Meeting the Newest FGD Wastewater Trace Contaminant Guidelines with Functionalized Activated Alumina

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ABSTRACT

In 2013, the USA EPA issued lower guideline targets for wastewater discharge from coal fired power plants that further limit the discharge of selenium, mercury, arsenic and other contaminants. In preparation for these new guidelines, a functionalized alumina adsorbent media was used to treat flue gas desulfurization and leachate wastewaters at twelve power plants. Selenium, mercury, and arsenic were removed to low levels across a broad range of water qualities. The media successfully removed the selenate species of selenium.

BACKGROUND

The recently issued USA EPA revised effluent limit guidelines for flue gas desulfurization (FGD) wastewaters tighten the discharge of trace contaminants in outfall waters. The new limits, derived from "technology-based" effluent limits researched for the Merrimack Station, NH (EPA, 2011), also reflect the evolving ability to analyze for toxic contaminants to lower detection limits. Three new target limits of special concern are:

Contaminant	Daily Max.	30 Day Avg.
Mercury	242 ng/L	119 ng/L
Arsenic	8 µg/L	6 µg/L
Selenium	16 µg/L	10 µg/L

This report is an investigation of functionalized activated alumina adsorbent technology (Hayes, 2013) as an effective, economical and easy to deploy method toward achieving these guidelines.

CHARACTERIZATION OF FGD WASTEWATERS

Treatment of FGD associated wastewaters to remove mercury, arsenic and selenium, which can be present at both mg/L (ppm) and at trace µg/L (ppb) levels, to target levels is not straightforward. FGD waters vary as a function of coal chemistry and station operating conditions. Factors such as the processes in the plant, the contaminant form, species, solubility, concentration and the level of competing contaminants all play a role in the ability to remove trace contaminants. In general, FGD wastewaters can be described as very high in total dissolved solids (TDS) containing calcium, magnesium, sulfate and chloride at levels well over 1000 mg/L each. Boron is often present at levels in the hundreds of mg/L. Due to forced oxidation scrubber operations, the wastewater is usually well oxidized driving selenium to the selenate form. Mercury will often be present in only a partially soluble form.

Through partnering relationships with EPRI and three electric utility companies operating power generation plants in the eastern, mid-western and southern regions of the US, twelve different FGD scrubber and FGD leachate/pond waters were evaluated for trace contaminant removal using functionalized alumina media deployed in flow-through vessels.

Table 1 summarizes the total and dissolved levels of trace contaminants contained in these waters before adsorbent treatment. In general, selenium and the majority of contaminants were present in a soluble form. Mercury is the exception and often was present partially as insoluble particles. Table 2 summarizes the levels of the common water ions present in these waters, including sulfate, hardness ions and chloride. There is significant variation in the water quality among the twelve. The leachate waters were found to be typically lower in total dissolved solids. Although specific selenium speciation was unknown for the water streams studied, the prevalent indication for FGD waters from prior research is that selenate is the majority species present along with some selenite and selenosulfate (Searcy, 2011).

Table 1 – Trace Contaminant Levels In The Twelve FGD Station Waters, Total - Dissolved

