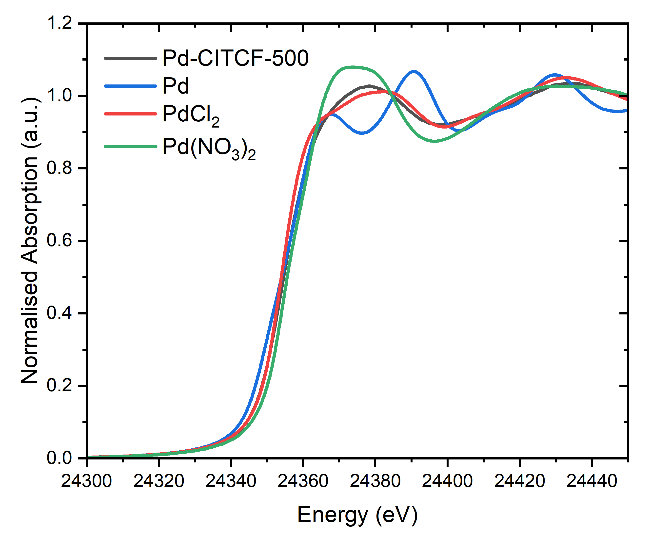
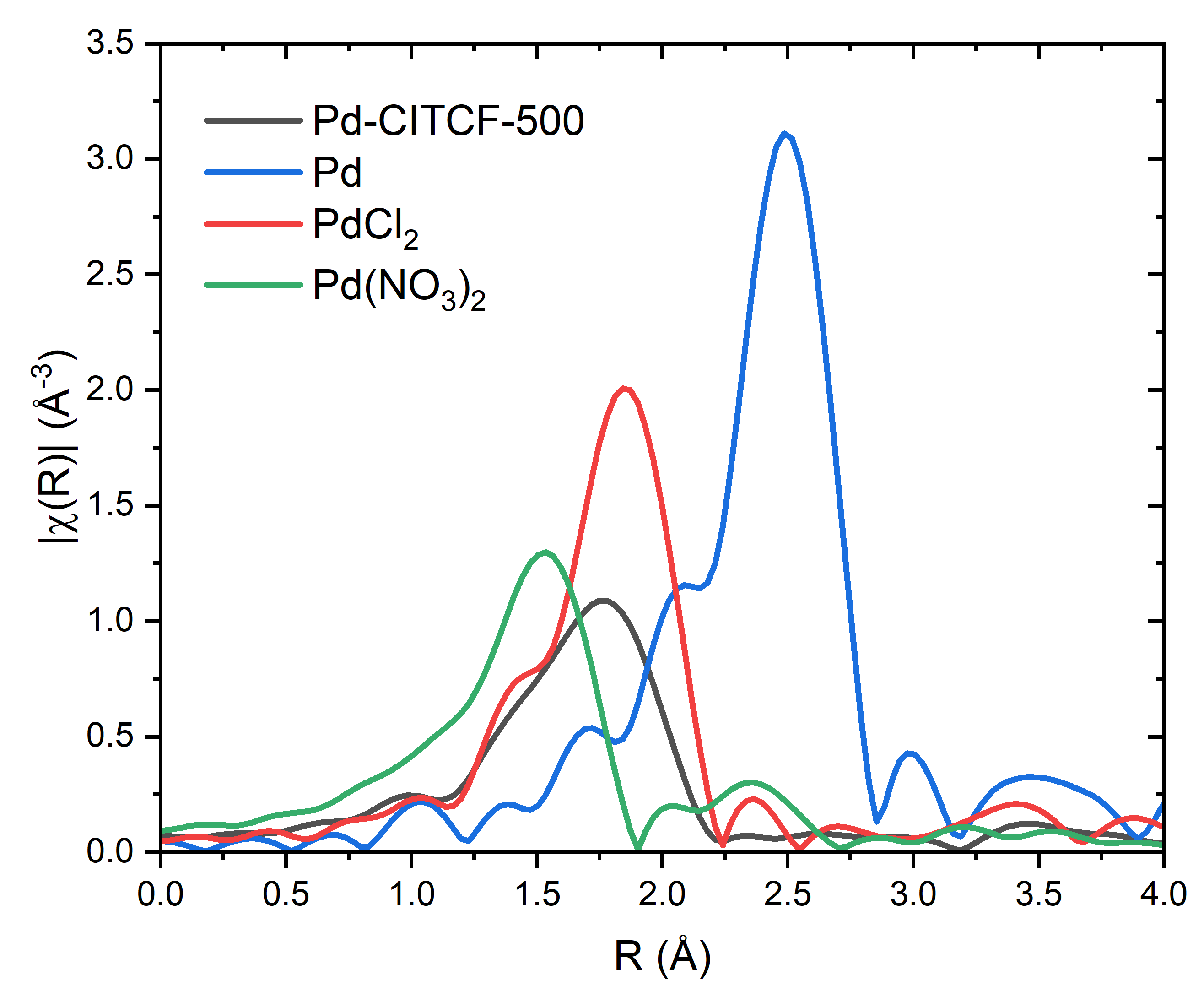
