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Selective recovery of silver from waste low-temperature co-fired ceramic and valorization through silver nanoparticle synthesis

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ABSTRACT

Considering the value of silver metal and silver nanoparticles, the waste generated during manufacturing of low temperature co-fired ceramic (LTCC) were recycled through the simple yet cost effective process by chemical-metallurgy. Followed by leaching optimization, silver was selectively recovered through precipitation. The precipitated silver chloride was valorized through silver nanoparticle synthesis by a simple one-pot greener synthesis route. Through leaching-precipitation optimization, quantitative selective recovery of silver chloride was achieved, followed by homogeneous pure silver nanoparticle about 100 nm size were synthesized. The reported recycling process is a simple process, versatile, easy to implement, requires minimum facilities and no specialty chemicals, through which semiconductor manufacturing industry can treat the waste generated during manufacturing of LTCC and reutilize the valorized silver nanoparticles in manufacturing in a close loop process. Our reported process can address issues like; (i) waste disposal, as well as value-added silver recovery, (ii) brings back the material to production stream and address the circular economy, and (iii) can be part of lower the futuristic carbon economy and cradle-to-cradle technology management, simultaneously.

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1. Introduction

Low-Temperature Co-fired Ceramic (LTCC) is made from multi-layer glass ceramic substrate co-fired with highly conductor metal (Ag or Cu) is a versatile material has a much wider range of electrical, mechanical and thermal properties than the silicon. Because of light, compact, high-speed and better functionality LTCC are especially suitable for the high frequency (RF) circuits those requires for high-speed data communications (Imanaka, 2006). For portable electronic devices, such as; the cellular phones, personal digital assistants (PDA), personal computers and devices used for wireless voice and data communication LTCC is an attractive material. LTCC has become an attractive technology for electronic components and substrates that are compact, light, and offer high-speed and functionality (Imanaka, 2006). The LTCC is a very selective and versatile junction material between microelectronics and microelectromechanical systems (MEMs) (Gongora-Rubio et al., 1999, 2001). The LTCC are being used for multi-chip ceramic modules for automotive, radio frequency (RF) microwave circuits, and various sensor and MEMs. The multi-chip modules application includes automotive in transmission control unit (TCU), electronic

power steering (EPS), electronic stability control (ESC), engine management system (EMS), various sensor modules, radar modules, pressure sensor, and radio frequency (RF) microwave circuits application includes power amplifier modules (PAM), LTE-advanced Modules (Maruta Manufacturing Co. Ltd, 2017; Global trade media, 2017). Application of LTCC continuously expanding (Claeys et al., 2004).

From industrial production and the manufacturing perspective, Asia-Pacific region is the largest producer of the LTCC product as well as industrial waste during manufacturing (marketreportsworld.com, 2017; QYResearchGroup, 2017). Considering production Asia-Pacific region is also producing the highest volume of the LTCC industrial waste during manufacturing. Massive production and demand lead to waste generation during the manufacturing process in the industry and end-of-life (EOL) will cause serious environmental problems. The scarcity of land, higher cost of land and stringent-landfilling policies motivate these wastes must be managed properly and the best answer for waste management should be a recycling of these LTCC. More importantly, these LTCC waste generated during manufacturing contains a significant amount of Ag, make these wastes a valuable commodity to be recycled. Considering energy and environment, the pyrometallurgical process for recovery of Ag from this LTCC industry waste-limiting the pyro process development interest. Hence,

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chemical metallurgy can be a suitable technology for recovery of these metals. Several authors have reported recovery of precious metals through recycling of e-waste or electronic industry waste by hydrometallurgy process (Behnamfard et al., 2013; Cayumil et al., 2016; Park and Fray, 2009). Akcil et al. have reviewed the precious metal leaching using cyanide and non-cyanide lixiviants (Akcil et al., 2015). Not only the precious metal recovery from e-waste but also environment-friendly technologies for recover/recycling resources from various e-waste has been reported in the literature (Ruan and Xu, 2012, 2016; Ruan et al., 2017b; Swain et al., 2015a, 2016). Several authors have reported various strategies for e-management or reviewed the status of e-waste in the different part of the world (Chung et al., 2011; de Souza et al., 2016; Dwivedy and Mittal, 2010; Favot and Grassetti, in press; Golev et al., 2016; Kaya, 2016; Li et al., 2014; Nnorom and Osibanjo, 2008; Nowakowski, 2017; Pant et al., 2012; Ruan et al., 2017 a; Singh et al., 2016; Song and Li, 2014). Whereas report regarding selective recovery of Ag from industrial LTCC waste never been reported. As leaching is the primary and essential stage for recovery of the metals these wastes were leached using mineral acid and the leaching process was optimized followed by valorization. Leached Ag was valorized through Ag nanoparticles (NPs) synthesis. Selective recovery of Ag through chemical metallurgy and valorization through one pot material synthesis relatively scarce.