Water	Type	Hg, ppt	Se, ppb	As, ppb	B, ppm	Cd, ppb	Cr, ppb	Cu, ppb	Th, ppb
1	FGD	470 - 40	300 - 300	6 – 6	88	ND	5 - 5	ND	_
2	FGD	26 - ND	1100 - 1100	4.6 – 4	520	0.9 – 0.9	10 -10	ND	-
3	FGD	911 - 218	503 - 422	9.4 - 3.9	232	ND	5.7 / 4.1	16 / 8	18.6 - 12.1
4	FGD	173 - ND	208 - 208	5.6 - 5.4	302	ND	5 - 5	6.2 – ND	17.6 - 15
5	FGD	14 - 14	353 - 353	5.5 – ND	173	2.6 / 2.3	9 / 8.3	8.2 - 7.7	25.5 - 24.0
6	FGD	20.8 T	1,700 - 1410	9.9 - 9.1	378	2.9 - 2.5	6.5 - 5.0	2.5 – ND	29.8 – 26.8
7	FGD	18,200 T	400 - 400	12 – 7.6	399	147 – 134	42 - 16	20 - 9	-
8	FGD	207 T	2800 - 2600	5.2 – 3.1	162	70 – 70	246 - 230	-	ND
9	Leachate	1100	10	150	9	ND	ND	ND	-
10	Leachate	78 - 53	550 - 520	2800	40	2.4 – ND	140 - 120	23 – 17	-
11	Leachate	8.9 - 8.9	0.3 - 0.3	6.2 - 2.8	26	ND	4 - 2.3	14 - ND	6.7 - 5.9
12	Pond	15.8 – 15.6* *oxidized	34 - 34	4.8 - 4.7	-	-	-	-	-

Table 2 - Primary Water Constituents in the Twelve FGD Station Waters, mg/L (ppm)

Water	Type	Calcium	Magnesium	Sulfate	Chloride	Sodium	Nitrate	Fluoride	SiO2	Bromide	Alkalinity
1	FGD	560	1200	6000	910	100	0.5	-	29	9	200
2	FGD	1500	2900	6100	7100	350	46	-	1.4	56	1800
3	FGD	979	2070	4440	4490	-	-	-	14	71	-
4	FGD	1350	2820	4750	6670	-	-	-	6	94	-
5	FGD	3320	2240	2060	9820	-		12	-	84	66
6	FGD	798	4650	8100	4480	-		102	-	76	-
7	FGD	842	4100	8810	5600	-	-	56	44	-	121
8	FGD	2910	2600	2430	4280	-	109	24	80	9	-
9	Leachate	1100	4.8	2400	1700	350	ND	-	13	20	270
10	Leachate	4600	1400	1700	110	1300	0.69	-	16	3	1400
11	Leachate	511	82	1310	9			0.2	23	4	-
12	Pond	368	1.3	1030	527		0.3	-	-	-	-

MATERIALS AND METHODS

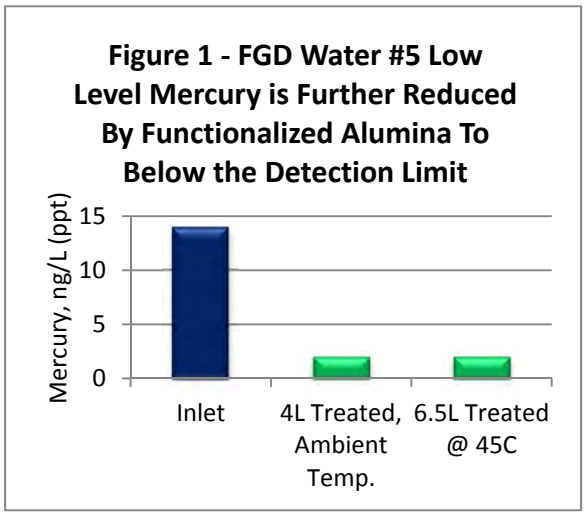
The performance of adsorbents is best determined under water flow conditions. This provides the most accurate prediction of % removal. To evaluate in water flow-through conditions, a packed bed of media in a vessel (tall, thin columns are suitable laboratory vessels) was utilized in which the hydraulic loading/flow flux was 1 to 6 gpm/ft^2 and the bed depth could be adjusted to provide contact times typically of 5 to 25 minutes. In laboratory evaluations, a minimum quantity of water equivalent to 10 empty bed volumes should be treated at a target contact time to establish a robust contaminant removal profile. Treating additional bed volumes can help more accurately identify the long-term capacity of the media. Up-flow or down-flow through the media packed bed has been found to produce no discernible effect on media performance. The flow-through test provides a prediction of long term adsorption capacity and defines the best contact time for optimum contaminant removal. Flow-through testing has a high correlation rate with performance in installed commercial systems.

