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Investigation of zinc recovery by hydrogen reduction assisted pyrolysis of alkaline and zinc-carbon battery waste

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ABSTRACT

Zinc (Zn) recovery from alkaline and zinc-carbon (Zn-C) battery waste were studied by a laboratory scale pyrolysis process at a reaction temperature of 950 °C for 15–60 min residence time using 5% $H_{2(g)}$ - $N_{2(g)}$ mixture at 1.0 L/min gas flow rate. The effect of different cooling rates on the properties of pyrolysis residue, manganese oxide particles, were also investigated. Morphological and structural characterization of the produced Zn particles were performed. The battery black mass was characterized with respect to the properties and chemical composition of the waste battery particles. The thermodynamics of the pyrolysis process was studied using the HSC Chemistry 5.11 software. A hydrogen reduction reaction of the battery black mass (washed with Milli-Q water) takes place at the chosen temperature and makes it possible to produce fine Zn particles by rapid condensation following the evaporation of Zn from the pyrolysis batch. The amount of Zn that can be separated from the black mass increases by extending the residence time. Recovery of 99.8% of the Zn was achieved at 950 °C for 60 min residence time using 1.0 L/min gas flow rate. The pyrolysis residue contains MnO and Mn_2O_3 compounds, and the oxidation state of manganese can be controlled by cooling rate and atmosphere. The Zn particles exhibit spherical and hexagonal particle morphology with a particle size varying between 200 nm and 3 μ m. However the particles were formed by aggregation of nanoparticles which are primarily nucleated from the gas phase.

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1. Introduction

Alkaline and zinc-carbon (Zn-C) batteries have been energizing various frequently used electrical tools since the 1960s and their consumption still dominates the portable battery market (Bernardes et al. 2004; Smith et al., 2014). At the end of their service life, the spent batteries are usually landfilled due to the low economical benefit of recycling them. However developed and developing countries enforce the collection, safe storage and/or recycling of the batteries by local regulations (Espinosa et al., 2004; Guevara-Garcia and Montiel-Corona, 2012; Lindhqvist, 2010; Terazono et al., 2015). Although several hydrometallurgical and pyrometallurgical methods were proposed for the recycling of batteries (Bernardes et al., 2004; Ferella et al., 2008), current processes have not been considered sufficiently economically viable for a widespread recycling of these batteries. The recycling of the alkaline batteries is challenging because the total cost of end-of-life recycling, including collection, sorting, transportation,

and recycling of the primary batteries currently exceeds the economic value of recovered materials from their waste (Turner and Nugent, 2016). Thus the industry needs innovative recycling approaches which are profitable as well as environmentally sound.

Alkaline battery waste contains high amounts of iron (Fe), manganese (Mn), and zinc (Zn). These metals can be recovered separately, and they can also be used as raw material in some industries. For example, spent alkaline batteries are added to molten metal to reclaim the Fe and Mn in the steel industry (Bernardes et al., 2004; Pareuil et al., 2010). Over the last decade, preparation of advanced materials using the spent alkaline batteries as a raw material has attracted scientific attention. Xi et al. (2006) prepared Mn-Zn ferrites by leaching of alkaline batteries and synthesis in a sol-gel process. However, they needed to adjust the concentration of the solution using commercial metal salts. Peng et al. (2008) used Mn-Zn dry batteries, waste scrap iron, and pyrolusite as raw materials to prepare Mn-Zn soft magnetic ferrite powders by a process that includes leaching, purifying, and co-precipitation steps. Kim et al. (2009) reported the production of Mn-Zn ferrite powder from Zn-C batteries by reductive acid leaching and oxidative alkaline co-precipitation. Nanostructured Mn-Zn ferrite particles were also produced by auto-combustion and microwave digestion methods using spent alkaline battery leaching solution

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Table 1
Chemical composition of the alkaline battery black mass; before (Ebin et al., 2016a) and after washing (Ebin et al., 2016b).

Elements	Alkaline battery waste before washing, % (w/w) ¹	Alkaline battery waste after washing, % (w/w) ²
Mn	28 ± 1	32.8 ± 2.6
Zn	25 ± 1	29.1 ± 2.8
K	4 ± 0.6	1.0 ± 0.5
Fe	0.83 ± 0.04	0.8 ± 0.1
Ni	0.1 ± 0.06	0.14 ± 0.03
Co	0.01 ± 0.004	0.01 ± 0.003
Cu	0.03 ± 0.01	0.03 ± 0.01
Cr	0.02 ± 0.005	0.01 ± 0.003
Pb	0.02 ± 0.002	0.02 ± 0.004
Cd	0.01 ± 0.003	0.01 ± 0.004
Hg	0.00	0.00

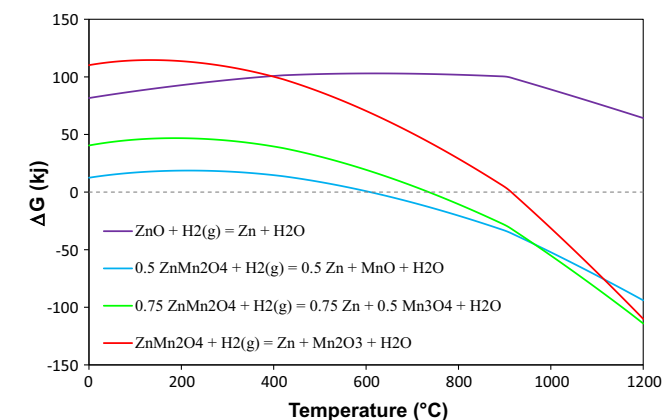
¹ Ebin et al. (2016a), Waste Manage.

