**Supplementary Information**

# Fly Ash Upcycling to Functional Silica Nanomaterials: Insights into Synthetic Strategies towards Efficient adsorbents for Water Purification

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**Table S1.** Elemental analysis of FA. The sample underwent microwave-assisted digestion in a mixture of nitric acid, hydrochloric acid, and hydrofluoric acid at elevated temperature. The total element content in the solution was then measured with Inductively Coupled Plasma Sector Field Mass Spectrometry (ICP-SFMS; Thermo Scientific Element XR)

| **Element** | **Amount (mg/kg dry FA)** |
|---|---|
| **Al** | 31900 |
| **As** | 16.6 |
| **Ba** | 984 |
| **Be** | 1.16 |
| **Ca** | 89700 |
| **Cd** | 2.55 |
| **Co** | 16.4 |
| **Cr** | 99.6 |
| **Cu** | 663 |
| **Fe** | 99600 |
| **Hg** | 0.0536 |
| **K** | 15200 |
| **Mg** | 21700 |
| **Mn** | 566 |
| **Mo** | 8.09 |
| **Na** | 7460 |
| **Nb** | 7.72 |
| **Ni** | 76.3 |
| **P** | 85000 |
| **Pb** | 142 |
| **S** | 3300 |
| **Sb** | 9.72 |
| **Sc** | 4.77 |
| **Si** | 165000 |
| **Sn** | 49.7 |
| **Sr** | 1510 |
| **Ti** | 4920 |
| **V** | 62.2 |
| **W** | 18.9 |
| **Y** | 15.3 |
| **Zn** | 2600 |
| **Zr** | 148 |
| LOI 1000°C | 1.52 % |

**Table S2.** Elemental analysis of FA residue after acid treatment. The sample underwent microwave-assisted digestion in a mixture of nitric acid, hydrochloric acid, and hydrofluoric acid at elevated temperature. The total element content in the solution was then measured with Inductively Coupled Plasma Sector Field Mass Spectrometry (ICP-SFMS; Thermo Scientific Element XR)

| **Element** | **Amount (mg/kg dry FA)** |
|---|---|
| **Al** | 38400 |
| **As** | 3.16 |
| **Ba** | 2350 |
| **Be** | 1.08 |
| **Ca** | 33400 |
| **Cd** | 0.145 |
| **Co** | 4.1 |
| **Cr** | 123 |
| **Cu** | 132 |
| **Fe** | 15800 |
| **Hg** | <0.05 |
| **K** | 21900 |
| **Mg** | 3550 |
| **Mn** | 119 |
| **Mo** | 1.66 |
| **Na** | 8850 |
| **Nb** | 14.4 |
| **Ni** | 20.2 |
| **P** | 7810 |
| **Pb** | 47.2 |
| **S** | 1620 |
| **Sb** | 9.15 |
| **Sc** | 2.79 |
| **Si** | 343000 |
| **Sn** | 19.4 |
| **Sr** | 550 |
| **Ti** | 9350 |
| **V** | 37 |
| **W** | 19 |
| **Y** | 9.67 |
| **Zn** | 316 |
| **Zr** | 238 |
| LOI 1000°C | 2.84 % |

**Table S3.** FA composition before and after Si extraction measured by EDS analysis

| | FA | Si extracted FA |
|---|---|---|
| Element | Atomic % | Atomic % |
| O | 58.88 | 57.12 |
| Si | 17.96 | 7.63 |
| K | 0.99 | 0.69 |
| Ti | 0.74 | 1.22 |
| Fe | 0.68 | 1.12 |
| P | 0.55 | 0.02 |
| Na | 0.48 | 13.47 |
| Mg | 0.30 | 0.19 |
| Al | 2.41 | 1.67 |
| Ca | 0.14 | 0.14 |
| C | 15.03 | 15.86 |
| N | 1.71 | 0.85 |
| S | 0.11 | 0.00 |
| Total | 100.00 | 100.00 |