The majority of results presented in this paper were obtained by flowing water received from each station up-flow through 1 inch diameter x 36 inch high glass columns packed with an average of 350 grams of functionalized alumina in 3/16 inch (4760 micron) pellet form. The average empty bed volume (BV) was 450 cm^3 at varied water-to-media contact times of 20 to 27 minutes.

Selenium, especially in the selenate form, is a contaminant that for removal by functionalized alumina media has shown a positive correlation with increasing contact time. It is theorized that the extra oxygen on selenate provides a larger molecular size that seems to benefit from a longer diffusion time into the porous media (MAR Systems, 2012). As a result for some waters, a fill-hold-drain batch approach results in much greater removal efficiency. Fill-hold-drain batch approaches were tested when flow-through results indicated that additional removal might be achieved from longer contact times. For fill-hold-drain testing, 110 grams of media were contacted with 110 mL station water and held for 30 minutes, 1 hour, or 2.5 hours. Following the hold time, the water was removed from the media with filtration through a 0.45µm syringe filter and analyzed for trace contaminant level. Often, the same media was then re-exposed to a new batch of water for the same hold time and this was repeated through numerous cycles of treatment.

PERFORMANCE of FUNCTIONALIZED ALUMINA ADSORBENT MEDIA IN FGD SCRUBBER and LEACHATE WASTEWATERS

MERCURY – For the EPA target limits for mercury of 119 ng/L (ppt) 30 day average and 242 ng/L (ppt) daily maximum discharge, six of the station's waters were already discharging well below the target and mercury was not a primary removal issue. At these low levels, mercury removal by functionalized alumina was still significant and typified by the removal shown in Figure 1 for FGD Water #5, where the mercury level was reduced from 15 ppt to below a detection level of 0.25 ppt



For the six other station waters that had mercury levels well above the target, functionalized alumina treatment of these waters "as received" resulted in the removal levels summarized in Table 3. When treating "as received", no attempt was made to modify the insoluble mercury that was present and the water was pumped through the media regardless of the level of soluble mercury. The results in Table 3 indicate variability in mercury removal efficiencies that are very typical when high levels of insoluble mercury particles are present. Adsorbent media are designed to remove soluble mercury. Soluble ionic inorganic mercury is free to form a chemical complex and bonds readily and permanently to sites on functionalized alumina for removal. Insoluble mercury will be filtered by media for removal but it will not be permanently bonded to the surface and removed by true adsorption. As a result, there will be variability in the amount of mercury removed and the removal may vary throughout the treatment.

Table 3 - Mercury Removal for FGD Waters "As Received"

	Inlet Hg ppt	% Soluble Hg	Outlet Hg ppt	% Removed
1	470	8.5%	220	53%
3	911	24%	ND	>95%
4	173	0%	93	46%
7	18,200	NA	550	97%
8	207	NA	91	56%
9	1100	<1%	53	95%

All six of the waters with mercury levels above the EPA target limits contained a low percentage of mercury present in a soluble form and a very high percentage of insoluble mercury. In particular, as shown in Table 3, 100% of the mercury in FGD Water #4 was insoluble. The mercury removal observed over the course of treatment for FGD Water #4 "as received" is shown in Figure 2. The average removal of mercury due to the media filtration effect was 46% but in reality, removal was very erratic and not efficient.

Figure 2 - FGD #4 Mercury Removal By Functionalized Alumina When All Mercury is Insoluble, 22 Min CT, Mercury ppt

