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Thin shell aerogel fabrication for FIREX-I targets using high viscosity (phloroglucinol carboxylic acid)/formaldehyde solution

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Abstract

Capsules with a thin aerogel shell were prepared by the $O_O/W/O_I$ emulsion process. (Phloroglucinol carboxylic acid)/formaldehyde (PF) was used as the water phase (W) solution to form the shell of the capsule. PF is a linear polymer prepared from phloroglucinol carboxylic acid. The viscosity of the PF solution can reach a high level of $9 \times 10^{-5} \text{ m}^2/\text{s}$ without gelation while resorcinol/formaldehyde (RF) gels at $\sim 3\text{--}4 \times 10^{-5} \text{ m}^2/\text{s}$. Using the viscous PF solution, capsule with a $17 \text{ }\mu\text{m}$ gel shell was fabricated. This thickness satisfies the specification of the first phase of Fast Ignition Realization Experiment (FIREX-I) at Osaka University. When PF gel was extracted to remove the organic solvent, shrinkage of 9% occurred. The final density of the PF aerogel was $145 \text{ mg}/\text{cm}^3$. Both the shell thickness and density can satisfy the specification of FIREX-I. The pore size of the PF aerogel was less than 100 nm while that of RF was $200\text{--}500 \text{ nm}$. The SEM showed that PF had particle-like foam structure while RF had fibrous-like foam structure.

Keywords: Fast ignition; Low density; Thin shell thickness; Fusion target; Phenol formaldehyde resin

1. INTRODUCTION

Fuel target technology (Nagai *et al.*, 2004a) is a key issue in fast ignition research (Yamanaka, 1983; Tabak *et al.*, 1994; Fernandez *et al.*, 2005; Hora, 2007; Nobile *et al.*, 2006). The invention of the hollow cone shell geometry (Kodama *et al.*, 2001; Nakamura *et al.*, 2006; Sakagami *et al.*, 2006) was critical in demonstrating the heating of a deuterated hydrocarbon plasma to a temperature of about 1 keV using Gekko XII and the petawatt laser at Osaka University (Kodama *et al.*, 2002, 2004; Kitagawa *et al.*, 2002). Even for the fast ignition scheme, highly compressed implosion is required (Azechi *et al.*, 1991), and fuel shell structure is necessary. In the first phase of fast ignition realization experiment (FIREX-I) (Tanaka *et al.*, 2004; Kodama *et al.*, 2004; Johzaki *et al.*, 2007), a cryogenic deuterium tritium (DT) fuel target (Norimatsu *et al.*, 2003, 2006; Mima *et al.*, 2007; Azechi *et al.*, 2006) was designed. The target includes a $\sim 500 \text{ }\mu\text{m}$ capsule which has a $20 \text{ }\mu\text{m}$ thick low density foam shell (Nagai *et al.*, 2005) to support liquid or solid DT (Iwamoto *et al.*, 2007) to develop a uniform, shell-shape-tailored fuel

layer. In FIREX-I, the density of this foam shell is allowed to be $100 \text{ mg}/\text{cm}^3$. Various materials have been studied for this support foam shell (Streit *et al.*, 2003; Khalenkov *et al.*, 2006; Nemoto *et al.*, 2006; Yamanaka *et al.*, 2007; Borisenko *et al.*, 2007) in which phenol-formaldehyde resin has been used and investigated because of its transparency in the visible region.

By using the $O_O/W/O_I$ emulsion process to fabricate capsules for these cryogenic targets, a high viscosity of the shell material allows for thin shell thickness (Ito *et al.*, 2006a). It is difficult to reach the high viscosity needed for thin shell fabrication with the traditionally used resorcinol/formaldehyde (RF) because of its cross-linking structure. Therefore, the thickness of RF shells are $50\text{--}100 \text{ }\mu\text{m}$ (Lambert *et al.*, 1997; Nikroo *et al.*, 2004; Ito *et al.*, 2006b; Paguio *et al.*, 2007). (Phloroglucinol carboxylic acid)/formaldehyde (PF), another phenolic resin, is a linear polymer, and the viscosity of its solution can reach a high value without gelation. In a previous study, PF was used as an additional precursor for RF solution to adjust its viscosity (Ito *et al.*, 2006a), but the properties of PF solution can satisfy the requirement better for the fabrication of thin shell capsules.

