Preparation and characterization of the composites based on hydridesilylated nanosilica and caffeic acid

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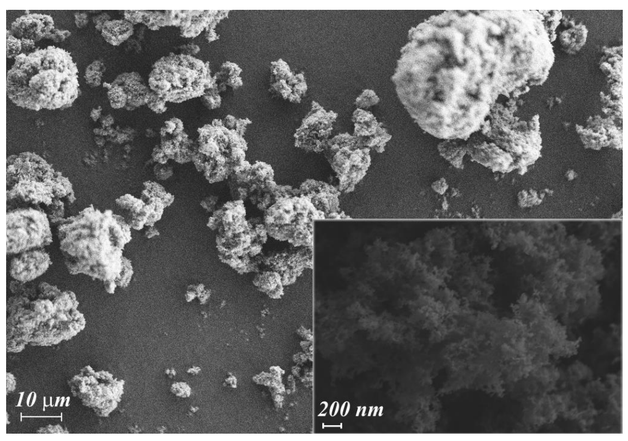
Introduction / Aim

Fumed silica (FS) is known to be efficient enterosorbent and carrier for biologically active molecules. Hydridesilylation of silica surface enlarges its functional capabilities, in particular, renders it with reducing properties in aqueous media. Caffeic acid (CA), being potent antioxidant, is a promising model object for deposition onto the silica surface to prepare the antioxidant composites. The aim of this work was to prepare and to study the composites based on pristine FS or hydridesilylated FS (HFS, hydride-silica) and CA with different CA concentration and using various methods of its deposition onto the silica surface.

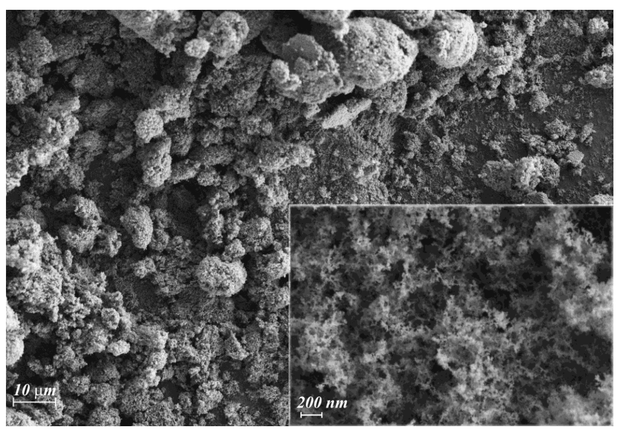
Experimental

HFS was obtained via FS treatment with triethoxysilane. The presence of grafted silicon hydride groups was confirmed by means of IR spectroscopy, and their concentration measured by titrimetric and spectrophotometric analysis was found to be about 0.4 mmol/g. FS-CA and HFS-CA composites were prepared either by CA deposition onto the respective silica surface from ethanol solution (impregnation with the solvent excess (C_{CA}=153 mg/g) or by sorptive modification under fluidized bed conditions (FBCs) (C_{CA}=23 mg/g) or by mechanical mixing of CA with the corresponding silica (C_{CA}=153 mg/g). The materials synthesized were characterized by scanning electron microscopy (SEM), and also studied by means of IR spectroscopy and thermogravimetric analysis (TGA).

