

Supporting information

Assessing the Effect of Acid and Alkali Treatment on Halloysite-Based Catalyst for Dry Reforming of Methane

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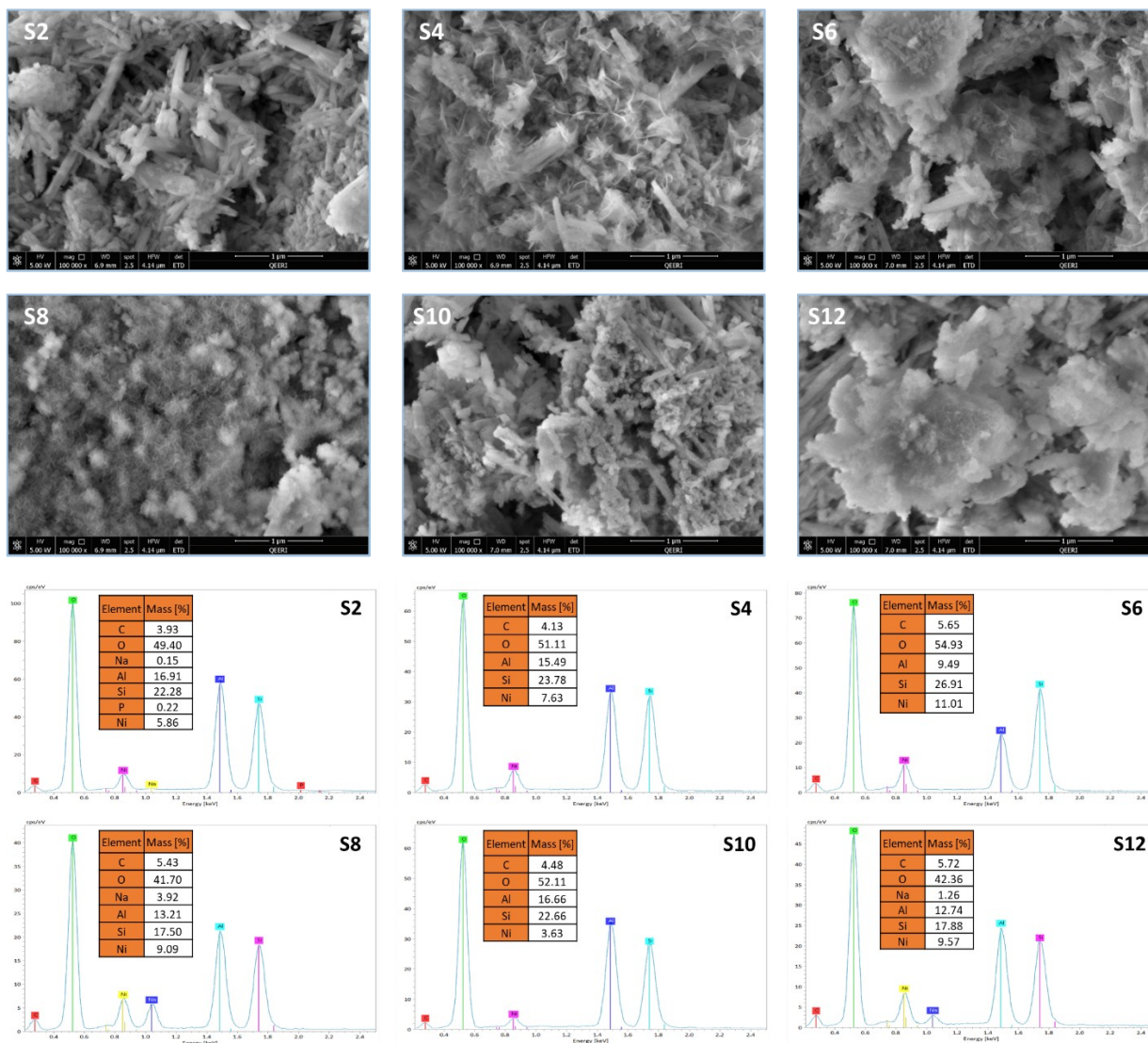


Figure S1. SEM-EDS images with corresponding elemental analysis of (S2) Ni-Raw HNT, (S4) Ni-AHNT (HNO_3), (S6) Ni-AHNT (H_2SO_4), (S8) Ni-AIHNT (ball milled), (S10) Ni-AIHNT (NaOH), and (S12) Ni-AIHNT (grinded).

