

# Sulfonated Nano Octaphenyl Polyhedral Oligomeric Silsesquioxanes Particles (SPOSS)

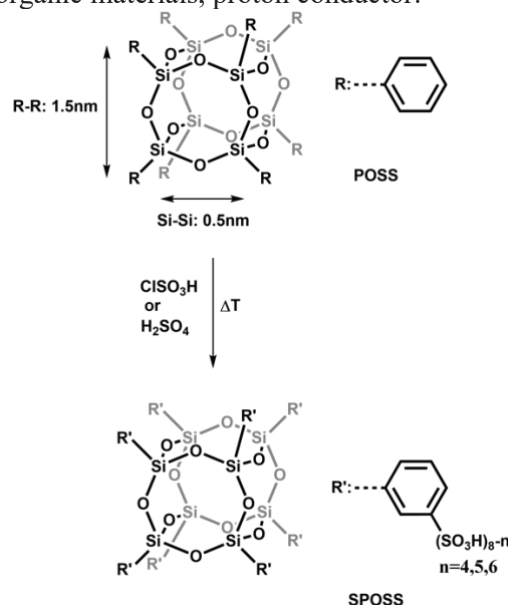
J. Kim<sup>1,\*</sup>

<sup>1</sup> Environmental Circulation Composite Materials Group, Functional Materials Field, Research Center for Electronic and Optical Materials, National Institute for Materials Science (NIMS), Tsukuba, Japan

## Abstract:

Polyhedral silsesquioxanes (POSS), nanosized particles (approximately 1-3 nm) with an  $(\text{RSiO}_{1.5})$  structure intermediate between silica ( $\text{SiO}_2$ ) and silicone ( $\text{R}_2\text{SiO}$ ), are being studied in ionics, electronics, photonics, and additives. POSS are network polymers or polyhedral clusters with an  $(\text{RSiO}_{1.5})_n$  structure, obtained by hydrolysis of trifunctional silanes. Each silicon atom is bonded to an average of 1.5 oxygen atoms (Sesqui) and one hydrocarbon group. These inorganic compounds have a cage-like framework consisting of up to eight organic functional groups and Si-O bonds. These stoichiometric compounds are known as organically compatible inorganics nanomaterials. By varying the cage structure and functional groups, materials with diverse properties can be created. In addition, by controlling the movement of polymer chains while maintaining the processability and mechanical properties of the base resin, it is possible to increase thermal and physical strength while also improving chemical and electrical properties [1]. Octaphenyl POSS (OPOSS), which has phenyl groups as organic functional groups, is a promising ionic conductor nanomaterial. Sulfonation can be performed on the eight phenyl groups. SPOSS has been studied as an ionic conductor and a conductivity-enhancing additive [2-5]. Most published studies have focused on qualitative evaluations of SPOSS, with few quantitative evaluations. To my knowledge, no quantitative analysis of SPOSS with eight sulfonate groups on eight phenyl groups has been performed. Conventional sulfonation methods ( $\text{ClSO}_3\text{H}$ , oleum acid,  $\text{H}_2\text{SO}_4$ ) can introduce four sulfonic acid groups to the octaphenyl groups. However, the POSS structure is prone to hydrolysis during the sulfonation process and the subsequent acid removal process, making the synthesis of SPOSS with all POSS structures challenging. In this study, I introduced two to four sulfonic acid groups into OPOSS using  $\text{ClSO}_3\text{H}$  and  $\text{H}_2\text{SO}_4$  (Figure 1). It was found that the introduction of sulfonic acid groups not only depends on the type of sulfuric acid, temperature, and time, but also on the process of removing the remaining acid so that POSS does not break down.

**Keywords:** octaphenyl polyhedral oligomeric silsesquioxanes (POSS), sulfonated POSS (SPOSS), nano particles, sulfonation, organic-inorganic materials, proton conductor.



**Figure 1:** Preparation of SPOSS: chemical structure of POSS and SPOSS.

## References:

1. SigmaAldrich.com
2. J. Kim, patent: Japan no. 6652766, EU EP3490043, US10868322.
3. N.A.M Nor, J. Jaafar, J. Kim. (2020), improved properties of sulfonated octaphenyl polyhedral silsesquioxane cross-link with highly sulfonated polyphenylsulfone as proton exchange membrane, *J. Solid State Electrochemistry*, 24, 1185-1195.
4. N.A.M Nor, J. Jaafar, J. Kim, A.F. Ismail, M.H.D. Othman, M.A. Rahman. (2020), effect of polyhedral silsesquioxane functionalized sulfonic acid groups incorporated into highly sulfonated polyphenylsulfone as proton-conducting membrane, *Arabian. J. Science and Engineering*, 23, November.
5. F.B. Fauzi, J. Kim. (2024), comprehensive studies on sulfonated octaphenyl polyhedral silsesquioxane (SPOSS) using sulfuric acid: structural analysis and composite crosslinked SPPSU/SPOSS membranes, *J. Mem. Sci.*, 702, 122756-122767.