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Effect of acid leaching time on pore diameter and volume of porous hollow glass microspheres

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ABSTRACT

Porous hollow glass microspheres have attracted attention in many applications because of their wall structure and storage ability. In this study, The effect of acid leaching time on porosity properties of porous microspheres was investigated. Hollow glass microspheres with wall thickness of 0.5–2 μm were fabricated from borosilicate glass frits in oxy-acetylene flame. Microspheres were heat treated and acid leached to produce porous hollow glass microspheres. Thermal analysis indicated appropriate temperature for the heat treatment and creation of adequate phase separation. According to the results, the various acid leaching times lead to different range of pore volume and sizes.

1. Introduction

Porous glasses are matrix which represents a rigid sponge-like SiO_2 framework that can be filled with different materials [1]. They are widely used in various fields of science and technology because of their unique properties (such as catalyst supports, photo catalysts, sorbents, gas sensors, etc.) [2,3]. This kind of glasses can be synthesized by different methods: in the conventional ways such as foaming or phase separation and leaching process, and also by the sol–gel method, or by geological alteration process of volcanic materials [4].

Porous hollow glass microspheres (PHGMs), with interconnected porosity in their thin outer walls, have attractive characteristics such as high and controllable permeability, low density and storage capacity [5]. Open channels allow the spheres to be a media for safe hydrogen storage of metal hydrides compounds [6,7]. Porous, hollow, glass microspheres as additives can be used to increase porosity in the electrodes for more electrolyte storage in lead acid batteries and enhance the energy performance of them [8]. Further application of PHGMs is as drug delivery vehicles with higher loading efficiency. In addition, they are favorable for cell attachment due to their larger surface area [9,10].

The structural properties of the HGMs are determined by the initial glass composition, the conditions of heat treatment (temperature, time) and the leaching condition [11]. The primary aim of this study is preparing hollow glass microsphere with acceptable quality and major object is to investigate effect of leaching time on porosity characteristics of sphere's wall.

2. Experimental

2.1. Fabrication procedures

Sodium borosilicate glass frits of $64SiO_2$-$22B_2O_3$-$8.5K_2O$-$0.5Li_2O$-$5.5Na_2O$ were prepared from silica (SiO_2), boric acid (H_3BO_3), potassium carbonate (K_2CO_3), sodium carbonate (Na_2CO_3) and lithium carbonate (Li_2CO_3) (all with purities of $>98\%$). Approximately 250 gm of precursor mixture was mixed with 25 gm

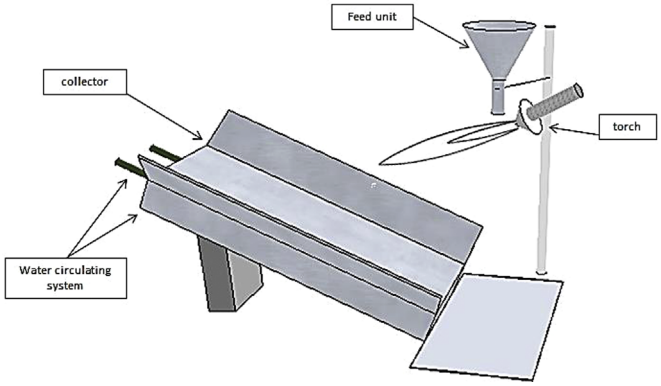

Fig. 1. Schematic of flame spheroidisation apparatus.

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sodium sulfate as a blowing agent and then melted in 1400 °C. After melting, the glass was quenched in water. The resulting frits were ground and sieved in the range of 38–45 μm. The glass feeds were dropped into oxy-acetylene flame to form spherical hollow particles. Solid and hollow spheres were separated by floatation method using distilled water.

To fabricate Porous hollow glass bubbles, the microspheres were heat treated at 580 °C for 8 h and then were acid leached with 3 M hydrochloric acid for 0.5, 1 and 2 h. After leaching, microspheres were rinsed with 0.5 M NaOH and distilled water,

