Hydrometallurgy 117-118 (2012) 108–115

Contents lists available at SciVerse ScienceDirect

Hydrometallurgy

journal homepage: www.elsevier.com/locate/hydromet

Recovery of vanadium from stone coal acid leaching solution by coprecipitation, alkaline roasting and water leaching

Puhong Ye, Xuewen Wang *, Mingyu Wang, Yeye Fan, Xiaoyan Xiang

School of Metallurgical Science and Engineering, Central South University, Changsha 410083, China

ARTICLE INFO

Article history:
Received 27 December 2011
Received in revised form 18 February 2012
Accepted 20 February 2012
Available online 28 February 2012

Keywords:
Stone coal
Acid leaching
Coprecipitation
Alkaline roasting
Water leaching

ABSTRACT

The recovery of vanadium from stone coal acid leaching solution by coprecipitation, alkaline roasting and water leaching was studied. A method used to recover vanadium from stone coal acid leaching solution was developed, and it primarily included vanadium coprecipitation with iron in the solution, vanadium extraction by alkaline leaching from the precipitate, and vanadium pentoxide preparation with the alkaline leaching solution. Experiments found that the vanadium in stone coal acid leaching solution can be effectively enriched in the precipitate obtained by adding 3.64 g $NaClO_3$ per liter solution with initial pH 1.73 under stirring for 0.5 h at 95 °C. By roasting the mixture of 25 g of the precipitate with 22.5 g NaOH at 170 °C for 1.0 h, and then water leaching the roasted mixture at 98 °C for 1.0 h under stirring with L/S ratio of 3.3:1 mL/g, 97.0% of vanadium was extracted from the precipitate. After purifying with $MgCl_2$, the vanadium pentoxide with purity 99.3% was obtained by adding NH_4Cl to precipitate ammonium vanadate from the solution at pH about 2.0, and then roasting the ammonium vanadate at 520 °C for 2.0 h. The essential components of the coprecipitate are $KFe_3(SO_4)_2(OH)_6$ and $HNaV_6O_{16}\cdot 4H_2O$.

1. Introduction

Stone coal is an important material for vanadium pentoxide production. In China, the gross reserve of vanadium in stone coal is 118 million tons, accounting for more than 87% of the domestic reserve of vanadium (Bin, 2006). To extract vanadium from stone coal, many processes were proposed (Zhang et al., 2011), such as NaCl roasting–water leaching (Wang et al., 2009b), oxidization roasting–alkaline leaching (He et al., 2007) or acidity leaching (Wang et al., 2009a), calcium salt roasting–acidity leaching (Li et al., 2007; Zou et al., 2001), direct acidity leaching (Li et al., 2008; Lu, 2002; Zhou et al., 2009) or pressure acidity leaching (Deng et al., 2010; Li et al., 2009; Wei et al., 2008), sulphation roasting–water leaching (Liu et al., 2009). Of the processes, the acid decomposition was dominantly adopted in the plants of vanadium pentoxide production with stone coal as the emission of HCl and Cl_2 in the NaCl roasting and a lot of Si existing in the alkaline leaching solution. Though both ion exchange (Xing et al., 2009; Zeng et al., 2009) and solvent extraction (Li et al., 2010, 2011; Yi et al., 1992) can be used to separate and concentrate vanadium from the acid leaching solution, the method commonly adopted in the plants is solvent extraction because there are many Fe ions in the solution, which will form the precipitate including vanadium when the solution is oxidized and adjusted the pH value to over 2.0 for ion exchange. However, the process of solvent extraction also presents many drawbacks, e.g. high-reagent consumption and discharge of the spent water. To avoid the interference of iron in solvent extraction, the vanadium was first reduced from V(V) or/and V(IV) to V(III) with the reduce of iron from Fe^{3+} to Fe^{2+} in the feed liquid, and then the vanadium was oxidized from V(III) to V(V) in the stripping solution. Too many reagents were added in the solution; the spent water cannot be recycled but be discharged in the process.

The motivation for this study was the need to avoid the interference of iron and to reduce the discharge of the spent water in the production of vanadium pentoxide. The work presents the process of vanadium recovery from stone coal acid leaching solution by coprecipitation, alkaline roasting and water leaching.

2. Experimental

2.1. Materials and analysis

The acid leaching solution used in this study was prepared by decomposing stone coal with sulphation roasting–water leaching. The composition of the solution after alum crystallization was listed in Table 1. Sulfuric acid, lime, ammonium sulfate, ammonium

---

* Corresponding author.
E-mail address: wxwcsu@163.com (X.-W. Wang).

0304-386X/$ – see front matter © 2012 Elsevier B.V. All rights reserved.
doi:10.1016/j.hydromet.2012.02.013

Table 1
Experiment results of vanadium recovery from stone coal acid leaching solution by coprecipitation, alkaline roasting and water leaching.

