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Estimation of retorted phosphor powder from spent fluorescent lamps by thermal process

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ABSTRACT

The degree of thermal stabilization of phosphor powder from spent fluorescent lamps (SFLs) manufactured by three companies (A, B, C) was estimated by examining mercury content in phosphor powder with retorting time, retorting temperature and rotational speed of drum. Mercury content of phosphor powders from spent fluorescent lamps manufactured by A, B and C companies as samples in thermal experiments was 4031 mg/kg, 3522 mg/kg and 3172 mg/kg, respectively. In the thermal experiments, the optimal conditions for retorting time, retorting temperature, and rotational speed were determined at 6 h, 400 °C, and 2.0 rpm, respectively. With thermal processing at the optimal conditions, mercury content of all samples for retorted phosphor powder was less than 3.0 mg/kg, while efficiency of thermal process to control mercury content was higher than 99.9%. Leaching tests such as Toxicity Characteristic Leaching Procedure (TCLP) and Korea Extraction Test (KET) were subsequently carried out to verify if retorted phosphor powder is hazardous waste. Leaching concentrations of mercury for all samples of retorted phosphor powder were satisfied with regulatory levels in both leaching tests. Hence, retorted phosphor powders at the optimal conditions are considered to be non-hazardous wastes.

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1. Introduction

Demand for fluorescent lamp in household, workplace and other settings is rising steadily due to the development of the electronics industry even though the market of LED (light emitting diode) lamp has grown recently. Spent fluorescent lamps (SFLs) with high content of mercury, if not managed thoroughly, could cause serious environmental problems. Thus, it is necessary to recycle spent fluorescent lamps to prevent environmental pollution.

In order to promote recycling of fluorescent lamps in Korea (Republic of), the government chose fluorescent lamp as an Extended Producer Responsibility (EPR) item in 2004 (Ministry of Environment in Korea, 2013a; Rhee et al., 2013). In 2010, 35.3 million spent fluorescent lamps were recycled at a recycling rate of about 28.8% (Korea Lighting Recycling Association, 2011). To improve recycling rates, government set a target for recycling obligation rate at 32.8% in 2013, while long-term target in 2017 was set and published at 39.4% (Ministry of Environment in Korea, 2013b). In the United States, SFL recycling rates are approximately 23% because mandatory recycling rules were applied to business

generators. Due to lamp recycling systems based on the EPR in other countries, much higher recycling rates were achieved: 80% in Austria, 95% in Switzerland, and 80% in Taiwan (Geraldo and Chang, 2010).

Fluorescent lamps can be generally divided into standard lamps and three-banded lamps. Phosphor powder from three-banded fluorescent lamps, unlike standard lamps, contains rare earth metals, which are separately recovered from phosphor powder (Michelis et al., 2011; Rabah, 2008). Recently, with the rapid increase in prices of rare earth metals, the price of phosphor powder is also skyrocketing. However, most mercury is contained in phosphor powder from SFLs so it should be removed using recovery technologies in order to obtain rare earth metals from phosphor powder.

There are mainly two kinds of mercury recovery technology from SFLs; dry technology and wet technology. Dry technology uses the low boiling point of mercury to roast mercury-containing material at a high temperature to volatilize mercury and then cool and condense again (Ma et al., 2014). With wet technology, mercury is oxidized using an absorbent to recover or remove it in mercuric oxide form. With respect to wet technologies, in general, large amounts of energy may be consumed in drying phosphor powders to apply the recovery technology (Rey-Raap and Gallardo, 2013). Lee et al. (2015) studied industrial recycling

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technologies and management strategies of end-of-life fluorescent lamps in Taiwan and other developed countries. In Taiwan, both subsidy and lamp processing fees are available to improve the recovery rate of end-of-life fluorescent lamps.

In order to recover mercury from phosphor powder, thermal stabilization among dry technologies can be used by heating at above 300–600 °C because the temperature in thermal stabilization should be higher than the boiling point (356.6 °C) of elemental mercury. To remove mercuric oxide form in phosphor powder, the temperature in thermal stabilization may be heated about 400–600 °C because the boiling point of mercuric oxide form is higher than that of elemental mercury (Raposo et al., 2003). In thermal stabilization, mercury vapor can be recovered via condensation using air or water with cooling temperature to collect liquefied mercury and mercury vapor, which is not condensed but passes through the condensation process, will be controlled by adsorption treatment with activated carbon in order to meet air quality standard.

Thermal stabilization can be explained by a modified retort process, which is described by a rotating retort drum with baffle to rotate phosphor powder without attaching to the wall of the drum. Using thermal stabilization, mercury-free phosphor powder and mercury can be recovered separately by evaporating mercury in phosphor powder and condensing mercury from vapor phase, respectively. For the thermal technique, removal efficiency of mercury significantly depends on temperature. Several studies reported that temperatures of 400–600 °C are not high enough to remove all the mercury from phosphor powder because part of the mercury is strongly bonded in mercuric oxide form to phosphor powder (Chang et al., 2009; Horvat et al., 1993; Rey-Raap and Gallardo, 2013).