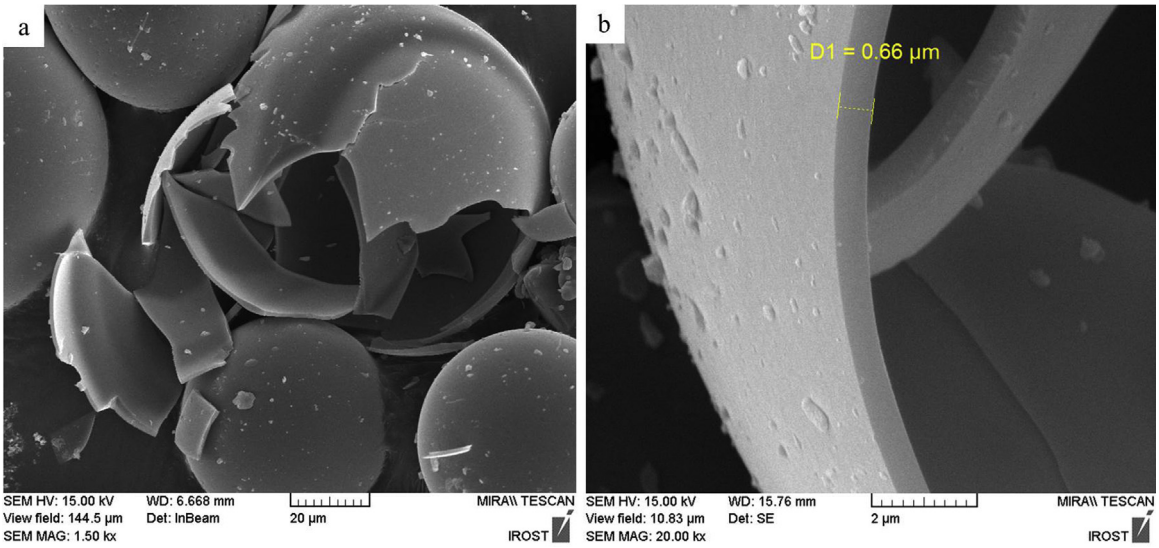

Fig. 2. SEM image (a) crushed HGMS (b) the cross section and wall thickness of HGM.

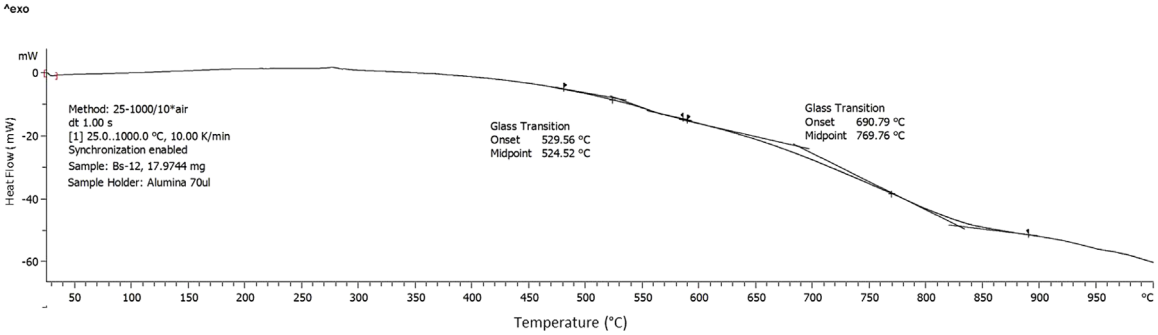

Fig. 3. DSC analysis curve of HGMs.

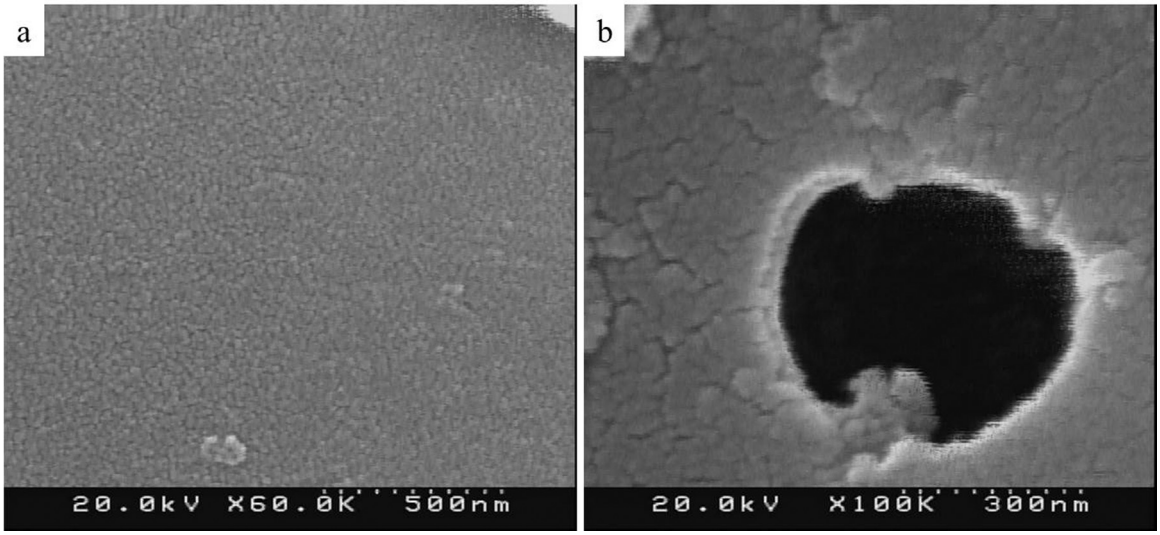

Fig. 4. SEM image of (a) surface of acid leached HGM for 30 min (b) pore in the wall of HGM.