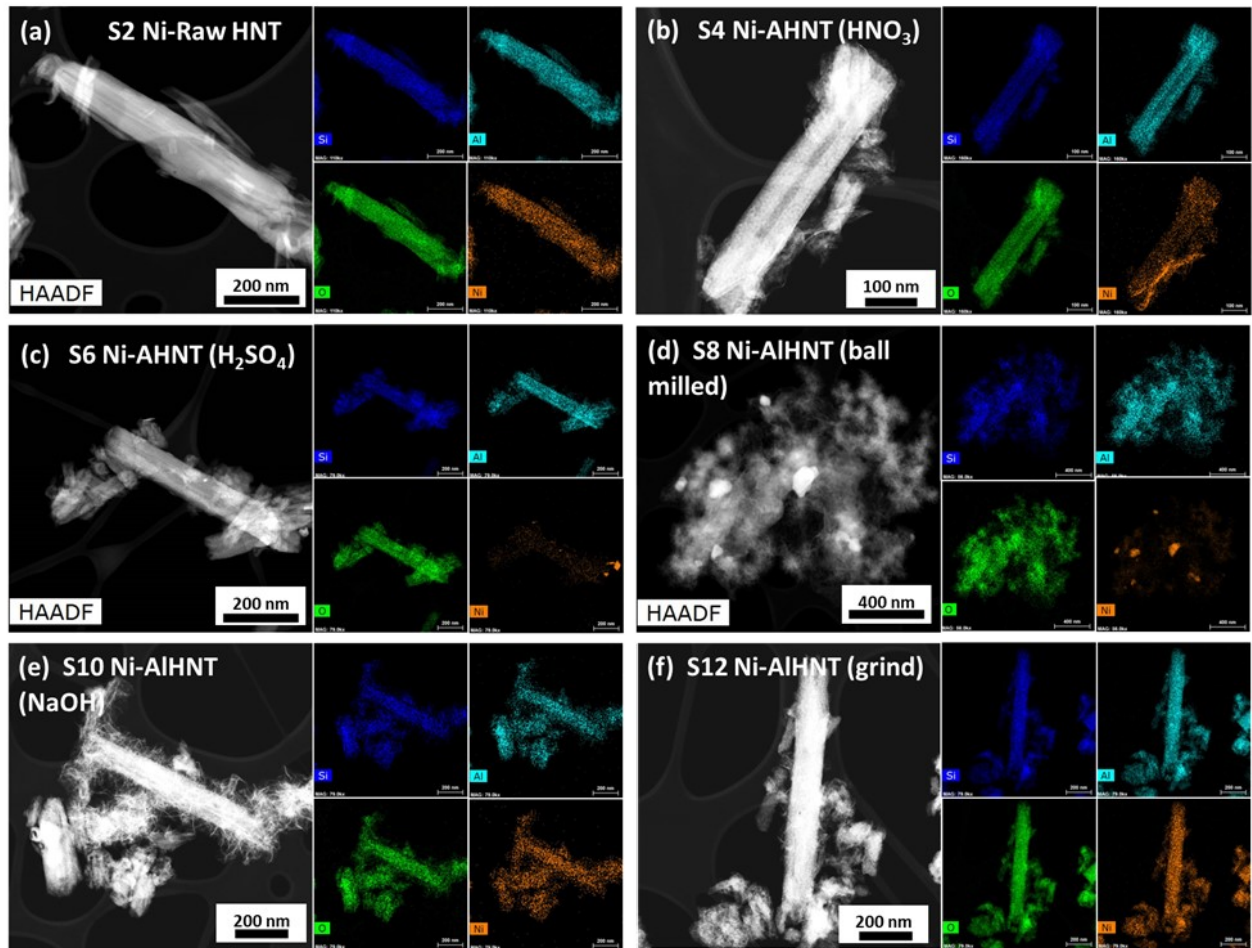


Figure S2. TEM-EDS elemental mapping of Si, Al, O, and Ni on (a) Ni-Raw HNT, (b) Ni-AHNT (HNO_3), (c) Ni-AHNT (H_2SO_4), (d) Ni-AIHNT (ball milled), (e) Ni-AIHNT (NaOH), and (f) Ni-AIHNT (grinded).