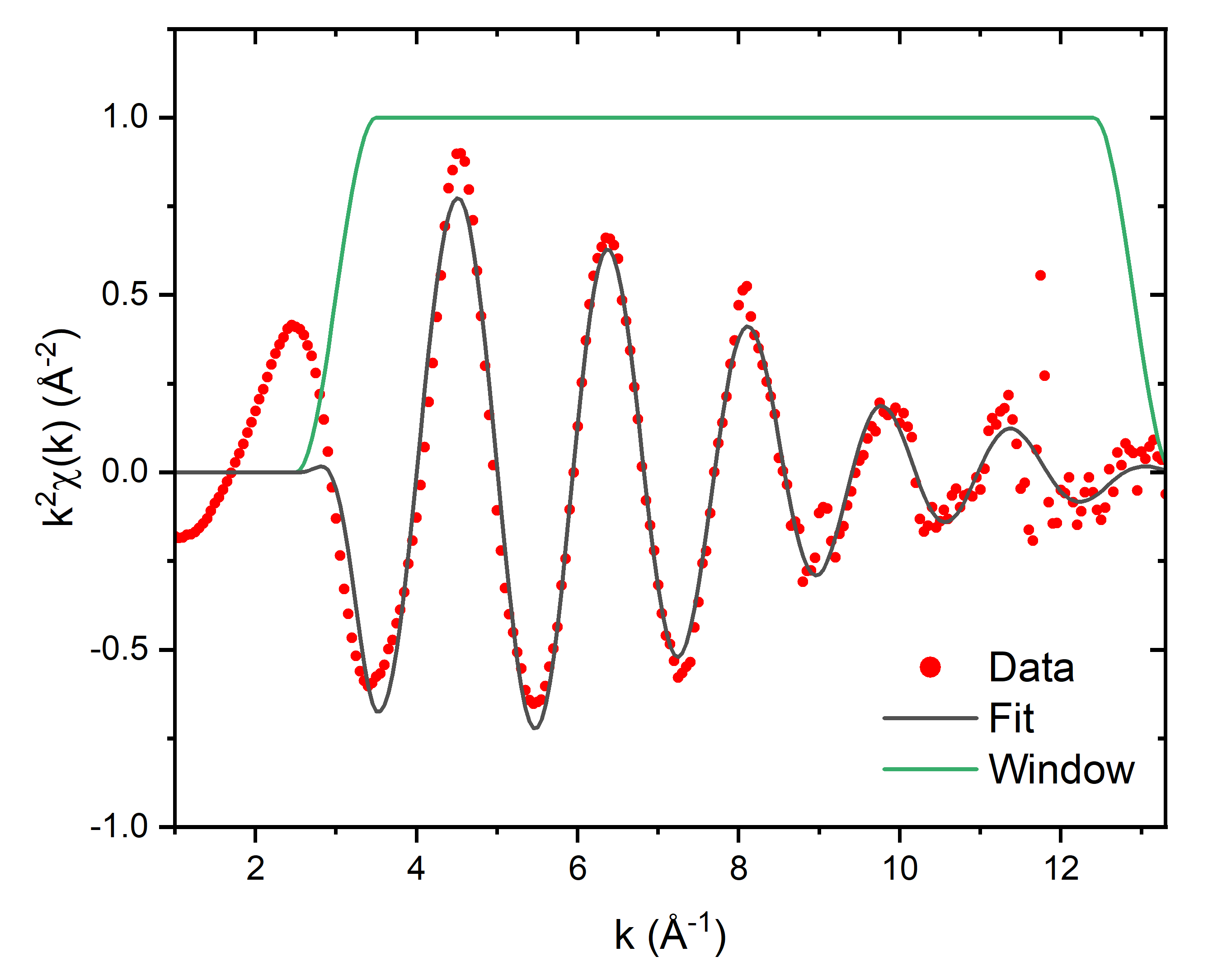
