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Synergetic interactions improve cobalt leaching from lithium cobalt oxide in microbial fuel cells

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HIGHLIGHTS

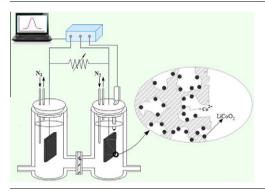
- Cobalt leaching from lithium cobalt oxide was successfully achieved in MFCs.
- Synergetic interactions were observed for cobalt leaching in MFCs.
- External resistor, solid/liquid ratio and solution conductivity affected performance.
- A pH of 1.0 and a temperature of 35 °C improved cobalt leaching.
- Apparent activation energy of 30.6 kJ/mol was estimated.

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G R A P H I C A L A B S T R A C T



ABSTRACT

Cobalt leaching from lithium cobalt oxide is a promising reduction process for recovery of cobalt and recycle of spent lithium ion batteries, but suffers from consumption of large amount of reductants and energy, and generation of excess secondary polluted sludge. Thus, effective and environmental friendly processes are needed to improve the existing process limitations. Here we reported microbial fuel cells (MFCs) to effectively reduce Co(III) in lithium cobalt oxide with concomitant energy generation. There was a synergetic interaction in MFCs, leading to a more rapid Co(III) leaching at a rate 3.4 times the sum of rates by conventional chemical processes and no-acid controls. External resistor, solid/liquid ratio, solution conductivity, pH and temperature affected system performance. This study provides a new process for recovery of cobalt and recycle of spent lithium ion batteries with concomitant energy generation from MFCs.

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1. Introduction

The decline of valuable metal resources, together with the increased future valuable metals demand gives an impetus for increased metal recovery from wastes such as fly ash, sewage sludge, spent batteries and electronic scrap materials, as well as the hydroprocessing catalysts (Marafi and Stanislaus, 2008). Lithium cobalt oxide (LiCoO₂) is the most commonly used cathode material in lithium ion batteries, which have been extensively



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applied as a power source for mobile phones, laptops and electronic devices due to their smaller, lighter, and no memory effect advantages (Xu et al., 2008). The higher contents of 5-15% cobalt and 2-7% lithium in these batteries than those found in natural ores or even concentrated natural ores makes recovery of the metals from spent lithium ion batteries more attractive (Xu et al., 2008). Since cobalt is a relatively expensive material compared to the other battery constituents such as lithium, its recovery is one of the primary objectives in the recycling of these wastes. Conventional techniques involving pyrometallurgical process, hydrometallurgical process or the combination of both can leach the valuable metals inside (Marafi and Stanislaus, 2008; Sun and Qiu, 2011; Xu et al., 2008). The great energy consumption, high cost and serious second pollution urge a turn to environmental friendly and cost-effective strategies. While metals bio-hydrometallurgical (bioleaching) processes have been intensively studied due to their lower cost and fewer requirements than hydrometallurgical process, challenges to this leaching approach in practice, however, are addition of chemical reductants, requirement of microorganism endurance to high metal concentration, and excess secondary polluted sludge generation (Mishra et al., 2008; Wang et al., 2012; Xin et al., 2009). New processes are needed that can achieve efficient cobalt leaching and improve the existing process limitations.

One possibility for improved cobalt leaching is to use microbial fuel cell (MFCs). An MFC is a device that uses microbes to convert the chemical energy stored in organic and inorganic compounds into electricity, providing a low-cost and low-maintenance reactor as well as a process that produces very little sludge (Logan, 2009, 2012). While a wide variety of wastes can be degraded in anode (Huang et al., 2011a, 2012a; Pant et al., 2010), the reducing environment in the MFC cathode, which is a sink for electrons generated in the anode, is an advantage for the treatment of oxidized pollutants (Huang et al., 2011b). Soluble organics including azo dye of acid orange 7 (Mu et al., 2009), nitrobenzene (Li et al., 2010), pentachlorophenol (Huang et al., 2012b) and aqueous heavy metals such as Cr(VI) (Li et al., 2008), V(V) (Zhang et al., 2009), Cu(II) (Ter Heijne et al., 2010), Se(IV) (Catal et al., 2009), Hg(II) (Wang et al., 2011) and Ag(I) (Tao et al., 2012) have been shown to be successfully reduced on the cathode, with concomitant energy recovery (Huang et al., 2011b). Considering the oxidative Co(III) in LiCoO₂, cathode in MFCs might be useful for the cobalt leaching, during which particle Co(III) is reduced to soluble Cr(II). However, the cobalt leaching process has not yet been examined in MFCs.

In this study, cobalt leaching from particle LiCoO₂ in MFCs was investigated. Kinetic parameters including external resistor, solid/ liquid ratio, solution conductivity, initial pH and temperature were examined. The performance of the system was evaluated in terms of cobalt and lithium leaching, current generation, and cathodic coulomic efficiency (CE_{ca}). A synergetic interaction of current generation with conventional chemical processes for cobalt dissolution was observed. Deeper insight into these aspects will enhance the performance of MFCs for efficient cobalt leaching from LiCoO₂ as discussed subsequently.

