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# Model and experimental investigations of aluminum oxide slurry transportation and vaporization behavior for nebulization inductively coupled plasma optical emission spectrometry

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ABSTRACT

We analyzed aluminum oxide ($Al_2O_3$) by slurry introduction inductively coupled plasma (ICP) optical emission spectrometry through modeling and experimentation. We also studied the relationship between the ICP nebulizer gas flow, the spray chamber geometry, and the particle size of $Al_2O_3$ in an attempt to minimize the need for correction factors by ensuring an efficient aerosol mass transport. A cut-off point for the particle size was implemented at approximately 7–10 μm for the sample introduction system. Based on modeling using a customized computer model and some experimental evidences, the maximum particle size for complete vaporization is approximately 7 μm. For a gas flow of 0.8 L $min^{-1}$, particles with a diameter of up to 8 μm can be evaporated with an efficiency of 68% and particles as large as 5 μm can be evaporated completely within the nebulization gas flow region. The $Al_2O_3$ sintering block was ground using a self-made alumina mortar combined with a mixer mill device for particle reduction. The sample slurry was prepared by directly dispersing powdered $Al_2O_3$ in an aqueous solution with an addition of 0.5 wt% poly (acrylate amine) ($NH_4PAA$) as the dispersant. The accuracy of the results was compared with the data obtained through high-pressure digestion with acid and with the value of a certified reference material, NIST SRM 699 alumina.

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## 1. Introduction

Slurry introduction combined with inductively coupled plasma optical emission spectrometry or mass spectrometry (ICP-OES/MS) is a feasible way to analyze powdery materials directly. Researchers have verified the effectiveness of the procedure for the analysis of powdery samples such as coal [1,2], geological materials [3,4], cement [5,6], biological materials [7,8] and size-tailored magnetic colloids [9]. This technique can also be used to characterize ceramic powders, which are being developed for numerous applications because of their desirable features. The increasing applications of high-tech ceramic materials in various fields of science and technology indicate that desirable properties of ceramics must be further investigated. These properties are often directly correlated with the contents of trace impurities. The direct analysis of powders by ICP-OES is desirable to avoid contaminations and losses of analytes as well as lengthy procedures associated with digestion. Therefore, powerful, rapid, and reliable analytical methods are required for material characterization. Several papers have reported the use of slurry introduction in ceramic analysis [10–21]. Analytical methods based on slurry introduction have several advantages, namely, ease of use and rapid analysis. However, the complexities of this approach, in which solid samples are directly analyzed in an aqueous slurry of a fine powdery material, are apparent. A highly stable and homogeneous slurry is required for precise and accurate analytical results [22]. In analyzing ceramic materials, the particle size of the starting powders is a critical factor. Many studies have reported that slurry particles larger than 5 μm (in several studies, 2 μm) do not reach the plasma, which results in signal loss [23]. In our previous study, we reported a particle size of titanium nitride powders in the μm level (80% of the powdered particles were less than 10 μm and had a mean diameter of 4.9 μm) by using slurry nebulization ICP for impurity analysis [24]. All analytical results were below the average compared with the results of fusion-prepared samples. Therefore, all sample results were multiplied by a correction factor of five to obtain a semi-quantitative method for rapid screening analysis. When refined aluminum nitride powders were used (most powdered particles were smaller than 7 μm with a mean diameter of 1.9 μm) and with the aid of dispersants, several elements with high boiling points, such as

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Si and Y, achieved a signal intensity of only 40–60% compared with equivalent aqueous standards [25].

The strong dependence of recovery on the particle size distribution of the slurry has particular implications for calibration. The use of a slurry standard or empirical correction factors for calibration is risky. Relatively subtle changes in the particle size distribution of the sample slurries are useless to the calibration procedure and can degrade the precision of the analysis [26]. Aqueous standards should be used for calibration to shorten the analysis time in slurry nebulization ICP-OES. This calibration should meet two requirements, namely, the analyte transport of slurry particle and the subsequent vaporization efficiency of that particle must be identical to the nebulization of the simple aqueous solution [10]. When the conditions are not met, the analytical accuracy of slurry nebulization ICP-OES decreases. Many attempts have been made to overcome these problems. Numerous studies have investigated the model transportation and vaporization behavior of aluminum oxide ($Al_2O_3$) and SiC-slurries that were introduced via pneumatic nebulization [27]. In addition, comparative investigations have described the nebulization of slurries and solutions with a Babington nebulizer by using different gas flows. McCurdy and Fry [28] have experimentally demonstrated that coal particles with sizes of up to 17 μm can still reach the plasma. However, Ebdon and Collier [29] reported that quantitative transportation and atomization of kaolin by slurry nebulization with a Babington nebulizer can only be performed when the maximum particle diameter is smaller than 6–8 μm.

