

Removal of selenium by reduction to selenite and surface complexation

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Abstract

Selenium is by now a well-known pollutant, present in various coal and hard-rock mines and some thermal power plant effluents through the world. Veolia has developed the Tracer™ Se process (patent pending), which is a new method for selenium removal based on biological reduction of selenium to selenite, its subsequent removal from water using surface complexation on ferric oxy hydroxide, and further biological oxidation of the treated water. The mechanisms by which selenium is understood to create toxicity in the environment will be reviewed, as well as some of the leading existing technologies for removing selenium. The Tracer Se process will then be described, and test results presented.

Brief Biography of the Author

Marc Laliberte is a water specialist with a background in process engineering and chemistry. He has 40 years of experience in mining, metallurgical and general industrial projects. He is a specialist in the treatment of industrial waste water, treatment of feed water, water management including using reuse, recycling, storage and water quality. His work in the last decade has focused on integrating biological and chemical treatment in treatment chains.

Introduction

Selenium, like sulfur, is widely present in the environment, albeit at usually low concentration. Like sulfur it is mostly present in the environment at higher oxidation states, i.e. as selenites and selenates (+4 and +6 oxidation states respectively), rarely as selenides (-2 oxidation state). It is almost never found as the element (0 oxidation state), but it is often produced in that state in biological treatment processes.

Selenites and selenates are generally soluble in water, more so than sulfites and sulfates. As such they cannot easily be removed from water by precipitation. Selenites can form insoluble surface complexes with

metal oxides such as ferric hydrous oxide and manganese hydroxide, and can therefore be removed that way. Elemental selenium can also be produced from selenites or selenates by using a reduction process, biological or otherwise. It is a solid and it is possible to remove it from water in that form. Because of their acute toxicity, selenides need to be handled with care, and if present can also form insoluble metal salts or, at low pH, be emitted as H₂Se, a noxious and highly toxic gas.

Organo-selenium species are important in understanding selenium toxicity. There are a great number of these species. Basically, any organic species that contains sulfur has an equivalent that contains selenium. As an example, the substitution of oxygen by sulfur in the cyanate ion gives thiocyanate, and the substitution of sulfur by selenium gives selenocyanate (OCN⁻, SCN⁻ and SeCN⁻ respectively). Of particular interest is the substitution of sulfur by selenium in the two sulfur-bearing amino acids, cysteine and methionine, giving selenocysteine and selenomethionine. Methylseleninic acid (CH₃SeO₂H) is another organo-selenium species which has been detected in water treated by biological processes and which is biologically active.

Selenium Toxicity - An Overview

The issue of selenium toxicity is complex but reducing toxicity is the main objective behind the development and commercialization of the Tracer Se process.

In recent years a number of large-scale bird mortality incidents have been associated with selenium. Examples include birds feeding and nesting at the Kesterson Reservoir in the San Joaquin Valley, California, between 1983 and 1985, eared grebes at the Salton Sea, California, in 1992 and, again, eared grebes at the Great Salt Lake, Utah in 2011. While selenium has been associated with the die-offs, the mechanism involved so is not well understood.

Acute Toxicity

To help understand toxicity data such as LC₅₀ (the concentration at which 50% of the test organism will die after a stated time), the US EPA (2021) uses five toxicity categories. We can use this system to characterize the acute toxicity of selenium for a few aquatic organisms, as shown in Table 1.

Table 1 - Acute toxicity of some selenium species

Species	Toxicological Endpoint	Selenium Form	Result in mg/L	Toxicity Category	Reference
<i>Cerodaphnia dubia</i>	LC ₅₀ 24 hr	Selenite	0.44	Highly toxic	Beatty 2014
<i>Cerodaphnia dubia</i>	LC ₅₀ 48 hr	Selenate	1 to 9.3	Moderately toxic	Brix 2001
<i>Daphnia magna</i>	LC ₅₀ 48 hr	Selenious acid (selenite)	0.43	Highly toxic	Beatty 2014