2. Methods

2.1. Fuel cell assembly

Two-chamber MFCs (duplicates) were used in all experiments, with the electrodes separated by a cation exchange membrane (CEM) (CMI-7000 Membranes International, Glen Rock, NJ) (2.5 cm in diameters). Graphite felt (Sanye Co., Beijing, China) $(3.0 \times 2.0 \times 1.0 \text{ cm})$ was used in each compartment and served as the anode and the cathode. Before the installation, the electrode materials were washed firstly in 32% HCl and then in 35% NaOH to remove metals from the surface (Clauwaert et al., 2007). This

procedure was repeated four times before they were rinsed four times in ultrapure water. A reference electrode (Ag/AgCl electrode, 195 mV versus standard hydrogen electrode, SHE) was used to obtain cathode and anode potentials, with all voltage reported here versus SHE. The net working volume of each chamber was 100 mL. All of the reactors were wrapped with aluminum foil to exclude light.

2.2. Inoculation and operation

A desired quantity of LiCoO₂ (Sigma–Aldrich, 99.8%) was added into 100 mL de-ionized water in one conical flask before a piece of graphite felt $(3.0 \times 2.0 \times 1.0 \text{ cm})$ was positioned and submerged inside. This flask was maintained under magnetic agitation for 15 min in order to uniformly spread and load the particles on the electrode surface. The catholyte was 0.01 M HCl aqueous solution. After transfer of the LiCoO₂ attached cathode, the catholyte was purged with nitrogen for another 15 min in order to remove oxygen. At the end of one cycle, the cathode electrode was treated as previously described (Clauwaert et al., 2007) and then used for another cycle. The medium used for the anode was the same as previously reported using acetate as a fuel (Huang et al., 2011c). The acclimation of bioanode was similar to previously reported (Huang et al., 2012a, 2012b; Sun et al., 2011), in which the bioanode was developed in a separate MFC (originally inoculated using primary sedimentation tank of the Lingshui wastewater treatment plant (Dalian, China) which contained (g/L) total COD, 0.38 ± 0.02 ; soluble COD, 0.20 ± 0.04 ; ammonia nitrogen, 0.22 ± 0.08 with pHs 6.6–7.2) that was operated for three months with acetate as the electron donor. While the origin from this wastewater can provide enough source for exoelectrogenic bacteria (Huang et al., 2012a, 2012b; Logan, 2009; Yates et al., 2012), this pre-established bioanode in an old MFC here not only accelerated the startup and avoided the time-consumed acclimation procedure for the new MFC reactor, but also benefited to the formation of exoelectrogenic biofilms with stable and high power generation (Cheng et al., 2011; Huang et al., 2012a, 2012b; Sun et al., 2011). Based on the community analysis using a polymerase chain reaction and denaturing gradient gel electrophoresis (Huang et al., 2012a; Sun et al., 2011), the composition of anodic biofilms was similar to previously reported (Huang et al., 2012a; Logan 2009; Sun et al., 2011; Yates et al., 2012) and mainly composed of Geobacter sulfurreducens (38%), Bacteroidetes sp. (14%), Pseudomonas sp. (11%), Burkholderiales sp. (8%), Comamonadaceae sp. (6%), and other (23%). All reactors were run in duplicate. Three controls were also operated: one was used for bare electrode processes and therefore the cathodic electrode was not attached by LiCoO₂ particles (bare MFCs); the second was run in the open circuit condition (OCC, conventional chemical process) to examine changes in leaching of cobalt and lithium in the absence of current generation; the third was operated with no presence of HCl in the cathode to exclude the role of HCl on cobalt and lithium leaching (no-HCl controls). In order to avoid the deficiency of solution conductivity caused by the absence of HCl, 0.1 M KCl was used to adjust to the indicated values.

The reactor was run in fed-batch mode at temperature of 20 ± 2 °C except the cases where temperature effects were examined at 4, 10, 35 and 50 °C, respectively. To examine the effect of external resistor on performance, the reactor was operated at each set resistor of 2000, 1000, 500, 200, and 100 Ω (multiple cycles). In tests on the effect of solid/liquid ratio on performance, four solid/liquid ratios were tested (50, 200, 500 and 1000 mg/L (w/v)).

In tests with solution conductivity effects, 0.1 M KCl was added directly to cathodic solution (pH: 2.0, conductivity: 4.5 mS/cm) to produce the desired conductivities of 9.8 and 17.6 mS/cm. In the investigation of pH effects, the initial de-ionized water was added by HCl to produce five sets of pHs (1.0, 1.5, 2.0, 2.5 and 3.0) and thus resulted in a range of solution conductivities from 1.6 mS/

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