



Research article

Removal of vanadium from industrial wastewater using iron sorbents in batch and continuous flow pilot systems

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ARTICLE INFO

Article history:

Received 27 July 2016

Received in revised form

25 November 2016

Accepted 26 December 2016

Available online 2 January 2017

Keywords:

Vanadium

Sorption

Iron sorbents

Industrial wastewater

Pilot study

ABSTRACT

This study investigated the removal of vanadium from real industrial wastewater by using six iron materials: commercial iron sorbent (CFH-12), commercial mineral sorbent (AQM), blast furnace sludge (BFS), steel converter sludge (SCS), ferrochrome slag (FeCr) and slag from a steel foundry (OKTO). Batch tests revealed that CFH-12 (ferric oxyhydroxide) removed vanadium most efficiently, which was explained by its high iron content and the amorphous form of the iron, and that the sorption followed the Langmuir isotherm. With a dosage of 10 g/l and an initial vanadium concentration of 58.2 mg/l, 91–94% removal rates for vanadium were achieved in the studied pH range (3–9). Other sorbents showed significantly lower efficiency than CFH-12, with the exception of BFS at acidic pH (93%). Based on the batch test results, CFH-12 was selected for a pilot study made on site. The pilot study demonstrated the feasibility of CFH-12 to remove vanadium at high temperature (80 °C) from concentrated industrial wastewater with fluctuating water quality (vanadium concentration varied from 51 to 83 mg/l, pH about 9 (at 25 °C)). Leaching of impurities (mainly S, Ca, Mg and K) into the effluent occurred during the first day, but subsequently good quality effluent was produced (e.g. <0.1 mg/l V). During the pilot study, the amorphous iron material of CFH-12 was crystallized into a hematite-like phase (Fe_{1.67} H_{0.99} O₃), and goethite (FeO(OH)) with a higher average pore diameter, probably due to the hot process conditions to which CFH-12 was exposed for over five days.

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1. Introduction

Vanadium is an important element for the steel industry since it is added to alloys to increase strength. Other important uses of vanadium chemicals are as catalysts and pigments. Vanadium is mainly supplied from steel plant slags, vanadium-bearing ores, and the slags and ashes formed in the processing of vanadium-bearing oil (Perles, 2014). The demand for vanadium may increase in the coming years, particularly in energy storage applications (Perles, 2014). The issue of vanadium demand have prompted even more research for vanadium recovery from waste streams. Besides solid streams, vanadium is present in high concentration in some industrial liquid streams, e.g. extracts of molybdenum minerals containing vanadium (Li et al., 2009) and wastewater from

synthesis gas scrubbers after the gasification of heavy oil (Keränen et al., 2015).

Recovery from liquid waste streams would also prevent vanadium pollution. At the moment, vanadium is on the USEPA (United States Environmental Protection Agency) Drinking Water Contaminant Candidate List (CCL3) due to its carcinogenic effects (ATSDR, 2012); the same list that includes for example pesticides, disinfection by-products and pharmaceuticals. The contaminants on this list are known or anticipated to occur in public water systems, but are currently not subject to any proposed or promulgated national primary drinking water regulations. Vanadium occurs mostly in its oxidation state +5 in an aquatic environment in the presence of oxygen. At low pH (<2–3), vanadium(V) occurs mostly as a cation (VO₂⁺), whereas the anionic species exist at higher pH values (>3): the decavanadate species (V₁₀O₂₆(OH)₂⁴⁻, V₁₀O₂₇(OH)₅⁻, V₁₀O₂₈⁸⁻) and mono- or polyvanadate species (e.g. VO₂(OH)₂, VO₃(OH)₂⁻, VO₄³⁻, and V₂O₆(OH)₃⁻, V₂O₇⁴⁻, V₃O₉³⁻, V₄O₁₂²⁻) (Guzman et al., 2002). Thus, anion exchange is one possible method for the removal of vanadium from wastewaters (Keränen et al., 2015). Furthermore, co-precipitation of vanadium with iron has also been demonstrated

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(Roccaro and Vagliasindi, 2015). Good sorption capacities have also been obtained using pine sawdust (103 mg/g, Leiviskä et al., 2015) as well as iron-based sorbents (65–165 mg/g; Lazaridis et al., 2003; Naeem et al., 2007). Sorption is commonly recommended for industrial applications, because of its simple operational procedure. However, cost-effective sorbents are required for huge water volumes. New uses for waste material that are currently discarded in landfills are needed. Moreover, studies on vanadium removal from real liquid waste streams are rare. The mixture of various components in wastewater may have a significant effect on the sorption process, and, in particular, the competitive effect of other anions may decrease efficiency and selectivity.

This study investigated the removal of vanadium from real industrial wastewater by using various iron materials. These materials included commercial iron sorbent, commercial mineral sorbent, and waste materials and by-products from industrial processes. The industrial wastewater was formed in the gas cleaning system of an oil gasification process. The specific objectives of this study were to

- i) determine vanadium affinity for selected iron materials in the presence of other contaminants in the wastewater,
- ii) demonstrate the performance of the best iron material on pilot scale with a continuous-flow system at high temperature (80 °C), and
- iii) determine the properties of materials before and after the sorption process.