Species	Toxicological Endpoint	Selenium Form	Result in mg/L	Toxicity Category	Reference
<i>Daphnia magna</i>	LC ₅₀ 48 hr	Selenite	0.55	Highly toxic	Maier 1993
<i>Daphnia magna</i>	LC ₅₀ 48 hr	Selenate	2.84	Moderately toxic	Maier 1993
<i>Hyallela azteca</i>	LC ₅₀ 96 hr	Selenite	0.34	Highly toxic	Beatty 2014
<i>Hyallela azteca</i>	LC ₅₀ 96 hr	Selenate	6 to 32	Slightly to moderately toxic	Brix 2001
<i>Oncorhynchus mykiss</i>	LC ₅₀ 96 hr	Selenate	4.5 to 9	Moderately toxic	Beatty 2014
<i>Oncorhynchus tshawytscha</i>	LC ₅₀ 96 hr	Selenite	3.5 to 13.6	Slightly to moderately toxic	Beatty 2014
<i>Oncorhynchus kisutch</i>	LC ₅₀ 96 hr	Selenite	8.1 to 23.4	Slightly to moderately toxic	Beatty 2014

As can be seen in Table 1, selenium is more acutely toxic for species lower in the food chain such as *C. dubia*, *D. magna* and *H. azteca* than for salmonids such as *O. mykiss*, *O. tshawytscha* and *O. kisutch*. In cases where acute toxicity data is available for both selenites and selenates, selenites are generally more toxic than selenates. Given that selenates are the dominant form of selenium in the aerobic aquatic environment, one could conclude that selenium is only slightly to moderately acutely toxic for most fish species, but can be highly toxic for small crustaceans such as *D. magna* and *C. dubia*.

Chronic Toxicity

Chronic toxicity is a different issue. Quoting Beatty (2014), “since ambient Se concentrations rarely reach levels that result in acute effects, the more common situation resulting in Se toxicity occurs at much lower chronic exposures.” The literature on selenium chronic toxicity is vast and confusing. Here are some key points from the Beatty paper:

“Bacteria, fungi, algae, and invertebrates are fairly tolerant to elevated Se concentrations, and the more important role these organisms play is in the rapid transformation and transfer of Se into the aquatic food web. However ... there is a high degree of variability in the toxic effects on algae and invertebrate taxa based on water Se concentrations, suggesting that Se uptake is very different among species at a given water concentration. ...

“Fish and bird species have the highest sensitivities to both Se-related embryo mortality and developmental deformity, although amphibians and reptiles may also be sensitive to Se. ... Reproductive and non-reproductive toxic effects may be seen in fish from chronic Se exposure.

Reproductive effects are those originating from the maternal transfer of Se, while non-reproductive effects refer to the direct toxic impacts Se may have on juveniles and adults. Both reproductive and non-reproductive effects result primarily from the dietary intake of Se. There is also evidence that waterborne Se can elicit non-reproductive effects albeit at higher aqueous concentrations. ...

“Many of the sublethal effects of Se in fish are similar to those found in birds... The more sensitive chronic effects in birds are related to reproductive impairment.”

One of the difficulties in assessing the chronic toxicity of selenium is that its uptake by living organisms depends on its speciation. Most lab studies have been done using selenites or selenates as these chemicals can easily be purchased and dosed at controlled concentration in the water. The issue is that the concentration of selenium in the tissues of living organisms does not depend on the total concentration of selenium in the water, but rather on the concentration of the various selenium species. Besser (1993) calculated the bioconcentration factor¹ of selenium for three organisms depending on the selenium speciation. All experiments used a total concentration of 10 µg Se/L in the water, but the selenium speciation varied:

Table 2 - Bioconcentration factors

Organism	Exposure	Selenium speciation	Bioconcentration factor
<i>Chlamydomonas reinhardtii</i> (a green algae)	24 hours	Selenate	428
		Selenite	1 440
		Selenomethionine	5 320
<i>Daphnia magna</i> (a crustacean)	96 hours	Selenate	293
		Selenite	570
		Selenomethionine	30 300
<i>Lepomis macrochirus</i> (a fish)	30 days	Selenate	20
		Selenite	20
		Selenomethionine	5 000

In the same paper, Besser also reported that the speed of selenium uptake by the organisms studied also varies with the selenium speciation, with selenomethionine uptake being much faster than selenite, and selenite uptake being faster than selenate.

A decade later, Amweg (2003) did another study that looked at the potential for bioaccumulation of various selenium species. In this case Amweg was looking at a process named Algal/bacterial selenium reduction (ABSR) where inorganic selenium is converted to elemental selenium by a combination of algae and bacteria and then is removed from the water. This process is not widely used nowadays but was considered for a time for treating agricultural waste water containing large concentrations of selenium.

¹ The bioconcentration factor, or BCF, is the ratio of an element in the tissue to the concentration of the same element in water.

Amweg's study "was intended to monitor Se concentrations in invertebrates found in the ABSR system and assess the effect of ABSR treatment on Se bioavailability."

