

Supplementary Information

Different Strategies to Anchor Organotin Methoxides on Silica and Their (Re)Use as Heterogeneous Catalysts for Transesterification Reactions

Roberta A. de Jesus,^{ib} ^a Poliana da Conceição,^a Jhosianna P. V. da Silva,^a Nívea S. Brainer,^a Simoni M. P. Meneghetti^{ib} ^a and Mario R. Meneghetti^{ib} ^{*a}

^aGrupo de Catálise e Reatividade Química, Instituto de Química e Biotecnologia, Universidade Federal de Alagoas, 57072-970 Maceió-AL, Brazil

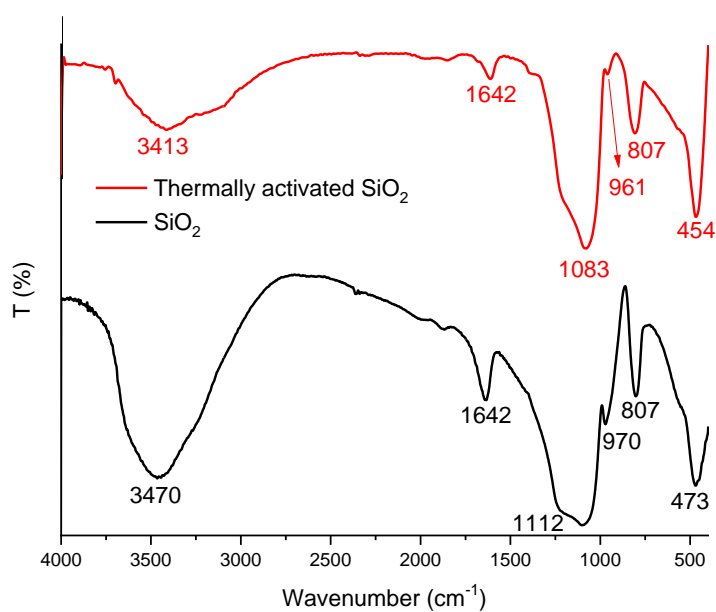
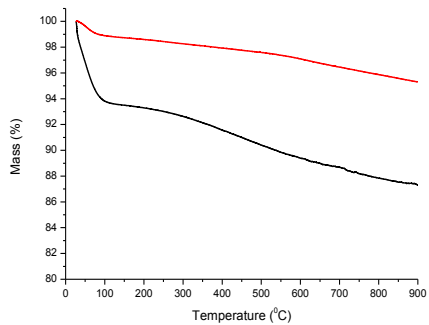
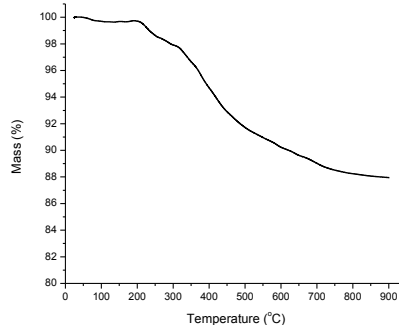


Figure S1. FTIR spectra of the untreated and thermally activated silica.

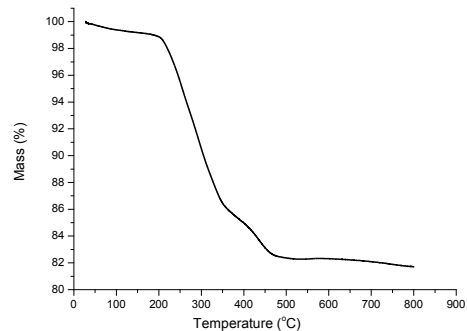
*e-mail: mrm@qui.ufal.br



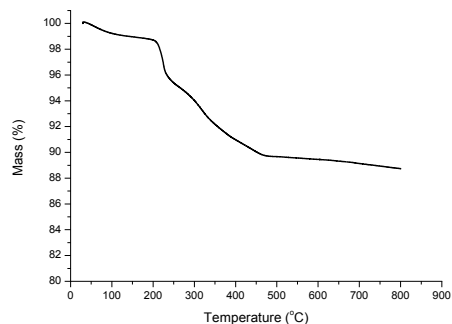
SiO_2 and thermally activated SiO_2 (red)