To recycle spent fluorescent lamps, phosphor powder can be collected using two kinds of methods. The first method is via air injection. After cutting the base cap in the spent fluorescent lamp, air is injected into a glass tube to collect phosphor powder. In the second method, mechanical separation, phosphor powder attached to a glass tube was completely separated by a mechanical process. As separation time increases, however, glass could be grinded by mechanical separation. Glass powder can be contained in phosphor powder as an impurity changing mercury content in phosphor powder.

In this study, the rotating retorted system is used to control mercury content in phosphor powder by using a pilot plant scale. The degree of thermal stabilization of phosphor powder from SFLs is estimated by examining mercury content in phosphor powder with retorting time, retorting temperature and rotational speed of drum. In order to analyze mercury content in phosphor powder, DMA (Direct Mercury Analyzer) method is used. After thermal stabilization for all samples of phosphor powder, leaching tests such as toxicity characteristic leaching procedure (TCLP) and Korea Extraction Test (KET) are carried out to verify if the stabilized phosphor powder is hazardous waste.

2. Material and method

2.1. Materials

Phosphor powders were collected from spent linear fluorescent lamps (40 W) manufactured by three different lamp companies (sample A, B, C) in Korea for thermal experiments at a pilot plant scale. Since 10 kg of phosphor powder was used in experimental tests, more phosphor powder should be collected. Hence, a 40 W lamp was used because about 4 g can be collected from each 40 W lamp and about 2 g from each 20 W lamp. All samples of phosphor powder came from fractured glasses of spent linear flu-

orescent lamps. The particle size of phosphor powder was estimated using a sieving device to show any degree of impurity, including any glass powder.

2.2. Experimental apparatus

The pilot plant scale thermal stabilization apparatus used in this study consists of a heating drum, cooling tower, and adsorption column as shown in Fig. 1. In the thermal stabilization experiments, a 10 kg sample of each phosphor powder was used in a batch system. The operating mechanism of thermal stabilization to control mercury content in phosphor powder can be described by the heating and rotating technique in a heating drum with baffles. The temperature in the heating drum can be raised up to 700 °C and the rotating speed can also be controlled up to 5.0 rpm to sustain the movement of phosphor powder in the heating drum.

Baffle plates placed in a heating drum ensures that the effect of mixing of phosphor powder in the heating drum remains homogeneous, which is suitable for treatment of mercury containing wastes. Mercury vapor is released from the heating drum and moved to the cooling tower for mercury, and then subsequently adsorbed onto mercury-specific activated carbon in adsorption tower to control the emission of mercury.

2.3. Experimental method

In the experiment using the thermal stabilization apparatus, the rotational speed of the heating drum, retorting temperature and retorting time were modified to estimate the mercury content in phosphor powder. Rotational speed was changed from 0.0 rpm to 3.0 rpm at a temperature of 400 °C and retorting time of 6 h. The retorting temperature was changed from 200 °C to 600 °C at rotational speed of 2.0 rpm and retorting time of 6 h. Finally, the retorting time was varied from 1 h to 8 h at a retorting temperature of 400 °C and rotational speed of 2.0 rpm.

In KET, the 100 g sample of components was used with the 1000 mL solution at an initial pH range of 5.8–6.3. In order to adjust pH, HCl was used a reagent. After making the solution, the sample and the solution were placed into a 1000 mL Erlenmeyer flask and shaken at a 4–5 cm vibration range continuously for 6 h at 200 rpm in room temperature. The shaken solution was filtered using a glass fiber filter with a pore size of 1.0 µm.

In TCLP, the 100 g sample of components was used with the 2000 mL solution. Two kinds of solution could be used in the test: fluid #1 and fluid #2. In this study, fluid #1, which has a pH of 4.93 ± 0.05 , was used as a leaching solution. If the pH of the solution is adjusted, acetic acid is used. After preparing the solution, the sample and the solution were placed into a 2000 mL Erlenmeyer flask and shaken continuously in a rotating shaker at 30 ± 2 rpm for 18 ± 2 h at 23 ± 2 °C. The shaken solution was filtered using a glass fiber filter with a pore size of 0.6–0.8 µm.

All mercury analyses for the parts of SFL were based on the principles of thermal decomposition, amalgamation and detection by atomic absorption as described in EPA Method 7473 (Mercury in Solid or Semisolid Waste – Manual Gold Amalgam Technique with atomic absorption spectrophotometer) using DMA 80 (Direct Mercury Analyzer) to analyze mercury content. In the analysis using DMA 80, the amount of sample used was about 3 g of phosphor powder. An Atomic Absorption Spectrometer (AAS) was used to analyze heavy metal concentrations in the leaching solution from KET and TCLP. Measurements were conducted 3 times for every sample and mercury concentration was estimated from their average value.

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