Elements	V_2O_5	Fe	Al	K	Ba	Mg	Ti	pH
Solution after alum crystallization, g/L	3.30	12.01	2.18	0.41	0.15	1.70	0.12	0.85
Solution after V precipitation, g/L	0.83	7.80	2.11	0.40	0.14	1.69	0.11	1.31
Precipitate containing V, %	13.02	32.56	0.44	1.52	0.10	0.05	0.63	–
Alkaline leaching solution, g/L	22.29	1.95	0.65	2.23	0.06	0.04	0.05	13.2
Alkaline leaching residue, %	0.73	58.62	0.10	0.40	0.10	0.05	1.21	–
Vanadium pentoxide, %	99.3	<0.01	<0.01	0.08	<0.01	0.05	<0.01	–

ferrous sulfite, urea, potassium permanganate, sodium nitrite and N-phenylanthranilic acid were used in the experiments.

Vanadium was titrated with ammonium ferrous sulfate (Chemistry department, Hangzhou University, 1982). Iron, aluminum, potassium and so on were determined by chemical methods and inductively coupled plasma emission spectroscopy (ICP) with a PS-6 PLASMA SPECTROVAC, BAIRD (USA). The pH was determined with ORION-230A which was made in USA; the degree of accuracy is 0.01.

2.2. Experimental procedure

The experiments were performed according to the flow sheet as shown in Fig. 1. To recover vanadium from the solution, the pH was adjusted to the pre-determined value with lime milk. After the required contact time, the supernatant was vacuum filtered and the cake was submitted to successive rinsing with water. In order to reduce vanadium loss, the cake was washed with the acid leaching solution under stirring and then filtered again when the pH was adjusted to over 2.0.

The vanadium in the solution can be oxidized from V(III) and/or V(V) to V(V) by $NaClO_3$, H_2O_2, $KMnO_4$ and so on. In the study, $NaClO_3$ was chosen as the oxidant. A pre-determined weight of $NaClO_3$ was added into 1000 mL of the adjusted pH solution under stirring at 95 °C to make the vanadium coprecipitated with iron. After the required reaction time, the precipitate was separated by filtering. The content of vanadium in the precipitate and the concentration of vanadium remaining in the filtrate were analyzed respectively.

All of precipitates obtained under different conditions were mixed by stirring in water, and then filtrated and dried. The composition of the mixed precipitate was showed in Table 1. The precipitate was treated by alkaline roasting and water leaching. A pre-determined weight of NaOH was added into 25 g of the precipitate. To make the NaOH contacted with the precipitate well, proper quantity water was mixed with them in a nickel crucible, and then the mixture was

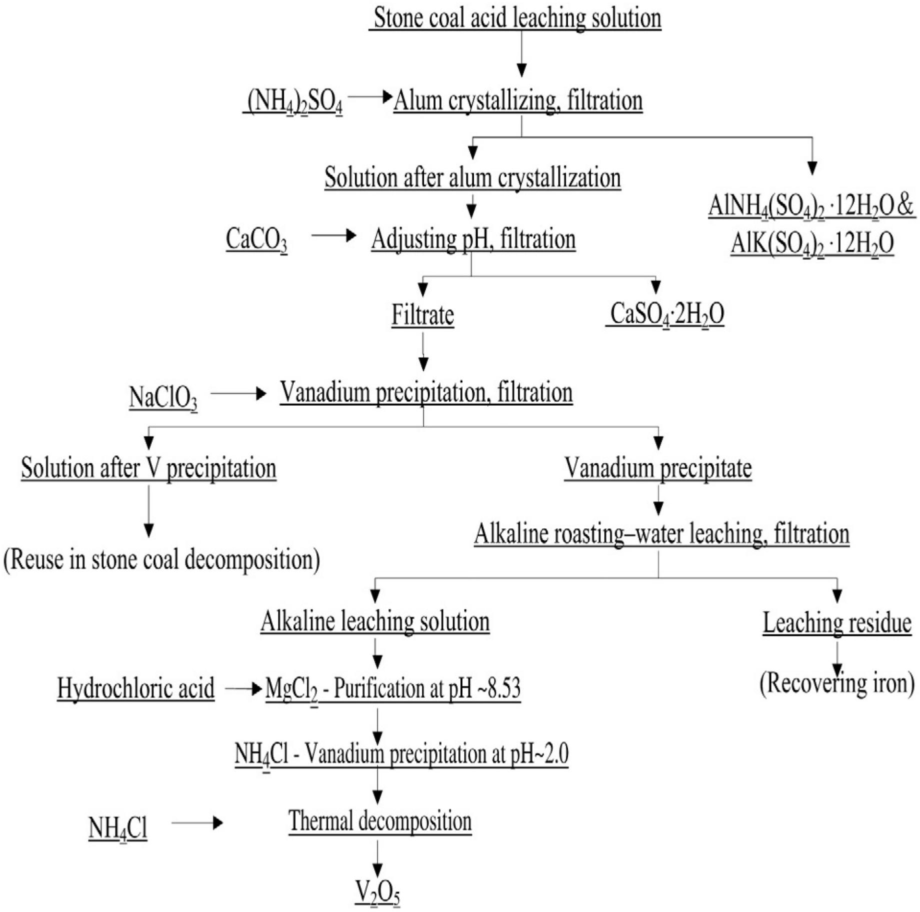

Fig. 1. Flow sheet of the process of vanadium recovery from stone coal acid leaching solution.