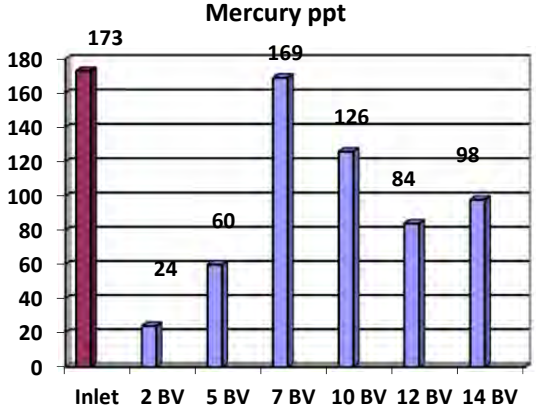
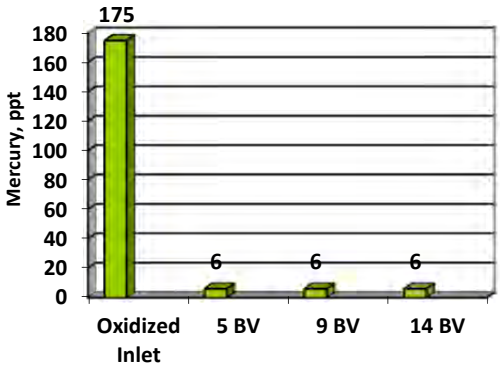


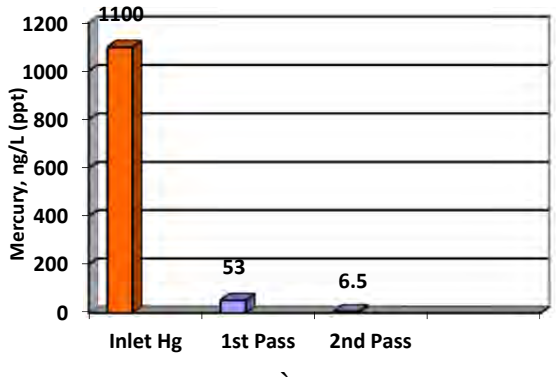
Figure 3 - FGD Water #4 Mercury Removal By Functionalized Alumina When All Mercury is Soluble, 22 Min CT, Mercury ppt



Importance of Soluble Mercury - To mitigate this concern, oxidation was used to solubilize mercury particles. Oxidation has been found to be effective in solubilizing mercury for removal not only in FGD waters but also in other types of industrial waters. As an example, FGD Water #4, which in Figure 2 had no soluble mercury and only 46% average removal, was pretreated with chlorine fed as sodium hypochlorite bleach to breakthrough of a free chlorine residual of 0.1 ppm and then held overnight to allow for reaction time. After chlorination and the hold time, essentially all of the mercury, 175 ppt, was found to be dissolved (defined as mercury after 0.45µm filtration). This chlorine pretreated water was then pumped through the functionalized alumina at a water-to-media contact time of 24 minutes. For oxidized FGD Water #4, functionalized alumina reduced the mercury by 96%, dropping it from 175 ppt to a constant 6 ppt through all 14 bed volumes treated as shown in Figure 3. A comparison of Figures 2 and 3 indicates the increased removal efficiency gained from pretreating the mercury to put it in a soluble form.

Contact Time - To achieve ultralow levels of mercury removal, such as the single digit ppt (parts per trillion) levels suggested by the EPA, longer contact times with functionalized alumina can be helpful. The benefit of longer contact time, which can be in the form of a second pass, additional media or fill-hold-drain applications, is shown in Figure 4 for Leachate Water #9. As listed in Table 3, Leachate #9 water initially contained 1100 ppt mercury. Removal by a single pass at 25 minutes of contact time reduced the mercury from 1100 ppt to 53 ppt (95% removal.) A second pass approach was used to increase the contact time. The treated water from the 25 minute contact time first pass was collected and pumped back through the used media effectively doubling the contact time to 50 minutes. The mercury dropped further from 53 ppt to 6.5 ppt, for a 99% total removal, as shown in Figure 4.

