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Technical note

Extraction of rubidium from gold waste: Process optimization

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ABSTRACT

The extraction of rubidium from gold waste from the Mouteh processing plant in Iran by a three-step process (acid washing, followed by salt roasting and water leaching) was optimized. The acid washing step for removal of impurities was found to be optimal at 85 °C and 5 h, using 5 M nitric acid. Factors effecting roasting operation were then optimized by a series of initial experiments, then using response surface methodology (RSM) based on a central composite design (CCD). Rb extraction of 90.95% was obtained with a mass ratio of GW/Na_2SO_4/$CaCl_2 \cdot 2H_2O$ of 1.00:0.11:0.45 at 910 °C for 30 min. Water leaching of the roasted mixture resulted in 97.14% Rb extraction under optimum conditions, viz. a liquid/solid ratio of 1.69 at 58.5 °C for 31.4 min. The proposed correlations using RSM showed good agreement with the experimental data.

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1. Introduction

The Mouteh gold plant is one of the largest gold processing projects in Iran. In this plant, gold is extracted from the ore using a cyanidation process. Due to the type of the ore and operation, some toxic compounds including arsenic and mercury are transferred to the wastewater. Pollutant accumulation in the tailings dam can result in serious environmental impacts in the long term. Nevertheless, these gold wastes (GW) contain valuable metals such as rubidium, titanium, cerium, neodymium, lanthanum, etc. In addition to creating value added products for the plant, extraction of these elements as by-products can reduce the considerable volumes of waste.

Rubidium (Rb) is a rare alkali metal in the first group of the periodic table, which was discovered in 1861 by German chemists R. W. Bunsen and G. R. Kirchhoff using flame spectroscopy. The most striking physical properties of this silvery-white element include softness, malleability and low melting point (39 °C). It is also the fourth lightest metallic element (De, 2003).

The application of Rb in ion engines for space vehicles, photocells, methanol and alcohol production, in analytical chemistry for identification of manganese, zirconium and noble metals, as well as many other applications, indicates the unique properties of this valuable element (Butterman and Reese, 2003; De, 2003).

The annual world production of Rb is limited to 2–4 tonnes per year due to the non-existence of a Rb-rich mineral. Because of the variety of applications, supply and demand of Rb has been constantly growing since 1990, and its price has increased in the international market (Butterman and Reese, 2003; Salazar and McNutt, 2013).

Rb is not found as main component in any mineral and is mainly produced together with cesium (Cs) as a by-product of lithium (Li) minerals processing (Butterman and Reese, 2003). The main commercially viable sources of Li include natural brines with a high LiCl content as well as pegmatite minerals such as lepidolite [$KRbLi(OH,F)Al_2Si_3O_{10}$], spodumene [$LiAl(SiO_3)_2$], petalite [$LiAlSi_4O_{10}$] and zinwaldite [$(K(Li,Al,Fe)_3(Al,Si)_4O_{10}F_2)$] (Amouzegar et al., 2000; Demirbaş, 1998; Kondas and Jandova, 2006; Sitando and Crouse, 2012; Wietelmann and Bauer, 2003). Most common Li minerals and typical compositions are shown in Table 1. According to this table, Rb and Cs are only found in Li minerals of lepidolite and zinwaldite (Garrett, 2004; Paukov et al., 2010).

The processing of Li minerals includes upgrading the ground ores using beneficiation techniques such as gravity concentration, flotation and wet magnetic separation (Siame and Pascoe, 2011), optical sorting (Brand and Haus, 2010) or heavy media separation (Galaxy Resources Ltd., 2008, 2010), chemical roasting of the concentrate using H_2SO_4,

Table 1
Typical compositions (%) of lithium minerals (Garrett, 2004; Paukov et al., 2010).

Minerals	Li_2O	Rb_2O	Cs_2O
Lepidolite	4.19	3.39	0.25
Spodumene	6	–	–
Petalite	4.73	–	–
Zinwaldite	2–5	0.45	0.03

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Table 2
Results of several published studies on the Rb extraction as by-product.