In this study, the viscous PF solution was used to fabricate thin shell capsules. The gel shell was extracted by CO_2

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supercritical fluid to remove the solvent, resulting in the foam structure filled only with air, which is called aerogel. The nano-structure of PF aerogel shell was compared with that of RF aerogel shell obtained the same way. The fine nano-structure endows PF and RF aerogel with not only the ability to serve as a fuel supporter, but also provides good transparency in visible region to observe fueling with DT and to allow the routine characterization of shells using optical techniques.

2. EXPERIMENT DETAILS

2.1. Materials

Phloroglucinol carboxylic acid (1.25 g, Tokyo Chemical Industry, Co., LTD) was uniformly dispersed into 12 ml of pure water by ultrasonication, and then 8 ml of 1 mol/l NaOH (Sigma Aldrich Japan) and 1.71 ml of 37% formaldehyde (Nacalai Tesque, Inc.) were added and stirred at 70°C. Sixty-five minutes later, the solution was cooled down in an ice bath for 30 min. Then the PF solution was kept at room temperature while waiting for the viscosity to increase.

The viscosity of the PF solution was checked during the gelation process. When the viscosity reached $9 \times 10^{-5} \text{ m}^2/\text{s}$, it was used as the water phase solution to fabricate capsules with the $\text{O}_\text{O}/\text{W}/\text{O}_\text{I}$ emulsion process. Encapsulation of the PF solutions to form a compound emulsion was accomplished using a triple-orifice droplet generator (Nagai *et al.*, 2000; Norimatsu *et al.*, 1998; Lambert *et al.*, 1997; Yang *et al.*, 2006). The fabrication conditions were as following: the flow rate of W, O_I , and O_O liquid are 0.083 ml/min, 0.130 ml/min, and 93 ml/min, respectively.

The shell of the obtained capsules made of the PF solution then gelled during the rotation in a drum. The PF gel shell was washed by $1 \times 10^{-6} \text{ m}^2/\text{s}$ silicon oil (Shinetsu, Japan) and then exchanged with acetone solutions ($V_{\text{acetone}}:V_{\text{water}} = 1:3$, $V_{\text{acetone}}:V_{\text{water}} = 1:1$, $V_{\text{acetone}}:V_{\text{water}} = 3:1$ and 100% acetone step by step) to remove water gradually (every solution for 1 h). The completely exchanged PF gel was extracted by supercritical CO_2 to remove the acetone in the gel porous structures to form the aerogel. The extraction condition was 80 atm and 333 K for 24 h.

2.2. Measurements

The viscosities of the PF solutions were measured by a digital viscometer, DV M-E, Tokyo Keiki Co. LTD, Japan. The SEM image was taken with a JEOL JSM 7400 FS. The PF gel was extracted by ISCO SFX 220 supercritical fluid extractor.

3. RESULTS AND DISCUSSION

3.1. The Viscosity Change and Minimum Shell Thickness

In order to get capsules with 20 μm thick shells, shell material solution with a viscosity higher than $9 \times 10^{-5} \text{ m}^2/\text{s}$ is required

in the $\text{O}_\text{O}/\text{W}/\text{O}_\text{I}$ emulsion process (Ito *et al.*, 2006a). The time-dependent viscosity of the PF precursor solution is shown in Figure 1. From the figure, we can see that after a reaction time of 40 h, the viscosity of PF solution reaches $9 \times 10^{-5} \text{ m}^2/\text{s}$.

The catalyst concentration of 0.4 mmol/ml was chosen to fabricate hollow spherical shells because the rate of viscosity increase with this catalyst concentration was suitable for capsule fabrication using $\text{O}_\text{O}/\text{W}/\text{O}_\text{I}$ emulsion process and the droplet generator. By using a PF solution with the viscosity of $9 \times 10^{-5} \text{ m}^2/\text{s}$, the minimum gel shell thickness obtained was 17 μm .

3.2. Microstructure Morphology of the PF Aerogel Shell

Although both RF and PF aerogel have three-dimensional (3D) nano-networks, there are two differences between their microstructures. First, the pore size of RF is about 200–500 nm (Fig. 2a) while the pore size of PF is smaller than 100 nm (Fig. 2b). Besides, the pore size uniformity of PF aerogel is better than that of RF aerogel (Fig. 3). The average diameter of the pores of RF and PF foam were $114 \pm 54 \text{ nm}$ and $32 \pm 10 \text{ nm}$.