² Ebin et al. (2016b), J. Anal. App. Pyrol.

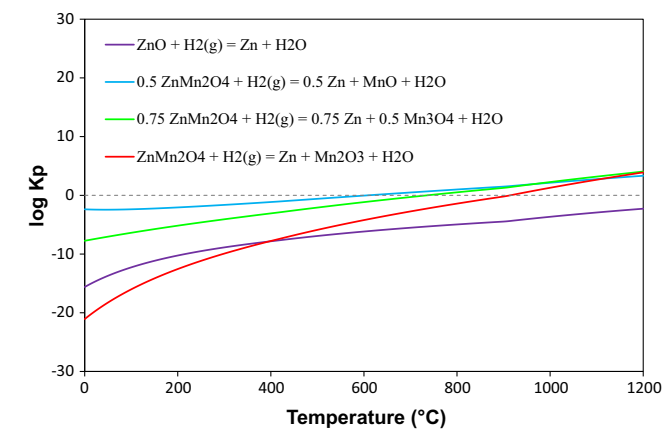
due to their interesting ferromagnetic properties (Hu et al., 2011; Yang et al. 2015). Tu et al. (2013) investigated the nano-absorbent properties of Mn-Zn ferrite particles prepared from battery waste. Qu et al. (2015) showed that spent alkaline batteries can be used to prepare photocatalytic $Zn_xMn_{1-x}O$ nanoparticles, which catalyzes the degradation of bisphenol A under solar light irradiation. ZnO nanoparticles have also been prepared from battery waste by a stepwise process consisting of leaching, liquid-liquid extraction and synthesis (precipitation and heat-treatment) steps (Deep et al., 2011; Deep et al., 2016). Preparation

of Zn nanostructured particles using vacuum separation at high temperatures (650–800 °C) and inert gas condensation from zinc manganese batteries was reported by Xiang et al. (2015).

After magnetic separation of the iron shells from the alkaline battery waste, Zn and Mn are the valuable elements to recover. Although fresh alkaline battery contains Zn and manganese dioxide (MnO_2), various oxide compounds such as hetaerolite

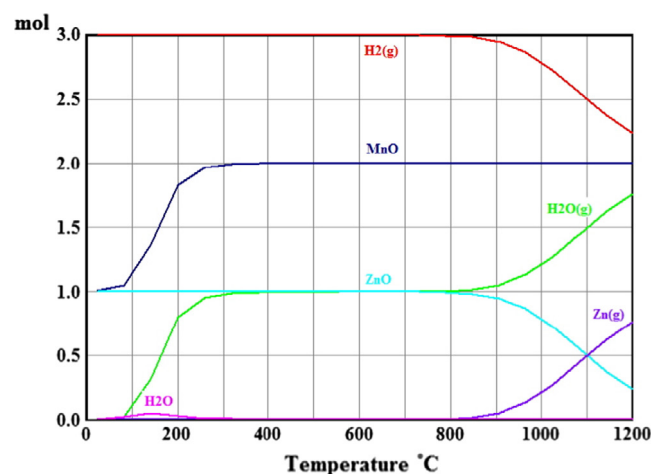


(a)

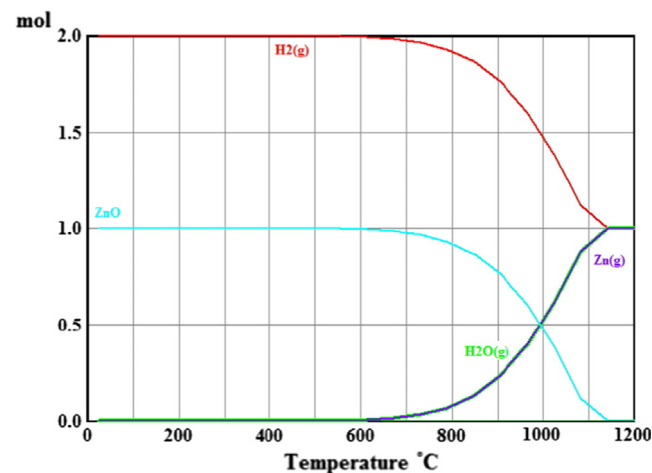


(b)

Fig. 1. Change of the Gibbs standard free energies and log value of the reaction equilibrium quotients (Keq) for hydrogen reduction of the alkaline battery black mass (a) ΔG° (kJ) and (b) $\log K_{eq}$.



(a)



(b)

Fig. 2. Phase equilibrium amount changes by temperature for (a) hydrogen reduction of $ZnMn_2O_4$ and (b) hydrogen reduction of ZnO .

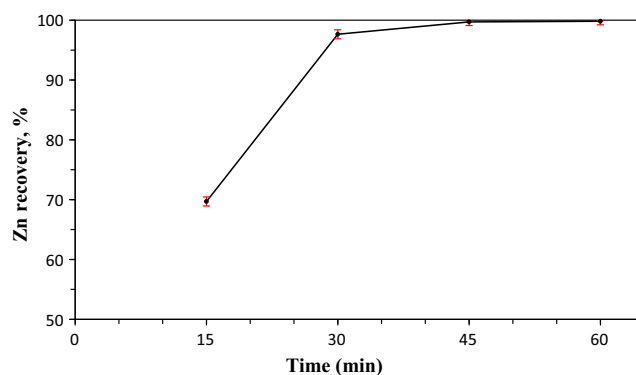


Fig. 3. Removal of zinc from alkaline and zinc-carbon battery black mass dependent on residence time at 950 °C under 1.0 L/min flow rate of 5% H_2 - N_2 gas mixture.

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