

# Laboratory-scale study of the advanced Fenton process for silica removal from brackish underground water in arid areas in Saudi Arabia

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#### ABSTRACT

Silica scaling of reverse osmosis (RO) membranes in water desalination poses a worldwide problematic issue in the cost-effective operations in seawater desalination plants. Underground water is one of the main water resources in the Kingdom of Saudi Arabia. Most of the inland cities and remote villages rely entirely on this supply mode for their domestic, industrial and agricultural purposes. However, this water resource is characterized by its relatively high silica contents. Therefore, pretreatment for silica removal from the RO feed is crucial in a system running on high silica waters. In this preliminary work, we investigated the effectiveness of advanced Fenton process (using hydrogen peroxide and zero valent metal iron Fe<sup>0</sup> as source of Fe<sup>3+</sup>) for the silica pretreatment of water from the Buwaib deep aquifer by its co-precipitation with ferric hydroxide. The influences of several reaction parameters, i.e., iron powder dosage, hydrogen peroxide concentration, pH effect, were investigated. In the optimum conditions, up to 70% of silica was removed. This preliminary study showed that this environmentally friendly process is an effective silica mitigation strategy prior to the RO desalination.

Keywords: Silica removal; Advanced Fenton process; Hydrogen peroxide effect; Iron dosage effect; pH effect

# 1. Introduction

## 1.1. Major water resources in Saudi Arabia

Saudi Arabia, with an area of around 2.1 million km<sup>2</sup> and an estimated population of 26 million in 2011, is an extremely arid country. The annual average precipitation during the period 1979–2009 was 93.5 mm [1]. There are no permanent watercourses or lakes. For its water consumption, the country relies mainly on seawater desalination and deep/shallow underground brackish aquifers [2], which are invaluable resources of drinking water in remote arid areas (Fig. 1). It is usually organic compounds and biological species-free.

#### 1.2. Groundwater from deep and shallow aquifers

Fig. 2 shows the estimated site of these huge and very deep sedimentary underground aquifers, formed from

the Paleozoic era (600 million years ago) to the Cenozoic era (present age) [3]. With a reserve estimated between 260 and 760 billion m<sup>3</sup> and with an average renewal rate of 900 million m<sup>3</sup>, they are rapidly depleting [4]. The surface water comes from the limited seasonal rainfall. Part of the runoffs recharges the shallow aquifers (Tuwail, Skaka, Khuff, Jauf, Jileh and Jubayla) while the remaining evaporates due to the extreme hot weather. Most of these deep and shallow aquifers are categorized as brackish, where they contain over 1,000 ppm of total dissolved solids, mainly silica [5].

#### 1.3. Seawater desalination

The state-owned Saline Water Conversion Corporation (SWCC) runs 28 desalination plants located on the western and eastern coasts of the country, with an estimated production of more than 4.6 million m<sup>3</sup>/d of potable water [6]. These plants use the reverse osmosis (RO) and the multistage flash distillation methods.

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Fig. 1. Water consumption by source in Saudi Arabia. The surface water sources indicated here are captured from infrequent extreme rainfall events through a vast network of dams.



Fig. 2. Major sedimentary deep underground aquifers in Saudi Arabia.

#### 1.4. Silica scaling

Seawater and brackish groundwater must be desalinated prior to their use. RO is the main adopted process in treating these water resources. However, this method is suffering a technical setback from scaling and fouling problems due to the presence of high concentrations of silica in the water resources. This will reduce the lifetime of RO membrane and cause a decline in recovery rate. In order to avert silica scaling, a minimum silica concentration of 10 mg/L in RO feed water is necessary for 95% water recovery rate [7].

Removing silica is very costly, up to 80% of the operating cost in a water treatment plant [8]. Various treatment processes for silica removal exist: coagulation with metal hydroxides [9,10], flocculation [11], nanofiltration [12] and using antiscaling chemicals [13]. In the past few decades, advanced oxidation process has proven its high efficiency in the water treatment processes [14–18]. Table 1 gives the performance in silica removal for various procedures.

