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#### Research article

# Enhanced alumina recovery from secondary aluminum dross for high purity nanostructured $\gamma$ -alumina powder production: Kinetic study

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#### ABSTRACT

The numerous ecological problems caused by the accumulation of secondary aluminum dross (SAD) as a hazardous waste generated in aluminum castings have necessitated a need for a sustainable recycling solution. This study proposes a novel and green leaching-based process for recovery of nano-alumina as a highly valuable material from SAD. The leaching phase was performed at atmospheric pressure and low temperature. To obtain optimum conditions for the recovery process, the dissolution kinetics and mechanism of SAD in hydrochloric acid were initially studied under a given liquid-to-solid ratio of 20 ml/ g, various reaction temperatures and times. It was found that the dissolution of SAD in HCl was controlled by layer diffusion with an apparent activation energy of around 10.49 kJ/mol. Alumina in the form of a high purity nanostructured powder from SAD was then recovered under optimum conditions of 85 °C, acid concentration of 5 mol/l, and reaction time of 120 min by the proposed process. Characterization of nano-alumina was performed using X-ray diffractometry (XRD), Fourier transform infrared (FTIR) spectroscopy, field emission scanning electron microscopy (FESEM), energy-dispersive X-ray spectroscopy (EDAX), and transmission electron microscopy (TEM) techniques. The results revealed that the as-produced alumina had a nano-crystalline structure, having the crystal size of 15.90 nm and consisting mainly of gamma phase. The microstructural studies disclosed the aggregations of roundedcorner shaped particles. Also, wet chemical analysis showed a purity of more than 98% for the produced alumina

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#### 1. Introduction

The improper management of various types of solid wastes (e.g. industrial, municipal and agricultural) results in serious environmental outcomes such as water toxicity (Melnyk et al., 2014). One way to manage the waste is to recycle or reuse it as the feedstock for production of valuable products (e.g. biodiesel, biofuel, etc.) (Hanifzadeh et al., 2017; Kim et al., 2017). One of the industrial solid wastes with many environmental issues is aluminum dross. It is a solid oxidic compound waste of aluminum smelting process formed when molten aluminum comes in contact with air in its outer surface (Dash et al., 2008; Sarker et al., 2015). For every 1000 kg of molten aluminum, an average of about 15–25 kg of aluminum dross is generated (Nai and Ming-Shean, 2013).

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Chemical and mineralogical composition of aluminum dross depend on raw material and metallurgical process used in production (Das et al., 2007). The main phases present in aluminum dross include aluminum metal, Fe<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub>, Na<sub>2</sub>O, AlN, Al<sub>2</sub>O<sub>3</sub>, NaCl, MgO·Al<sub>2</sub>O<sub>3</sub>, and traces of KCl, cryolite (Na<sub>3</sub>AlF<sub>6</sub>), aluminum carbide (Al<sub>4</sub>C<sub>3</sub>), and small amounts of fluoride and chloride compounds. In other studies, however, components such as MgF<sub>2</sub>, NaAlCl<sub>4</sub>, KAlCl<sub>4</sub>, MgO, KMgF<sub>3</sub>, and K<sub>2</sub>NaAlF<sub>6</sub> have also been reported in addition to the aforementioned chemicals (Dash et al., 2008; Sarker et al., 2015). Aluminum dross can also be generated from treatment of molten aluminum scraps such as drinking cans with molten salt flux (Manfredi et al., 1997). The molten salt flux accumulates on the surface of the molten metal resulting in the formation of dross or skim (Adeosun, 2014). There are two types of aluminum dross including primary dross (also called white dross or wet dross) (Abdulkadir et al., 2015) and secondary dross (other common names: black or dry dross) (Adeosun, 2014; Yoo et al., 2011). These two types of dross are different from each other in terms of aluminum metal content and the amount of soluble salts and oxidic







