Experimental report

25/01/2024

Proposal: 6-07-115 **Council:** 4/2023

Title: Structural and vibrational properties of sulfur confined into porous carbon

Research area: Chemistry

This proposal is a new proposal

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Samples: Sulfur
Porous Carbon
S@Porous carbon composites

Instrument	Requested days	Allocated days	From	To
IN1 LAG	6	3	04/09/2023	07/09/2023
D4	4	4	23/09/2023	27/09/2023

Abstract:

The general context of our studies is the confinement of sulfur in nanoporous materials for applications in the field of energy (batteries and gas adsorption). In this proposal we focus on the confinement of sulfur into meso and microporous carbons. Our objective is to provide new insight on the structural and dynamical properties of confined sufur.
We plan to monitor the structure on D4 and the vibrational density of states on IN1-Lagrange. These measurements will allow to validate our hypothesis on amorphous phase stabilization upon confinement and to analyse the S/C interaction as a function of temperature and pore network.

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Sulfur was incorporated in meso- and microporous carbon by impregnation methods. Thermodesorption analyses enabled us to shown that sulfur is more strongly retained in micropores than in mesopores. Recent differential scanning calorimetry measurements suggested that in micropores, amorphous sulfur can be stabilized while strong effect of confinement on the sulfur melting peak are observed under mesoporous confinement.

The aims of the experiment was to monitor the structure and the density of states of the confined phase and their evolution as a function of temperature in order to (i) to validate our hypothesis on amorphous phase stabilization upon confinement, (ii) to analyse the S/C interaction as a function of temperature and pore network. Two porous carbon matrices were measured during the experiments: a mesoporous graphitic one (Cmeso) and a microporous amorphous one (Cmicro).

The following samples were measured:

- Cmeso and S@Cmeso
- Cmicro and S@Cmicro

The samples were measured at 77K and 300K in the energy range 200-3500cm-1 by using the Cu220 configuration (Figure 1). All the spectra were normalized to monitor counts and the quartz empty cell is substracted. This measurement allowed to analyze the dependence of the S/C interaction as a function of the pore network.

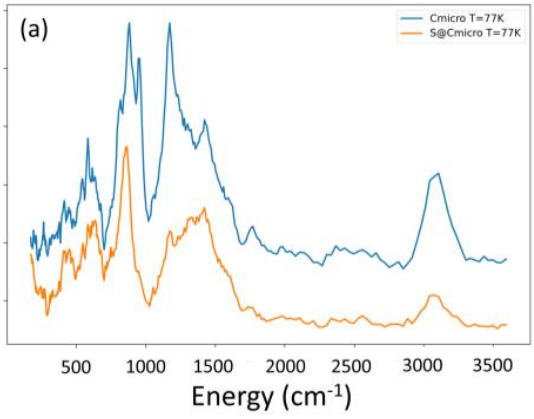

Figure 1: Generalised Density of States (GDOS) of Cmicro and S@Cmicro at 77K.