Along with the conventional use of Ag, currently, Ag nanoparticles (NPs) are being used in numerous technologies and incorporated into an extensive array of consumer products to take advantage of their desirable optical, conductive, and antibacterial properties (Li et al., 2010; Lubick, 2008). The Ag NPs application includes diagnostic, antibacterial, conducting coating, conducting ink, plasmonic antennas, molecular sensing (Li et al., 2010; Lubick, 2008; Zhang et al., 2000). The Ag NPs are also used to harvest light, enhanced optical spectroscopies including metal-enhanced fluorescence (MEF) and surface-enhanced Raman scattering (SERS). Several authors have reported the synthesis of Ag NPs through wet chemical reduction using a reductant and stabilizing/surfactants/capping reagent (Ajitha et al., 2016; Jacob et al., 2007; García-Barrasa et al., 2011; Lubick, 2008; Silvert et al., 1996; Tejamaya et al., 2012; Zielińska et al., 2009). Investigation reveals that Polyvinylpyrrolidone (PVP) used as a stabilizer and Polyethylene glycol (PEG) used as reducing reagent as well as stabilizing reagent to control size and shape of the Ag NPs (García-Barrasa et al., 2011). Considering the application of Ag NPs, development of cost-effective environment-friendly process, the waste management strategy through valorization of Ag NPs synthesis has been applied in our reported process. In the valorization process; followed by leaching, the Ag was selectively precipitated as AgCl and from AgCl precipitate homogeneous Ag NPs about 100 nm size has been synthesized and reported in this paper. In this investigation, Ag leaching from the industrial waste of LTCC was optimized followed by selective recovery by the valorization of Ag as NPs has been optimized for economy, environment perspective. Both selective quantitative recovery of Ag and high pure Ag NPs about 100 nm size could be achieved by the reported process. The importance and novelty of the reported waste management process for waste LTCC explained below.

1. The LTCC industrial waste recycling signifies a process from waste to wealth through simplest recycling and valorization process.
2. The process can be a cradle-to-cradle waste management strategy, where, in the same industry from a waste LTCC, Ag can be recover and valorized as NPs, which can be used for remanufacturing of LTCC material. As the process is simple and required no special facilities or specialty chemicals, hence, through small

scale industrial facility the waste can be converted to product/raw material for the same industry.

3. Most of the Ag NPs preparation reported in the literature are from the pure chemicals but in the investigation waste, LTCC has been used as the precursor.
4. Our process offers a versatile and flexible approach for mass production capability.
5. As reported waste recycling process is a simple, versatile, greener process, which is easy to implement, requires minimum facilities and no specialty chemicals. Through which semiconductor manufacturing industry can address various issues like; (i) waste disposal, as well as value-added Ag recovery, (ii) brings back the material to production stream and address the circular economy, and (iii) can be part of cradle-to-cradle technology management and lower the futuristic carbon economy, simultaneously.

2. Materials and methods

2.1. Materials

The Ag-rich LTCC waste was collected from the industry, followed by the various shaped and sized waste material was powdered using mortar and pestle. Other chemicals like HNO₃, HCl, NH₄OH, and NaBH₄ were of analytical grade supplied by Dae-Jung chemical and metal Co, Ltd, the Republic of Korea. Polyvinylpyrrolidone (PVP) and Polyethylene glycol (PEG) were supplied by Sigma-Aldrich, Korea.

2.2. Apparatus

A leaching reactor reported elsewhere (Swain et al., 2015b) was used for leaching of the Ag-rich LTCC industrial waste. The main reactor vessel is 500 ml round-bottomed three-necked flask equipped with a condenser and thermocouple. A heating mantle was used for heating, equipped with a thermostat to control the reactor temperature. A thermocouple equipped with digital measurement of temperature during continuous operation of the reactor was used to monitor temperature during leaching. A magnetic stirrer was used for stirring.

2.3. Leaching and Ag NPs recovery procedure

All the leaching experiments were carried out using the reactor mentioned above. The HNO₃ was used as lixiviant. The required volume of lixiviant was poured into the reactor and allowed to reach thermal equilibrium, and then the weighted amount of waste LTCC was added to the reactor and agitated with require speed simultaneously controlling speed and heating. Two ml of the leach samples was drawn periodically at the desired time interval. Various process parameters such as; lixiviant concentration, pulp density and temperature were optimized. Through filtration, solid-liquid (residual solid and leach liquor) was separated. The Ag was precipitated as AgCl selectively from leach liquor using HCl and characterized by XRD and ICP-AES. Followed by, the AgCl powder was dissolved in NH₄OH and reduced to pure Ag NPs using NaBH₄ as a reductant. NaBH₄ was used for wet chemical reduction of AgCl to metallic Ag and PVP, PEG was used as control the morphology and size of the Ag nanoparticles (NPs).

2.4. Analytical procedure

Concentrations of Ag, Al, Fe, Sr and Zn after leaching were measured using ICP-AES (Agilent 4200, USA) after suitable dilution using 5 v/v% of HNO₃. The maximum deviations permitted were

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