

SUPPORTING INFORMATION

Novel non-aqueous sol-gel route for the *in situ*
synthesis of high loaded silica-rubber nanocomposites

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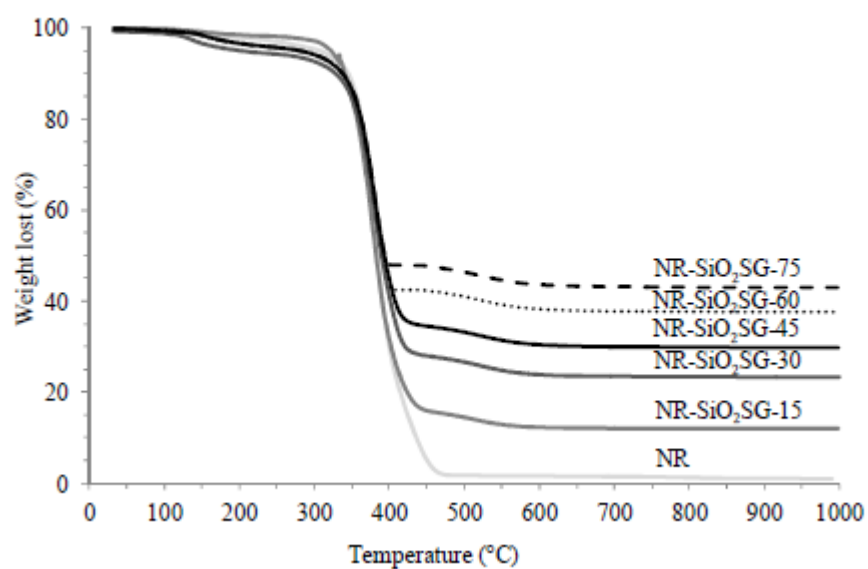


Figure S1: Comparison of TGA profiles of natural rubber (NR) and NR-SiO₂SG-X nanocomposites

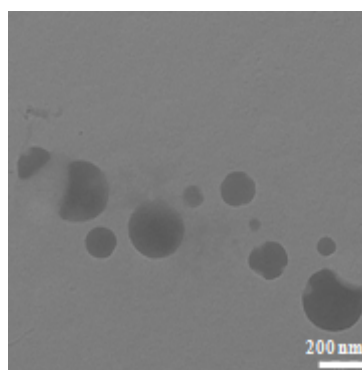


Fig. S2: TEM image of SiO₂SG powder

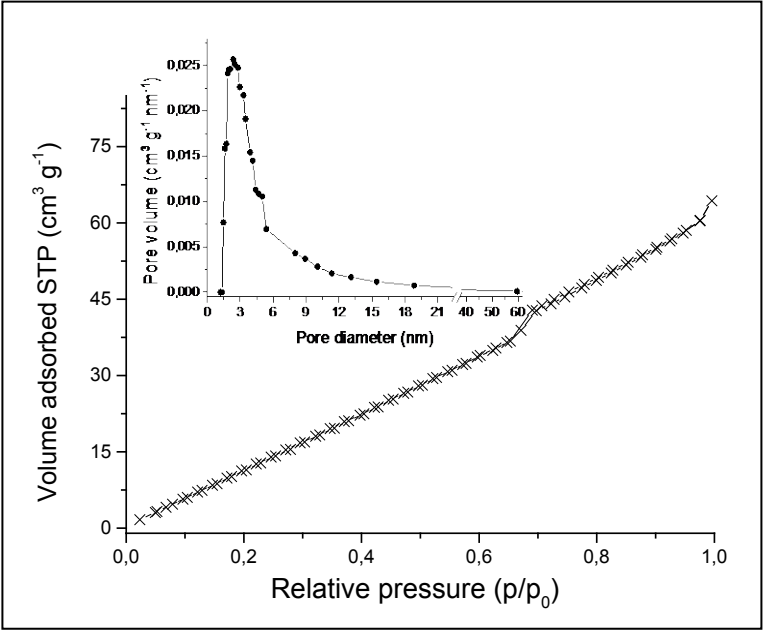


Figura S3: Nitrogen adsorption-desorption isotherm for SiO₂SG silica powder. In the inset the pore distribution.