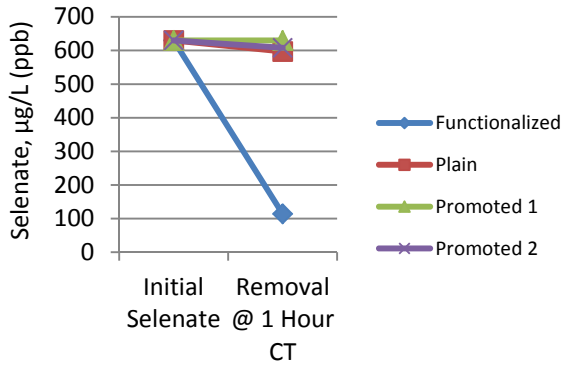
Figure 4 - Increasing the Media-to-Water Contact Time Removed Mercury to Single Digit ppt Levels for Leachate Water #9



SELENIUM – The application of functionalized alumina to remove selenium from FGD water is similar to mercury with the water-to-media contact time being the primary control parameter. However, selenium does differ markedly from mercury in both solubility and species type and this presents its own particular set of issues. In water, selenium typically is present as an oxyanion. As an anion, it would utilize different adsorption sites on the media than cationic mercury. Selenium also tends to be highly soluble and does not benefit from oxidation. In fact oxidation is not recommended as it can drive the oxyanion species from selenite (SeO_3) to selenate (SeO_4). It is generally recognized that selenate is the more difficult species to remove and that for FGD waters, forced oxidation drives speciation to be predominantly selenate (Searcy, 2011).

Removing Selenate - There are limited approaches and materials effective for selenate removal. Among various activated alumina materials, only the functionalized alumina, which contains a proprietary reaction chemistry throughout its pore structure, was effective in removing selenate. Figure 5 shows the significant reduction in selenate that occurs quickly with functionalized alumina over plain and two different types of promoted activated alumina.

Figure 5 - Selenate Removal Capabilities of Various Activated Alumina Adsorbents Functionalized Alumina = 82% Removal

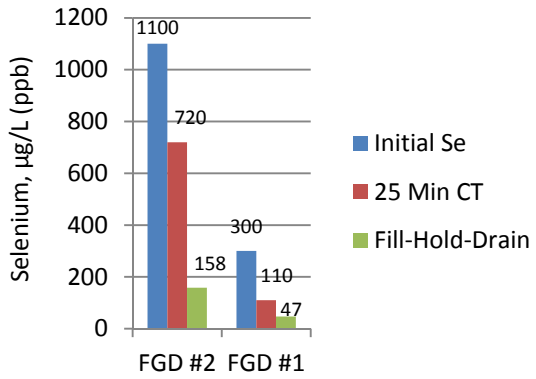


Functionalized Alumina On First Pass - The application of functionalized alumina to selenium in FGD waters results in significant removal of selenium. Table 4 is a summary of the selenium removal from all twelve station waters tested “as received.” The initial selenium concentrations varied across the waters from <1 ppb to 2800 ppb. Results are reported for 25 – 28 minute contact times with one pass through the media and show an average selenium reduction of 51%.

Table 4 – Selenium Removal Summary for Twelve FGS Station Waters First Pass Results at a 25 Minute Water-to-Media Contact Time			
Water	Initial Se, ppb	Treated Se, ppb	Removal
1 FGD	300	110	63%
2 FGD	1100	720	35%
3 FGD	503	337	33%
4 FGD	208	70	66%
5 FGD	353	125	65%
6 FGD	1700	220	87%
7 FGD	400	160	60%
8 FGD	2800	2300	18%
9 Leach	10	5	50%
10 Leach	550	400	27%
11 Leach	0.3	0.12	60%
12 Pond	0	-	-
Average % Selenium Removal @ 25 Min CT			51%

The Importance of Contact Time - Table 4 indicates that there is variability to selenium removal in these waters and it is believed to be due to the variations in selenium species present and the water quality. As noted in Table 4, average selenium removal was 51%. One approach that increases selenium removal with functionalized alumina media is to increase the contact time. This can be accomplished by adding additional media, by running the water through the media for a second time or by slowing down the water flow to a fill-hold-drain approach. Examples of the benefit of longer contact time are shown in Figure 6 for FGD Waters #2 and #1, where a fill-hold-drain treatment approach was tested. A 2.5 hour retention time of the fill-hold-drain treatment approach provided a significant reduction in selenium over the 25 minute contact time in the flow-through system for both waters. Removal in FGD Water #2 increased from 35% to 86% and FGD Water #1 saw a 20% increase from 63% to 83% selenium removal.