Amweg found that while the treatment managed to reduce the total selenium in the water by 60%, the bioconcentration factor for two algae species was actually increased by an order of magnitude after treatment. Overall, "[r]esults indicate that Se within and discharged from the ABSR was more bioavailable than that in the untreated drain water, and that except in the algal bioaccumulation test, organisms accumulated more Se exposed to ABSR-treated water than if exposed to untreated water." Amweg conclusions are damning for the ABSR process:

"We believe that production of organo-Se by microbial activity..., and reduction of selenate to selenite which is then accumulated by algae ... and incorporated into algal selenoproteins, caused the increased Se bioavailability seen after ABSR treatment. Higher organisms generally accumulate Se through their diet ... and this Se-rich algal biomass presumably served as a Se source for the other invertebrates of the ponds. ... Increased Se bioavailability is inherent in the system design due to its dependence on microbial Se reduction. ... {G}iven that organic Se forms are approximately 1000 more bioavailable than selenate ..., the system would have to be extraordinarily effective to achieve less bioaccumulation from the effluent than from the influent. Hypothetically, if the influent contained entirely selenate and the effluent contained entirely organic Se, the system would have to achieve greater than a 99.9% reduction in total Se concentration in order to be judged a success by the criterion of less bioaccumulation from the effluent."

An adage of modern toxicology is that "solely the dose determines that a thing is not a poison"²: there is a relationship between the concentration of a substance and its effect. What is important, however, is not the concentration in the environment, but the concentration in the tissues.

Selenium is only moderately acutely toxic in water, especially when present as selenate. Because of the widely different values for the bioconcentration factor of the various selenium species and the lack of information on the selenium speciation in most papers looking at chronic toxicity, its chronic toxicity is difficult to assess. For biological treatment at least, the question of bioavailability of the selenium in the treated water is highly relevant in assessing the performance of the treatment system.

Even neglecting the effect of selenium on the reproduction success, the well-publicized die-off events mentioned above are a good example of selenium toxicity. It is however likely that these die-offs are complex events caused by bioaccumulation of selenium through the food chain up to a point where the selenium concentration became high enough that it caused acute toxicity in the predators. It is also likely

² Paraphrased from Paracelsus' "Alle Dinge sind Gift, und nichts ist ohne Gift, allein die Dosis macht dass ein Ding kein Gift ist", which can also be translated to "All things are poison, and nothing is without poison, the dosage alone makes it so a thing is not a poison."

that many species have issues with reproduction success because of a similar chain of events, even if the selenium concentration in the tissues is not sufficient to cause acute toxicity.

Current Approaches for Selenium Removal in Water

Given the toxicity of selenium and the difficulty of removing it, it is not surprising that many competing technologies have been developed to try to remove it from waste water. We will try to broadly describe the main approaches that are currently in use, but the interested reader can refer to the excellent review by Golder (2020) titled “State-of-Knowledge on Selenium Treatment Technologies”, which itself refers to numerous papers on the subject.

Physico-chemical Treatment Options

Physico-chemical treatment options include reverse osmosis, ion exchange and chemical reduction. Because they are not biological they produce very little or no organo-selenium species, thereby reducing potential toxicity issues.

Reverse osmosis separates molecules based on their size. Selenites and selenates being relatively large molecules, it is quite easy to separate them. In mine effluents there are, however, many large molecules. In the presence of scaling molecules such as silica and gypsum, it becomes difficult to achieve a high recovery, that is to have a large fraction of the water produced as clean water (permeate). The other issue with reverse osmosis is that it only move selenium to a more concentrated stream. Given that in typical mine water the concentrated stream is 30% of the feed flow, one ends up with 70% of the feed being clean water ready for discharge and 30% of the flow having three times the selenium concentration that needs to be disposed of.

Ion exchange is similar to reverse osmosis, except that separation is based on the charge of the molecules rather than their size. Ion exchange resins have been developed that can bind selenites and selenates quite effectively, but they are subject to competition from other molecules, notably nitrates and sulfates. This increases the requirements for column regeneration, increasing the volume of waste to reject. In water with low nitrates and sulfates this volume can however be managed. Electro-reduction has been used with some success for this situation, but other techniques could also be used. In water with high nitrate or sulfate, the volume of waste water produced rapidly becomes unmanageable.

