PNNL-38400, Rev. 0
DVZ-RPT-123, Rev. 0

Batch Test Analysis of Potential Treatment Technologies for Cyanide Groundwater Remediation

September 2025

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Prepared for the U.S. Department of Energy
under Contract DE-AC05-76RL01830

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Summary

In addressing groundwater contamination, several remediation technologies are widely employed, including ion exchange (IX) resins and other adsorbents in pump-and-treat (P&T) facilities. However, many sites do not have the complex nature of the Hanford Site – a legacy production site that is part of the U.S. Department of Energy nuclear weapons complex. The Hanford Site covers an area of nearly 1,500 km^2 and has various inorganic and organic contaminants. One contaminant of concern in the Hanford Site's Central Plateau is cyanide, particularly in the recalcitrant form of ferrocyanide. While ferrocyanide is often considered a stable complex, it can release free cyanide under certain conditions and presents challenges when testing for cyanide as a total cyanide analysis cannot differentiate between complexed and free cyanide.

The Hanford Site already uses P&T to mitigate groundwater contamination across the site, notably removing radionuclides technetium-99 and uranium from groundwater at the 200 West Area (200W) P&T facility and chromium at the five 100 Area P&T facilities. The 200W P&T facility specifically uses two IX resins, Dowex 21K to remove uranium and Purolite A532E to remove technetium-99. To evaluate the potential of IX resin and other adsorbents for ferrocyanide treatment at the Hanford Site, this study aimed to determine which commercially available IX resins/sorbents are likely to take up the most ferrocyanide under batch conditions (high liquid to solid ratio) using synthetic groundwater (SGW) of a similar composition to that at the 200W P&T facility. This work did not include evaluation involving Tc, U, or Cr, it did include common groundwater anions and nitrate, which is also a contaminant of concern. In addition to the targeted treatment of ferrocyanide Dowex 21K and A532E were investigated as they may incidentally uptake ferrocyanide during P&T activities. Both resins were able to uptake ferrocyanide at a concentration of 1.5 g/L, with Dowex 21K taking up more (nearly 100%) than Purolite A532E (approximately 50%). Both resins also took up nitrate under testing conditions.

In addition to the competitive nature of anion uptake by the resins already in use at the 200W P&T facility, various other commercially available resins were also evaluated and were shown to uptake ferrocyanide along with nitrate. At a concentration of 1.5 g/L ferrocyanide, almost all tested commercially available resins removed all ferrocyanide from the system within 72 hours. However, when exposed to a concentration of excessively high ferrocyanide, one resin – Purolite A500 – outperformed the others. At the higher concentration (10.5 g/L ferrocyanide), only one of the resins (A532E) tested took up any nitrate, suggesting that A532E has a high affinity for nitrate despite the high concentration gradient for ferrocyanide uptake.

These findings emphasize the need for site-specific testing to optimize treatment technologies for ferrocyanide removal at the 200W P&T facility. Purolite A500 emerged as the best performing IX resin for ferrocyanide removal, with SBG1 and Dowex 21K also demonstrating significant uptake. While lower concentrations of ferrocyanide were effectively removed by most IX resins, this process simultaneously interacts with anions of interest (e.g., TcO_4^-) and groundwater constituents. Dowex 21K showed high uptake of ferrocyanide and nitrate, while A532E exhibited moderate performance, indicating that increased concentrations of influent cyanide could disrupt treatment efficiency due to competition. To address these knowledge gaps, future testing with one-dimensional flow columns of Dowex 21K, A532E, and Purolite A500 would further constrain cyanide uptake and its influence on contaminant removal efficiency, along with the addition of other contaminants of concern (e.g., TcO_4^-).

Acknowledgments

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Acronyms and Abbreviations

1-D	one dimensional
200W	200 West Area
ASTM	American Society for Testing and Materials
BSN	bismuth subnitrate
DOE	U.S. Department of Energy
EPA	U.S. Environmental Protection Agency
HDPE	high-density polyethylene
GAC	granular activated carbon
ICP-OES	inductively coupled plasma – optical emission spectroscopy
IX	ion exchange
NQAP	Nuclear Quality Assurance Program
OU	operable unit
P&T	pump-and-treat
QA/QC	quality assurance/quality control
SGW	synthetic groundwater

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1.0 Introduction

This report aims to support the understanding, treatability, and necessary decision-making processes related to cyanide contamination and its remediation at the U.S. Department of Energy (DOE) Hanford Site (Richland, WA). Specifically, the ability of commercially available resin were examined under batch test conditions with ferrocyanide added to synthetic groundwater containing nitrate, a common co-contaminant. The ferrocyanide was investigated at two concentrations, 1.5 g/L which was selected based on the highest total cyanide reported at the Hanford site, and 10.5 g/L, an exaggerated concentration used to determine which resin had the highest capacity. Additionally, the tests described herein were designed to begin assessing the potential impacts of cyanide on the performance of existing treatment technologies used at the Hanford Site's 200 West Area (200W) pump-and-treat (P&T) facility. This report outlines the current knowledge gaps in cyanide remediation and impacts on new and ongoing use of ion exchange (IX) resins for groundwater remediation in the presence of cyanide species, with a focus on the ongoing cleanup mission at the Hanford Site.

Section 1.0 provides an overview of ongoing remediation activities at Hanford and cyanide chemistry in subsurface environments. Section 2.0 details the methods used in this study. Section 3.0 describes the results and discusses their significance. Section 4.0 summarizes the findings of this work and future research opportunities to fill remaining technical knowledge gaps. Section 5.0 lists references for further information.

1.1 Quality Assurance

This work was performed in accordance with the Pacific Northwest National Laboratory Nuclear Quality Assurance Program (NQAP). The NQAP complies with the DOE Order 414.1D, *Quality Assurance*. The NQAP uses NQA-1-2012, *Quality Assurance Requirements for Nuclear Facility Application*, as its consensus standard and NQA-1-2012, Subpart 4.2.1, as the basis for its graded approach to quality.

This work emphasized acquiring new theoretical or experimental knowledge. The information associated with this report should not be used as design input or operating parameters without additional qualification.

All reagents used were of analytical grade or higher unless otherwise noted. All water used was deionized water meeting ASTM requirements for Type II water or higher (ASTM International 2024).

1.2 Hanford Pump-and-Treat Operations

During decades of operation as a production site, the Hanford Site experienced releases of various chemical and radioactive waste that contaminated soil and groundwater (DOE 2023). This includes historical releases of ferrocyanide $[Fe(CN)_6]^{4-}$, which was used as a scavenger for Cs-137 and Sr-90. Various successful technologies have been used for remediation at Hanford, including permeable reactive barriers, source treatment (e.g., excavation, *in situ* immobilization), and the use of P&T facilities that treat over 2 billion gallons of water per year (DOE 2023).

The Hanford Site operates six groundwater P&T facilities, including one located in the 200 Area, which is one of the four original areas at Hanford included in the U.S. Environmental Protection Agency (EPA) National Priorities List. The 200 Area is further divided into the 200 East and 200 West areas to better address cleanup concerns. The 200 Area P&T facility is located in the 200 West Area (200W) and, for example, currently treats perched water and groundwater that has been contaminated with inorganic (i.e., uranium, technetium-99) and organic (carbon tetrachloride) contaminants (EPA 1995). There are also five

P&T facilities located in 100 Area operable units (OUs) (100 HX, 100-DX, 100-KW, 100-KX, and 100-KR4) along the Hanford river corridor that are used to treat chromium contaminated groundwater (DOE 2024).

The P&T facilities at Hanford use various IX resins to remove contaminants prior to reinjection. For example, Purolite A530E and A532E resins have been used to remove technetium-99; however, analysis has shown that A530E also has an affinity for radioiodine, cobalt, and untargeted groundwater constituent ions such as calcium, iron, silicon, and sulfate (Levitskaia et al. 2017). SIR-700 (ResinTech Inc.) is currently used in the P&T facilities in 100 Area and has been proposed for use in 200W for the removal of Cr (Saslow et al. 2023a). Cyanide, which is not currently a contaminant of interest at the 200W P&T facility, may interfere with the uptake of other anionic contaminants or may become a contaminant of interest in the future, which would require an understanding of the chemistry and mobility of the cyanide species and the interactions with the IX resins. Dowex 21K, which is used on the Hanford Site to treat uranium, has been shown previously to uptake cyanide complexes, notably ferrocyanide (Campbell et al. 2018).

1.3 Cyanide – Distribution, Behavior, and Treatment Technologies

Cyanide is found in Hanford Site groundwater, primarily because of previous discharges and unplanned releases of scavenging waste. The Hanford Site Groundwater Monitoring Report for 2023 (DOE 2023) recounts the current distribution of cyanide across two primary OUs: 200-BP-5 and 200-ZP-1. A total of 222 samples, including samples from three BP-5 extraction wells and 74 unconfined aquifer groundwater wells, were analyzed for total cyanide. Results showed that total cyanide was detected in 183 of the 222 samples (82%), with concentrations ranging from 1.71 to 1,120 µg/L. Notably, 28 samples from nine well locations exceeded the drinking water standard of 200 µg/L, with the highest concentration (1,120 µg/L) detected in well 299-E33-44. A 200-BP-5 OU plume map is shown in Figure 1.