Table 1	
Silica removal performances for various procedures	

Method	Percentage	Ref.
	silica removal	
Lime softening	47%	[19]
Electrocoagulation	60%-80%	[20]
Coagulation/ultrafiltration	65%	[21]
Magnesium compounds at high pH	Up to 85%	[22]
Coagulation with Al salts	76%	[23]

To the best of our knowledge, the advanced Fenton process (AFP), based on the formation of the powerful hydroxyl radicals when  $H_2O_2$  reacts with Fe<sup>0</sup>, for the removal of silica from water was rarely investigated. This environmentally friendly wastewater treatment method is mostly attractive due to the fact that the hydrogen peroxide and the non-toxic iron are low cost and largely available.

The aim of this preliminary study is to assess the efficiency of this alternative technology for the silica removal from the water feed RO system at Buwaib water treatment plant near the capital city Riyadh.

#### 1.5. Chemistry of silica: silica scaling

Although the kinetics of silica scaling is very slow, it is a major limiting factor in high-recovery RO technology for the desalination of brackish and seawater. Chemistry of silica is quiet complex. Silica found in surface water (1–20 ppm), well water (10–50 ppm) and brackish well water (120–150 ppm) can be present in amorphous form of solubility 6 ppm or crystalline form of solubility 100–150 ppm at 25°C [24]. It results from the chemical weathering of silica containing rocks such as albite [25].

$$2NaAlSi_{3}O_{8(sol albite)} + 2H^{+} + 9H_{2}O \rightleftharpoons Al_{2}Si_{2}O_{5}(OH)_{4(sol kaolinite)} + 4Si(OH)_{4(aq silica)} + 2Na^{+}$$
(1)

It is present as monosilicic, polysilicic or colloidal silica particles. In aqueous solutions, the monosilicic acid exists as orthosilicic acid Si(OH)<sub>4</sub>. It is the soluble form found in low concentration in all natural waters. It is a weak acid ( $pK_a \sim 9.8$ ) where the four hydroxyl groups are bonded tetrahedrally to the silicon atom [26]. Silica solubility increases with an increase in temperature (up to 300 ppm at 70°C) and an increase in the pH (from 150 ppm at pH 4 to 380 ppm at pH 10) [27]. Successive hydrolysis of the orthosilicic acid produces different anionic species:

$$Si(OH)_4 + H_2O \rightleftharpoons SiO(OH)_3^- + H_3O^+$$
(2)

 $SiO(OH)_{3}^{-} + H_{2}O \rightleftharpoons SiO_{2}(OH)_{2}^{2-} + H_{3}O^{+}$  (3)

$$\operatorname{SiO}(\operatorname{OH})_{2}^{2-} + \operatorname{H}_{2}\operatorname{O} \rightleftharpoons \operatorname{SiO}_{3}(\operatorname{OH})^{3-} + \operatorname{H}_{3}\operatorname{O}^{+}$$

$$\tag{4}$$

$$\operatorname{SiO}_{3}(\operatorname{OH})^{3-} + \operatorname{H}_{2}\operatorname{O} \rightleftharpoons \operatorname{SiO}_{4}^{4-} + \operatorname{H}_{3}\operatorname{O}^{+}$$
(5)

with a net dissociation reaction:

$$H_4 SiO_4 + 4H_2 O \rightleftharpoons SiO_4^{4-} + 4H_3 O^+$$
(6)

Silica is known to condense with a variety of metal ions possessing OH groups [28]. In particular, it aggregates on the ferric hydroxide to form a hydrocomplex, with Fe–O–Si bridge formation [29], which is then removed from the bulk by settling or filtration:

$$Si(OH)_4 + Fe(OH)_3 \rightarrow [SiO_2.Fe(OH)_3] + 2H_2O$$
(7)

#### 1.6. Advanced Fenton process

Under acidic conditions, the AFP uses zero-valent metallic iron with hydrogen peroxide,  $H_2O_2$  (Fe<sup>0</sup>/ $H_2O_2$ ) [30], to generate ferrous ion. It is considered as a very attractive eco-friendly redox agent for sustainable water treatment [31].