After use, the vanadium-rich iron material could be supplied to the steel industry in case of low-cost and suitable iron material. The second option would be the recovery of vanadium from the material in the regeneration process and reuse of the material in wastewater treatment. Successful completion of the research will provide an overview of sorbent materials for metal recovery from industrial wastewater along with their potential risks in use.

2. Materials and methods

2.1. Materials

The six materials (Table 1) chosen for this study included commercial iron sorbent from Kemira Oyj (granulated ferric oxyhydroxide, CFH-12, d50% 1.4 mm, 98% <2.0 mm (Backman et al., 2007)), one commercial mineral sorbent from Aquaminerals Finland Ltd (AQM PalPower K10 PM = AQM), two industrial by-products from Outokumpu Chrome Oy (OKTO-JT slag 0–4 mm = OKTO and FeCr granulated slag 0–6 mm = FeCr) and two sludge materials from SSAB Raahe (blast furnace sludge = BFS and steel converter sludge = SCS). OKTO is an air-cooled by-product

from the steel foundry having low metal content. FeCr slag is a water-cooled by-product from ferrochrome production, which produces several slag products mainly for the road and civil construction purposes. BFS and SCS are formed in the wet cleaning of blast furnace and steel converter gases. BFS and SCS were pre-treated for moisture removal by oven drying (24 h at 50 °C). The particle size distribution of materials was measured by sieving (with 2 repeats, Table 1).

Industrial wastewater was obtained from a chemical plant synthesis gas scrubber. The pH of the wastewater was 5.8 in the batch experiments and 8.8–9.2 in the pilot study. The variation in pH was due to process changes made at the plant. The concentration of vanadium was 58.2 mg/l in batch experiments (52.2 mg/l by graphite furnace atomic absorption spectrophotometry (GFAAS)) and varied between 51 and 83 mg/l in pilot experiments. The characteristics of the wastewater can be seen in Table 2. Besides vanadium, the wastewater contained a high amount of sulphur (approximately half of which was in the form of sulphate), ammonium and nickel. The original wastewater also contained some solids. In the batch experiments, the solids of the wastewater were allowed to settle for a few hours after sampling, and the supernatant was used in the batch experiments. The aim was to study the removal of dissolved vanadium. In pilot experiments, a pre-filtration system was designed for solids removal (see section 2.4).

2.2. Material characterization

2.2.1. XRF analysis

A Bruker AXS S4 Pioneer X-ray fluorescence (XRF) spectrometer was used to determine the chemical composition of the fresh materials. AQM sorbent was characterized in its original state (powder). Fresh CFH-12, BFS, SCS, OKTO and FeCr were subjected to a grinding process prior to XRF analysis. An amount of 20 g was ground to a particle size of <0.01 mm. For grinding, a mortar and pestle was used for CFH-12, and a planetary ball mill (Retsech PM200) was used for BFS, SCS, OKTO and FeCr. For comparison, XRF analysis of the CFH-12 sorbent used in the pilot study was also performed. A similar grinding procedure was performed as for fresh CFH-12.

For the XRF analysis, 13.16 g of ground sample (AQM as such in powder form) was taken and mixed with 0.84 g of C-wax. A WC/Co mortar HERZOG pulverizing mill was utilized to obtain a homogeneous dispersion and uniform particle size of the resultant mixture (sorbent and C-wax). Pressed pellets for XRF analysis were prepared from the mixture (7–8 g) using boric acid as binder and applying a hydraulic pressure of 10 metric tons to compress the sample.

Table 1
Description of materials used.

Sorbent name	Abbreviation	Supplier	Particle size distribution (%)			
Ferric oxyhydroxide	CFH-12	Kemira Oyj.	>1 mm	1 mm–500 µm	500–250 µm	<250 µm
AQM PalPower K10 PM	AQM	Aquaminerals Finland Ltd.	87.6 ± 0.3	11.8 ± 0.1	0.1 ± 0.1	0.5 ± 0.1
Blast furnace sludge	BFS	SSAB Raahe	>90 µm	90–63 µm	63–45 µm	<45 µm
Steel converter sludge	SCS	SSAB Raahe	2.3 ± 0.2	8.7 ± 0.4	44.6 ± 2.2	44.3 ± 2.2
OKTO-JT slag 0–4 mm	OKTO	Outokumpu Chrome Oy	>250 µm	250–125 µm	125–90 µm	<90 µm
FeCr granulated slag 0–6 mm	FeCr	Outokumpu Chrome Oy	18.0 ± 9.7	11.9 ± 0.2	8.2 ± 1.1	71.0 ± 1.7
			>250 µm	250–125 µm	125–90 µm	<90 µm
			75.3 ± 1.1	10.2 ± 0.8	3.2 ± 0.1	11.3 ± 0.3
			>90 µm	90–63 µm	63–45 µm	<5 µm
			35.6 ± 0.9	9.0 ± 0.7	15.2 ± 0.3	40.2 ± 0.6
			>1 mm	1 mm–500 µm	500–250 µm	<250 µm
			83.1 ± 4.8	16.3 ± 4.6	0.2 ± 0.0	0.0 ± 0.0

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