Figure 6 - The Positive Impact of Increasing the Contact Time on Selenium Removal

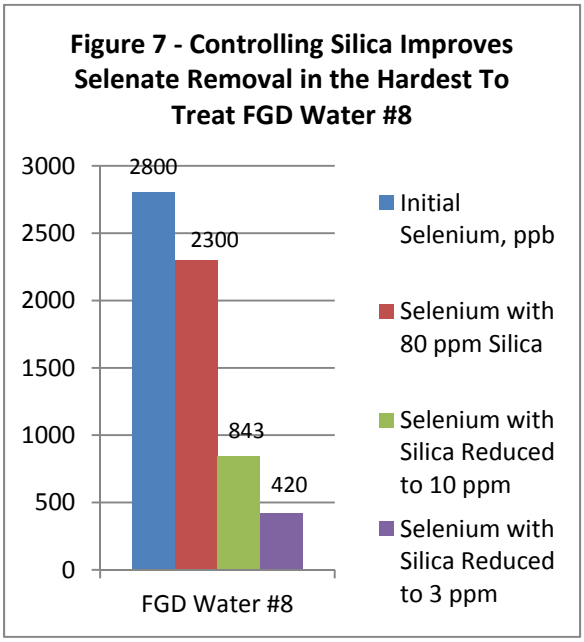


In total, eight of the station waters were investigated at longer contact times and all eight saw a large increase in selenium removal with increased contact time. A 150 minute (2.5 hour) empty bed contact time (equivalent to a 30 minute hydraulic retention time) was tested by a fill-hold-drain application and in every case, the selenium removal increased by a minimum of 20%. The increased contact time results are summarized in Table 5 and indicate an average increase of 31% over the 25 minute contact time results shown in Table 4. A maximum 64% improvement in selenium removal was found for Leachate #10 water.

Table 5 – Increased Selenium Removal With 150 Minute Contact Time vs. Table 4				
Water	Initial Se,	Se, ppb at 2.5	Removal	Δ%
1	280	47	83%	+20%
2	1100	158	86%	+51%
3	503	100	80%	+47%
4	208	30	86%	+20%
5	353	3	99%	+34%
8	2800	1050	63%	+45%
9	10	3	70%	+20%
10	550	51	91%	+64%
Average % Selenium Removal @ 2.5 Hour			82%	+31% avg.

Understanding Competing Ions - It is recognized that increasing the contact time is not the only answer to improve selenium removal. To reach single digit selenium removal levels for all FGD waters when using functionalized alumina, a pretreatment of the water may be necessary due to other factors, especially competing anions. The high ionic strength of FGD waters makes them vulnerable to competing ions. Sulfate, fluoride, nitrate, alkalinity, orthophosphate and silica all have the potential to compete with the selenate anion for adsorption sites on medias capable of adsorbing anions. It is a complicated relationship between the chemistry of the adsorption site, kinetics and rate of bond formation/complexation to the media adsorption site, ion exchange mechanisms and the ion pairing potential of all anions with the counter ions present in the water.