The following reactions sequence occurs:

Oxidation via two electron oxidation of  $Fe^0$  with generation of ferrous ion:

$$Fe^0 + 2H^+ \rightarrow Fe^{2+} + H_2 \tag{8}$$

Catalytic decomposition of ferrous ion by hydrogen peroxide, via Fenton reaction, which generates the hydroxyl free radical OH•:

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^{\bullet} + OH^{-}$$

$$\tag{9}$$

This radical is one of the most reactive and non-selective oxidizing species when present in aqueous solution [32]. Its standard oxidation potential is 2.7 V in acidic solution and 1.8 V in neutral and alkaline solution [33]. This species can oxidize numerous organic and inorganic contaminants at rates close to diffusion controlled [34,35].

Oxidation by hydroxyl radical, via two electron transfer, of the metal:

$$Fe^0 + 2OH^{\bullet} \rightarrow Fe^{2+} + 2OH^{-}$$
(10)

$$Fe^{2+} + OH^{\bullet} \rightarrow Fe^{3+} + OH^{-}$$
(11)

Ferric ion hydrolysis as ferric hydroxide:

$$Fe^{3+} + 3H_2O \rightarrow 3H^+ + Fe(OH)_{2 \text{ col}}$$

$$(12)$$

Catalytic regeneration of ferrous ion:

$$Fe^{3+} + H_2O_2 \rightarrow Fe^{2+} + HO_2^{\bullet} + H^+$$
 (13)

#### 2. Materials and methods

All chemicals used in this work were Analar grade from BDH and were used without any further treatment. Iron metal powder (<10  $\mu$ m, 99.99% pure) was from Sigma-Aldrich. Standard solutions of silica were prepared by dissolving sodium metasilicate nonahydrate Na<sub>2</sub>SiO<sub>3</sub>·9H<sub>2</sub>O (99% purity) in purified and deionized water obtained from Milli-Q water purification system. The pH solution was measured using the Jenway 3510 glass electrode pH-meter and was adjusted when necessary by adding sodium hydroxide or perchloric acid (99% purity).

In this laboratory-scale test, underground raw water samples from the Buwaib water desalination plant fed by the Minjur and Dhruma deep aquifer near the capital city Riyadh (Fig. 2) were selected to investigate the AFP for the silica removal. The conventional jar-test method using a Phipps & Bird Six-Paddle Stirrer (Fig. 3) with six 1-L beakers was performed at room temperature to simulate the mixing and settling conditions found in water treatment plants [36]. The coagulation testing cycle (Fig. 4) was as follows: 5 min continuous rapid stirring at 100 rpm of the water sample mixed with Fe<sup>0</sup> and H<sub>2</sub>O<sub>2</sub>, at initial pH, to enhance the homogeneity throughout the 1-L beaker, followed by a slow stirring for 15 min at 20 rpm at equilibrium pH, to allow an efficient coagulation and up to 3-h settling time. For the sake of reproducibility, all the jar tests were performed six times.

Åfter the settling, 5 ml aliquots were withdrawn from the reactor and analyzed for silica content by the silicomolybdate analytical standard protocol [37]. This analytical method is based on the reaction in acidic medium of soluble silica with ammonium heptamolybdate to form a yellow silicomolybdate complex according to the following reaction:

$$7[Si(OH)_{4}.2H_{2}O] + 12(NH_{4})_{6}Mo_{7}O_{24}.4H_{2}O + 36H_{2}SO_{4} \rightarrow 7H_{4}SiMo_{12}O_{40}.14H_{2}O + 36(NH_{4})_{2}SO_{4}$$
(14)



Fig. 3. Diagram of the jar test apparatus with the six 1-L beakers.