Second, the network morphology of RF and PF aerogel is different. The pore of RF is formed by strings of the gel network (Fig. 2a). This structure has clear junctions (Ito *et al.*, 2006a, 2005). The pore of PF was formed by polymer particles sticking together. There is no fibrous-shaped structure but just particle-like structure (Fig. 2b). This is because the gelation mechanism of RF and PF is different. Resorcinol has only four subsistent groups and thus it is possible to form cross-links with formaldehyde *via* methylene groups ($-\text{CH}_2-$) (Pekala *et al.*, 1989; Ruben *et al.*, 1992) which are inert. Phloroglucinol carboxylic acid has only two subsistent groups on the benzene ring and can only form a linear polymer with formaldehyde. The linear polymers are linked *via* non-covalent interactions such as

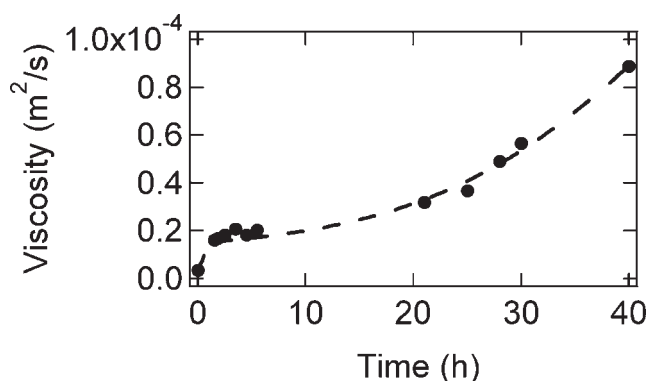


Fig. 1. Shown is the viscosity change of PF solution at room temperature as a function of time. The precursor PF solution was obtained by stirring the uniform mixture of 1.25 g phloroglucinol carboxylic acid, 1.71 ml formaldehyde, 12 ml H_2O and 8 mmol NaOH at 70°C for 65 min.