Separately, free cyanide analysis was conducted on 185 samples, comprising three BP-5 extraction wells and 42 unconfined aquifer groundwater wells. Free cyanide was detected in 100 of the 185 samples analyzed (54%), with concentrations ranging from 1 to 10.3 µg/L. Sixteen samples from eight monitoring wells reported concentrations above the Model Toxics Control Act Method B threshold of 4.8 µg/L [(WAC 173-340-730 (Washington State Legislature 2023)], with the highest free cyanide concentration of 10.3 µg/L recorded in well 299-E33-47.

Additionally, 161 samples from four ZP-1 extraction wells, one proposed site well, and 39 unconfined aquifer groundwater wells were analyzed for total cyanide. Total cyanide was detected in 54 of the 161 samples (34%), with concentrations ranging from 1.76 to 545 µg/L. A 200-ZP-1 plume map is shown in Figure 1. Among these, 27 samples from just two monitoring wells exceeded the drinking water standard threshold of 200 µg/L, with the highest concentration (545 µg/L) collected from well 299-W14-18. For free cyanide, the same 161 samples were analyzed, revealing detection in 35 samples (22%) with concentrations ranging from 1 to 61.9 µg/L. Twelve samples from four monitoring wells exceeded the threshold of 4.8 µg/L, with the maximum concentration (61.9 µg/L) found in well 299-W10-26.

The environmental behavior of cyanide in groundwater systems is governed by speciation, complexation, and geochemical conditions such as pH and redox potential. Cyanide occurs in various forms, each of which exhibits different mobility and persistence (Figure 2).

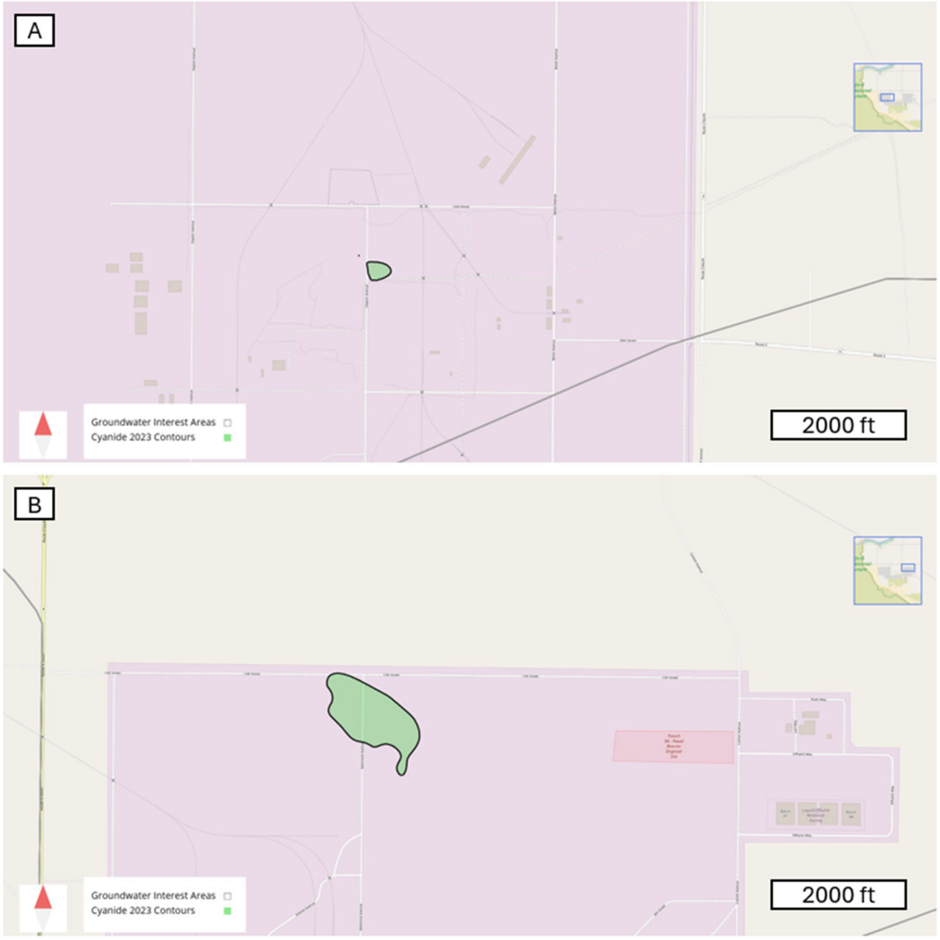


Figure 1. Map of (A) the total cyanide plume in the 200-ZP-1 and the (B) 200-BP-5 OUs. The green area indicates testing in excess of 200 mg/L.

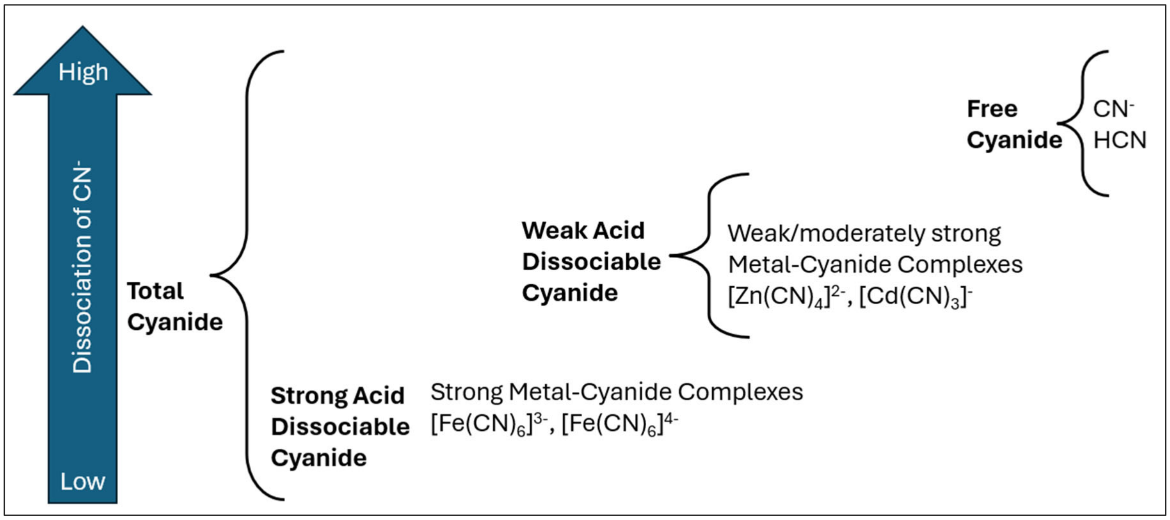


Figure 2. Diagram of cyanide species showing relative dissociation potential of free cyanide and the three primary groups of cyanide complexes: strong acid dissociable, weak acid dissociable, and free.

Free cyanide (top right portion of Figure 2) persists as either hydrogen cyanide (HCN) or the cyanide ion (CN^-) (Kjeldsen 1999). At a pH greater than 9.2, cyanide persists in its ionic form, while as protons become more abundant (i.e., having a lower pH), the equilibrium is driven further toward the generation of HCN because HCN is volatile and can evolve, as a gas, from solution. Hydrogen cyanide is known as a rapidly acting toxin and is considered a hazard (Hogod 1979). Comparatively, CN^- is highly mobile in aqueous solution, though it is generally considered unstable as it will complex, biodegrade, and undergo chemical oxidation.

Weakly complexed cyanide coordinates with transition metals such as copper, zinc, or cadmium. These complexes are thermodynamically stable; however, they are kinetically labile, meaning they will dissociate in mildly acidic conditions or in some cases when exposed to light (Kjeldsen 1999). These species can extend the life of a cyanide plume in groundwater systems as they periodically take up and release free cyanide, potentially acting as continuing sources under changing geochemical conditions. This distinction is not made using free and total cyanide testing carried out on the Hanford Site.

Strongly complexed cyanides such as the hexacyanoferrates, ferrocyanide $[Fe(CN)_6]^{4-}$, and ferricyanide $[Fe(CN)_6]^{3-}$ are highly stable cyanide complexes (Young and Jordan 1995). Both are stable in groundwater conditions, with ferrocyanide being more stable in reducing conditions and ferricyanide being more stable under oxic conditions. Ferricyanide, however, is also photolabile, indicating that sunlight may promote dissociation, but this of minimal concern in groundwater systems. Both hexacyanoferrates are mobile in groundwater; however, as they are negatively charged, they can sorb to positively charged mineral surfaces such as iron oxides or aluminosilicates, which can prolong the life of groundwater plumes. The hexacyanoferrates and other strongly complexed cyanide species are measured as total cyanide as they can only be measured as cyanide after analytically separating the CN^-.

Due to the complexities of cyanide speciation and subsurface geochemistry, it is important to identify which target cyanide compounds are present and which technologies are best suited for site-specific conditions (e.g., geochemistry, alkalinity). There are various technologies that can be used to treat cyanide in groundwater systems, which are highly dependent on cyanide speciation, concentration, and site-specific conditions. Free cyanide is readily treatable using chemical oxidation, air stripping, biological processes, or IX. Weakly complexed cyanides require stronger oxidants or a combination of approaches. The hexacyanoferrates are generally considered the most recalcitrant and require specialized oxidation, IX resins, or long-term sequestration strategies.