**Table S4.** Elemental composition of SSS measured by EDS analysis. The SSS solution was dried on the carbon tape prior the measurement

| | SSS solution |
|---|---|
| Element | Atomic % |
| C | 23.87 |
| O | 53.80 |
| Na | 20.54 |
| Si | 1.29 |
| P | 0.15 |
| N | 0.34 |
| Total | 100.00 |

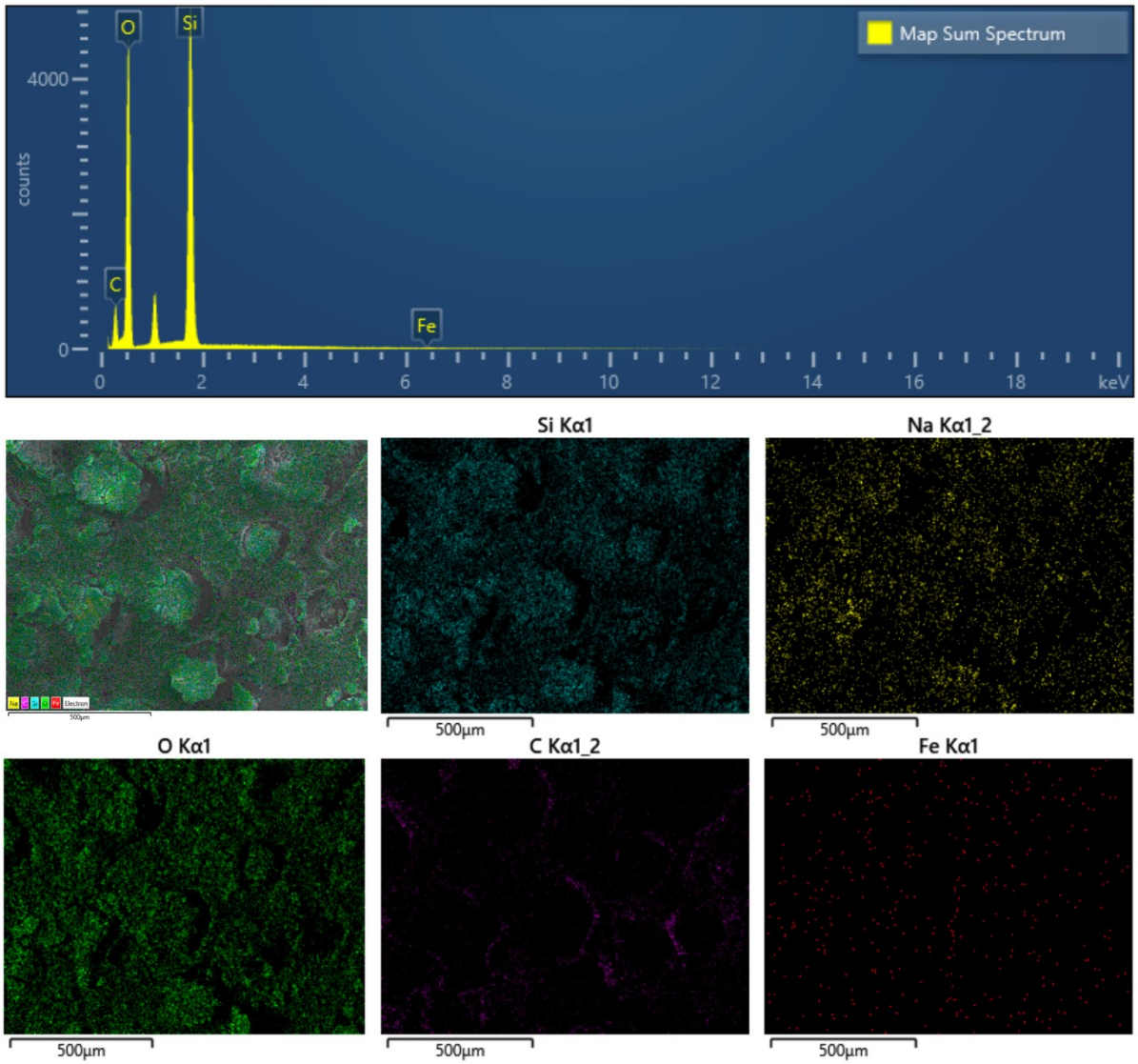



**Fig. S1** EDS analysis of dense $SiO_2$ NPs derived from FA

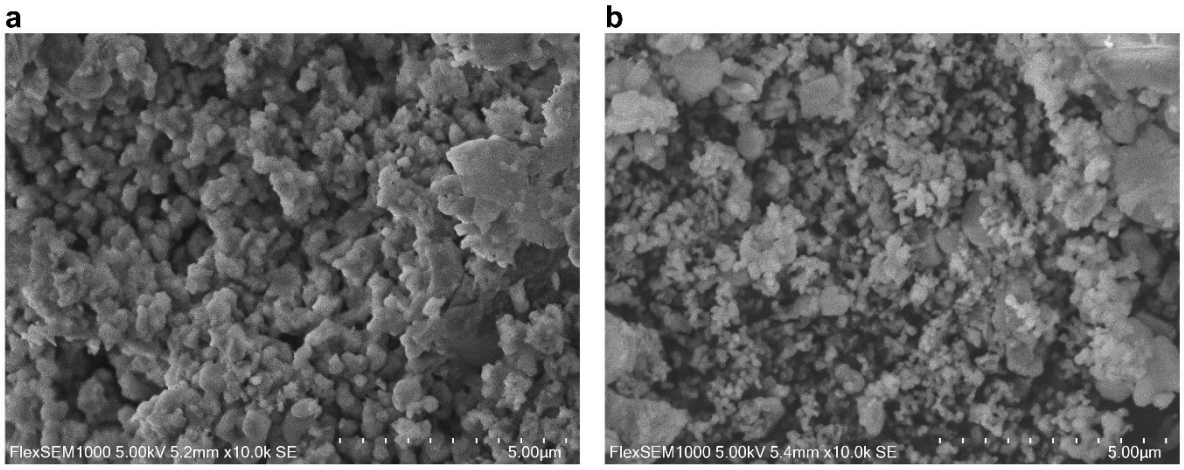
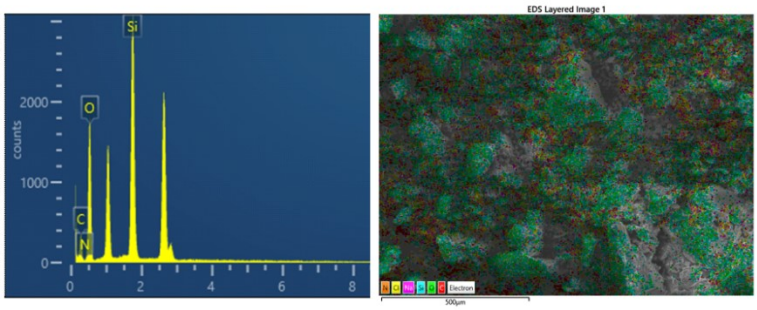
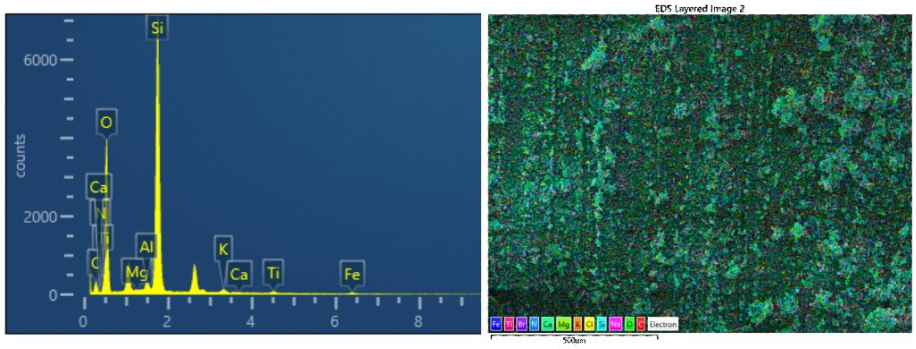