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compounds (Yoo et al., 2011). Primary dross contains approximately 80% of aluminum metal and small amounts of oxidic and salt constituents in the form of clump (Abdulkadir et al., 2015), whereas secondary dross contains about 5-10% of aluminum metal and large amounts of oxidic and salt compounds (Adeosun, 2014; Nai and Ming-Shean, 2013). Primary dross is generally returned to smelter for the purpose of recycling of remaining aluminum metal. At this stage, the salt flux is used to minimize oxidation (Dash et al., 2008; Nai and Ming-Shean, 2013). Salt flux is mainly prepared based on an equimolar combination of NaCl-KCl with about 10% of fluoride additives such as NaF, cryolite, or KF to improve coagulation and therefore the melting process (Ramesh and Yogeshwar, 1997). The oxides present in dross form a long continuous network in which aluminum metal has been trapped. The molten flux smashes this framework, thereby releasing the metal trapped inside. As a result, coalescence of molten aluminum droplets is facilitated and eventually, the droplets flow down into the aluminum bath. In most cases, salt flux is applied to maximize the recycling of aluminum metal and also to reduce the amount of impurities in final aluminum products (Das et al., 2007; Dash et al., 2008). In some cases, various alloying elements are utilized along with the salt flux for the production of aluminum alloys. The dross obtained at this stage is known as secondary dross or alloy dross (Dash et al., 2008).

Disposal of the secondary dross generated in the process of aluminum smelting is a global issue and a serious challenge for aluminum manufacturers from the view point of ecological problems (Adeosun, 2014; Das et al., 2007). A large portion of the produced secondary dross is discarded in the vicinity of factories that more likely causes the toxic metal ions to enter the surface and



Fig. 1. The experimental setup used for the SAD dissolution tests.

underground water consequently resulting in serious concerns of water pollution (Meshram and Singh, 2018). In addition, when aluminum dross comes in contact with moisture or water vapor, toxic and flammable gases such as NH<sub>3</sub>, CH<sub>4</sub>, PH<sub>3</sub>, H<sub>2</sub>, and H<sub>2</sub>S etc. may be generated (Das et al., 2007; Murayama et al., 2012). Due to the aforementioned issues, the adoption of an appropriate recycling solution is of particular importance.

There are two possible ways to manage secondary dross: pyrometallurgical and hydrometallurgical processes. The former is faced with the problem of high energy consumption and usually requires relatively high temperature treatments and reducing agents (Unlu and Drouet, 2002). Since the latter is a method with low capital and production costs, together with the high efficiency and favorable environmental aspects (Havlik, 2008), it can be a promising route for the future and an interesting way to recover and process secondary dross. Hydrometallurgical processes can be performed through alkaline or acidic routes (Tsakiridis et al., 2013). An accurate understanding of the kinetics of dissolution is required in order to interpret the complex behavior of leaching process, and to optimize the performance of hydrometallurgical operation (Irena et al., 2016). Therefore, the study of dissolution kinetics of secondary aluminum dross in acid or base can be of great importance, especially for the purpose of the production of nanostructured alumina powder as a very high valuable material. The dissolution kinetics of aluminum dross in hydrochloric acid and the ratedetermining step (RDS) of the dissolution reactions have not been reported in the literature, vet. The first aim of this work is to investigate these issues for the first time. As the second aim within the scope of this study, the preparation of nanostructured alumina powder using a green leaching-based process will be discussed.

#### 2. Materials and methods

#### 2.1. Materials

Secondary aluminum dross (SAD) was supplied from an aluminum company located in Arak province, Iran. Hydrochloric acid (37 w/w%), sodium hydroxide (99% purity), and ammonia 25% were purchased from Merck (Darmstadt, Germany) and used without any additional purification. Distilled water was used at all stages of the experiment.

#### 2.2. Experimental procedure

#### 2.2.1. Dissolution experiments

After sampling by quartering and coning method (Brittain, 2002) and before performing any dissolution test, the sample was dried in an oven at 105 °C. This is done to neutralize the effect of the moisture content of the sample in the dissolution efficiency calculations. For the purpose of dissolution experiments of the SAD in hydrochloric acid, the experimental setup (shown in Fig. 1) including a three-neck glass reactor, Graham condenser, water bath, and magnetic stirrer equipped with a temperature probe (IKA C-MAG HS7 Digital) was used. After carefully weighing, 5 g of the SAD was poured into the reactor at the specified temperatures and atmospheric pressure. Then, 100 ml of HCl with optimum concentration was added to it. The reaction between the SAD and hydrochloric acid begins under a certain stirrer speed (500 rpm). After any dissolution experiment at the given temperature and time, the suspension was filtered through an ash-less filter paper of Whatman to separate the insoluble residues. After burning the filter paper inside the crucible, the solid residues were washed several times with distilled water and then dried at 105 °C in an oven. Finally, after the weight of the crucible was fixed, the amount of solid residues was obtained. For temperatures higher than ambient Download English Version:

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