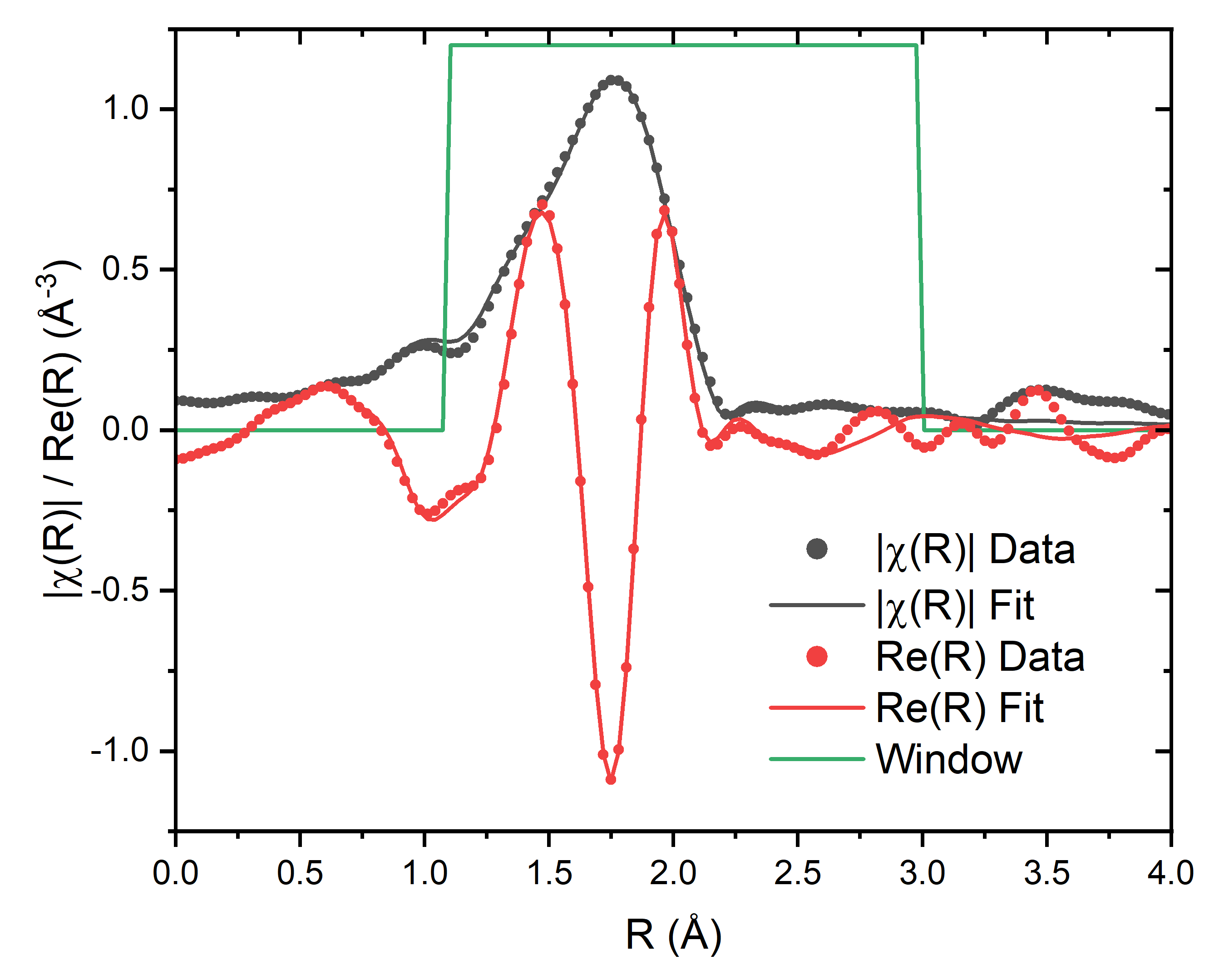
Pd500-CITCF EXAFS analysis