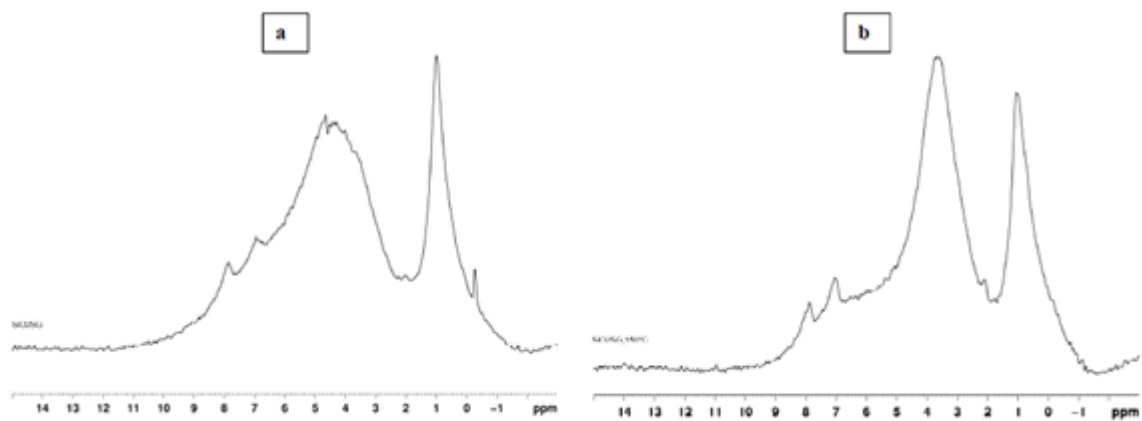


Figure S4: ¹H CP MAS NMR spectra of samples a) SiO₂SG and b) SiO₂SG dried at 150°C

Table S1: Assignments and chemical shifts of ¹H NMR

δ (ppm)	Functional group	
7.9	H-COO	Formic acid/ Ethyl formate
7.0	C-H aromatic	toluene
4.5 broad	O-H /CH ₂	water/ Ethyl formate
3.6 broad	O-CH ₂	EtOH, ethoxyde
2.1	CH ₃	toluene
1.0	CH ₃	EtOH, ethoxyde

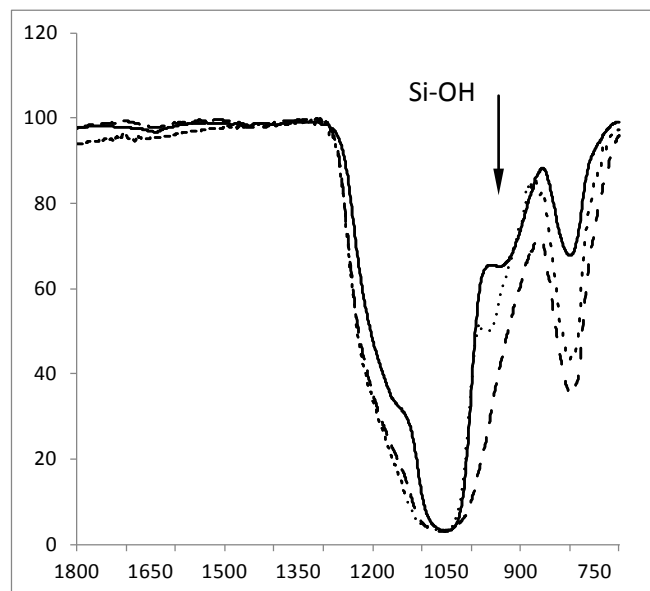


Figura S5: ATR-FTIR spectrum of SiO₂SG (bold line), SiO₂AQ (dotted line) and SiO₂R (dashed line), dried at 150°C in air. The intensity of the spectra is normalized referring to the intensity of the stretching mode Si-O-Si at $\sim 1100\text{ cm}^{-1}$ ⁵¹