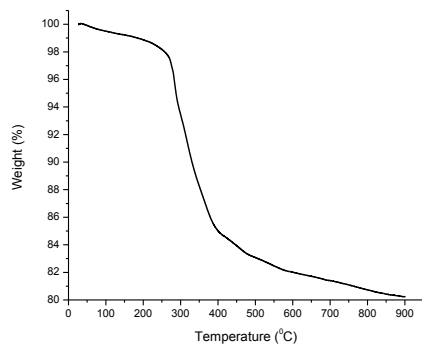
$\text{SiO}_2\text{-SH}$



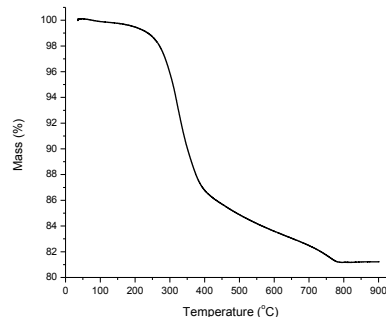
$\text{SiO}_2\text{-SnBu}_2$



$\text{SiO}_2\text{-SnBu}_3$

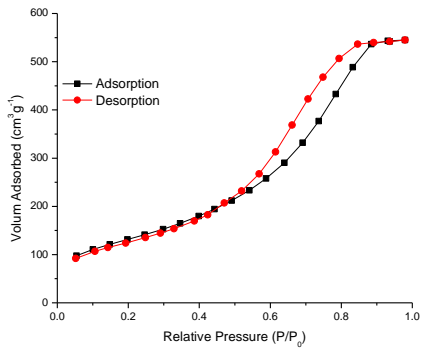


$\text{SiO}_2\text{-S-SnBu}_2$

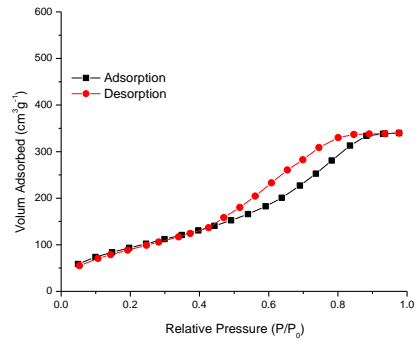


$\text{SiO}_2\text{-S-SnBu}_3$

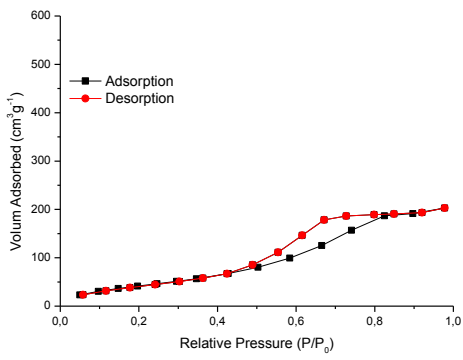
Figure S2. Thermograms of materials investigated in the present work before reactions.