Of the potential competing anions, silica is of high interest for functionalized alumina media because activated alumina has been shown in prior research to remove silica from waters through formation of aluminum silicate complexes (Bouguerra, 2007). Aluminum is also one of the chemistry sites on functionalized alumina that could be capable of forming a complex with selenate. Limited silica data is available for the twelve FGD waters in this study because silica was not an investigative focus when the evaluations were conducted. The silica reported in Table 2 was measured after the evaluations but that analysis does indicate that some low to moderate levels of silica were generally present in these FGD waters. In all cases, the level of silica present was in excess of the concentration of selenium present. The highest silica and lowest percent selenium removal was found for FGD Water #8. Enough of this water remained for an evaluation of the effect of removing silica from the water prior to using functionalized alumina media for selenate removal. For this study, a column of a proprietary media that adsorbs silica but not selenium (<10% selenium removal was measured by the silica pretreatment media) was employed ahead of the functionalized alumina column as a pretreatment. FGD Water #8 containing 80 ppm silica and 2600 ppb selenium was pumped through a column of the pretreatment media directly into a column of the functionalized alumina at a 26 minute contact time for each column. When silica was reduced by pretreatment media, the selenium removal by functionalized alumina for FGD Water #8 increased significantly, Figure 7. 85% of the selenium was removed by functionalized alumina when the silica was reduced to 3 ppm. This is significant in comparison to the result of only 18% removal achieved when 80 ppm silica was present in the water. The selenium removal as a function of decreasing silica concentration appears to be dramatic in Figure 7.



Understanding the impact of all competing anions for each of the selenium species will better enable the development of tailored selenium removal treatment programs that meet the requirements of individual water streams. Continuing research is in progress to identify and quantify all of these impacts on selenium. It appears that simple pretreatments and extensions of contact time for functionalized alumina can significantly close the gap toward selenium discharge targets.

ARSENIC - Arsenic was present in most of the twelve waters at low levels. The exception was Leachate Water #10, which contained arsenic at the ppm level. As shown in Table 5, arsenic was removed simultaneously with selenium and mercury by functionalized alumina at an average removal rate of 70%. For Leachate Water #10, arsenic was reduced from 2800 ppb to 430 ppb with a 25 minute contact time. A longer contact time would be expected to further reduce this level to achieve the target limits for arsenic.

Table 5 – Simultaneous Removal of Arsenic Along With Mercury and Selenium By Functionalized Alumina Treating FGD Waters

Water	Initial As, ppb	Treated As, ppb	Removal
1	6	ND	>95%
2	4.6	2	56%
3	9.4	5.6	40%
4	5.6	3.9	30%
5	5.5	ND	>95%
6	9.9	5.9	40%
7	12	6	50%
8	5.2	ND	>95%
9	150	47	69%
10	2800	430	85%
11	6.2	ND	>95%
12	4.8	ND	>95%
Average % Arsenic Removal @ Varied Contact Times			70%

COMPARATIVE COST TO TREAT

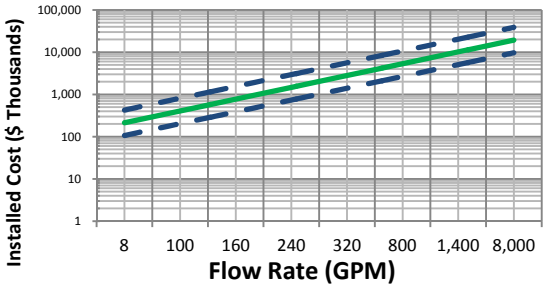
Functionalized alumina has very favorable economics compared to other technologies that are often considered for meeting new wastewater discharge limits.

Figure 8 and Figure 9 provide cost estimates for installed equipment and operating costs for functionalized alumina, respectively. The functionalized alumina

system costs include the following assumptions:

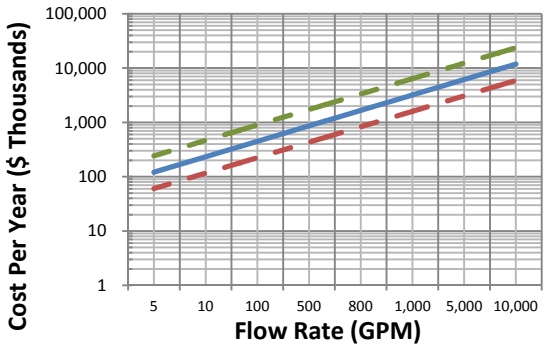
- Media capable of simultaneously removing selenite, selenate, and other FGD trace contaminants
- Influent water multi-media filtration
- Primary treatment vessels capable of processing full flow at average contact time followed in series by a duplicate set of treatment vessels
- Operating costs include media replacement and disposal costs at four month intervals

