adsorption capacities were as follows: \(GEH = 167.8\ \mu g/g\), \(CFH12 = 105.7\ \mu g/g\) and \(Bayoxide\ E33 = 90.9\ \mu g/g\).

According to the material balance of antimony in these experiments, the Figure 8 shows the amount of adsorbed antimony depending on the \(V/V_0\) ratio, as well as the amount of adsorbed antimony in adsorption media when the limit concentration \(Sb\) (5 \(\mu g/L\)) was achieved at the outlets from the adsorption column.

![Figure 8](image)

**Figure 8.** The amount of adsorbed \(Sb\) in adsorption media in dependence on the filtration time (left) and amount of adsorbed nickel in sorption materials when was reached the limit concentration 5 \(\mu g/L\) of \(Sb\) at the outlet from the columns (right)

### 4. Conclusions

Performed technological experiments with the model water with arsenic or nickel in laboratory and groundwater of springs in locality Dúbrava shown that it is possible to decrease the arsenic, nickel and antimony content in the water down to the values which are limited by the Decree of the Ministry of Health of the Slovak Republic No. 247/2017 on requirements for drinking water and monitoring of drinking water quality with the tested selected sorption materials.

The results obtained support the published findings of foreign writers and our so-far research, according to which these sorption materials are 10x more effective for the removal of arsenic compared to antimony and nickel removal. The results at this point have proved that filtration (sorption) with iron-based materials can also be used for antimony and nickel removal too.

Based on the results obtained under these test conditions and for the quality of the treated water, it can be stated that Bayoxide E33 has a higher efficiency for removal of arsenic from water than CFH12.

Performed technological tests of ground water from the spring in the locality of Dúbrava showed that the investigated sorption materials can decrease the content of antimony in water to the limited value for drinking water. Based on the pilot operation tests, the most suitable sorption material for antimony removal from water is GEH. The results proved that the materials CFH12 and Bayoxide E33 can be also used to decrease the concentration of \(Sb\) in drinking water below the limit value of 5 \(\mu g/L\). The adsorption capacities and \(V/V_0\) ratio are lower for these sorption materials.
Based on the laboratory tests, the most suitable adsorbent for nickel removal is Bayoxide E33. However, its effectiveness increases with decreasing water pH. For this material it is better to treat the water at lower pH, i.e. 6.5 to 7.5.

The results proved that the materials CFH12, CFH18 and GFH can be also used to decrease the concentration of Ni in drinking water below the limit value of 20 µg/L (Decree No. 247/2017). The adsorption capacities and V/V₀ ratio are lower for these sorption materials.

It is necessary to optimize filtration conditions (e.g. filter media height, filtration rate, contact time of raw water with filter material in the column, etc.) and to monitor the effect of pH, water quality etc. to the effect of removing heavy metals from water.

Filtration rate affects the achieved efficiency, when the rate is lower, the period of the contact with water is longer and hence the efficiency of heavy metals removal from water is increases.

The composition of the water had no adverse effects on the adsorption materials, and no release of iron into the treated water was observed. Moreover, no effect on the pH value of the treated water was observed when the water passed through the adsorption media.

This method of water treatment is especially suitable in localities where it is not possible to use coagulation, sedimentation and filtration for treatment processes. Filtration using an appropriate sorption material is preferred for small water resources or in emergency situations. Reliability, efficiency, the filtration rate and the simplicity of the operation are among the major advantages of this technology, mainly because of the wide range of sorption materials that can be used in this process.

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