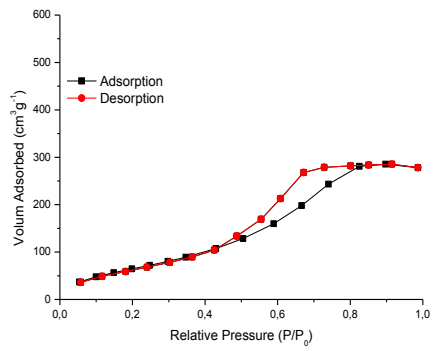
SiO_2



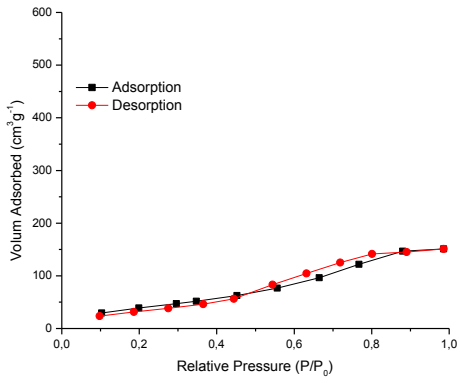
$\text{SiO}_2\text{-SH}$



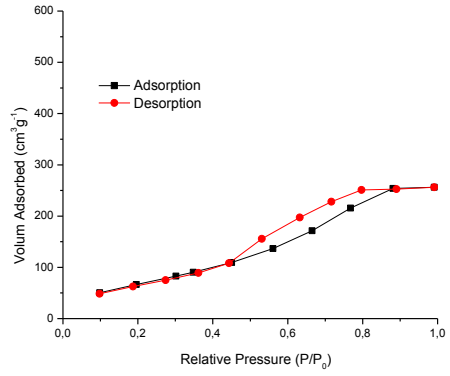
$\text{SiO}_2\text{-SnBu}_2$



$\text{SiO}_2\text{-SnBu}_3$



$\text{SiO}_2\text{-S-SnBu}_2$



$\text{SiO}_2\text{-S-SnBu}_3$

Figure S3. Textural analysis of materials investigated in the present work.

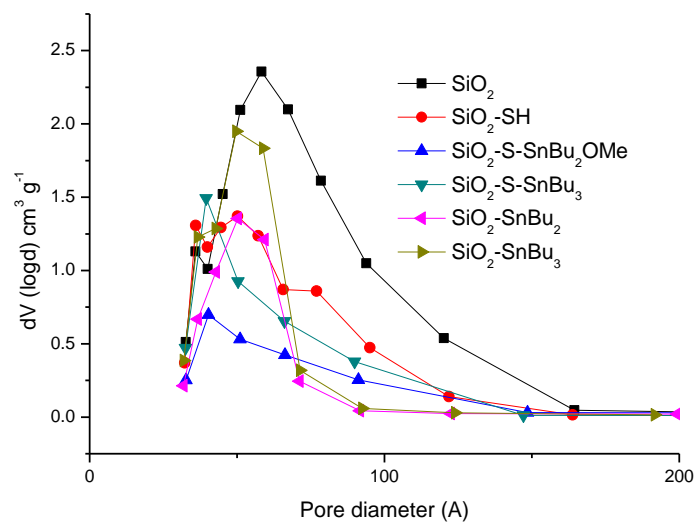


Figure S4. Pore diameter distribution of materials investigated in the present work.