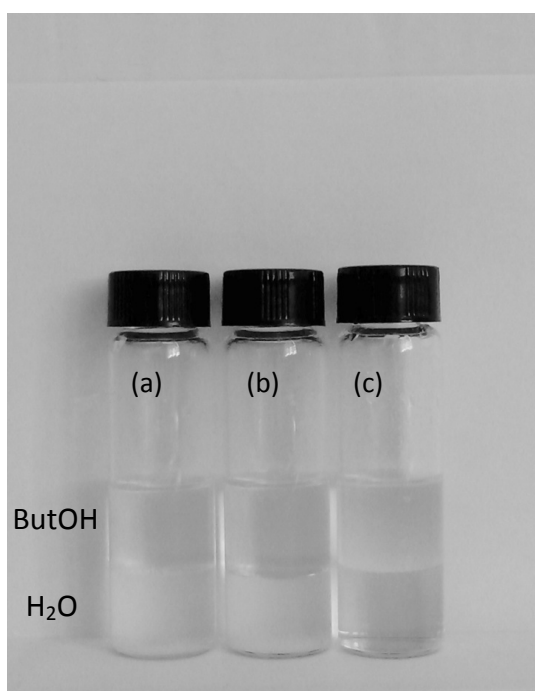


Figura S6: Mixing test of (a) $\text{SiO}_2\text{-R}$; (b) $\text{SiO}_2\text{-AQ}$ and (c) SiO_2SG in water/buthanol immiscible phases. $\text{SiO}_2\text{-SG}$ is dispersed in buthanol while $\text{SiO}_2\text{-R}$ and $\text{SiO}_2\text{-AQ}$ in water.