Chemical reduction with zero-valent iron (ZVI) was studied extensively for a time and has been installed at some sites. With this approach selenates are reduced to selenites, producing ferrous iron, and are then co-precipitated on iron hydroxide flocs. There are a number of variations on this process, including the use of sulfur-modified iron. While the process works, there are a number of practical considerations that make it quite complex. First, ZVI iron will also reduce nitrates to nitrites. This both consumes part of the ZVI and produces nitrites, a by-product toxic to many fish species. Secondly, ZVI effluent contains a lot of

soluble iron, mainly ferrous iron, another toxic element which must be removed before discharge. Finally, ZVI media has had a tendency to plug, reducing the flow in the reactor to a point where the media need to be replaced entirely. Newer media formulations are said to be less affected by this issue.

Biological Treatment Options

Biological treatment systems are based on the fact that bacteria and archaea can, in the right conditions, reduce selenites and selenates to elemental selenium, which is essentially insoluble in water. Elemental selenium can then be removed to produce selenium-free water.

All biological treatment options share some characteristics:

- Nitrates compete with selenites and selenates and when present in high enough concentration must be removed from the water before selenium reduction can occur effectively;
- Elemental selenium particles are both very small and not electrically charged, which makes their removal from water challenging;
- Elemental selenium particles are easily oxidizable and care must be taken through the process to prevent this from occurring;
- Reaction kinetics are slow, which translates into large reactors and hence high costs;
- By their nature all these processes will convert a fraction of the selenium to organo-selenium, with the potential toxicity issues that this may cause;
- Biological treatment systems have difficulty *consistently* achieving the 10 µg/L of total selenium which will possibly become the new selenium criteria for Canadian coal mines (ECC Canada 2022, Golder 2020).

There are many biological reduction processes available, and biological reduction is by far the most common selenium treatment process. The reader is referred to Golder (2020) for a more detailed introduction to these processes and some case studies.

Veolia's Tracer Se Process

In this section, Veolia's Tracer Se process will be presented. We will then present preliminary results and some advantages of this process.

The Process

Veolia's Tracer Se process combines biological reduction of selenates to selenites, removal of selenites and biomass under reducing conditions, and reoxygenation of the selenium-free water. A process schematic is shown below:

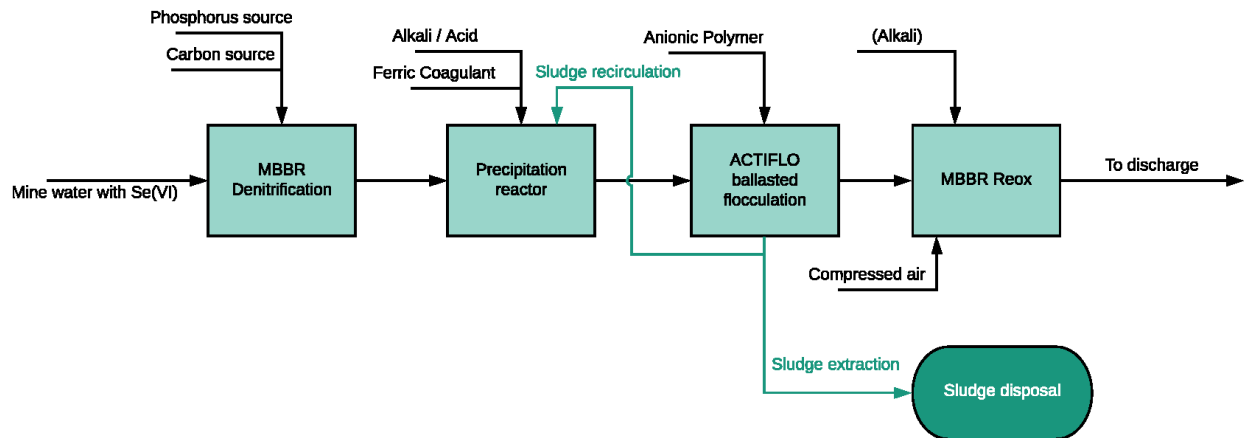


Figure 1 - Tracer Se Schematic Diagram

First, water is directed to a biological reduction reactor containing biomass and operated under anaerobic or anoxic conditions. A Moving Bed Biofilm Reactor (MBBR) can be used for this purpose. In this reactor, selenates are biologically reduced by the biomass to selenites or absorbed on the biomass. At the same time nitrites and nitrates, if present, are being reduced to nitrogen gas. The conditions in this reactor are controlled to minimize further reduction of selenites.