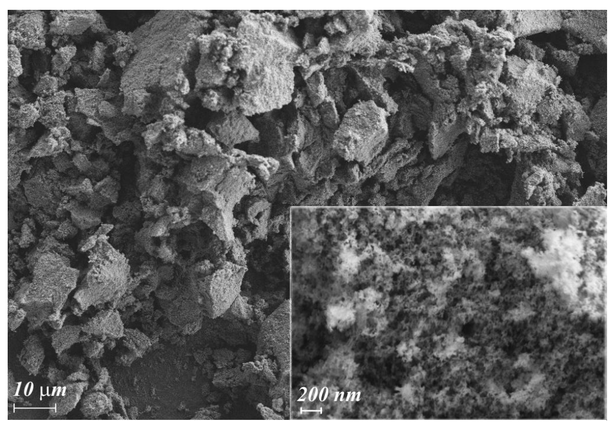
Results



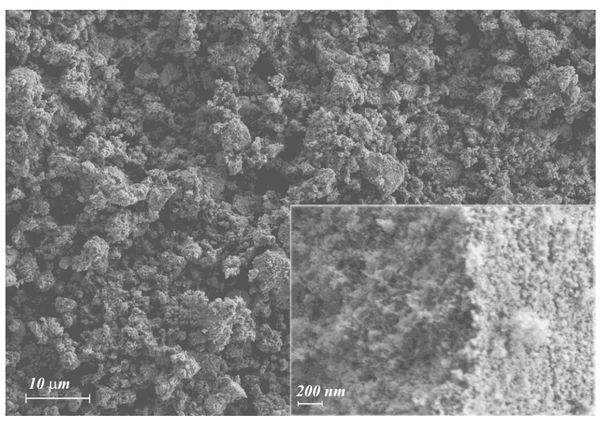
Fumed silica (FS)



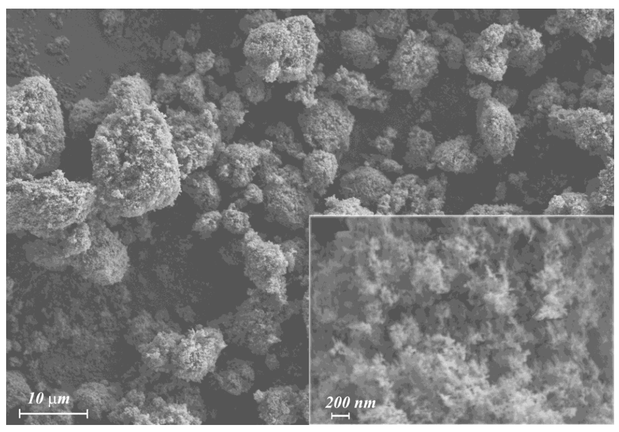
FS-CA (fluidized bed conditions)



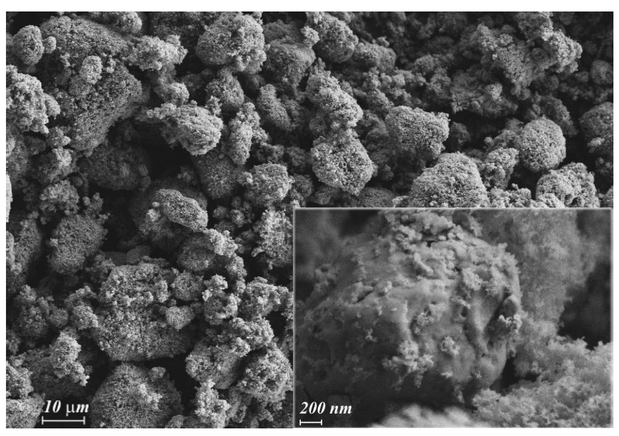
FS-CA (impregnation)



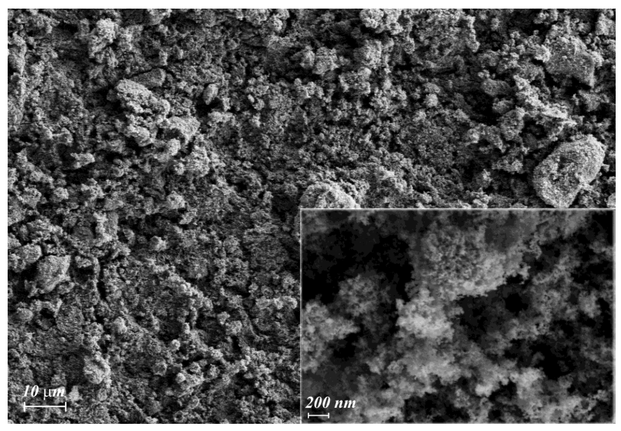
FS-CA (mechanical mixing)



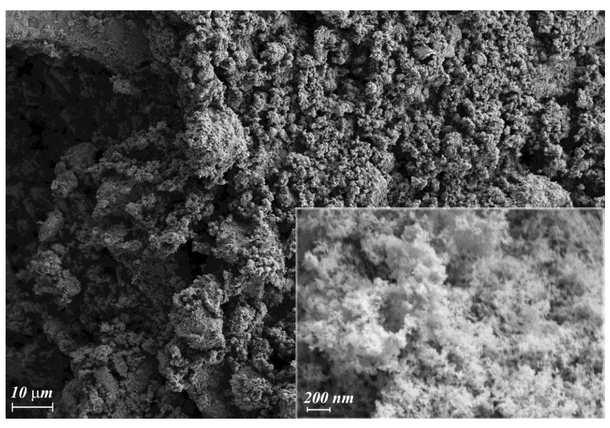
Hydridesilylated fumed silica (HFS)