These strategies are summarized in Table 1. This table focuses on the treatment technologies as they relate to free cyanide and the previously discussed hexacyanoferrates. The ability of each technology to treat the contaminant of interest in ideal conditions is decided into four possible categories of effectiveness: high, moderate, low, and negligible. Generally, oxidative or reductive technologies are more effective for treating free cyanide, which is more reactive, as the hexacyanoferrates are more recalcitrant.

Selection of appropriate treatment technologies requires understanding the site specific hydrogeochemical conditions in which the plume persists, and the cleanup levels established for permissible residual cyanide in groundwater. As 200W P&T has proven to be successful in cleanup efforts using IX resins, this report focused primarily on these and similar IX resin technologies while considering several other sorbents that could easily be applied in the existing P&T system. Chemical and oxidative treatment technologies, while efficient and effective would require significant additional testing to determine the effects on other contaminants such as comingled radionuclides which make them less attractive treatment options.

Table 1. Summary of treatment options for cyanide in groundwater and their uses for free and weak cyanide and hexacyanoferrates.

Treatment Method	Free CN^-/HCN	Hexacyanoferrates	Notes	Reference
Air stripping	High	Negligible	Requires off-gas treatment	Yang et al. (2022)
Activated carbon	Low	Moderate	Best for polishing	Adams (1994)
Alkaline chlorination	High	Low–moderate	Widely applied, generates residuals	Dobson (1947)
H_2O_2/ozone/UV oxidation	High	Moderate (with UV/ozone)	Lower chemical residuals	Sarla et al. (2004)
SO_2/air	High	Low	Mining industry standard	Devuyst et al. (1989)
Biological treatment	High	Low	Inhibited at high concentrations	Alvillo-Rivera et al. (2021)
Constructed wetlands	Moderate	Negligible	Passive, low concentration polishing	Gessner et al. (2005)
Oxidants (e.g., persulfate, permanganate, ozone)	High	Low	Best for pump-and-treat or hotspot	Mudliar et al. (2009)
Ion exchange/P&T	Moderate	High	Effective with strong-base resin, widely used	Chu et al. (2020)

1.4 Knowledge Gaps and Research Aims

The challenges outlined in Section 1.3 begin to highlight some of the knowledge gaps that must be addressed before a treatment technology, such as IX resins and other sorbents of interest to this work, can be deployed for cyanide remediation on the Hanford Site. One of the most significant uncertainties concerns the selectivity of the treatment in natural groundwater systems. While IX resins may exhibit high affinity for cyanide complexes in clean laboratory solutions, the presence of competing anions such as nitrate, sulfate, chloride, and even bicarbonate can reduce capacity, along with the likelihood of fouling in industrial scale-up (Cyganowski and Dzimitrowicz 2020). The magnitude of this effect cannot be systematically quantified from other groundwater systems and must be tested for each composition. In addition to the natural anions in solution, contaminants that are anionic may also interfere with targeted remediation of cyanide. Conversely, cyanide complexes may interfere with the remediation of other anions, e.g., technetium-99 removal by A532E and uranium by Dowex 21K.

Understanding the cyanide speciation will also be fundamental for making the appropriate choices in remediation strategy and treatment technology. In groundwater, cyanide speciation can shift in response to changes in pH, redox potential, and even microbial activity. The potential effects of these shifts on resin/sorbent loading and capacity are unknown. Additionally, pilot/field-scale validation would be required to determine if treating cyanide via P&T using IX resin would be necessary prior to application. The 200W P&T facility has shown high performance in treating groundwater for other contaminants despite changing hydraulic conductivity, fluctuating groundwater chemistry, and variable flow regimes; however, the effect of cyanide complexes in this system is poorly understood.

To evaluate the potential application of IX resin and other adsorbents for cyanide treatment at the Hanford Site, this study aimed to determine which commercially available IX resins/sorbents are likely to take up the most cyanide under batch conditions (high liquid to solid ratio) using synthetic groundwater (SGW) of a similar composition to that processed at the 200W P&T facility. While this work did not include the additional contaminants (Tc, U, or Cr), it did include comingled common groundwater anions and nitrate. Two different concentrations of ferrocyanide were used to determine the extent of uptake by each treatment technology, identify candidates for additional testing based on highest cyanide loading/uptake, and determine whether nitrate and/or other anions would interfere with total cyanide uptake.

2.0 Methods

This initial study focused on batch tests to systemically evaluate the behavior of ferrocyanide in SGW comingled with nitrate when treated with IX resins or other commonly used adsorbents. This study, as part of a phased approach, began with batch testing that will later advance to one dimensional (1-D) column testing. This methodology is designed to incrementally build understanding, moving from a more controlled, more simplified system to a more complex and dynamic system that will better simulate field environments.

Batch testing serves as the foundational step providing critical insights into equilibrium conditions, reaction kinetics, and sorption capacities under well-mixed, static conditions. The controlled nature of batch systems allows additional testing in shorter periods than more complex and dynamic testing, which is often accomplished by column tests. Once this foundational information is well understood by adding flow dynamics, other important data can be collected, such as breakthrough, mass transfer limitations, and how the reaction kinetics change under flow conditions.

2.1 Treatment Preparation

2.1.1 Materials

Eight IX resins were evaluated in this study along with bismuth subnitrate (BSN), alumina, and granular activated carbon (GAC) sorbents. Table 2 summarizes the characteristics of these materials. Six of the resins were specifically selected for their potential to remove cyanide complexes based on manufacturer -reported use cases. Two of the resins, Dowex 21K and A532E, were also tested since these resins are currently used in the 200W P&T facility. Materials were tested at high and low concentrations of ferrocyanide.

Table 2. Summary of characteristics of the tested materials.

Material	Manufacturer	Type	Functional Group(s)	Structure	Manufacturer Reported Exchange Capacity (mEq/mL)
SBG 1	Resin Tech	Type 1 Strong Base	Trimethylamine	Gel	1.4
SBG 2	Resin Tech	Type II Strong Base	Dimethylethanolamine	Gel	1.4
Amberchrom 1X8	DuPont	Type 1 Strong Base	Trimethylammonium	Gel	1.2
Amberlite IRA-9000	DuPont	Type 1 Strong Base	Trimethylammonium	Macroreticular	1
Purolite A830	Purolite	Weak Base	Complex Amine	Macroporous	2.7
Purolite A500	Purolite	Type 1 Strong Base	Type I Quaternary Ammonium	Macroporous	1.1
Dowex 21K	DuPont	Type 1 Strong Base	Trimethylammonium	Gel	1.4
A532E	Purolite	Type 1 Strong Base	Trihexylamine, Triethylamine	Gel	0.7
Activated charcoal, granular	Sigma-Aldrich	N/A	N/A	Granular	N/A
Bismuth Subnitrate	Sigma-Aldrich	N/A	N/A	Powder	N/A
Alumina	Sigma-Aldrich	N/A	N/A	Powder	N/A

Three solid phase amendments were chosen for analysis as well. GAC (i.e., charcoal) was selected for both batch tests, while the potential for BSN and alumina was evaluated in the high concentration batch test only. The granular activated charcoal was purchased at ≤ 5 mm size and size-reduced to 20 mesh

(0.841 mm). Bismuth subnitrate, also called bismuth oxynitrate, has been examined for remediation of other contaminants previously and its structure has been shown to incorporate different contaminant oxyanions (Boglaienko et al. 2024). Alumina and GAC have also been widely used as remedial amendments at other sites due to their high sorption capacities (Jjagwe et al. 2021; Mahesh et al. 2023).

2.1.2 Chloride Loading IX Resin

To guarantee that all resins were loaded the same and that each free site was occupied, each IX resin was loaded (approximately 7.42 mL equivalent volume) into separate low-pressure columns (BioRad, 1.5 cm × 5 cm, cross-sectional area 1.77 cm^2), with a 5% NaCl solution pumped through the packed columns using a Kloehn syringe pump for 1 hour. This corresponds to 600 g of NaCl per liter of resin. Then, ultrapure water (EMD Millipore double deionized, 18.2 MΩ·cm) was passed through the columns to rinse excess NaCl from the resins until the effluent Cl^- concentration had a measured conductivity ≤50 µS/cm using an Orion VersaStar Pro meter with an Orion 013010MD conductivity probe (corresponding to less than 5 mg/L NaCl). The resins were removed from the columns and stored in high-density polyethylene (HDPE) bottles until used.

2.1.3 Moisture Content

The moisture content of each material (as received and after chloride loading, if loaded) was measured using a modified EPA Method 1314 (EPA 2017). Approximately 1 g of each resin was added to a pre-weighed aluminum tray with a lid (in triplicate) and placed into a preheated oven at 105 °C overnight. The following day, the mass of the tray with the lid and resin was recorded. The trays were returned to the oven for further drying until the dry mass of the resin was within 0.5 % of the previous mass measurement. Moisture content was used to determine the dry weight of chloride-loaded resin.