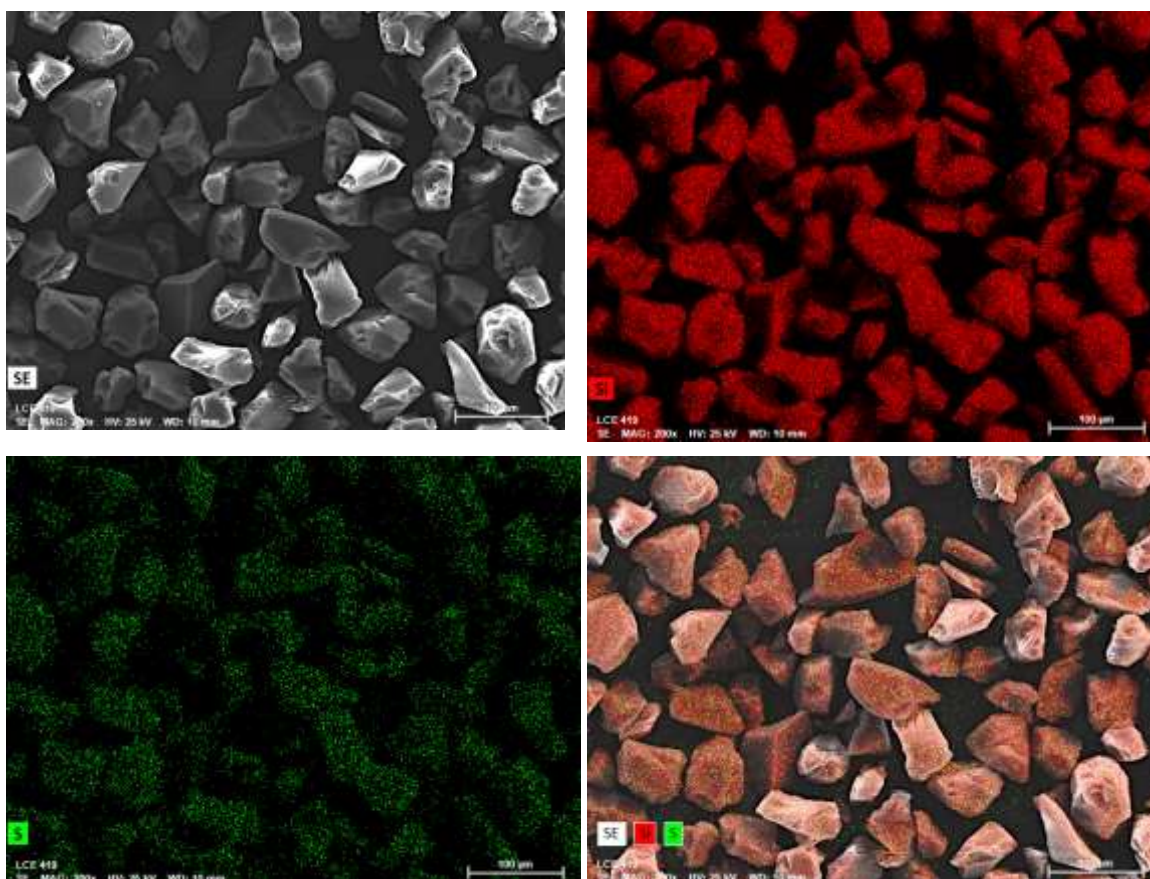


Figure S5. SEM-EDS of $\text{SiO}_2\text{-SH}$.