Both samples that were measured appear to be within tolerance of being identical. Analysis here is presented for the Pd500-CITCF sample.





Comparison of the XANES region for the sample compared to relevant standards shows a remarkable similarity to a PdCl2 standard demonstrating Pd2+ oxidation state and a similar average local coordination. From the R space it is evident that the Pd coordinates predominantly with Pd-S/Cl from the appearance of the peak centered at 1.8 Å (not phase corrected) with a should to lower R likely due to bonding with light scattering atoms (probably a small contribution of Pd-O).



Fitting of the EXAFS yields a high agreement with the data when using a combination of Pd-S and Pd-O interactions. The results of the fitting are given in the table below. The R-factor for the achieved fit was 0.009 as reported by Artemis.

Scattering Path S02 N σ2 (Å2) e0 (eV) R (Å)

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Pd-S 0.880 2.9 ± 0.3 0.0058 ± 0.001 1.5 2.31 ± 0.01

Pd-O 0.880 0.8 ± 0.2 0.0025 ± 0.001 1.5 2.05 ± 0.02

Methods text:

X-ray absorption spectroscopy (XAS) experiments were performed at the SuperXAS beamline of the Swiss Light Source at the Paul Scherrer Institute in Villigen, Switzerland.[1] The Swiss Light Source operates in top-up mode at 400 mA and 2.4 GeV. Radiation from a 2.9 T bending magnet was collimated using a Si-coated collimating mirror at 2.9 mrad (which also served to reject higher order harmonics) subsequently monochromatized by a Si(111) channel-cut monochromator. Focusing of the beam to a spot size of 1.0×0.2 mm on the sample was achieved by a Rh-coated toroidal mirror. XAS spectra were collected using samples pressed to 13 mm diameter pellets formed of 40 mg of as-received sample mixed with approximately 20 mg cellulose. The sample measurements were performed in quick fluorescence mode using a PIPS detector.[2] Simultaneous measurement of the reference Pd foil was performed using 20 cm long ionization chamber filled with 1 bar N2 and 1 bar Ar. Spectra were collected with 1 Hz scanning speed and 300 spectra were averaged per sample. The data were processed using ProQEXAFS,[3] to calibrate, normalize and average the obtained XAS spectra with subsequent EXAFS analysis performed within the Demeter software package.[4] The amplitude reduction factor used during EXAFS refinements was obtained from fitting to the reference Pd foil and was determined to be 0.88.

[1] https://doi.org/10.1107/S1600577515018007

[2] https://doi.org/10.1107/S1600577520002350

[3] https://doi.org/10.1107/S1600577519017053

[4] https://doi.org/10.1107/S0909049505012719