2.1.4 Solution Preparation

SGW was used as the background solution in batch tests. The target solution pH for all solutions used was 6.8 and was achieved by incrementally adding HCl, or NaOH, if necessary. The SGW was prepared based on representative groundwater conditions for 200W, previously described (Lawter et al. 2021). To evaluate the effect of competitive nitrate (NO_3^-), SGW batches were prepared with NO_3^- (SGW + NO_3^-). The SGW consisted of 0.133 g/L $NaHCO_3$, 0.012 g/L $KHCO_3$, 0.044 g/L $MgSO_4 \cdot 7H_2O$, 0.052 g/L $MgCl_2 \cdot 6H_2O$, and 0.157 g/L $CaCl_2$ added in the order listed to double-deionized water. The solution was agitated until all solids were dissolved. It was amended with approximately 0.118 g/L (1.9 mM) NO_3^- added as $NaNO_3$ after the other SGW chemicals.

2.2 Batch Testing

All batch tests were conducted with SGW+NO_3^- in duplicate at room temperature and a solution:solid ratio of 200 mL:1 g dry resin. A total of 100 mL of the desired solution was added to 125 mL HDPE sample bottles, followed by the required dry resin mass (0.50 g). Two rounds of batch testing were conducted, one with a lower concentration of potassium ferrocyanide (1.5 g/L) and one at a higher concentration (10.5 g/L) of potassium ferrocyanide, which was prepared prior to addition of the resin. The lower concentration of ferrocyanide was relevant to the capacity reported by the manufacturers (Table 2), while maintaining some relevance of the highest reported concentrations of total cyanide across the site. The higher concentration is far more than any anticipated concentration but was prepared to exaggerate discernable differences between the resins. The addition of alumina and BSN in the high concentration testing efforts necessitated the fabrication of a second batch of SGW+NO_3^-. Sample bottles were placed on a table shaker set to 125 rpm for 72 hours. At the end of the batch test, the final pH was measured, and an aliquot of solutions was collected for analysis. The remaining materials were allowed to dry in their bottles with the lid loose under ambient conditions in a fume hood. The pH of each sample was tested before and after sampling using a Thermo Orion STAR A211 benchtop pH meter with a Thermo Orion 9107BNMD epoxy pH triode.

2.3 Analytical Chemistry

2.3.1 Inductively Coupled Plasma – Optical Emission Spectroscopy

Metals in solution were analyzed by inductively coupled plasma – optical emission spectroscopy (ICP-OES) following EPA Method 6010D and standard quality assurance/quality control (QA/QC) protocols, including internal standards, duplicate samples, blanks, and certified reference material. A PerkinElmer Optima 8300 dual view with a PerkinElmer S-10 auto-sampler interface was used for ICP-OES measurements. Iron measurements were used as a proxy for ferrocyanide in solution. Due to the high binding strength of the ferrocyanide complex, all iron in the samples is assumed to be associated with the $[Fe(CN)_6]^{4-}$ polyatomic anion.

2.3.2 Ion Chromatography

Anion concentrations were analyzed following EPA Method 300.0 using standard QA/QC protocols, including internal standards, duplicate samples, blanks, and certified reference material. Analysis was performed using a Dionex Reagent Free Ion Chromatography System 5000 (RFICS-5000) with an AS-AP auto-sampler. The RFICS-5000 instrument was calibrated using a multi-component anion solution made by Inorganic Ventures.

2.3.3 Total Cyanide Analysis

Batch samples were shaken at each termination point, allowed to settle, and decanted into tubes. Samples were filtered through a 0.2-micron syringe filter (25 mm Acrodisc). Samples were preserved by adjusting the sample pH to 12 using 6 N NaOH and stored at 4 °C in the dark until the samples were shipped to GEL Laboratories, LLC for analysis. Sample analysis of total cyanide followed SW-846 Test Method 9010C, *Total and Amenable Cyanide: Distillation*, and SW-846 Test Method 9012B, *Total and Amenable Cyanide (Automated Colorimetric, With Off-Line Distillation)* / (SW-846 9010C/9012B) (EPA 2017).

3.0 Results and Discussion

Two different batch tests were conducted, each using the same nitrate-doped SGW. Results and discussion below are presented for pH changes after 72 hours and analytical chemistry, including anion and hexacyanoferrate chemistry. The data are separated by the concentration of ferrocyanide added (i.e., low and high concentrations).

3.1 pH Changes

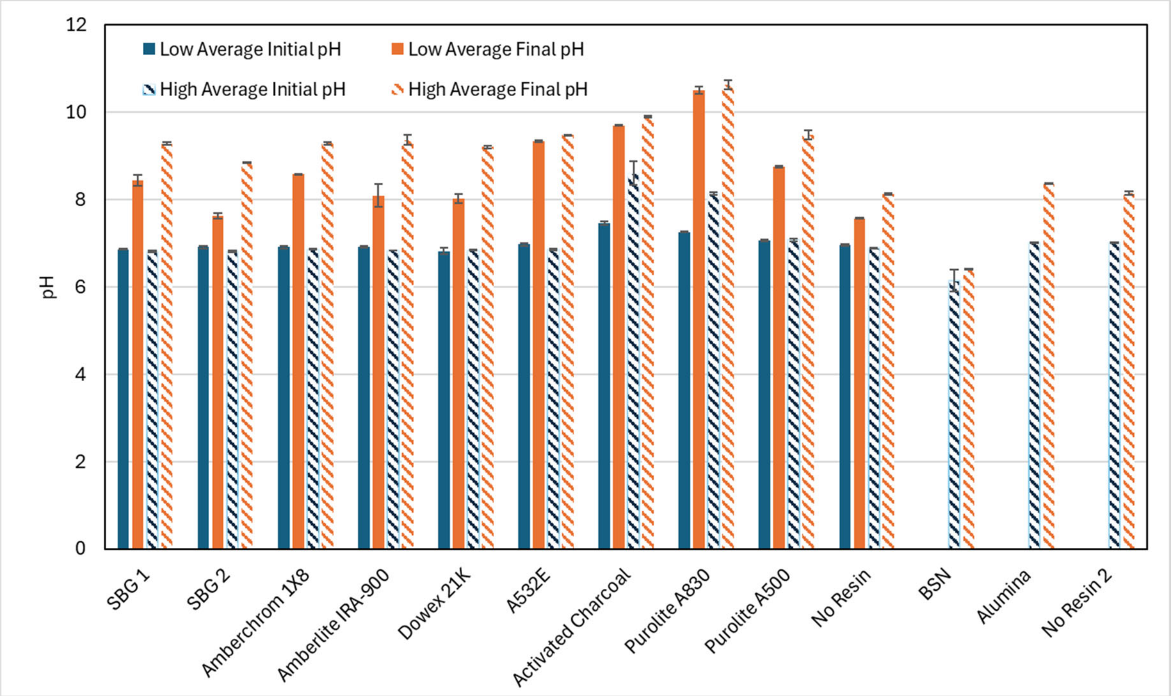
In all tests, both with the low and high concentrations of ferrocyanide in SGW with added nitrate, the pH of samples increased after incubating for 72 hours. In the low-concentration batch tests, summarized in Table 3, the greatest increase after 72 hours (37%) was in the sample with Purolite A830 resin. The next highest notable changes were the A532E and activated charcoal, which increased by 29% and 26%, respectively. Note that the SGW with nitrate and ferrocyanide increased by 8% over time as well, with the lowest change with the addition of resin being 10% for the SBG1.

Table 3. Low-concentration (1.5 g/L) ferrocyanide sample pH changes after 72 hours.

Sample	Average Initial pH[(a)]	Average Final pH[(a)]	Percent Difference after 72 Hours (%)
SBG 1	6.86	8.44	21
SBG 2	6.91	7.63	10
Amberchrom 1X8	6.91	8.59	22
Amberlite IRA-900	6.92	8.09	16
Dowex 21K	6.82	8.02	16
A532E	6.98	9.34	29
Activated charcoal	7.46	9.70	26
Purolite A830	7.26	10.51	37
Purolite A500	7.06	8.75	21
No resin	6.96	7.57	8

(a) Unless otherwise stated all error between measurements is less than 0.1 units.

In the high-concentration batch tests, summarized in Table 4, the greatest increase after 72 hours (32%) was A532E. However, many samples had higher increases after the 72 hours. SBG 1 and Amberlite IRA-900 increased 31%, Amberchrom 1X8 increased 30%, and Purolite A500 and Dowex 21K increased 29%.



shows both sets of values as bar graphs with error bars of the individual averages. Samples including the controls (i.e., no resin samples), which are only SGW amended with nitrate and ferrocyanide, show increases in pH over time as well. Sample 'no resin 2' serves as the control BSN and alumina as a second batch of SGW+NO_3 was required for those samples.