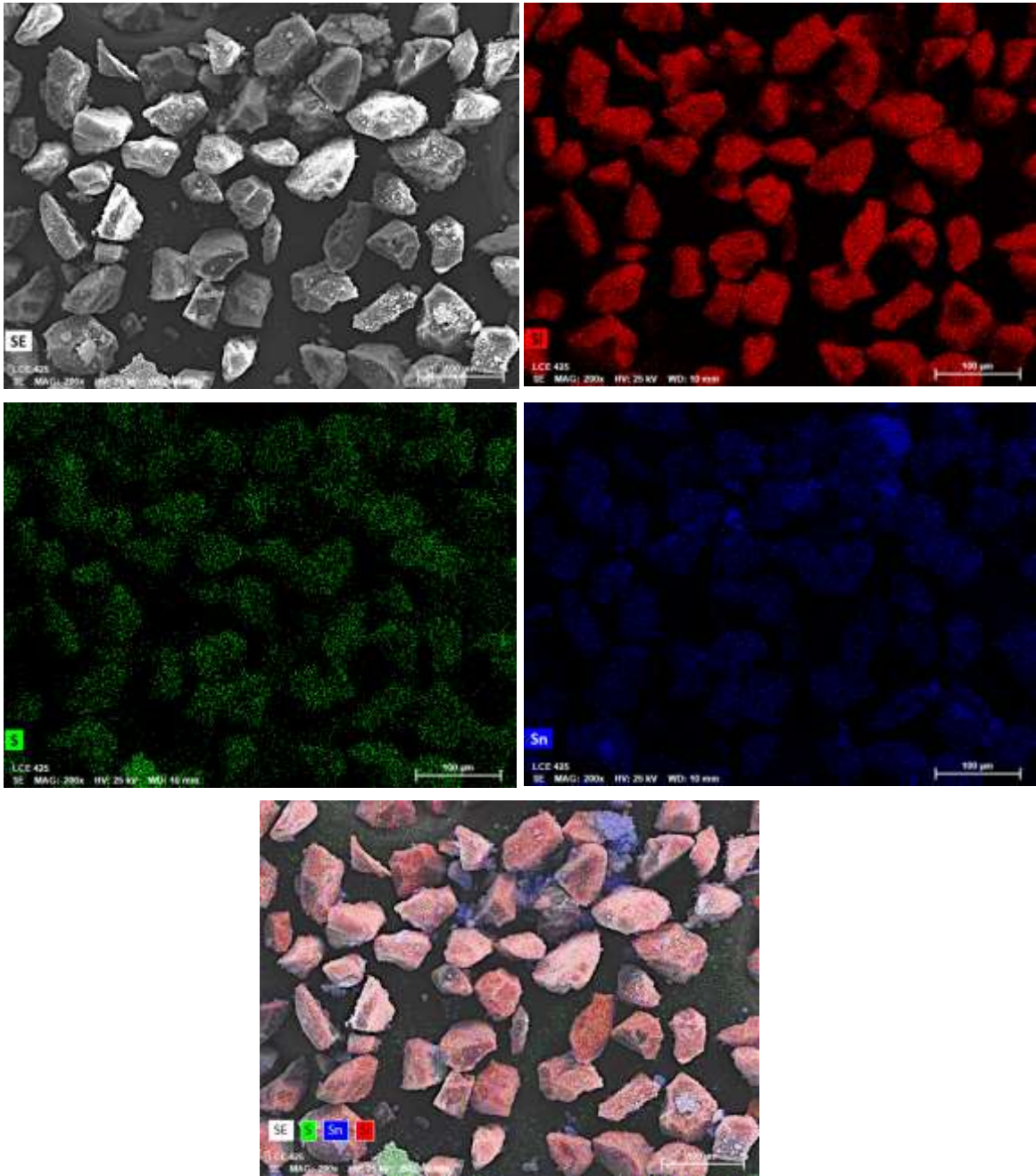


Figure S6. SEM-EDS of $\text{SiO}_2\text{-S-SnBu}_2$.