The water containing the selenites and any sloughed biomass is then directed to a precipitation reactor. While maintaining reducing conditions, a coagulant such as a ferric or aluminum salt is mixed with the water. By controlling the pH and using sludge recirculation to age the sludge, solids having adsorption sites are formed. Selenites are adsorbed onto these sites. Thereafter, the solids with adsorbed selenites in addition to the sloughed biomass containing adsorbed selenium are separated from the water. A ballasted flocculation settler such as Veolia’s Actiflo is well suited for this application.

The water is further treated in a second biological reactor under aerobic conditions where the water is subjected to reoxygenation. In the process reduced selenium species present in the water are oxidized to selenate, thus reducing the final toxicity of the final effluent.

Preliminary Results

Two laboratory studies have been completed so far. The first study, a proof-of-concept test, was conducted on a coal mine effluent. No detailed speciation was conducted and the test was stopped once removal of selenium was confirmed. Note that in this test the sequence was Reduction > Reox > Selenium removal

Table 3 - Proof-of-concept test results

Parameters	Units	Raw Water	After treatment
Se total	µg Se/L	344	38.4
Se dissolved	µg Se/L	311	-

Parameters	Units	Raw Water	After treatment
Selenites	µg Se/L	< 1	8.1
Selenates	µg Se/L	308	< 1

These results were encouraging, hence a second test was started. In this trial water from a hard rock mine was used. The second test was longer in duration and detailed analyses were conducted to try to understand the mechanisms involved in each reactor. The reactor sequence was also changed to reduction > solid removal > reox, hoping that this would improve selenium removal and decrease the concentration of selenites in the treated water.

For total recoverable and dissolved Se, each sample was digested in a closed vessel (bomb) with nitric and hydrochloric acids. The resulting digests were analyzed for Se content via inductively coupled plasma triple quadrupole mass spectrometry (ICP-QQQ-MS). Particulate selenium was calculated by difference. The sample analyzed for dissolved selenium was filtered on a 0.45 µm filter. It should be noted that a significant fraction of any elemental selenium present in the water will pass through such a filter.

For the selenium speciation, each aqueous sample was analyzed using ion chromatography inductively coupled plasma collision reaction cell mass spectrometry (IC-ICP-CRC-MS). In this process, selenium species are chromatographically separated on an ion exchange column and then quantified using inductively coupled plasma collision reaction cell mass spectrometry (ICP-CRC-MS).

Two detailed speciation campaigns were undertaken. The first campaign was initiated three months after the start of the test and focused on the reduction and solid removal steps. No sampling of the Reox reactor was done in this campaign.

Table 4 - First speciation campaign's results

Parameters	Units	Raw Water	Reduction	After Solid Removal
Macro Parameters				
Total recoverable selenium	µg Se/L	47.1	6.13	3.36
Particulate selenium (calculated)	µg Se/L	3.5	4.21	0.78
Dissolved Selenium	µg Se/L	43.6	1.92	2.58
Speciation				
Sum of dissolved selenium species (calculated)	µg Se/L	45.32	1.126	2.762
Dimethylselenoxide	µg Se/L	< 0.01	0.024	< 0.01
Methylseleninic acid	µg Se/L	0.046	< 0.01	< 0.01
Selenites	µg Se/L	0.219	0.377	0.186

Parameters	Units	Raw Water	Reduction	After Solid Removal
Selenates	µg Se/L	45	0.17	2.27
Selenocyanate	µg Se/L	< 0.01	< 0.01	< 0.01
Selenomethionine	µg Se/L	< 0.01	< 0.01	< 0.01
Selenosulfate	µg Se/L	< 0.01	< 0.01	< 0.01
Unknown Se speciation	µg Se/L	< 0.01	0.515	0.256

The main observations from this first speciation are the following:

- The measured dissolved selenium concentrations are close to the calculated total of the speciation species detected in the sample for all samples. This would be indicative of a low concentration of elemental selenium³;
- Selenium “disappears” in the reduction stage. This suggests that the system was not yet stabilized and that there was significant absorption of selenium on the biomass;
- Most of the total selenium remaining in the reduction stage is in particulate form;
- Selenium speciation in the reduction stage shows a high concentration of unknown selenium species. The organoselenium concentrations are low;
- Most of the selenium remaining after solid removal is in dissolved form, and mostly as selenate;
- After selenium removal organoselenium concentrations are below detection limits. Some unknown selenium species are still present in the clarified water.