Table 4. High concentration (10.5 g/L) ferrocyanide sample pH changes after 72 hours.[(a)]

Sample	Average Initial pH	Average Final pH	Difference (%)
SBG 1	6.82	9.29	31
SBG 2	6.81	8.85	26
Amberchrom 1X8	6.86	9.29	30
Amberlite IRA-900	6.82	9.36	31
Dowex 21K	6.84	9.20	29
A532E	6.85	9.48	32
Activated Carbon	8.61	9.90	14
Purolite A830	8.14	10.62	26
Purolite A500	7.08	9.48	29
BSN	6.14	6.41	4
Alumina	7.01	8.37	18
No Resin	6.89	8.13	16
No Resin 2	7.01	8.15	15

(a) Unless otherwise stated, all error between measurements is less than 0.1 units.

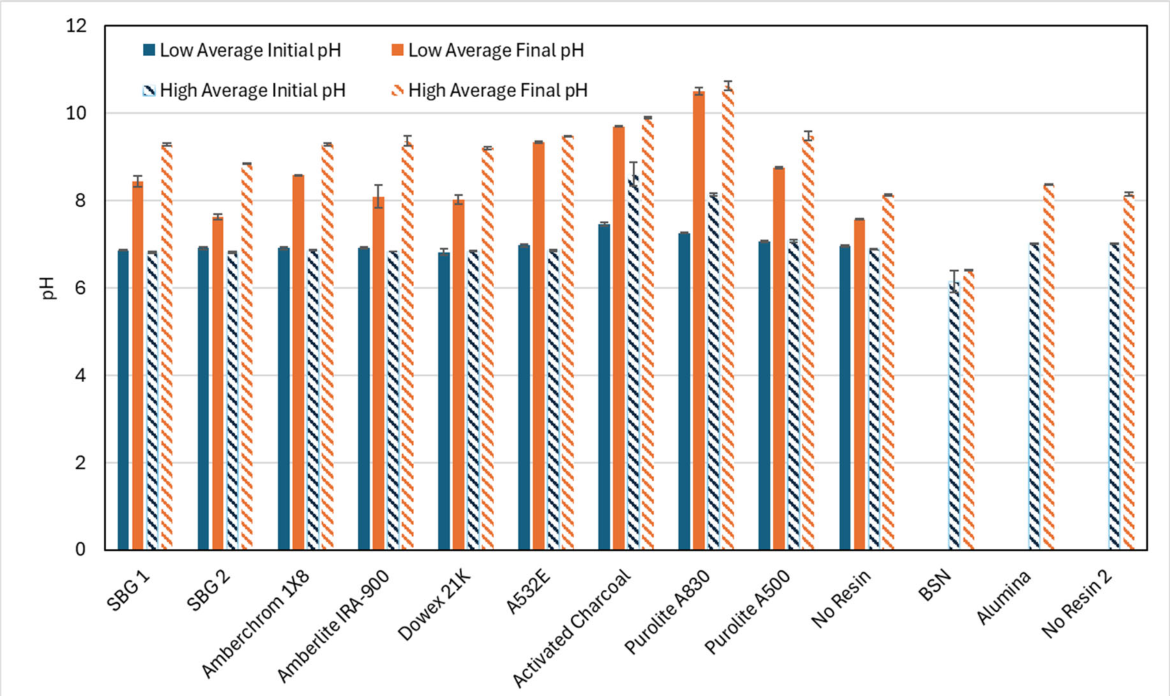


Figure 3. Comparison of the initial and final pH levels of low and high concentrations of ferrocyanide after 72 hours. All bars show error of the standard deviation of duplicate samples.The pH of each set of batch experiments increased after a 72-hours incubation period, while the initial starting pH levels for most tests were consistent within ± 0.2 around the projected pH of the SGW, 6.8. The exception to this was seen with BSN, activated charcoal, and Purolite A830. For BSN, this is consistent with previous studies (Boglaienko et al. 2024; Christensen and Lebech 2012). Activated charcoal often has residual alkalis (e.g., NaOH, KOH) from production, which increases pH as well but would likely be resolved quickly in a system with flow. This marked decrease in pH is consistent with the known hydrolysis behavior of BSN. BSN is not fully hydrolyzed during the synthesis process, which leads to additional hydrolysis (Christensen and Lebech 2012). The hydrolysis of the bismuth nitrate into bismuth subnitrate also accounts for the increase in the nitrate seen in the anion results for the BSN samples (**Error! Reference source not found.**).

Anion exchange resins typically increase the pH of groundwater because strong base anion resins exchange anions for hydroxide (OH^-) ions, which increase the pH. However, these reins were chloride-loaded prior to the batch tests, and while there may be a small amount of residual OH^-, this would unlikely account for the pH increase alone. Similarly, many of the functional groups used on the resins are alkaline when in solution, but this should have limited, if any, effect on the batch tests.

One potential explanation is that the ferrocyanide is a Brønsted-Lowry base. A Brønsted-Lowry base is defined as a chemical substance that accepts a proton (H^+) from another substance. In an acid-base reaction, a Brønsted-Lowry base receives this proton from a Brønsted-Lowry acid, forming a conjugate acid (Gillard 2003). The ferrocyanide ion $[Fe(CN)_6]^{4-}$ consists of a central Fe^{2+} ion coordinated to six cyanide ions (CN^-) (Almeida et al. 2003). The CN^- are coordinated through their lone pairs of electrons on the carbon atom, creating a complex with an overall negative charge of -4, yielding a potential of 4 OH^- for each ferrocyanide anion, as described in Eq. (1).

$$[Fe(CN)6]^{4-} + H_2O \rightarrow H_n[Fe(CN)_6]^{(4-n)} + nOH^- \quad (1)$$

While the ferrocyanide is complexed, the CN^- can still act as a Brønsted-Lowry base by donating a pair of electrons to accept a proton from water, leading to the formation of hydroxide ions. This results in an increase in the pH as the solution becomes more basic due to the release of hydroxide ions. This would suggest that while the ferrocyanide is exchanged onto the resin surface, that it takes some of the protons from the water with it to charge balance, which would account for the increase in pH. In batch experiments, the pH is more susceptible to fluctuation and drift because the system is closed, and a chemical reaction occurs in a single vessel over time. By contrast, flowing systems (e.g., 1-D columns) can maintain consistent pH levels by continuously introducing fresh reagents.

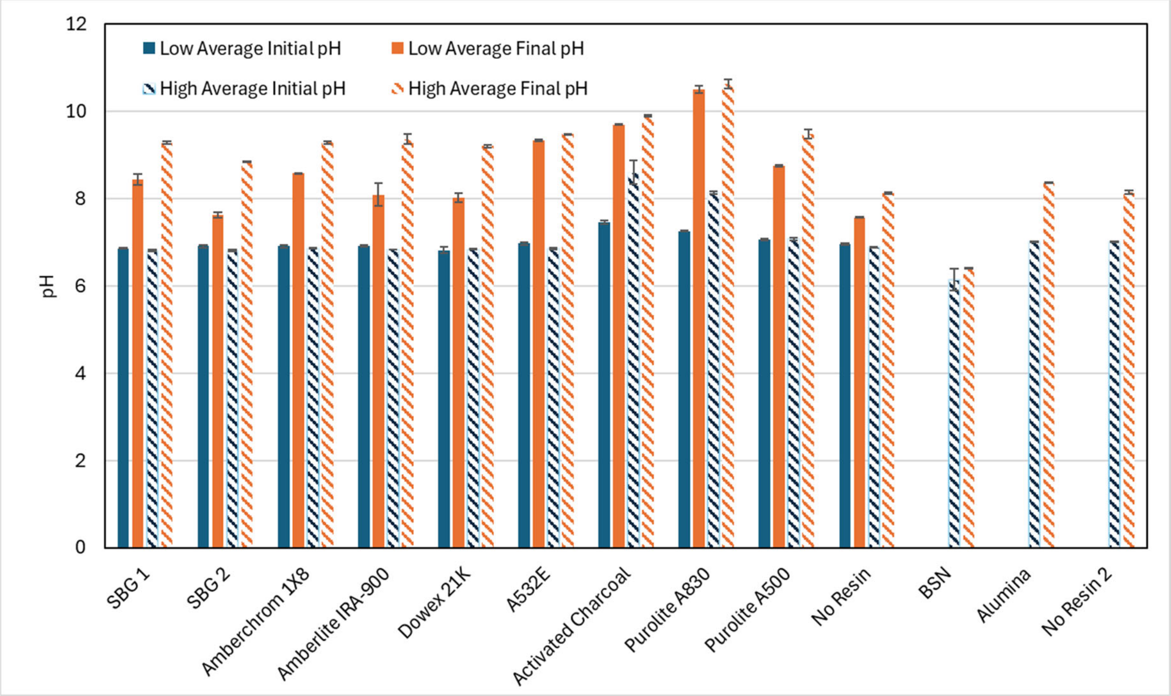


Figure 3. Comparison of the initial and final pH levels of low and high concentrations of ferrocyanide after 72 hours. All bars show error of the standard deviation of duplicate samples.