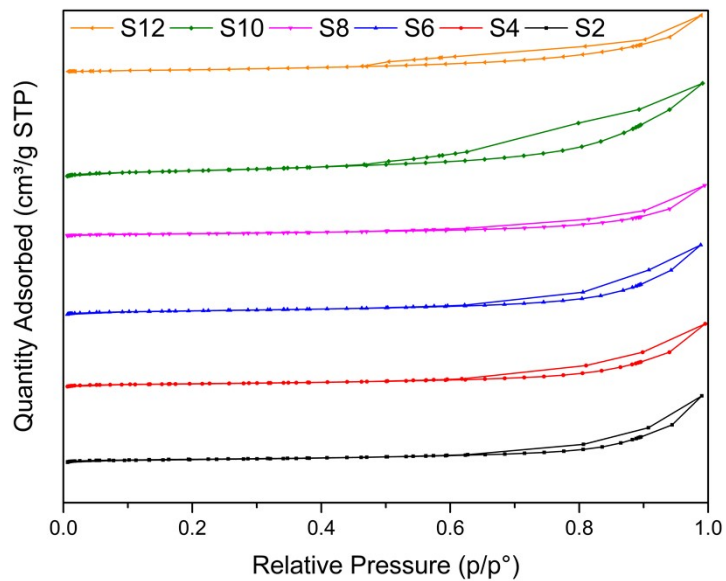


Figure S3. Isotherms for the developed catalysts measured by N_2 -sorption analysis.

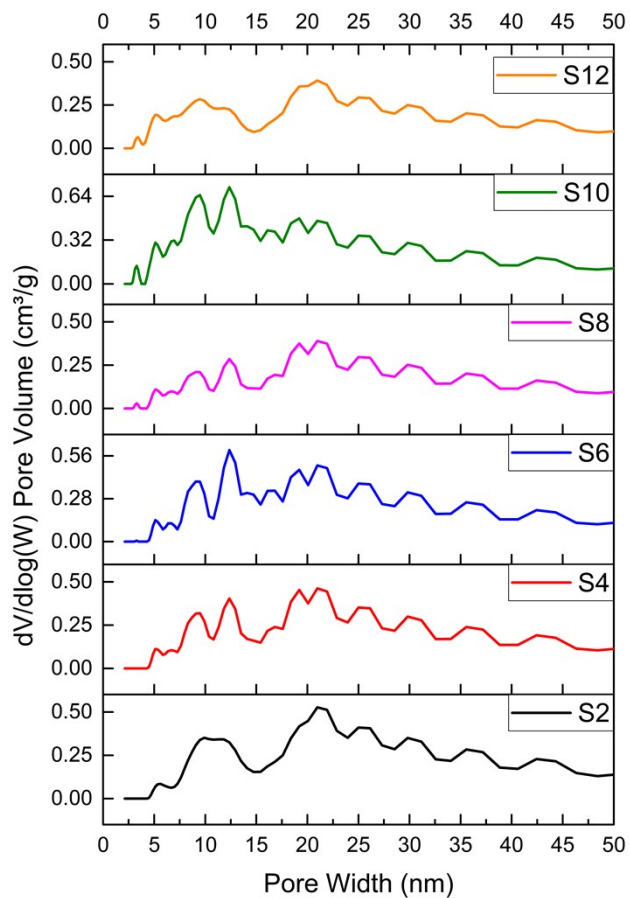


Figure S4. Pore Size Distribution for the developed catalysts measured by N_2 -sorption analysis.

