Report on the outcomes of a Short-Term Scientific Mission[1]

Action number: CA20126

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Details of the STSM

Title: Characterization and testing of waste-based magnetic activated carbon materials

Start and end date: 17/10/2022 to 28/10/2022

Description of the work carried out during the STSM

The group led by María Victoria Gil and Covadonga Pevida has three main lines of research, all involving adsorption in the gas phase and the use of biomass: the production of hydrogen through Sorption Enhanced Steam Reforming (SESR), the CO_2 capture at low temperatures (0-50 ºC) from different gas mixtures and the gasification of biomass for H_2 production (partial combustion using several types of biomass, steam and O_2 in limiting amounts). I had the chance to follow one cycle of SESR, a recent variation of the conventional steam reforming method, in which biomass (in this case – usually methane) and steam are used to produce H_2 and CO_2. This novel approach introduces in-situ CO_2 capture using adsorbents (and a catalyst) to shift the equilibrium towards the H_2 production, achieving conversion rates usually above 90 % (conventional steam reforming conversion rate is around 60 %).

The collaboration with INCAR started as they provided N_2 isotherm analysis of our activated carbon samples for the determination of BET (Brunauer–Emmett–Teller) porosity parameters like specific surface area, pore volume, size and distribution. I had the possibility of performing the analysis myself in a Micrometrics ASAP 2420 equipment. Two samples were degassed (120 ºC) overnight prior to adsorption measurements. Then, each sample tube was filled with N_2 at different relative pressures and adsorption occurs until the equilibrium is reached (-196 ºC), which for my samples could be for up to 20 h (this depends on the temperature and the microporosity of the sample – some samples can take up to 3 days to reach equilibrium). Parameters were calculated from the BET isotherm using the built-in equipment software, making sure the fit is good. After the experiment the material can be recuperated and used for other tests, once it is degassed again, including performing CO_2 adsorption isotherms in a Micromeritics TriStar analyser.

I had the chance to follow the development process of carbon adsorbents for CO_2 capture from combustion (or other) gases. Once materials are produced, the first step is to test the materials performance in CO_2 adsorption by TGA analysis. The materials are heated at 120 ºC under N_2 flow to remove any water adsorbed, the temperature is lowered to 50 ºC to simulate that of combustion gases and CO_2 is added to the chamber at a predefined percentage. The equipment continuously measures small variations in weight related with adsorbed CO_2 and after equilibrium the CO_2 percentage is incremented, and the sequence repeats until finally using 100 % of CO_2. The same evaluation

[1] This report is submitted by the grantee to the Action MC for approval and for claiming payment of the awarded grant. The Grant Awarding Coordinator coordinates the evaluation of this report on behalf of the Action MC and instructs the GH for payment of the Grant.

described is performed using steam mixed with the CO_2 stream but only once the right microporosity (micropore diameter < 0.7 nm) of the material is confirmed (evaluation in CO_2 using a surface area and porosity analyser). The kinetics of steam adsorption are usually quite slower than those of CO_2, hence allowing the separation of both fractions although sometimes steam can change the dynamics of CO_2 adsorption.

Description of the STSM main achievements and planned follow-up activities

The goal of the STSM was to gain experience in performing N_2 and CO_2 adsorption isotherms of activated carbons, as well as briefly accessing the potential of specific materials for CO_2 capture. The referred goals were achieved successfully. Two materials were characterized regarding the N_2 adsorption isotherms: activated carbon produced from primary paper mill sludge using microwave pyrolysis (ACMW) and ex-situ magnetic activated carbon (MACX4). BET parameters were determined and are presented as follows:

		ACMW	MACX4
N_2	BET surface area ($m^2\ g^{-1}$)	1055.2 ± 0.5	825.98 ± 0.08
	Dubinin-Astakhov micropore volume ($cm^3\ g^{-1}$)	0.495 ± 0.002	0.387 ± 0.001
	Dubinin-Astakhov limiting pore width (nm)	1.67	1.67
	Dubinin-Astakhov limiting micropore capacity ($cm^3\ g^{-1}$)	320.29	250.23

For the CO_2 adsorption isotherms two different materials were analysed: activated carbon produced from spent brewery grains (ACSBG) and a one-step magnetic activated carbon produced primary paper mill sludge (S17). The determined parameters are represented as follows:

		ACSBG	S17
CO_2	Dubinin-Radushkevich surface area ($m^2\ g^{-1}$)	1144.07	225.12
	Dubinin-Radushkevich micropore volume ($cm^3\ g^{-1}$)	0.437 ± 0.004	0.086 ± 0.001
	Dubinin-Radushkevich limiting micropore capacity ($cm^3\ g^{-1}$)	227.71	44.81

Pore width distribution was also determined as this is an important factor governing adsorption of CO_2. Pore widths above 0.7 nm tend to decrease selectivity towards the CO_2 molecule. Overall, the tested materials were not ideal for CO_2 adsorption as they presented a wide range of pore width distribution, well over 0.7 nm.

I had the opportunity to attend a group's weekly meeting, where I presented my PhD work so far with valuable inputs from the team considering their different approach to adsorption. It was also very productive to participate in the discussion of the group's activities in the past week. The STSM allowed to share ideas and knowledge between members of two international groups in the field adsorption and porous materials, which otherwise would not be possible. It also strengthened collaborations ties as other opportunities for common work arose during my stay, namely using TPD (temperature programmed desorption) to better characterize functional group composition on the surface of the activated carbons and in this way shed some light into possible mechanisms of adsorption.