3.2 Low Concentration Ferrocyanide Batch Test

Anion data is presented in Figure 4. As shown in the figure, bromide concentrations are residual from impurities in the salts used for preparation of the SGW and are only shown for completeness, with a total concentration never exceeding 0.2 ppm. Phosphate is similarly low for all samples, staying at 0.5 to 1.25 ppm for all samples except the activated charcoal, which may have released a small amount of phosphate into solution. Chloride concentrations are similarly shown for completeness due to the resins having been chloride-loaded. The chloride concentrations for all resins except Purolite A830 exceed 400 ppm, while the activated charcoal, no resin control, and SGW blank are all at approximately 100 ppm. Curiously, the Purolite A830 was only slightly higher than the SGW blank. Sulfate and nitrate both show competitive uptake by the resins. For sulfate, compared to the SGW and no resin controls, Amberlite IRA-900 and Dowex 21K showed similar uptakes, reducing the concentration of sulfate in solution by about half (i.e., ~ 15 ppm). Purolite A500 reduced the concentration of sulfate by ~ 10 ppm. Nitrate update followed trends similar to those for Purolite A500 and Amberlite IRA-900, removing the most (~75 ppm), while SBG1, Dowex 21K, and A532E removed ~ 70 ppm, 70 ppm, and 55 ppm, respectively.

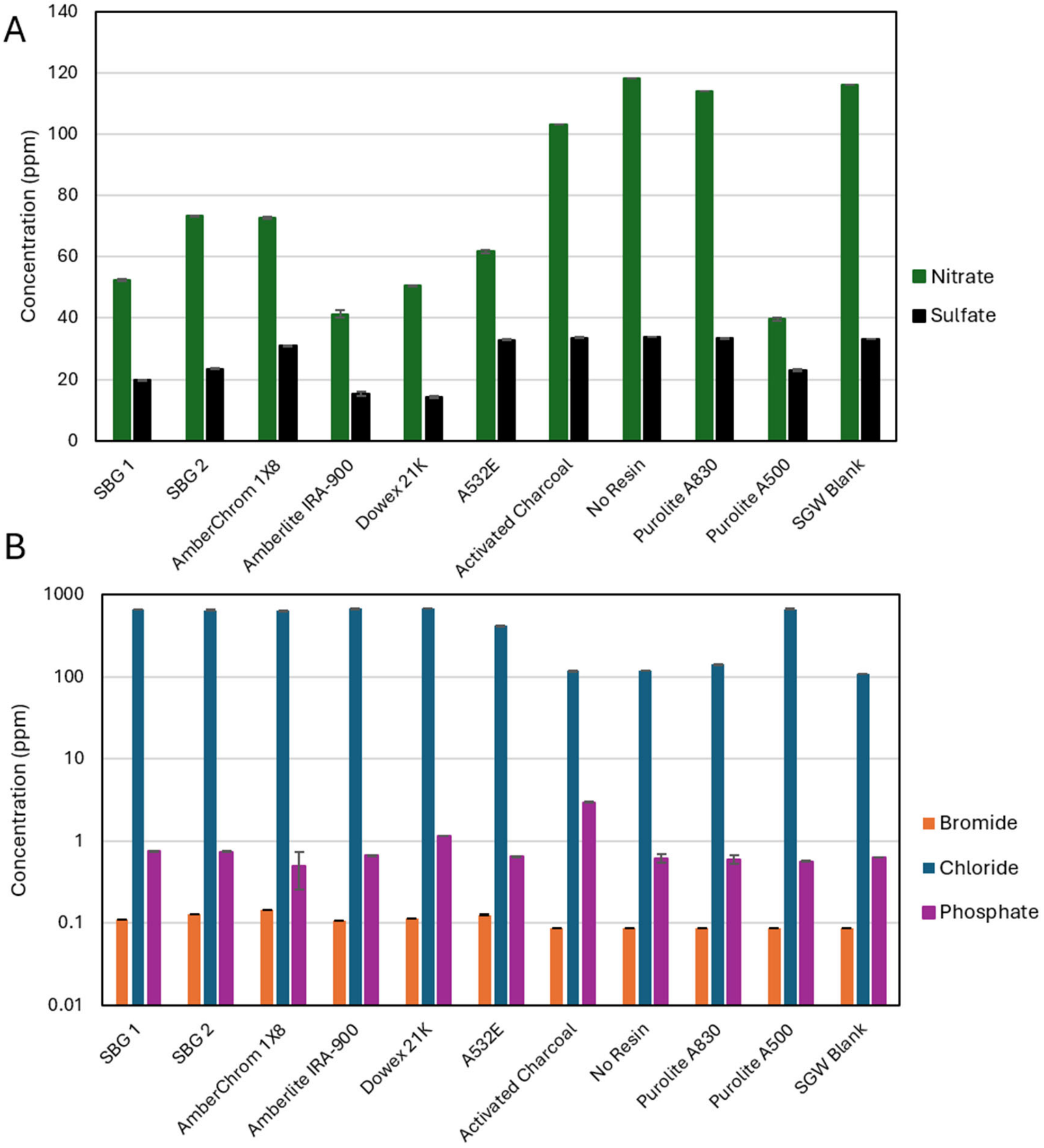


Figure 4. Anion concentrations for (A) nitrate and sulfate and (B) bromide, chloride, and phosphate in SGW with nitrate and potassium ferrocyanide (1.5 g/L) in solution after 72 hours of reaction time with various amendments. Figure B is reported on a logarithmic scale. Error bars show standard deviation of duplicate samples.

Iron concentrations used as a proxy for the ferrocyanide polyatomic anion (Figure 5) showed that overall, at the lower concentration of 1.5 g/L potassium ferrocyanide in solution, almost all treatment options performed well at ferrocyanide removal. SBG1 and SBG2, Amberchrom 1X8, Amberlite IRA-900, Dowex 21K, and Purolite A500 had no measurable iron in solution. A532E showed an uptake of 55%. Of the IX resins, Purolite A830 showed the least uptake, only removing 18%. The granular activated charcoal demonstrated negligible uptake.

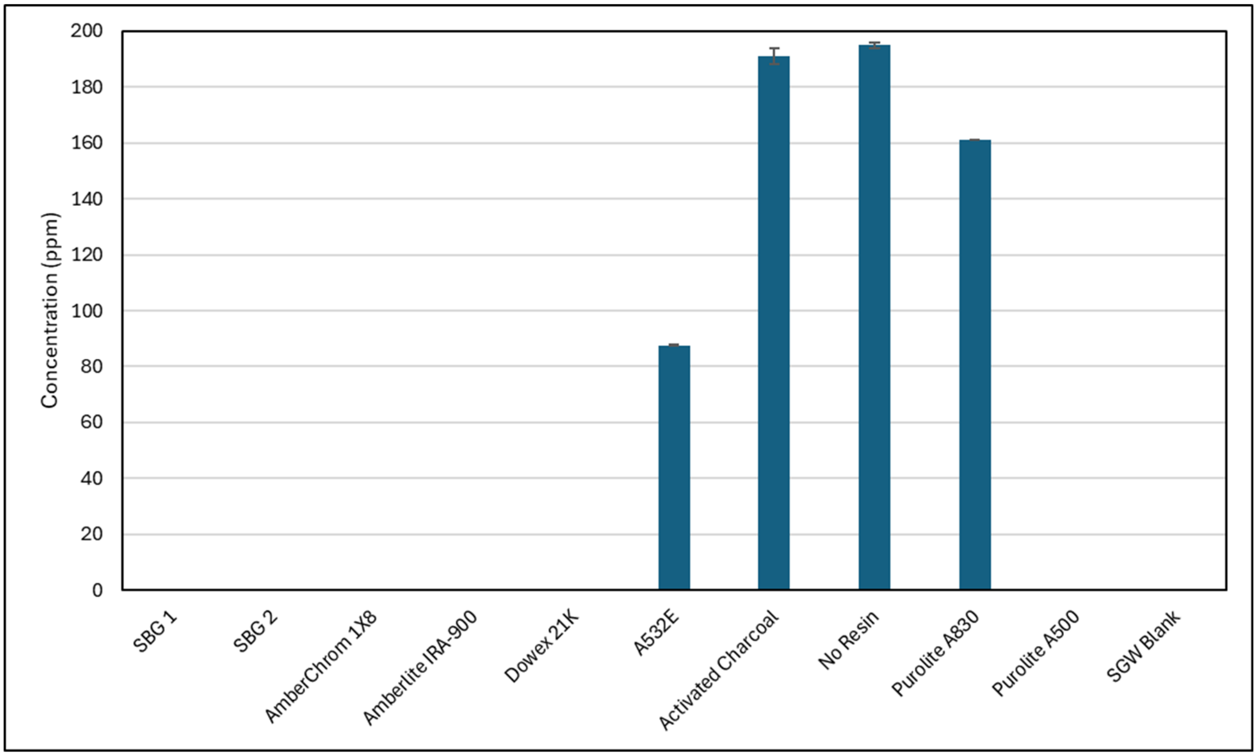


Figure 5. Iron concentration in SGW with nitrate and potassium ferrocyanide in solution after 3 days of reaction time with various amendments.