Table S1. BET and Pore Size analysis for undoped samples.

| Sample | S_{BET} [m²/g] | Micropore area [m²/g] | V_t [cm³/g] | Pore Size [nm] | Si/Al ratio |
|---|---|---|--|---------------------------|------------------------|
| S01: Raw HNT | 65 | 7 | 0.276 | 14.7 | 1.02 |
| S03: HNO₃-HNT | 40.5 | 8 | 0.23 | 14.1 | 1.26 |
| S05: H₂SO₄-HNT | 62.5 | 9 | 0.354 | 14 | 1.45 |
| S07: Alkaline-HNT NaNO₃ ball milled | 19 | 6 | 0.105 | 19.5 | 0.99 |
| S09: Alkaline-HNT NaOH | 50.19 | 5.55 | 0.3208 | 19.25 | 0.99 |
| S11: Alkaline-HNT NaNO₃ grinded | 47 | 7 | 0.277 | 13.8 | 1.04 |

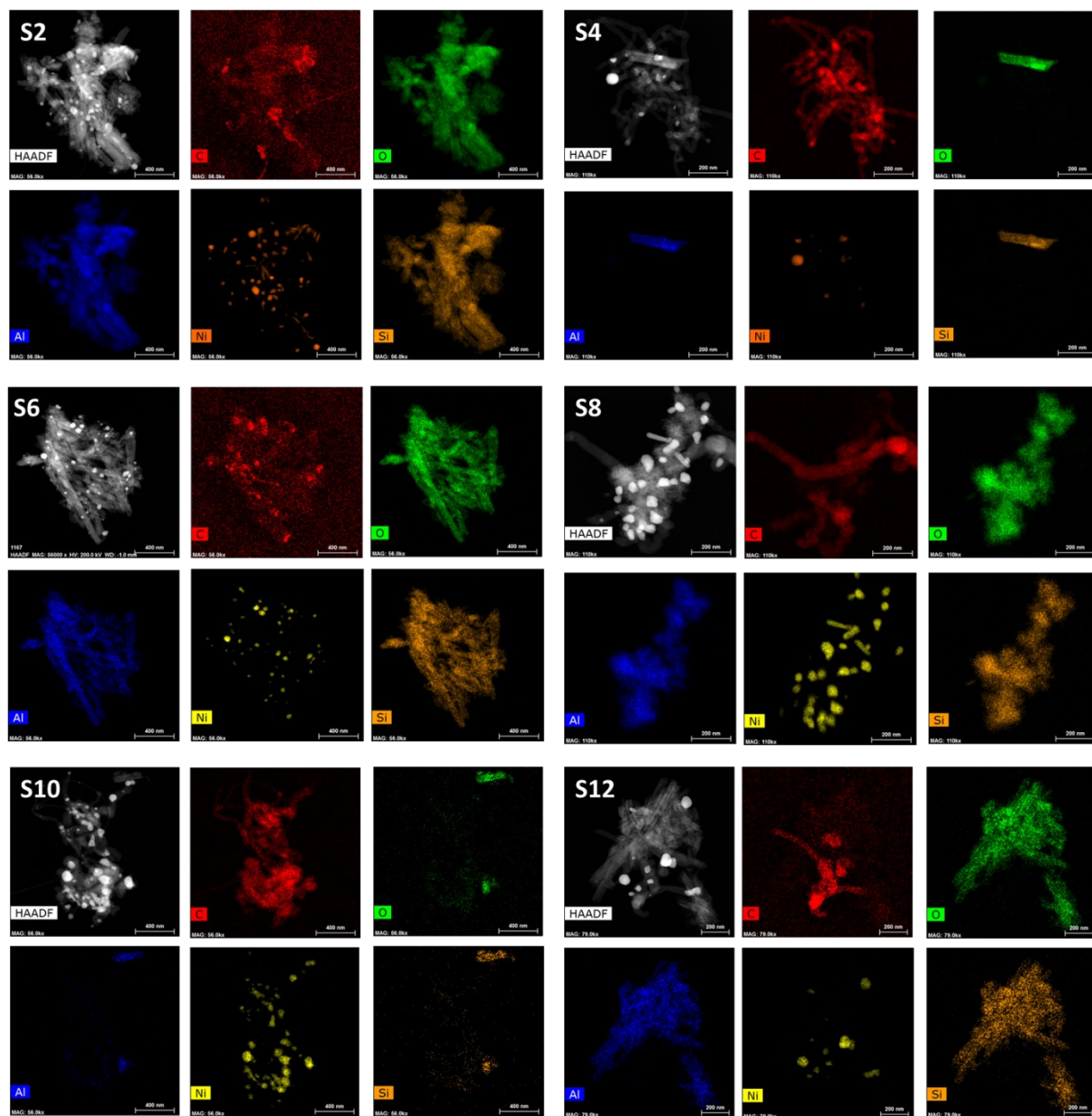


Figure S5. TEM-EDS elemental mapping of Si, Al, O, C and Ni on spent catalysts S2- Ni-Raw HNT, S4- Ni-AHNT (HNO_3), S6-Ni-AHNT (H_2SO_4), S8-Ni-AIHNT (ball milled), S10-Ni-AIHNT (NaOH), and S12-Ni-AIHNT (grinded).

Table S2. Particle size analysis for both fresh and spent catalyst, calculated from TEM images.

| Sample | Mean Particle Size [nm] | Standard Deviation | Average Ni particle size increase due to sintering [nm] |
|-----------|-------------------------|--------------------|---|
| Fresh S2 | 7.05 | 6.64 | +42 |
| Spent S2 | 49.22 | 25.07 | |
| Fresh S4 | 7.35 | 9.23 | +22 |
| Spent S4 | 29.57 | 19.69 | |
| Fresh S6 | 9.24 | 12.71 | +33 |
| Spent S6 | 42.58 | 21.77 | |
| Fresh S8 | 30.43 | 24.16 | +17 |
| Spent S8 | 47.44 | 33.63 | |
| Fresh S10 | 20.88 | 18.04 | +29 |
| Spent S10 | 49.97 | 38.02 | |
| Fresh S12 | 13.67 | 15.89 | +54 |
| Spent S12 | 67.24 | 37.29 | |