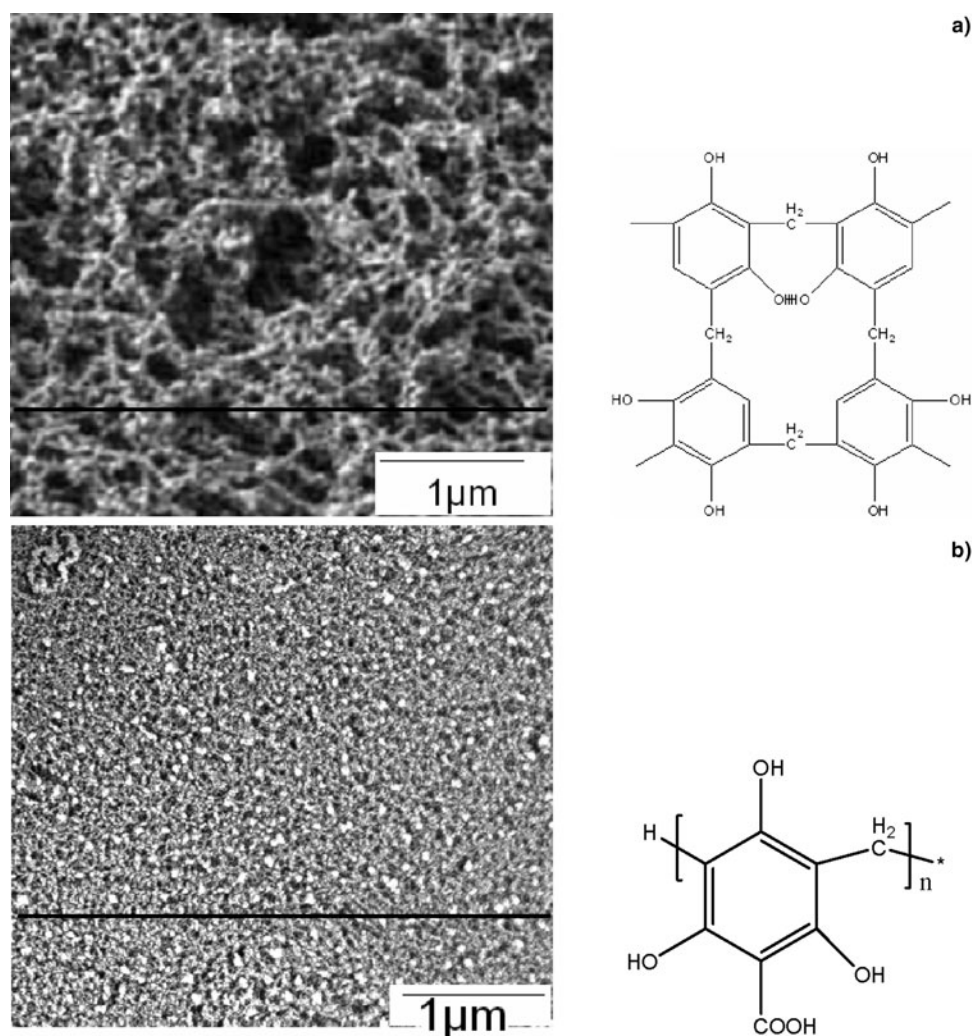


Fig. 2. SEM images: (a) RF aerogel (b) PF aerogel. The pore size of PF aerogel is smaller than 100 nm. The pore size of RF aerogel is 200–500 nm.

hydrogen bonding which is labile and thus it is possible to reorient the 3D network structure. In general, particle-like structures are more stable because they minimize the surface energy between the phases of polymer and solvent. Therefore, in the case of labile bonding network, such a particle structure would be more stable than a fibrous structure.

Such particle-like structure has been observed in the more labile bonding case (van der Waals interaction) of PMP (poly (4-methyl-1-pentene)) aerogel (Nagai *et al.*, 2004b). Pores of 100 nm minimize light scattering so that the final dried gel shell is transparent, allowing one to observe the fuel layer formation optically.

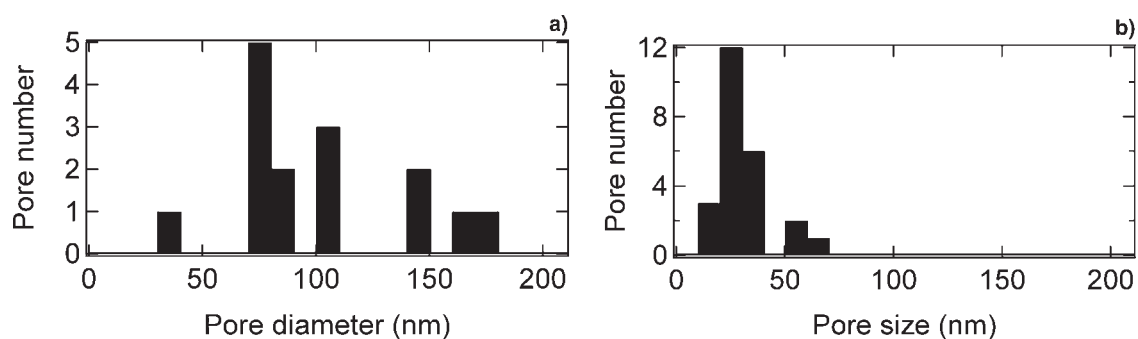


Fig. 3. Shown are the distribution of the pore diameters of (a) RF foam and (b) PF foam along linear lines of 4 μm.

3.3. The Volume Shrinkage During Organic Solvent Extraction

We monitored the volume change during extraction. Here is shown one example. Before the extraction, the gel volume was 2.45 mm^3 while after the extraction the aerogel volume was 2.28 mm^3 . The shrinkage due to extraction was about 9% in volume. The density was calculated to be 145 mg/cm^3 from the volume and the mass (0.33 mg). This shrinkage rate was smaller than that of RF aerogel.

4. CONCLUSION

(Phloroglucinol carboxylic acid)/formaldehyde is a linear polymer and linked by non-covalent interactions at gelation. Therefore, the viscosity of the precursory PF solution before gelation can be controlled at a high value of $9 \times 10^{-5} \text{ m}^2/\text{s}$. By fabricating capsules using such a viscous solution, the shell thickness can be reduced to $17 \text{ }\mu\text{m}$ which satisfies the requirement for fast ignition cryogenic targets. The PF gel during extraction had shrinkage of 9% in volume resulting in a final density of 145 mg/cm^3 . The SEM of PF aerogel showed a fine uniform porous structure with a pore size smaller than 100 nm . Because of different crosslink bonds in the RF and PF foam structure, the inert bond of methylene in RF and the labile bond of hydrogen bonding in PF, RF has a string-like gel network while PF has a particle-like structure.

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