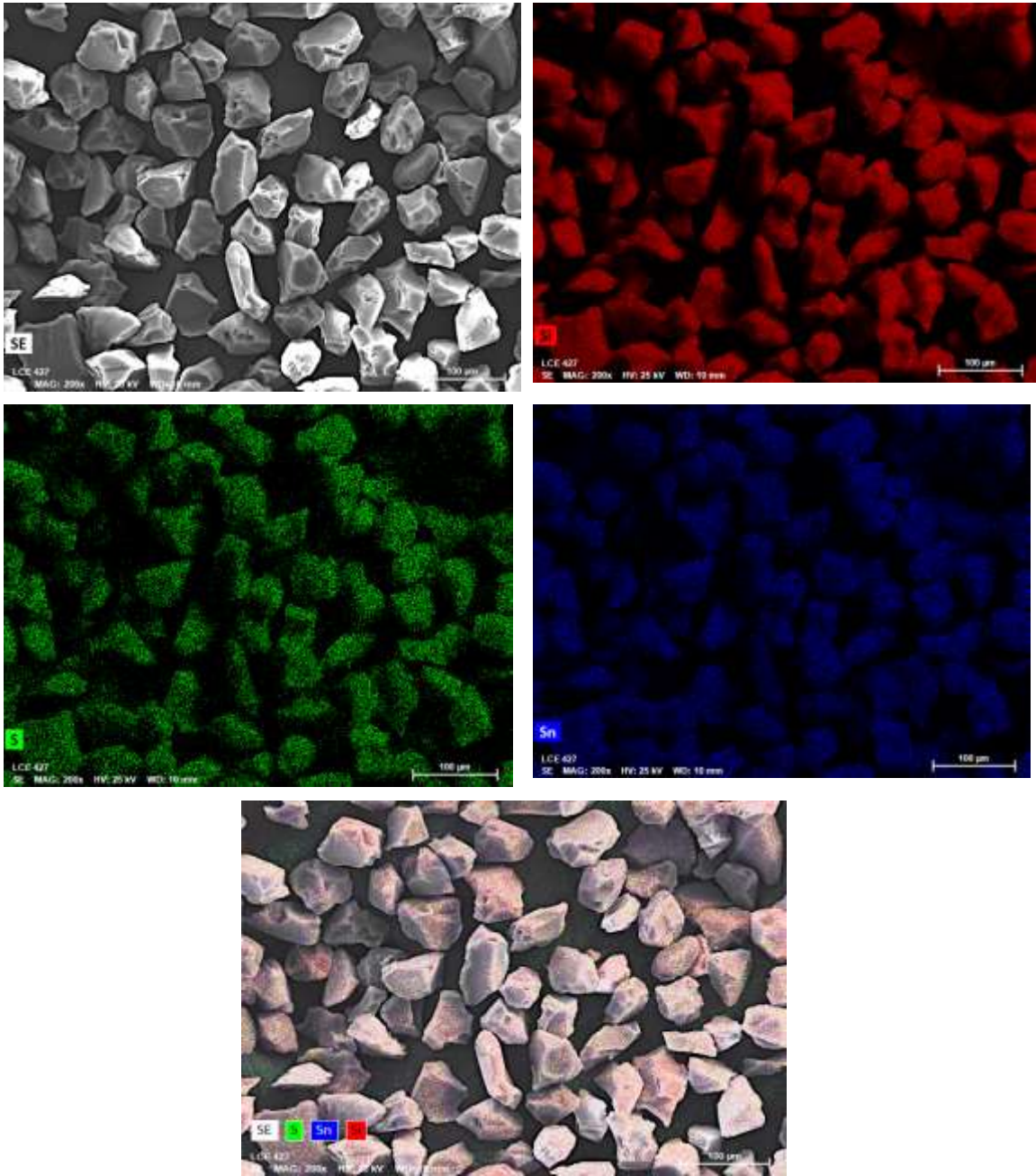
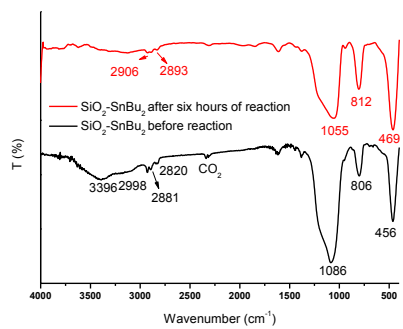
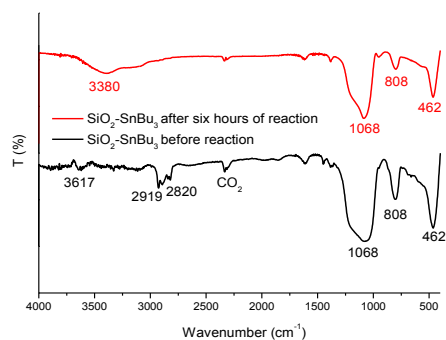