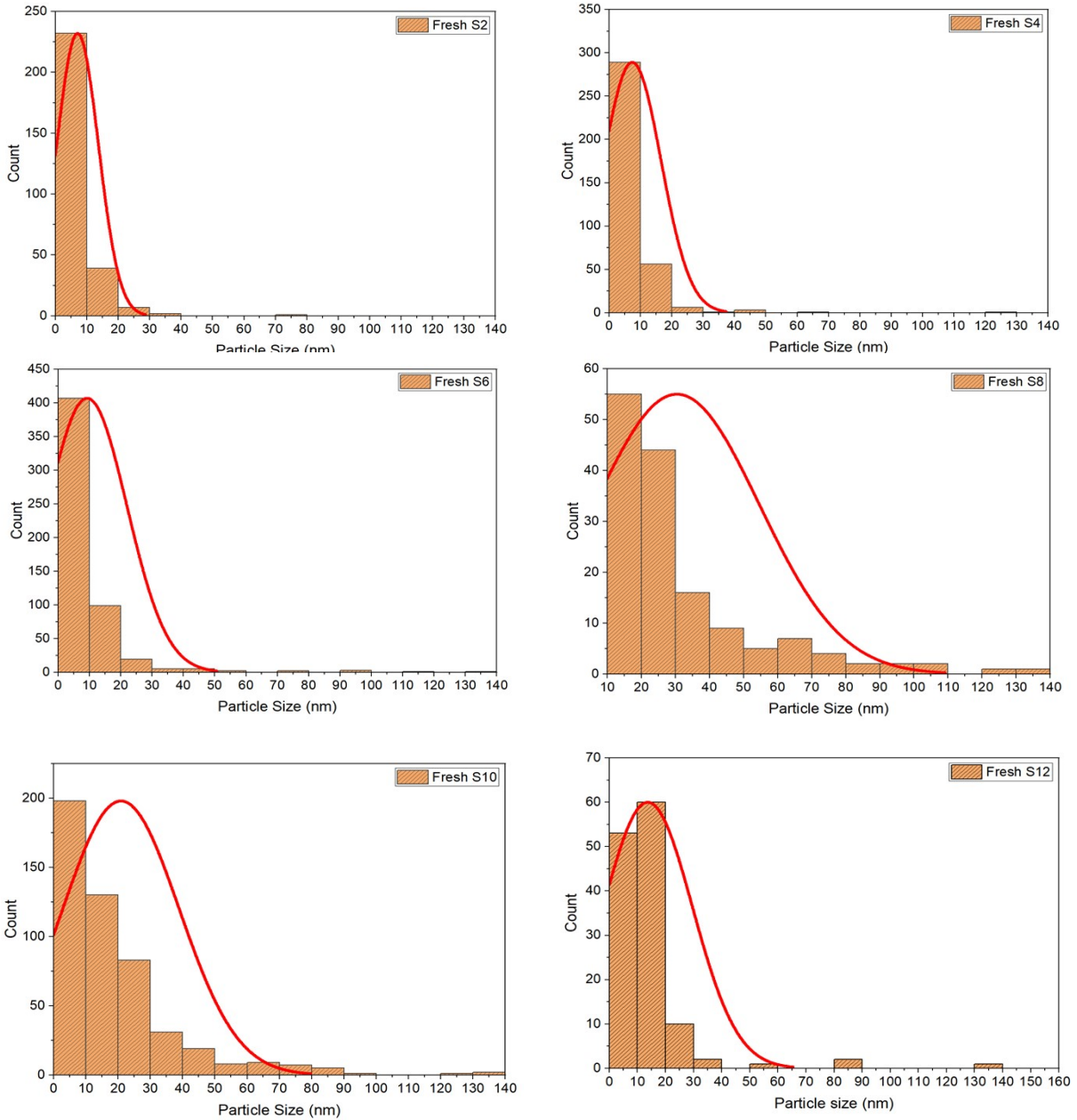


Figure S6. Particle size distribution for the fresh catalysts, calculated from TEM analysis.