Mineral tested	Lepidolite	Zinwaldite	Zinwaldite	Lepidolite	Zinwaldite	Zinwaldite	Lepidolite
Best roast temp, °C	1000	850	1050	880	825	825	850
Roasting time, h	0.5	1	1	0.5	1	1	1.5
Additives	Na_2SO_4	Na_2SO_4	Gypsum	Na_2SO_4 $CaCl_2$	$CaCO_3$	$CaCO_3$	$FeSO_4 \cdot 7H_20$ CaO
Best leach temp, °C	85	85	85	Ambient	90–95	95	Ambient
Leaching time, h	3	0.5	0.5	0.5	0.5	1	1
Water/roasted ratio	1:15	1:10	1:10	1:0.8	1:5	1:10	1:1
Max. Li extract., %	90.4	97	84	92.86	85	84	93.3
Max. Rb extract., %	27.3	23	14	93.60	85	91	33.2
Max. Cs extract., %	23.5	–	–	93.01	–	–	21.6
Reference	Luong et al. (2013)	Siame and Pascoe (2011)	Siame and Pascoe (2011)	Yan et al. (2012b)	Jandova et al. (2010)	Vu et al. (2013)	Luong et al. (2014)

HCl (Luong et al., 2013; Sitando and Crouse, 2012; Wietelmann and Bauer, 2003; Yan et al., 2012a), limestone, gypsum as well as alkali salts or sulfates (Jandova et al., 2009; Jandova et al., 2008) with the aim of converting Li minerals into soluble form for the subsequent leaching stage (Luong et al., 2013; Sitando and Crouse, 2012). Table 2 summarizes the results of several published studies relevant to Rb extraction during Li processing.

Shan et al. (2013) roasted the muscovite [$KAl_2(AlSi_3O_{10})(OH)_2$] mineral under different conditions with the aim of extracting Rb. According to their results, the best Rb extraction (90.1%) was achieved for the mixture roasted with mass ratio of muscovite/NaCl/$CaCl_2$ of 1.00/0.25/0.25 at 850 °C for 30 min.

The present study was undertaken with the aim of achieving maximum rubidium extraction from GW from an environmentally friendly process. An attempt was made to optimize the extraction conditions of potentially valuable metals in a three-step process (acid washing, followed by salt roasting and water leaching) using response surface methodology (RSM) based on central composite design (CCD).

2. Experimental

2.1. Characterization of the gold waste

The representative sample was obtained from six trenches each 2 m depth, excavated in the tailings dam of the Mouteh gold plant. The sample was first subjected to dry sieving, and D70 of up to 150 microns was obtained. In order to prevent the re-absorption of metal ions in the leaching operation by the activated carbon remaining in the GW, the sample was passed through a 150 micron sieve. Rubidium content and other metal elements were analyzed by ICP (OES-VARIAN 735) and ICP-MS (HP 4500). The chemical composition of the GW sample is listed in Table 3.

According to XRD analysis, GW consisted of major phases of quartz (SiO_2) and albite ($NaAlSi_3O_8$) and minor phases of chlorite [$(Mg,Fe)_6(Si,Al)_4O_{10}(OH)_8$], muscovite [$KAl_2(AlSi_3O_{10})(OH)_2$], orthoclase ($KAlSi_3O_8$), dolomite [$CaMg(CO_3)_2$], gypsum ($CaSO_4 \cdot 2H_2O$) and pyrite ($FeS_2$), all predominantly silicate and carbonate compounds insoluble in the cyanidation process.

Table 3
Chemical composition of GW sample (mg metal/kg solid).

Fe	Na	Ca	V	Mn	Li
41684.24	17546.40	9441.28	51.08	303.76	19.15
Rb	**Cs**	**Mg**	**Ce**	**La**	**Nd**
120.12	9.42	9861.62	87.54	49.22	52.34
Al	**K**	**Ti**	**Sc**	**Sr**	**Y**
38482.44	1087.36	2426.06	11.30	63.66	5.74

Table 4
Metal extractions (%) from GW using 5 M acids at room temperature for 2 h.