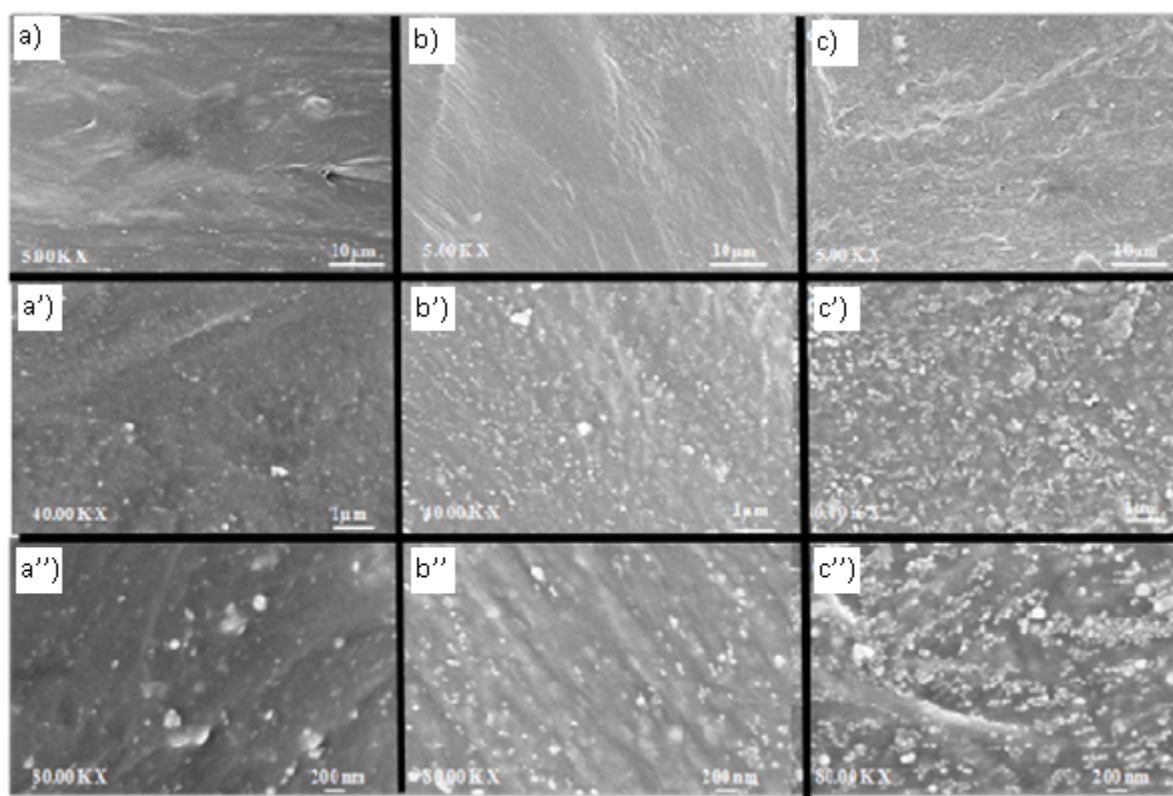


Figure S7: FE-SEM micrographs of in-situ composites at different silica content: a) NR-SiO₂SG-15; b) NR-SiO₂SG-30; c) NR-SiO₂SG-45. The superscript ‘ and ‘’ stands for the high magnification.

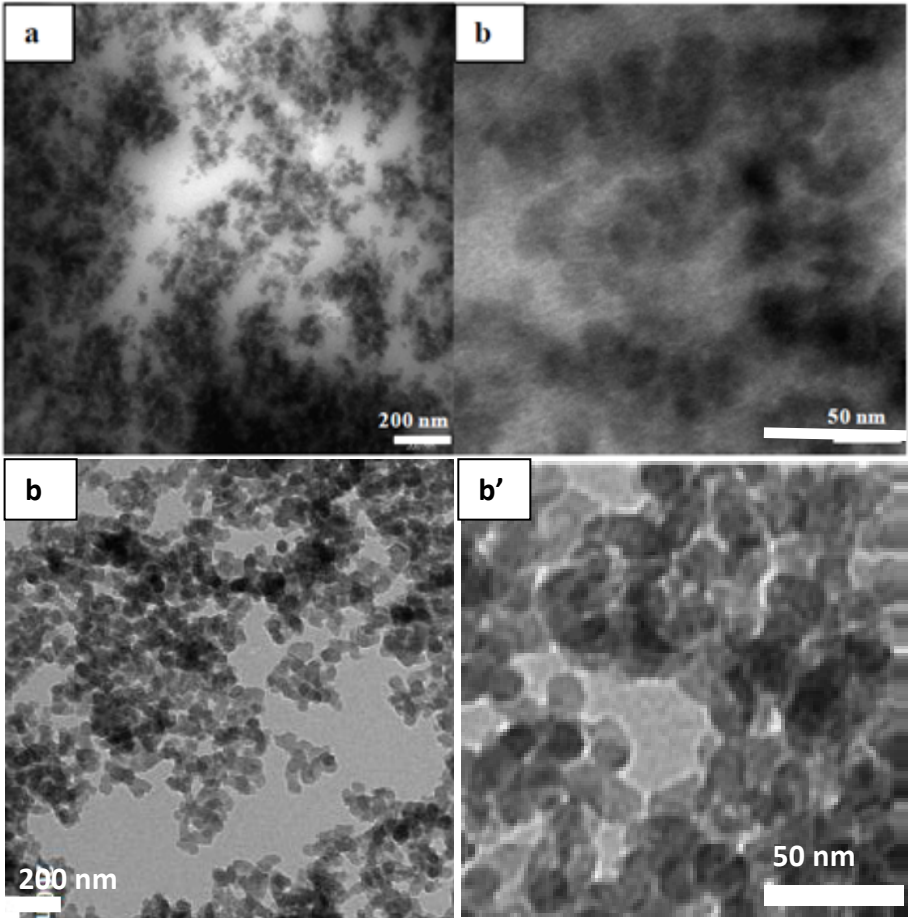


Figura S8 TEM image of a) NR-SiO₂R-30 and b) NR-SiO₂AQ-30 composites. The superscript ‘ stands for higher magnification.