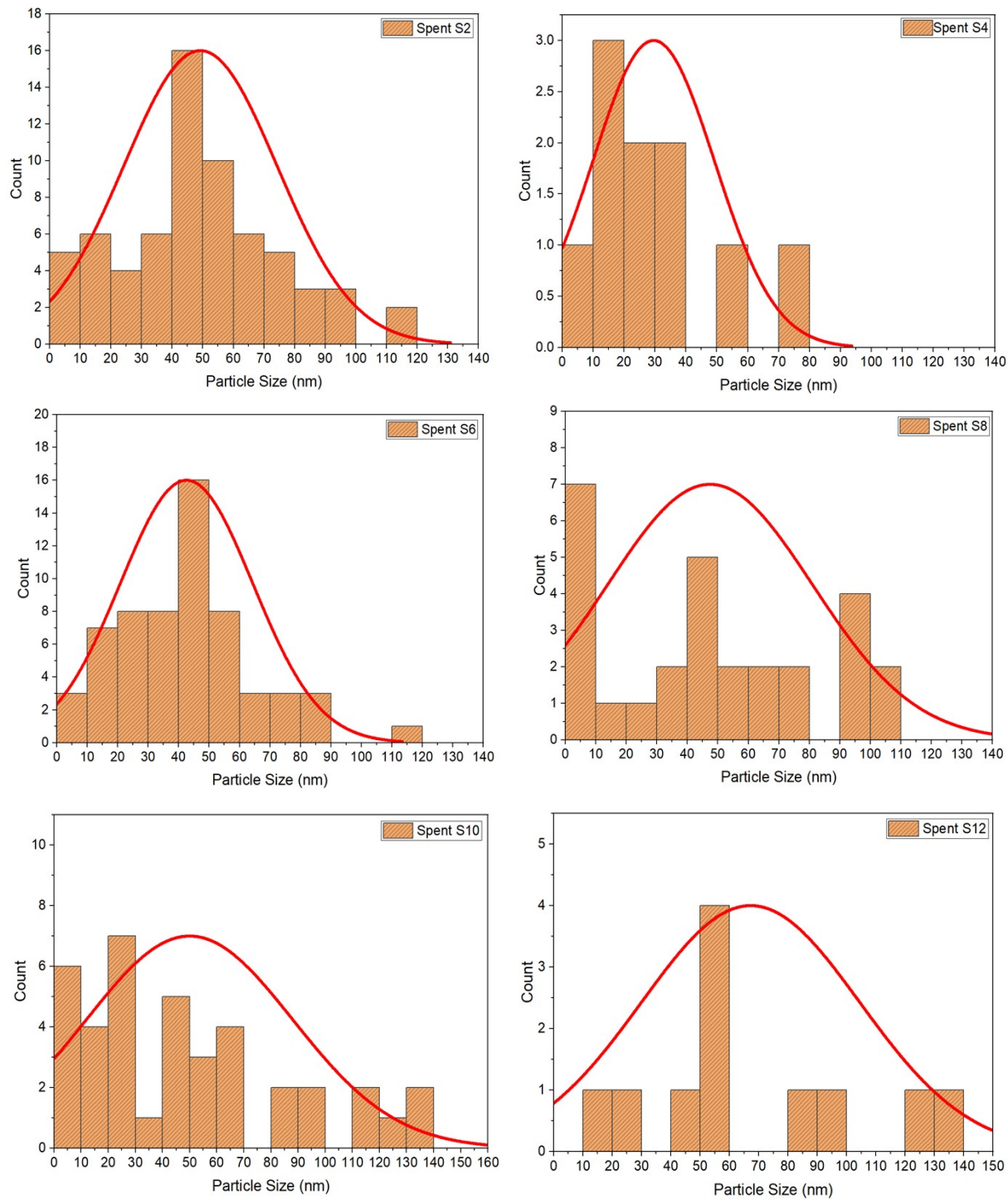


Figure S7. Particle size distribution for the spent catalysts, calculated from TEM analysis.

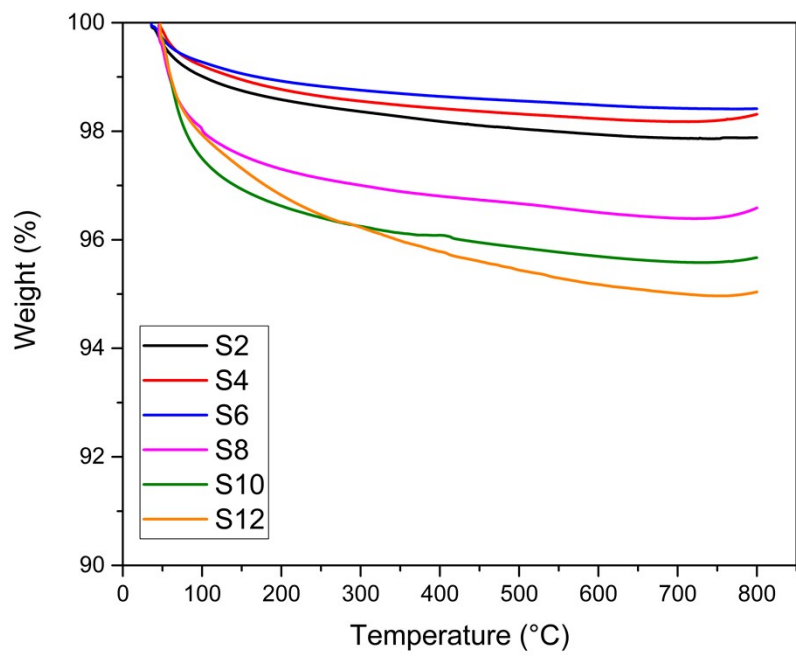


Figure S8. TGA analysis for the reduced catalysts