The low-concentration batch tests follow primarily the predicted behaviors of anion exchange resins. SBG 1 and 2, Amberchrom 1X8, Amberlite IRA-900, Dowex 21K, and Purolite A500 removed all the ferrocyanide from solution after 72 hours. This is consistent with being an anion exchange resin that operates by removing negatively charged contaminants from solution by exchanging their fixed anions (e.g., chloride) for targeted anions through electrostatic attraction. Interestingly, many of these also took up some of the nitrate in solution, meaning that there was remaining capacity available in the low-concentration tests, which was used for the uptake of additional anions from SGW. A532E did not take up all of the ferrocyanide from solution but did remove more than 50% of the available nitrate, suggesting that the removal of nitrate may be more favorable for this particular resin. The granular activated charcoal did not perform well for any anion uptake, possibly because it was used as provided by the manufacturer while most activated charcoal is used at a finer mesh than what was purchased or size reduced. (Randtke and Snoeyink 1983). Activated charcoal has been shown in the literature to adsorb various anions (Azam et al. 2022); however, a smaller mesh size increases the availability of sorption sites; thus, any future investigations may need to use a smaller size.

This test was unable to benchmark performance for most of the tested resins as it was not capable of differentiating between many of them, so an additional test was performed at a higher concentration to force the distinction between their performance.

3.3 High Concentration Ferrocyanide Batch Test

Anion data is presented in Figure 6. As shown in the figure, bromide concentrations are residual from impurities in the salts used for preparation of the SGW and are only shown for completeness, with a total concentration never exceeding higher than 1 ppm. Phosphate is similarly low, remaining below 0.5 ppm for all samples except the activated charcoal, which may have released a small amount of phosphate into solution as also observed in the low concentration batch test. Chloride concentrations are shown for completeness because the resins had been chloride loaded. The chloride concentrations for all resins, except Purolite A830, exceed 400 ppm, while the activated charcoal, no resin control, SGW blanks, BSN, and alumina are all at approximately 100 ppm. The Purolite A830 chloride concentration was only slightly higher than the blank. This matches the trends from the low ferrocyanide concentration test. Sulfate and nitrate both saw very little uptake. The only uptake for sulfate is the BSN, which removed 55% of the sulfate from solution. The only resin that showed some notable uptake of nitrate in the presence of 10.5 g/L ferrocyanide was A532E, which removed 33% of the nitrate. The BSN released nitrate into solution, increasing the concentration to 751 ppm.

Iron concentrations were used as a proxy for the ferrocyanide polyatomic anion (Figure 7) showed that overall, at the higher concentration of 10.5 g/L none of the materials were able to take up all the ferrocyanide. This test was designed to ensure that the amendments would become completely saturated, which is shown by the lack of any treatment option removing all iron from solution. SBG1 and 2, Amberchrom 1X8, Amberlite IRA-900, Dowex 21K, and Purolite A500 removed 20%, 16%, 15%, 17%, 19%, and 18% respectively. The A532E showed an uptake of 8%. Of the IX resins, Purolite A830 showed the least uptake, 3%. The two solid phase non-polymeric substrates BSN and alumina removed 5% and 10%, respectively. The granular activated charcoal demonstrated negligible uptake.

Total cyanide measurements (Figure 8) suggest that all the resins have taken up approximately 40% of the ferrocyanide in solution. The Purolite A500 removed the most total cyanide from solution and has the smallest error bars of the duplicate samples measured. Note that the second SGW blank and the second resin control are for comparison to the BSN and alumina. The BSN and alumina both show minimal uptake, but higher-than-expected results, as does the second no resin control for comparison. Based on the consistency of the iron data, we suspect that there is an error unaccounted for in these sample sets; however, other data confirms that both BSN and alumina are of limited use in the tested system.

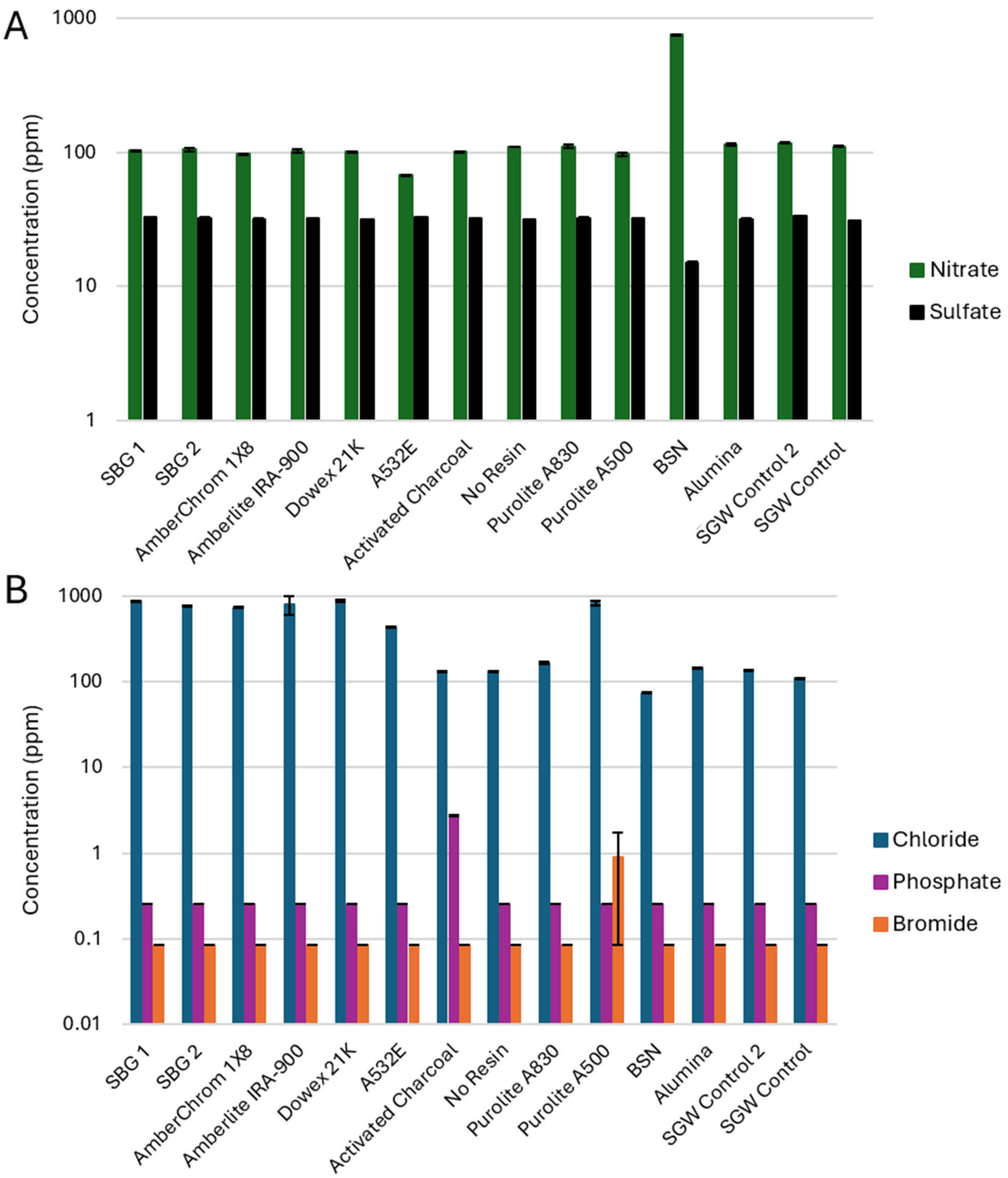


Figure 6. Anion concentrations for (A) nitrate and sulfate and (B) bromide, chloride, and phosphate in SGW with nitrate and potassium ferrocyanide (10.5 g/L) in solution after 3 days of reaction time with various amendments. Both graphs are shown on a log scale for clarity. Error bars are shown for duplicate samples. The phosphate data for the A500 was measured as a single replicate.

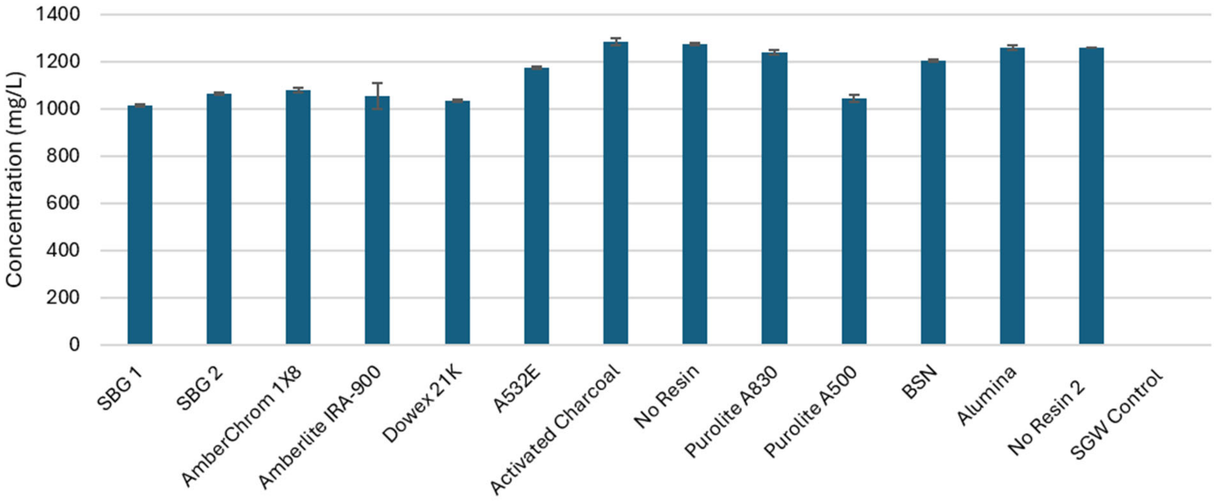


Figure 7. Iron concentration in SGW with nitrate and potassium ferrocyanide (10.5 g/L) in solution after 3 days of reaction time with various amendments. No Resin 2 is the second batch of SGW, which has been doped with the ferrocyanide. Error bars show standard deviation of duplicate measurements.

