

**TOPLINSKA SVOJSTVA ALKILNO MODIFICIRANIH
FENIL-SILSESKVIOKSANA**
**THERMAL PROPERTIES OF ALKYL-MODIFIED
PHENYL-SILSESQUIOXANES**

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Phenyl-silsesquioxanes are hybrid organic-inorganic compounds that can thermally soften, and whose softening temperature depends on the level of condensation, which can be controlled by conditions of synthesis [1]. Phenyl-silsesquioxane particles find application in preparation of transparent thick films or matrices for functional thin films. Other polysilsesquioxanes also can have softening temperature, which has been shown to depend on the size of organic substituent [2]. In order to control the softening temperature and thermal stability of the phenyl-silsesquioxane particles, they were modified with alkyl chains of varying lengths. Polysilsesquioxane hybrid particles were prepared by two-step acid-base catalysed sol-gel process, with ethanol as solvent.

Alkyl-triethoxysilanes, $R-Si(OC_2H_5)_3$, $R = CH_3, C_2H_5, C_5H_{11}$ and C_8H_{17} , were mixed with phenyl-triethoxysilane, $C_6H_5-Si(OC_2H_5)_3$, in a constant 1:4 ratio, hydrolysed with aqueous solution of HCl and then condensed with aqueous solution of NH_4OH . Resulting particles were dried, investigated by scanning electron microscopy (SEM), differential scanning calorimetry (DSC) and combined differential thermal and thermogravimetric analysis. Pentyl- and octyl- modified particles were found to have less homogeneous size distribution, presumably because long alkyl chains were less miscible with other components resulting in less homogeneous initial mixture. Increased size of alkyl chains also presents greater sterical hindrance to condensation of phenyl-silsesquioxanes, so the softening temperature decreases from 380 °C for methyl-modified particles to 50 – 100 °C for octyl-modified ones. Thermal stability of alkyl-modified particles was expected to decrease, since alkyl-silsesquioxanes degrade at much lower temperatures. Methyl- and ethyl-modified particles start to degrade at 400 °C, same as unmodified phenyl-silsesquioxane, while pentyl- and octyl-modified particles start to degrade at 250 °C, which corresponds to degradation of pentyl- and octyl-silsesquioxanes. Therefore it can be supposed that longer alkyl chains are exposed on the surface of the particles while shorter ethyl and methyl groups are in some way shielded by phenyl groups.

- [1] K. Takahashi, K. Tadanaga, A. Matsuda, A. Hayashi, M. Tatsumisago, *J. Sol-Gel Sci. Techn.* **41** (2007) 217.
[2] K. Tadanaga, K. Takahashi, A. Hayashi, M. Tatsumisago, *ICG 2007 Proceedings*, 2C-61.