**Fig. S2** SEM images of synthesized M_$SiO_2$ NPs (a) and 1 pot_M_$SiO_2$ (b)




| Map Sum Spectrum | |
|---|---|
| Element | Atomic % |
| C | 31.99 |
| O | 41.81 |
| Na | 7.79 |
| Si | 10.23 |
| Cl | 8.18 |
| N | 0.00 |
| Total | 100.00 |

**Fig. S3** EDS analysis of M_SiO2 NPs derived from FA




| Map Sum Spectrum | |
|---|---|
| Element | Atomic % |
| C | 23.24 |
| O | 52.37 |
| Si | 15.06 |
| Cl | 1.87 |
| Na | 2.78 |
| K | 0.26 |
| Al | 0.48 |
| Ti | 0.22 |
| Fe | 0.24 |
| Mg | 0.00 |
| Ca | 0.01 |
| N | 3.47 |
| Total | 100.00 |

**Fig. S4** EDS analysis of 1 pot_M_SiO2 NPs derived from FA

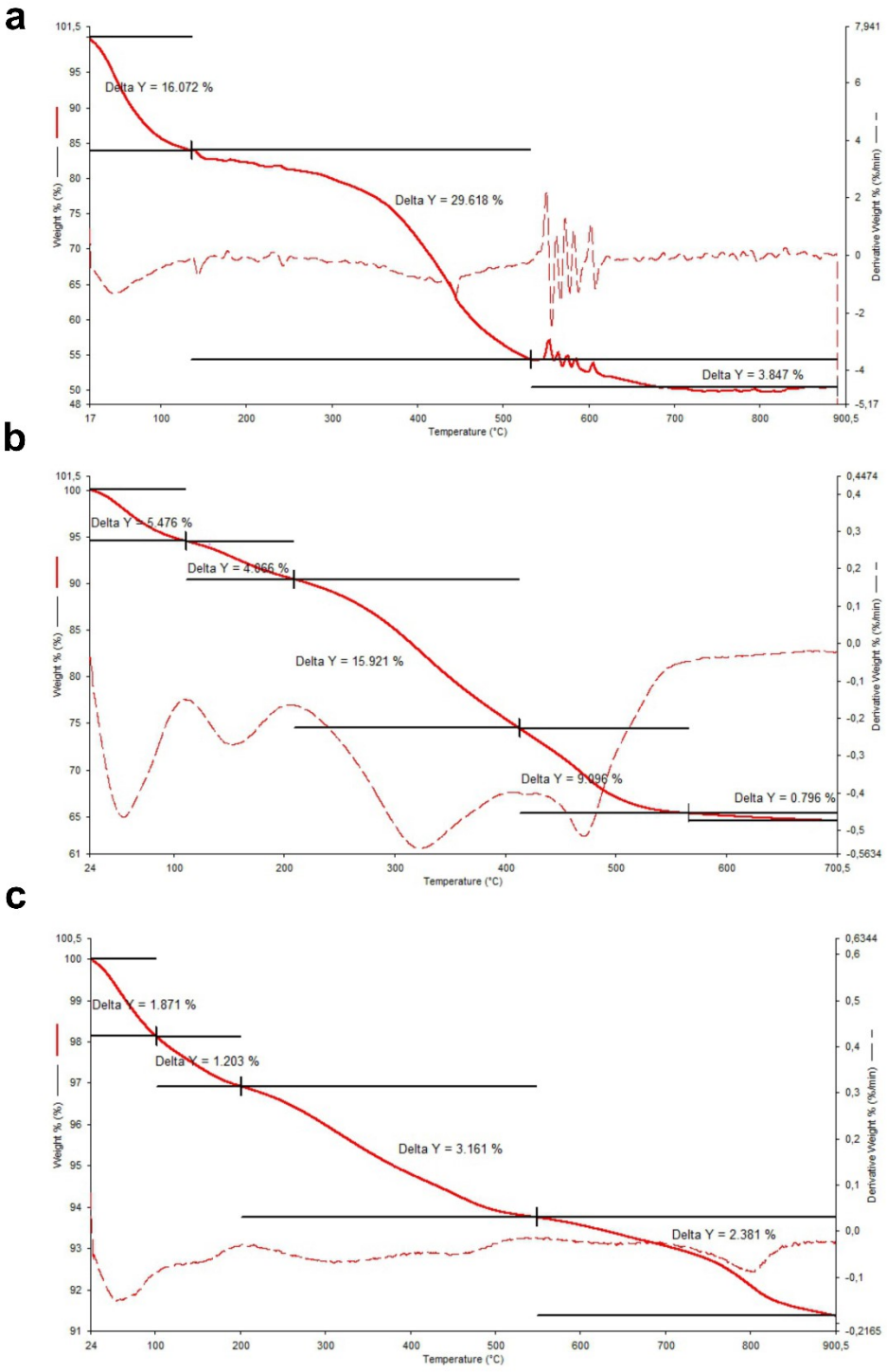



