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# Designing of hollow AgI spheres by ultrasonic spray pyrolysis

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ABSTRACT

A theoretical model which describes the mechanism of droplet formation, structures of the complexes used in the precursor solution and hollow AgI particles prepared by the process of ultrasonic spray pyrolysis (USP) was investigated. Theoretical approaches described very well the properties of silver iodide particles collected and aged in 2-propanol after the USP process. The morphology of hollow silver iodide particles aged in 2-propanol was found to be connected with the mechanism of surface precipitation caused by low precursor density. In addition, the structures of small complexes used are directly connected with structural changes in the synthesized hollow AgI particles. The proposed model and experimental results imply that the structure of the starting thermodynamically stable AgI complexes, used for the first time as a precursor solution, affects the final particle structure.

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## 1. Introduction

The ability to manipulate matter and to assemble structures on the nanometer scale has been the goal of many researchers in materials sciences for a long time. Nanostructures of a few nanometers are collections of assemblies of atoms (Jayanthi et al., 1993;Ichinose et al., 1992). For aerosols, these include structures formed by deposition of individual nanometer-size particles onto "nanophase" materials (Gurav et al., 1993). In fact, the particle processing using aerosol-assisted self-assembly techniques to fabricate particles with controllable size and morphology have been extensively studied (Nandiyanto & Okuyama, 2011;Eslamian & Ashgriz, 2006; Milosević et al., 2009; Wang et al., 2011).

Recently, it has been shown that AgI particles synthesized during the process of ultrasonic spray pyrolysis (USP) and collected in different solvents such as water, 2-propanol and toluene exhibit extraordinary structural and morphological properties with aging (Validžić et al., 2008). Aqueous suspensions of thermodynamically stable silver iodide clusters were used as starting materials in the process of USP (Mladenović et al., 2003; Validžić & Kegel, 2004). The scanning electron microscopy showed that morphologies of AgI particles are different for the samples aged in different solvents (hexagonal/ triangular shape and hollow spheres were formed). The X-ray diffraction analysis revealed the tetragonal high-pressure AgI modification besides the cubic and hexagonal (Validžić et al., 2008). Further in the paper we shall concentrate on the AgI particles collected and aged in 2-propanol.

In this paper, two approaches (Lang, 1962; Peskin & Raco, 1963) were used which describe the mechanism of droplet formation and its transformation into hollow spheres of silver iodide particles, prepared by the process of ultrasonic spray pyrolysis. Comparing various theoretically calculated results for average particle diameters with those obtained experimentally, it was possible to estimate the average wall thickness of the hollow spheres produced through surface

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precipitation of the USP droplets. Using conditions that provide only the surface precipitation of the droplets inside the furnace, the main idea was to attempt to realize very soft contacts between the clusters during the pyrolysis, which would influence the final particle structure of the system. It is well known that when the droplets are transported into the furnace tube the surface precipitation of the dissolved substances within the droplet occurs during the particle formation, which causes the formation of the hollow structure (Zhang et al., 2007).

It was found that the theoretical model of structural design successfully described the system with respect to its basic design element, in our case, small thermodynamically stable complexes of $Ag_4I_6^{2-}$. The morphology of hollow silver iodide particles aged in 2-propanol was found to be connected with the processes occurring during and after the USP, most likely with surface precipitation. On the other hand, the nature and structure of the starting complexes allow us to design the experiment and reach the desired morphology.

## 2. Experimentation

All chemicals-silver nitrate ($AgNO_3$), potassium iodide (KI) and 2-propanol (pure grade)—were purchased from Merck and were used without further purification. Aqueous solutions of thermodynamically stable AgI clusters, used as a starting material in the process of USP, were prepared as described in the literature (Mladenović et al., 2003). Briefly, 1.25 M $AgNO_3$ was combined with 5 M KI. After precipitation of AgI, supernatant solution containing thermodynamically stable AgI clusters was separated and used in further work.

The AgI powders were obtained in the process of USP, using solutions containing AgI clusters as a starting material. Briefly, laboratory setup for USP (see Fig. 1) consists of an ultrasonic atomizer (GAPUSOL-RBI-91-012) operating at a frequency of 1.7 MHz for aerosol generation, and a horizontal electric furnace with a quartz tube and a vessel for particle collection. The effective heating length of the reactor tube was 1.25 m and the temperature was 300 °C. In fact, the reaction furnace consisted of five heating zones, each 0.2 m long. The temperature of each heating zone was controlled within 5 °C using a temperature controller; $T_1$, $T_2$, $T_3$, $T_4$, and $T_5$ indicate the wall temperatures in the middle of each heating zone. Setting five zones provides the desired temperature distribution. The flow rate of air was 30 L/h. The flow rate of aerosol droplets was assumed to be equal to the flow rate of air carrier, and the residence time of aerosol droplets in the furnace was found to be 1 min.

The obtained AgI powders were collected in 2-propanol. In the first series of experiments, AgI particles were separated from the solvent containing excess KI immediately after the synthesis, while in the second set of experiments the aging time of AgI particles was two weeks. The separation process was performed using ultra-filtration through a Millipore membrane with a pore size of 10 nm.

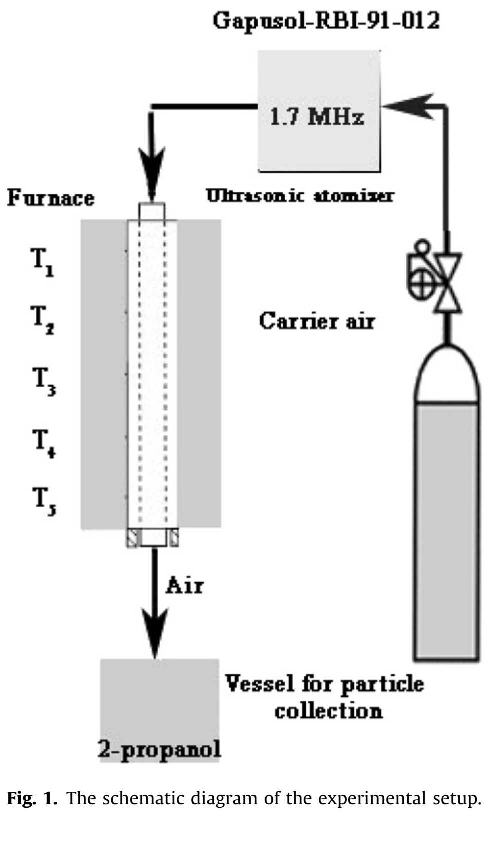


**Fig. 1.** The schematic diagram of the experimental setup.

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