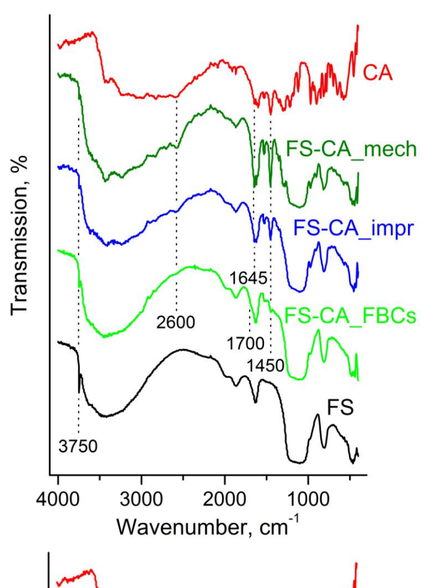
HFS-CA (fluidized bed conditions)

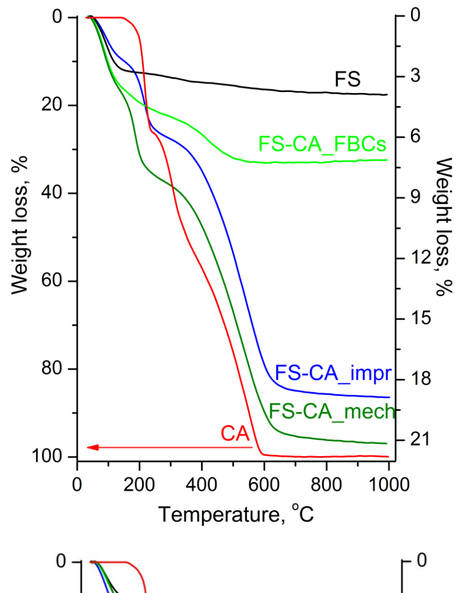


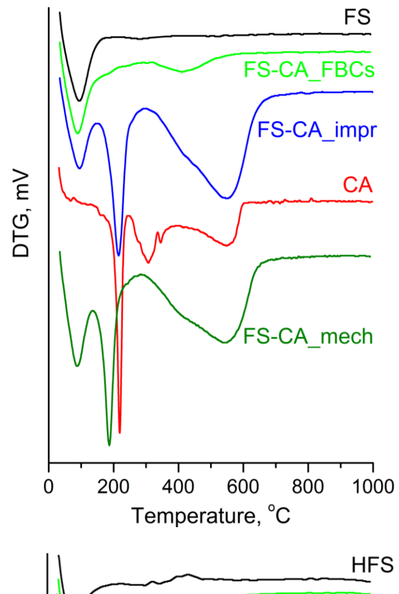
HFS-CA (impregnation)

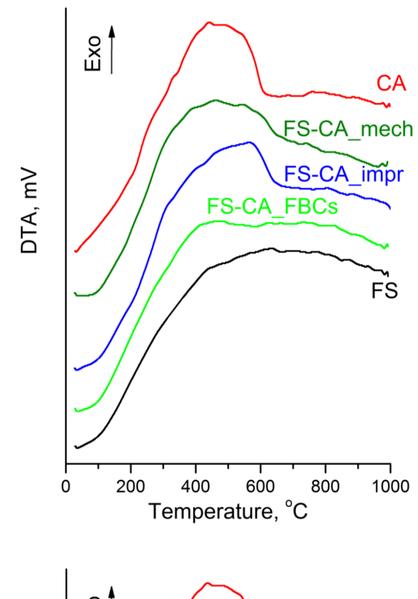


HFS-CA (mechanical mixing)









Conclusions

SEM studies revealed that CA-silica composites are composed of round-shape tens-micron-size agglomerates/particles comprising of or covered with the network of nanoparticles. IR spectroscopic studies confirmed the presence of ≡SiH groups (2255 cm^{-1}) on the surface of hydride-silica in HFS-CA composites, and it was found that CA is present on the FS and HFS surface in the form of dimers and probably associates; its molecules being involved in hydrogen binding with surface silanols. The TGA data has shown that silica surface affects the mechanism (and in the case of HFS also the kinetics) of CA thermo-oxidative destruction.