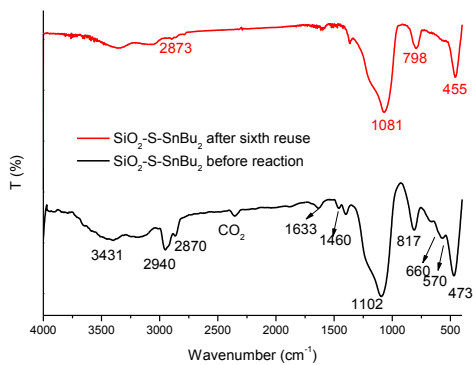
Figure S7. SEM-EDS of $\text{SiO}_2\text{-S-SnBu}_3$.



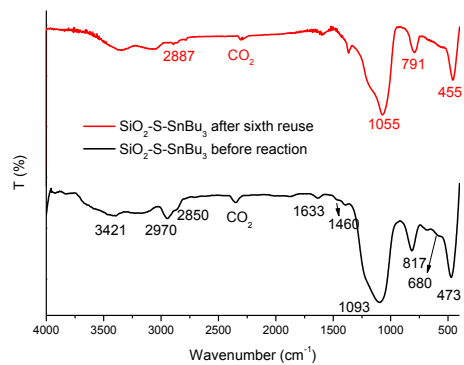
$\text{SiO}_2\text{-SnBu}_2$



$\text{SiO}_2\text{-SnBu}_3$

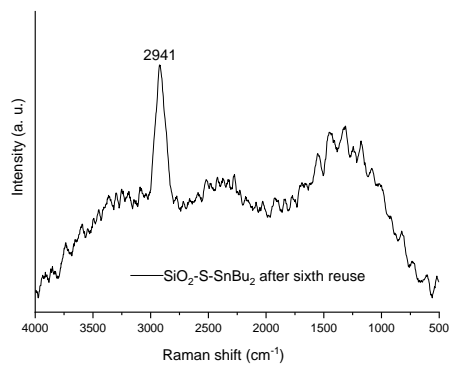


$\text{SiO}_2\text{-S-SnBu}_2$

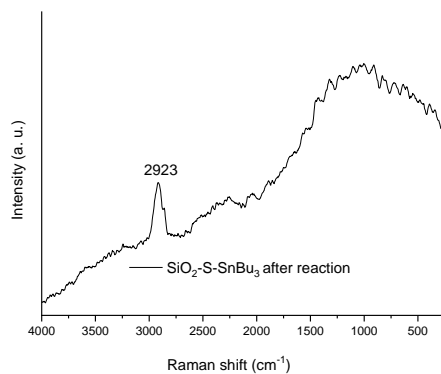


$\text{SiO}_2\text{-S-SnBu}_3$

Figure S8. FTIR spectra of the solids after use.

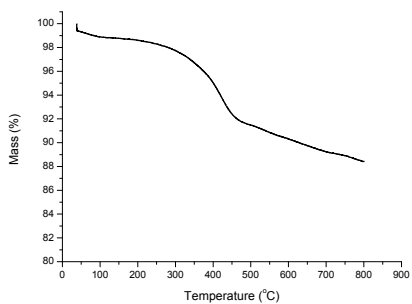


$\text{SiO}_2\text{-S-SnBu}_2$

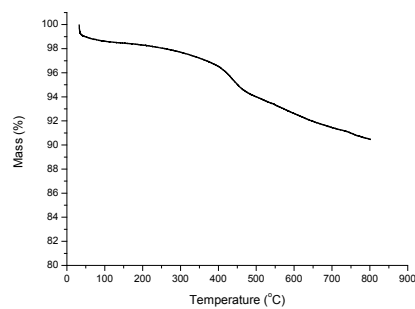


$\text{SiO}_2\text{-S-SnBu}_3$

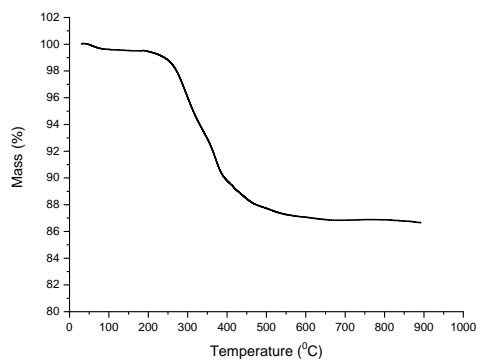
Figure S9. Raman spectra of the solids after sixth reuse.