In the current paper, we investigated the behavior of alumina slurry particles in the transportation and vaporization process of suspension nebulization ICP-OES to determine the size requirement of slurry nebulization in ICP. A simple and easy method for grinding $Al_2O_3$ sintering blocks in our laboratory is presented. The analytical results obtained using slurry nebulization ICP-OES with aqueous standard calibrations are evaluated in comparison with the data obtained by high-pressure digestion with acid in a sealed vessel and with the value of a certified reference material, NIST SRM 699 alumina.

## 2. Experimental

### 2.1. Instrumentation

All analyses were performed using a VISTA AX ICP-OES spectrometer with an axially viewed configuration (Varian, USA). The sample introduction system consists of a V-groove nebulizer and a reduced-volume Sturman-Masters Type spray chamber made of polytetrafluorethylene. The operating parameters and the selected analytical lines are listed in Table 1. The prepared sample solution or slurry was introduced by a cross nebulizer with a V-groove and was measured by an axially viewed ICP-OES spectrometer. Mixer mill MM40, which has a grinding jar (tungsten carbide, 25 mL, screw top design) (Retsch GmbH Co.), was used for particle reduction.

### 2.2. Transport behavior of slurry particles

Researchers have determined the diameters in which a particle can be transported to the plasma [26,27,30–32]. The upper limit of the particle size that can be transported into the plasma torch and the effects related to the transport behavior of particular materials from those caused by vaporization were determined by both the particle size distribution of the original ceramic powders and after the pneumatic nebulization of the slurries. A self-made device shown in Fig. 1 was used to investigate the aerosol produced by pneumatic nebulization of the slurry. The aerosol that left the spray chamber and/or crossed the injector torch was trapped in water and collected by the particle collection device.

**Table 1**
Instrumentation, operating conditions and selected analytical lines.

| Spectral range | 167–785 nm | | |
|---|---|---|---|
| Viewing | Axial | | |
| RF generator | 40 MHz | | |
| Torch | All-quartz | | |
| Injector tube diameter | 2.3 mm | | |
| Power | 1.25 KW | | |
| Plasma flow | 15 L/min | | |
| Auxiliary flow | 1.5 L/min | | |
| Nebulizer flow | 0.65 L/min | | |
| Sample uptake rate | 0.8 mL/min | | |
| Analytical lines | | | |
| Element | Spectral line(nm) | Element | Spectral line(nm) |
| Ca | 317.933 | Na | 589.592 |
| Cr | 206.149 | Ni | 231.604 |
| Cu | 324.754 | Pb | 220.353 |
| Fe | 238.204 | V | 309.310 |
| Ga | 294.363 | Si | 251.611 |
| Li | 610.365 | Ti | 336.122 |
| Mg | 285.213 | Zn | 213.857 |
| Mn | 257.610 | | |

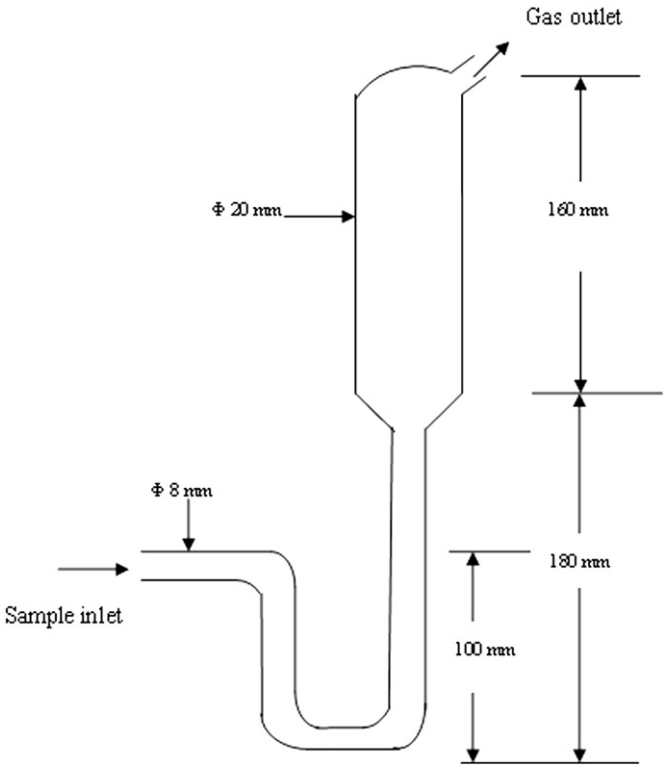


**Fig. 1.** Particle collection device.

### 2.3. Vaporization behavior of slurry particles

$Al_2O_3$ is a material with extreme thermal properties (melting temperature: 2303 K, vaporization temperature: 4548 K) [27,33,34]. On the other hand, the complete vaporization of $Al_2O_3$ in the plasma is difficult to achieve because of its stability. Thus, a negative deviation occurs when obtaining the results. In this paper, we estimated the vaporization behavior of the slurry particles by using the model developed by Merten and Broekaert as basis [27]. The

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