Fig. 4. Block diagram of the jar test method.

which is then reduced to the so-called silicomolybdenum complex blue by ascorbic acid. This complex exhibits a spectrophotometric absorption at 810 nm with a molar extinction coefficient of 44,700 L mol<sup>-1</sup> cm<sup>-1</sup>, and its concentration was measured using its optical absorption at by the UV/visible double beam spectrophotometer Biochrom Libra S32 PC with 1.8 nm bandwidth. It should also be mentioned that this method is only valid to measure soluble silica [37].

# 3. Results and discussion

The silica concentration in the Buwaib water samples was found to be 54.2  $\pm$  2.1 mg/L. At this concentration, the polymerization of silica does not occur, and the dominant species is Si(OH)<sub>4</sub> [26]. The percentage removal of silica was computed using the following equation:

% 
$$R(t) = \frac{C_0 - C(t)}{C_0} \times 100\%$$

where  $C_0$  is the silica concentration before its removal, and C(t) is its concentration at time *t* after its removal.

# 3.1. Effect of initial pH on silica removal

The initial pH is one of the key factors in the AFP reaction sequence due to its role in governing the stability of  $H_2O_{22}$ , the



Fig. 5. Effect of  $pH_{initial}$  on the percentage removal of silica: (•)  $pH_{initial} 2$ , (•)  $pH_{initial} 3$ , (•)  $pH_{initial} 4$ , (•)  $pH_{initial} 5$ .  $[H_2O_2] = 15 \text{ mM}$ ,  $[Fe^0] = 8 \text{ g/L}$ , equilibrium pH 8.

catalytic action of Fe<sup>2+</sup> and the iron speciation [38]. Removal of silica was determined under initial pH values of 2, 3, 4 and 5, while hydrogen peroxide and iron metal concentrations were fixed to 15 mM and 8 g/L, respectively. Fig. 5 illustrates the % R(t) of silica dependence on the initial pH of the water sample. The removal reached a maximum value of ~70% after a contact time of 3 h for a pH in the range 2. The decreased removal efficiency at higher pH can be attributed mainly to the deficiency of the Fe<sup>2+</sup> catalyst (reaction (10)) and to a lesser extent to the disproportionation of hydrogen peroxide into oxygen and water [39]. To initiate the AFP, a low pH is crucial for the formation of ferrous ions (reaction (1)). During the reactions sequence of AFP, a slight increase in the pH is observed and is attributed to reactions (2)–(4) upon release of hydroxide ions.

#### 3.2. Effect of equilibrium pH on silica removal

Removal of silica was determined under various equilibrium pH in the range 3–11 with an initial pH of 2,  $[H_2O_2] = 15 \text{ mM}$  and  $[Fe^0] = 8 \text{ g/L}$  (Fig. 6). At low equilibrium pH, the adsorption by weak van der Waals attraction of silicic acid on ferric hydroxide results in ligand exchange in which the hydrogen atom the hydroxyl group of the ferric ion is replaced of the silanol group (=Si–OH) of dissolved silicic acid through Fe–O–Si bonds [29]. This leads to the formation of the silica–iron complex according to the stoichiometric reaction:

$$Si(OH)_4 + Fe(OH)_3 \rightarrow [(OH)_3Si - O - Fe(OH)_2] + H_2O$$
(15)

As the equilibrium pH is increased toward the  $pk_a$  of the silicic acid, this latter dissociates (reaction (6)) into negative silicate anions that adsorb on the ferric cations complex by electrostatic attraction leading to a removal increase reaching a maximum at a pH about 8, which is close to the  $pk_a$  of the silicic acid. However this removal decreases for pH > 8 due to increasing competition of hydroxyl anions with silicate anions for positive surface sites of the ferric complex. This is in agreement with the works of Sanciolo et al. [40] and Salvador Cob et al. [9].