The second campaign was conducted a month later, four months after the test was started. For this test, the reox was operated for a week with water from the selenium removal. Prior to this the reox was fed directly from the reduction step. This time the selenium mass balance is much better:

Table 5 - Second speciation campaign's results

Parameters	Units	Raw Water	Reduction	After Selenium Removal	Reox
Macro Parameters					
Total recoverable selenium	µg Se/L	47.9	47.8	3.55	6
Particulate selenium (calculated)	µg Se/L	1.2	43.71	0.97	0
Dissolved Selenium	µg Se/L	46.7	4.09	2.58	6.03

³ Because of the small size of elemental selenium particles, the measured “dissolved” selenium will include a large fraction of any elemental selenium present. However, elemental selenium being electrically neutral, it is not measured in the speciation analysis.

Parameters	Units	Raw Water	Reduction	After Selenium Removal	Reox
Speciation					
Sum of dissolved selenium species (calculated)	µg Se/L	45.015	3.709	1.145	2.908
Dimethylselenoxide	µg Se/L	< 0.01	0.067	0.045	0.026
Methylseleninic acid	µg Se/L	0.136	0.014	0.031	0.03
Selenites	µg Se/L	0.529	1.49	0.324	1.71
Selenates	µg Se/L	44.3	0.039	0.01	0.926
Selenocyanate	µg Se/L	< 0.01	0.767	0.501	0.141
Selenomethionine	µg Se/L	< 0.01	0.168	< 0.01	< 0.01
Selenosulfate	µg Se/L	< 0.01	0.202	0.034	0.041
Unknown Se speciation	µg Se/L	< 0.01	0.962	0.19	0.024

The main observations from this second speciation are the following:

- The measured dissolved selenium concentrations are close to the sum of the dissolved species detected in the sample for the raw water and in the reduction step, which is indicative of a low concentration of elemental selenium;
- The total selenium concentration in the reduction step is the same as in the raw water. The reduction reactor now seems at equilibrium, at least for selenium;
- Most of the total selenium in the reduction step is in particulate form and is likely associated with sloughed biomass.
- Selenites are the main dissolved selenium species in the reduction step;
- After selenium removal most of the selenium remaining is in dissolved form. Selenocyanates and selenites are the most common species identified;
- The total selenium concentration in the reox reactor is higher than in the water after selenium removal. This is likely due to the reox not being at equilibrium;
- Most of the selenium remaining in the reox is in dissolved form, and mostly as selenite and selenate. Organoselenium species concentrations in the reox step are lower than after selenium removal, suggesting at least a partial oxidation of these species in the reox.

Advantages

Compared to existing treatment processes the Tracer Se has the following advantages:

- Nitrites and nitrates are not detrimental to the process, and might in fact be required;
- Kinetics are much faster than with a process where there is biological reduction to elemental selenium. This should translate into smaller size and lower costs;
- The process minimizes the production of the more toxic forms of selenium;
- Preliminary results indicate that the process will be able to meet the proposed Canadian Coal Mine Effluent Regulation of 10 µg/L. Pilot tests will be required to confirm the process performance.

Future work

At this point we believe that the Tracer Se process shows a lot of potential, but further work is required before it can be commercialized.

A third laboratory trial is being planned. In this trial, the reox reactor will be fed on a continuous basis with water after solid removal, which should solve the equilibrium issues we had in the preceding trial. This will allow us to better understand the change in selenium speciation in the reox reactor.

Laboratory trials are useful, but at some point pilot tests will have to be undertaken. A few sites have manifested an interest, but setting up these tests is a long process, and it is likely that these tests will not start until fall of 2022, maybe spring of 2023.

An important aspect of the pilot tests will be to monitor the long-term system performance when the raw water characteristics change, such as changes in concentration or temperature.

Like most treatment processes, the sludge produced in the Tracer Se process is susceptible to oxidation. It is not really possible to characterize the sludge at laboratory scale, a pilot is required to produce enough sludge to allow testing. Once the sludge is available various options for safe long-term sludge storage can be studied.

Conclusion

Selenium in wastewater effluent can be a major issue due to the complexity of its removal as well as the possibility to result in a more toxic effluent after treatment, due to the modification of the bioavailability of the selenium through different processes. A new treatment process for removal of selenium, the Tracer Se process, which combines biological and physico-chemical processes, is being developed by Veolia. While the development is still in the early phases, preliminary results have shown that there is potential for excellent performance, both in terms of total selenium and low concentration of known bioavailable, hence toxic, organo-selenium forms in the treated water. Tests are ongoing and we will publish new results as they become available.

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