Extraction (%)	Fe	Na	Ca	Ba	Mn	Li	Al	K	Ti
Acid type									
H_2SO_4	23.41	5.19	8.80	0.00	37.31	7.62	1.72	27.20	0.34
HCl	19.16	4.63	95.12	4.58	40.74	10.34	1.48	26.13	1.35
H_3PO_4	21.33	0.25	93.37	0.37	25.79	7.62	1.11	16.28	0.09
HNO_3	28.57	5.96	96.05	3.56	50.88	9.61	1.24	28.56	0.10

Extraction (%)	Rb	Cs	Mg	Ce	La	Nd	Sc	Sr	Y
Acid type									
H_2SO_4	0.60	0.85	43.72	4.92	3.29	7.55	12.39	9.57	27.35
HCl	0.38	1.27	41.82	6.41	3.78	8.65	18.94	15.52	63.59
H_3PO_4	0.19	1.70	39.87	3.86	0.41	5.54	13.36	22.81	36.24
HNO_3	0.32	6.26	42.90	2.52	1.79	8.98	14.60	29.12	45.64

2.2. Procedures and analytical instruments

The roasting operation was conducted in a muffle furnace (KSL-1200X-M, MTI corporation) at temperatures ranging from 500 °C to 950 °C. The surface morphology of samples using two different roasting temperatures was examined using SEM (KYKA-EM3200, China). The phases of the mixture roasted under optimum conditions were identified using XRD. After heating, water leaching of the mixture was done at ambient temperature and a liquid/solid (L/S) ratio of 1 for 30 min.

Table 5
The effect of temperature on metal extractions (%) from GW using 5 M HNO_3 for 5 h.

Extraction (%)	Fe	Na	Ca	V	Mn	Li	Al	K	Ti
Temp.									
Ambient	41.46	5.99	97.31	4.39	80.07	13.26	3.43	47.89	0.18
85 °C	76.72	6.02	99.86	6.23	94.07	19.53	6.91	81.09	0.48

Extraction (%)	Rb	Cs	Mg	Ce	La	Nd	Sc	Sr	Y
Temp.									
Ambient	0.63	6.90	65.67	4.24	2.26	9.69	15.66	29.41	48.08
85 °C	1.62	9.66	91.97	4.93	2.91	11.67	16.73	31.61	58.36

Table 6
The effect of roasting chemical agents on the Rb extraction.

Mixture	Mass ratio	Rb extraction (%)
GW/Na_2SO_4/$CaCl_2 \cdot 2H_2O$	1.00:0.10:0.50	85.11
GW/Na_2SO_4/$CaCl_2 \cdot 2H_2O$	1.00:0.25:0.50	68.21
GW/Na_2SO_4/$CaCl_2 \cdot 2H_2O$	1.00:0.50:0.50	44.14
GW/Na_2SO_4/$CaCl_2 \cdot 2H_2O$	1.00:0.50:0.25	36.76
GW/NaCl/$CaCl_2 \cdot 2H_2O$	1.00:0.10:0.50	82.08
GW/NaCl/$CaCl_2 \cdot 2H_2O$	1.00:0.25:0.50	61.98
GW/NaCl/$CaCl_2 \cdot 2H_2O$	1.00:0.50:0.50	38.77
GW/NaCl/$CaCl_2 \cdot 2H_2O$	1.00:0.50:0.25	29.13
GW/$CaCO_3$/$CaCl_2 \cdot 2H_2O$	1.00:0.10:0.50	44.78
GW/$CaCO_3$/$CaCl_2 \cdot 2H_2O$	1.00:0.25:0.50	38.54
GW/$CaCO_3$/$CaCl_2 \cdot 2H_2O$	1.00:0.50:0.50	30.04
GW/$CaCO_3$/$CaCl_2 \cdot 2H_2O$	1.00:0.50:0.25	23.26

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