Figure 8
Functionalized Activated Alumina
Total Installed Cost



Dashed lines represent +100% / -50% Variance

Figure 9
Functionalized Activated Alumina
Operations and Maintenance Cost,
50 µg/L (ppb) Selenium



Dashed lines represent +100% / -50% Variance

Table 6 provides a comparison of equipment and operating costs for a functionalized alumina treatment system versus other selenium removal technologies. Selenium was chosen for this example because recent third-party cost comparisons are available from the NAMC White Paper Report Addendum prepared by CH2M HILL (CH2M Hill 2013).

The cost comparisons presented in Table 6 generally represent systems with pretreatment and post-treatment processes that are appropriate for the specific technology. Since the NAMC White Paper report compares operating costs for these technologies at various selenium concentrations, a common level of 50 ppb was chosen.

Table 6 - Cost Comparison for Functionalized Alumina vs Alternate Selenium Removal Technologies at 100 GPM with 50 ppb Selenium

			Variance vs. Functionalized Alumina	
			Total Installed Cost	Annual Operating Cost
Technology	**Total Installed Cost**	**Annual Operating Cost**	**$000's**	**$000's**
Functionalized Alumina	800	600		
Advanced Biologic Metals Reactor	14,000	600	13,200	-
Fluidized Bed Reactor	9,000	700	8,200	100
Ion Exchange	10,000	1,500	200	900
Zero Valent Iron	9,000	1,500	8,200	900

As shown in Table 6, functionalized alumina has an installed equipment cost that is estimated to be 20% to 90% less than the

compared technologies. This variance is primarily due to functionalized alumina using industry-standard filtration tanks that are significantly less expensive than the custom delivery systems used by many of the comparative technologies.

Table 6 also provides estimates that indicate functionalized alumina annual operating costs can be as much as 90% less than the compared technologies. These lower operating costs are reflective of the straightforward implementation of this adsorptive media that does not require significant manpower for chemical dosing and system monitoring, significant levels of power consumption, or the handling of large volumes of ancillary waste that can be generated in large volumes using the compared technologies in a FGD application.

While the comparison presented in Table 6 is for a 100 gpm system, comparison of Figure 8 with the total installed cost curves in the NAMC White Paper report illustrate the cost advantage of functionalized alumina versus the compared technologies increases at a greater rate as the treatment system flow rate increases.

The operating cost advantages of functionalized alumina are dependent on the contaminant concentration. The operating cost will increase with increasing contaminant concentration but will maintain a similar cost advantage to other concentration dependent technologies shown in Table 6.

The operating cost advantage versus the biological technologies in Table 6 will be reduced at higher concentrations. However it should be noted, that functionalized alumina provides a treatment technology that is less prone to system upsets associated with equipment failure or biological media impairment that can occur with biological treatment solutions.

Selenium was the primary contaminant considered in this cost analysis but as previously stated, the functionalized alumina will simultaneously remove other trace contaminants common in FGD wastewater streams. This trace contaminant removal will generally be complementary to selenium removal as the selenium breakthrough will most often dictate functionalized media exchange requirements.

CONCLUSIONS

Functionalized alumina significantly addresses the key contaminants cited in the EPA report. It simultaneously removed mercury, selenium and arsenic from all twelve of the FGD and Leachate power plant waters investigated.

There are application factors that can further boost the performance of functionalized alumina adsorbent. Longer water-to-media contact times and pretreatments for insoluble metals and competing silica ions significantly increased mercury and selenium removal in these waters.

Functionalized alumina can provide an economic advantage to alternative technologies for removing trace contaminants from FGD wastewaters. Capital installation costs are significantly lower than other treatment alternatives. Annual operating costs are generally equivalent or less than other treatment alternatives.

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