Fig. 6. Equilibrium pH effect on silicate removal by adsorption on ferric hydroxide: initial pH: 2,  $[H_2O_3] = 15 \text{ mM}$ ,  $[Fe^0] = 8 \text{ g/L}$ .



Fig. 7. Effect of  $[H_2O_2]$  on the silica removal:  $[Fe^0] = 8 \text{ g/L}$ , initial pH = 2, equilibrium pH = 8.

#### 3.3. Effect of H<sub>2</sub>O<sub>2</sub> dosage

The effect of hydrogen peroxide concentration on the removal of silica was investigated for different amounts of  $H_2O_2$  ranging from 0 to 25 mM, while keeping the iron dosage at 8 g/L, initial pH at 2 and equilibrium pH at 8. As it can be seen in Fig. 7, hydrogen peroxide affects greatly the percentage removal of silica. At lower concentrations, the removal increases with increasing  $H_2O_2$  concentration. However, at higher concentration, the hydrogen peroxide self-decays (into water and oxygen) and competes with Fe<sup>2+</sup> for OH• radicals as reported by other workers [41–43]:

$$OH^{\bullet} + H_2O_2 \rightarrow H_2O + HO_2^{\bullet}$$
(16)

OH• is then consumed, leading to a decrease in the ferric ion concentration and consequently to a reduction in the percentage removal of the silica. It should be noted that  $HO_2^{\bullet}$  peryhydroxyl radical has a less oxidizing effect on Fe<sup>0</sup> and Fe<sup>2+</sup> than OH• [32].

# 3.4. Effect of iron metal dosage

Removal of silica at different iron metal dosage was investigated in the range 0-10 g/L by keeping the concentration of hydrogen peroxide to 15 mM with an initial pH = 2 and an



Fig. 8. Effect of metallic iron on the silica removal:  $[H_2O_2] = 15$  mM, initial pH = 2, equilibrium pH = 8.

equilibrium pH = 8. As shown in Fig. 8, higher amount of Fe<sup>0</sup> increases the concentration of Fe<sup>2+</sup> ions and consequently the concentration of Fe<sup>3+</sup> (following reactions (10) and (11)), which provides a more surface area for silicate co-precipitation for its removal. However, as shown in Fig. 6, the increase in percentage removal is less pronounced at concentration of iron above 2 g/L. A plateau value of around 70% is reached at iron dosage ~6 g/L suggesting a saturation in the silica coagulation taking place in the bulk solution as it was found by other workers [44–46]. This saturation in the removal may be attributed to the catalytic regeneration of ferrous ion as in reaction (13).

For a percentage removal as high as 70% obtained in this work, the optimum conditions of the main reaction parameters studied here were the following: Fe<sup>0</sup> dosage: 8 g/L,  $[H_2O_2]$ :  $1.5 \times 10^{-2}$  mol/L, initial pH: 3 and equilibrium pH: 8. It should be noted that Al-Rehaili [47] reached 56% silica removal when using at neutral pH a dose combination of lime, soda ash and sodium aluminate as coagulant aid.

# 4. Conclusion

This laboratory-scale preliminary study investigated the viability of the AFP for the silica removal from the Buwaib water desalination plant using hydrogen peroxide and zerovalent iron as the source of catalytic ferric iron.

Under the experimental conditions used in this study, the effect of four reaction parameters considered showed that the percentage removal:

- decreased with increasing initial pH the optimum initial pH is 2;
- increased with increasing equilibrium pH up to the *pk<sub>a</sub>* = 8 of the silicic acid, then decreased at higher pH;
- increased with hydrogen peroxide dosage; however, an excess in hydrogen peroxide lowers the percentage removal; and
- reached a plateau at a concentration around 8 g/L of iron dosage.

This method of water treatment would be viable in arid regions of Saudi Arabia where there is a limited access to freshwater. However, implementation of this promising method on a larger scale will depend on many factors especially operational and economical.

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