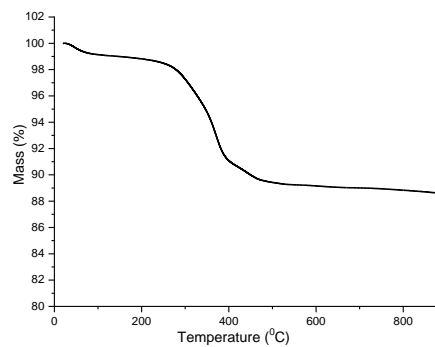
$\text{SiO}_2\text{-SnBu}_2$



$\text{SiO}_2\text{-SnBu}_3$

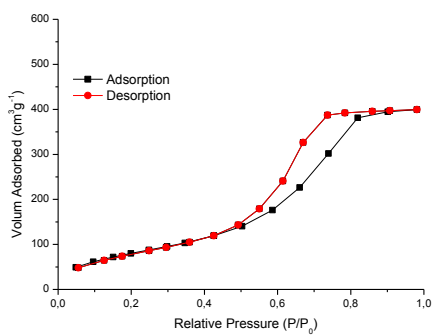


$\text{SiO}_2\text{-S-SnBu}_2$

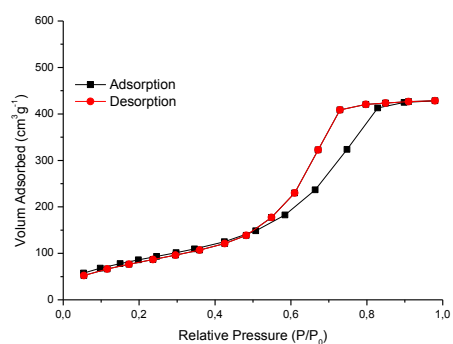


$\text{SiO}_2\text{-S-SnBu}_3$

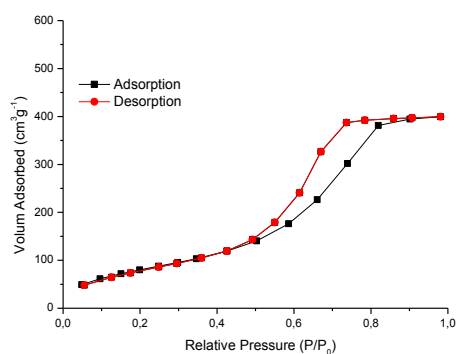
Figure S10. Thermograms of materials after 6 h of reaction.



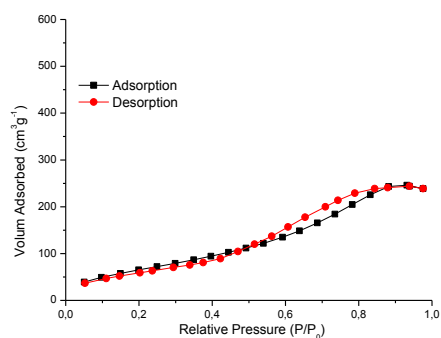
SiO₂-SnBu₂ 6 h of reaction



SiO₂-SnBu₃ after 6 h of reaction



SiO₂-S-SnBu₂ after sixth reuse



SiO₂-S-SnBu₃ after sixth reuse

Figure S11. Textural analysis of materials after use.

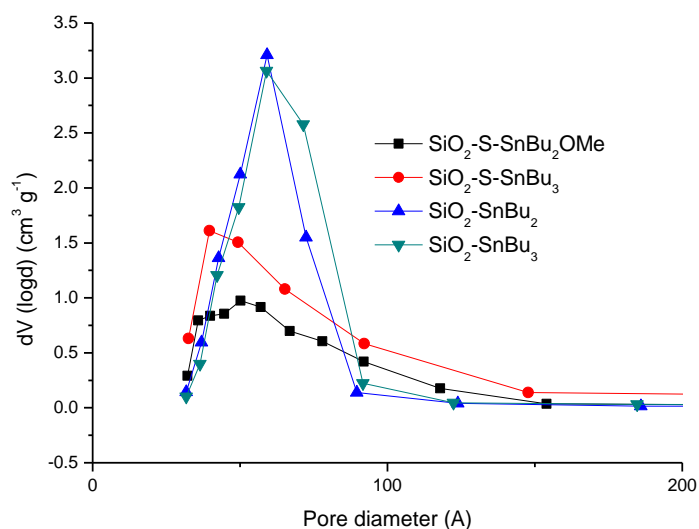


Figure S12. Pore diameter distribution of materials after reuse.

Table S1. Percentage of total mass loss before and after the reaction transesterification

Catalyst	Total mass loss / %			
	Before reaction		After reaction ^a	
	30-150 °C	150-800 °C	30-150 °C	500-800 °C
Non-activated SiO ₂	6.6	5.5	–	–
Activated SiO ₂	1.2	2.8	–	–
SiO ₂ -SnBu ₃	1.5	10	4.5	6
SiO ₂ -SnBu ₂	0.5	17	1.2	10
SiO ₂ -SH	0.3	10	–	–
SiO ₂ -S-SnBu ₃	0.3	18	1.0	10
SiO ₂ -S-SnBu ₂	0.9	18	0.5	12

^aAfter 6 h.

Table S2. Textural and structural properties of the solids investigated after reuse

Sample	S _{BET} / (m ² g ⁻¹)	V / (cm ³ g ⁻¹)	D _{BJH} / nm
SiO ₂ -SnBu ₃ ^a	327	0.7	5.9
SiO ₂ -SnBu ₂ ^a	312	0.7	5.9
SiO ₂ -S-SnBu ₃ ^b	325	0.6	4.0
SiO ₂ -S-SnBu ₂ ^b	261	0.4	4.6

^aSix hours of reaction; ^bafter sixth reuse. S_{BET}: Brunauer-Emmett-Teller (BET) surface area; V: pore volume; D_{BJH}: Barrett-Joyner-Halenda (BJH) pore diameter.