**Fig. S5** TGA curves of synthesized $SiO_2$ nanomaterials grafted with TMSPDETA ligand: (a) M_$SiO_2$, (b) D_$SiO_2$ and (c) 1 pot_M_$SiO_2$ NPs

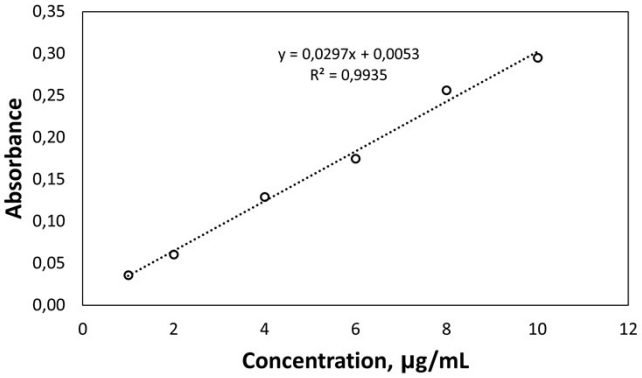



**Fig. S6** Calibration curve DFC measured by UV-Vis with DFC standard concentrations varying between 1-10 µg/mL

**Table S5**. Amount of grafted TMSPDETA ligand on synthesized $SiO_2$ NPs

| Sample | Grafted TMSPDETA (mmol/g) |
|---|---|
| D_$SiO_2$ | 0.94 |
| M_$SiO_2$ | 1.12 |
| 1 pot_M_$SiO_2$ | 0.2 |

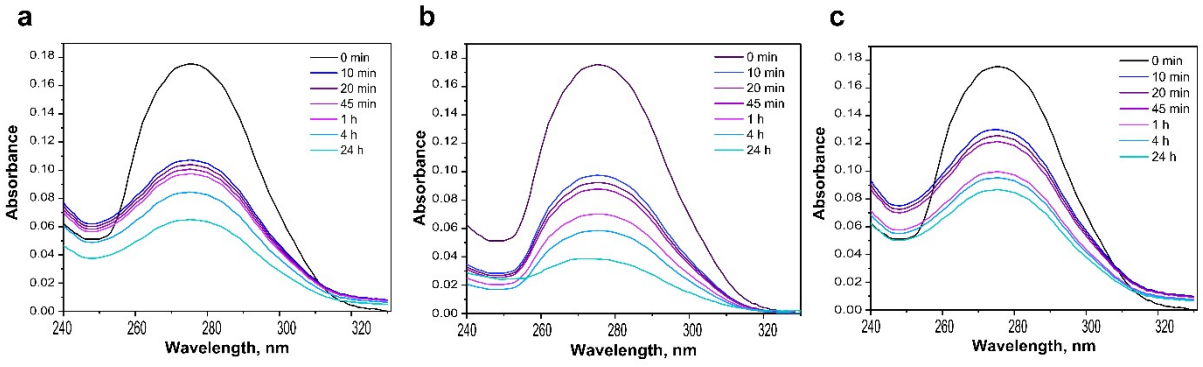



**Fig. S7** Adsorption kinetics of DFC on $SiO_2$ nanomaterials: (A) D_$SiO_2$, (B) M_$SiO_2$ and (C) 1 pot_M_$SiO_2$ NPs