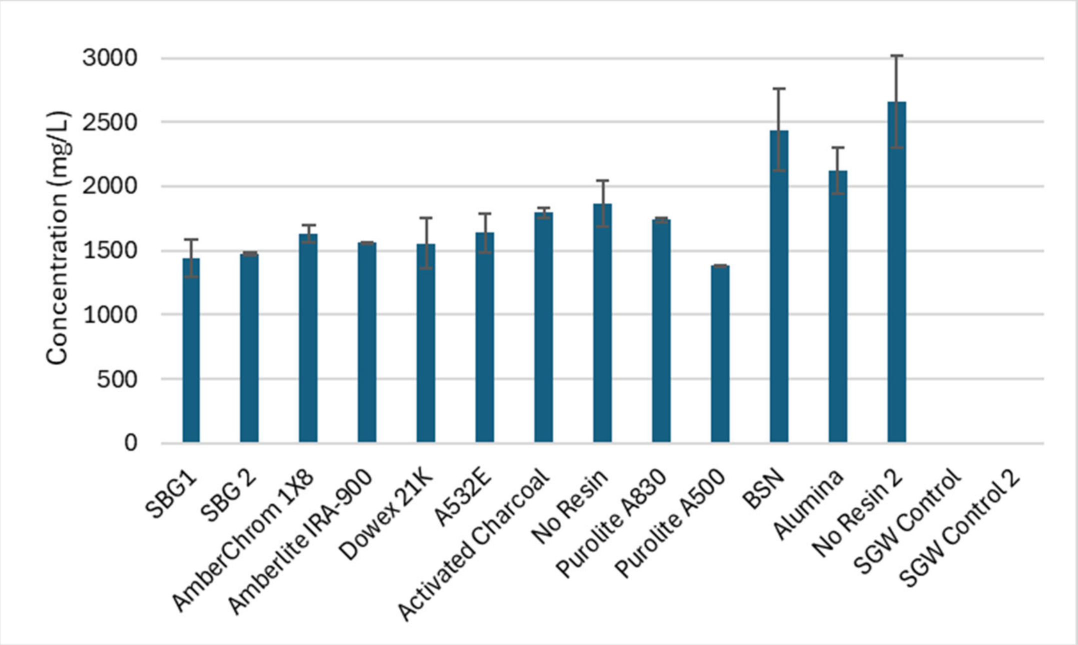


Figure 8. Total cyanide concentration in SGW with nitrate and potassium ferrocyanide (10.5 g/L) in solution after 3 days of reaction time with various amendments.

The higher concentration batch tests revealed that the Purolite A500 removed the most ferrocyanide in a saturated batch system after 72 hours. However, SBG 1 and 2, Amberchrom 1X8, Amberlite IRA-900, and Dowex 21K performed nearly as well as Purolite A500. This grouping of top-performing resins is reflected in both the total iron measurement via ICP-OES and the total cyanide analysis. While both methods are valuable for determining the total cyanide in solution, due to the high levels, the total cyanide analysis required considerable dilution (i.e., 5000x), which increases the analytical error considerably, while ICP-OES analysis required less dilution and is more suited for measurements of high concentrations. The remaining amendments performed noticeably worse for cyanide removal from this SGW, which included the solid absorbents, GAC, BSN, and alumina, and the Purolite A830 and A532E IX resins. Of this second-tier performing group, the A532E performed marginally better than the other treatment technologies.

Table 5. Summary of removal of nitrate, sulfate, iron, and total cyanide for all tested technologies and both low (1.5 g/L) and high (10.5 g/L) ferrocyanide concentrations.

Material	Removal Nitrate (%)	Removal Sulfate (%)	Removal Iron (%)	Removal Total Cyanide (%)	Ferrocyanide Concentration
SBG 1	56	41	100	N/A	Low
SBG 2	38	30	100	N/A	Low
AmberChrom 1X8	38	8	100	N/A	Low
Amberlite IRA-900	65	55	100	N/A	Low
Dowex 21K	57	58	100	N/A	Low
A532E	48	3	55	N/A	Low
Activated Charcoal	13	0	2	N/A	Low
Purolite A830	3	1	17	N/A	Low
Purolite A500	66	32	100	N/A	Low
SBG1	8	-4	18	23	High
SBG 2	5	-1	14	21	High
AmberChrom 1X8	12	0	13	13	High
Amberlite IRA-900	8	-1	15	16	High
Dowex 21K	9	1	17	17	High
A532E	39	-3	5	12	High
Activated Charcoal	9	0	-4	4	High
Purolite A830	0	-2	0	7	High
Purolite A500	13	-1	16	26	High
BSN	-539	55	4	8	High
Alumina	2	5	0	20	High

Table 5 presents a summary of removal of the primary constituents of interest, nitrate, sulfate, iron, and total cyanide. Error is not reported in this table but is reported in figures for each technology and reported concentrations. When comparing the performance of the two groups of treatment technologies, it is immediately apparent that the top-performing technologies were all SBA IX resins, either Type 1 or Type II, with a single functional group. The outlier to this observation is Purolite A532E, which is also an SBA resin, but contains two functional groups and did not perform as well as the other SBA resins assigned to the top-performing group of candidate technologies.

The two functional groups on the A532E resin include a triethylammonium group, which is responsible for the rapid uptake of NO_3^-, and the trihexylammonium group, which is engineered to remove weakly hydrated anions like pertechnetate (Saslow et al. 2023b). With a fraction of the total exchange capacity for this resin designed to remove NO_3^-, cyanide may not be as competitive at the trihexylammonium sites. This is supported by the persistent uptake of NO_3^- even at elevated cyanide concentrations. It is also important to note that for the poorest performing technologies for cyanide removal, there is less tunability in the materials' performance, whereas for the absorbents, one is limited to the secondary functionalization step to improve uptake or adjusting the particle size/available sorption site surface coverage to increase cyanide uptake. Lastly, in the case of BSN, there is less certainty about what will form given its mineralogical transformation's sensitivity to groundwater chemistry (Pearce et al. 2025).

The nitrate data is also fundamentally important for consideration of the high-concentration batch tests. In all samples, limited nitrate was removed from solution except for a small amount of removal by the A532E. This suggests that the ferrocyanide ion is more competitive than nitrate for all of the resins except A532E. While the concentration of ferrocyanide is also considerable in these tests, A532E was still able to take up a small amount, and previous research suggests that nitrate is competitive for resins in use at the 200 W P&T facility (Saslow et al. 2023b; Saslow et al. 2023c). This suggests that should ferrocyanide enter the system, it may influence the order of selectivity for anion removal.

4.0 Conclusions – Batch Testing

The findings from this study highlight the importance of site-specific testing when selecting treatment technologies to remove ferrocyanide from groundwater in a P&T facility. Overall, the Purolite A500 was the best performing tested material, with SBG1 and Dowex 21K also showing considerable uptake at both concentrations. (Table 5). At lower concentrations of ferrocyanide, almost all IX resins removed all ferrocyanide and removed anions from solution. This may cause complications for radionuclides, which are transported as anions (e.g., TcO_4^-) as well as constituent anions of the groundwater. Additionally, Dowex 21K showed considerable uptake of ferrocyanide along with comingled nitrate, while A532E showed moderate uptake, which suggests that ferrocyanide, if in concentrations higher than the incidental concentrations studied previously, could be taken up at the 200W P&T facility if it was in the influent water, impacting efficiency.

Further testing of 1-D flow columns of Dowex 21K and A532E along with Purolite A500 will help close some of the remaining knowledge gaps with respect to cyanide treatment and impacts to the 200W P&T facility. Specifically, while small amounts of ferrocyanide would likely only be incidentally removed in the presence of the target contaminants technetium and uranium, if a significant increase in the groundwater influent cyanide concentration passes through the A532E and/or Dowex 21K treatment vessels, competition between cyanide and target contaminants may occur. These column tests will help determine if the ferrocyanide is a competitor for uptake by IX site in the IX resins currently used at the 200W P&T facility.

An additional area of research that was not addressed herein is the treatment of free cyanide by IX resin. While there are a few groundwater extraction wells across the Hanford Site that have historically had measurable concentrations of free cyanide, most of the cyanide on site is predicted to be in the form of ferrocyanide. While both ferrocyanide and cyanide are anions the selectivity of an IX resin may be fundamentally different. This may be due to the complexation with iron or the significantly different charge on the anion and additional work to explore the importance of species specific IX resins is recommended.

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