

Cost Action CM1404

CHEMISTRY OF SMART ENERGY CARRIERS AND TECHNOLOGIES: SMARTCATs

Report of the STSM with code COST-STSM-ECOST-STSM-CM1404-030417-083754

STSM Title

"Adsorption tests as diagnostic tool for pollutants emissions control in SECs combustion"

STSM duration

4 weeks

STSM period

3-28 April 2017

STSM applicant

Valentina Gargiulo, Research fellow at the Istituto di Ricerche sulla Combustione IRC-CNR of Naples

Host institution

Institute of Conditions Extrêmes et Matériaux: Haute Température et Irradiation, CEMHTI, belonging to the Centre National de la Recherche Scientifique (CNRS) in Orléans, France.

CEMHTI has a highly recognized international experience on the characterization of the physicochemical properties of materials under extreme conditions, through the understanding of the fundamentals of their structure at atomic scale with a special concern in the study of materials for energy applications and environmental remediation.

Host group

ADPOR Group under the coordination of Dr. Conchi O. Ania.

The host group has a long-standing interest and a well-known experience on research activities related to exploring precursors and developing advanced nanostructured materials for their use in high-tech applications, covering energy storage and conversion and environmental protection.

Working group

WG3

Keywords

Diagnostic, gaseous pollutants, CO₂, gas adsorption measurements

Motivation of the STSM

Nowadays the interest in natural and renewable forms of energy, also referred as smart energy carriers (SECs), is progressively increasing over the classical fossil fuel sources which are severely diminishing. Such carriers include hydrogen and natural gas. At the same time great attention is focused in monitoring pollutants emissions in SECs combustion. Adsorption techniques are a reliable diagnostic tool from both the technological and economical points of view (high energy efficiency, ease of control, low capital investment costs) to face the issues involved in SECs combustion (i.e. the presence of CO, CO₂, SO₂, NH₃ and VOCs in combustion exhaust causes combustion chamber corrosion and overall environmental problems). Separation and quantification of pollutants from gas exhaust is thus inevitably demanded in order to minimize the impact of SECs combustion. In a view of exploiting adsorption on solid matrix as diagnostic tool to monitoring gaseous pollutants emission in SECs combustion, an ideal sorbent should exhibit high selectivity toward CO, CO₂, SO₂, NH₃ and light hydrocarbons (i.e. CH₄) with respect N₂, possibly acting as potential pollutant storage sink.

Purpose (objectives) of the STSM

The topic of the research developed by Dr Gargiulo during the STSM was focused on exploring the fate of gases emitted to the atmosphere in typical SECs combustion processes (i.e., CO₂, CH₄, CO, H₂ among others). The approach was based on studying the differences in affinity of these gases and their mixtures in meso-nanoporous materials used for monitoring their control and in the storage, as well as the interactions in the confined pore voids to maximize physisorption and/or separation. The goal of these adsorption tests on new materials based on carbon, ideally produced via inexpensive, low-energy consumption methods, using renewable resources as precursors, with flexible morphologies, pore structures and functionalities, was the identification of the optimal shape, size, geometry and chemistry of the pores as ought to be for a given application like those described above.

Description of the work carried out during the STSM

Samples

A selection of chemically tailored materials prepared at IRC-CNR, differing in terms of textural, chemical and structural characteristics, was tested at the host institution where volumetric gas adsorption analyzers are available. The materials, prepared at IRC and analyzed at CEMHTI, are listed in the Table 1 along with the nomenclature used within the text to identify them.

The materials object of the experimental campaign of measurements performed during the STSM can be gathered into four groups: i) reference carbonaceous material (carbon black), ii) inorganic materials (nFeM, Ti-MIL125), iii) carbonaceous materials derived from biomass thermochemical conversions -carbonized rice husk (RH), biochars from cellulose (Char C), biochars from populus nigra wood (Char P)- and iv) chemical modified materials obtained by modifying carbonaceous materials derived from biomass thermochemical conversions (RH NaOH, Char P NaOH, Char P nFeM and Char P NH₂). The selected biochars differ not only for the type of biomass used for their preparation (cellulose fibers and populus nigra wood) but also for

the pyrolysis temperature employed (530, 650, 700 °C for cellulose fibers, 480 and 600 °C for populus nigra wood). A so great variability of samples was chosen to cover a large range of chemical and textural properties.

Table 1. List of samples and their nomenclature

Group	Sample	Description	Label
i	Carbon black	Furnace black carbon, selected as reference material	CB
ii	MIL-125 (Ti)	Metal organic framework made by Ti^{4+} and 1,4-benzene dicarboxylic acid (BDC) [1]	Ti-MIL125
	Magnetite nanoparticles	Ferric oxide nanoparticles prepared in presence of a surfactant in order to prevent agglomeration [2]	nFeM
iii	Biochars from cellulose pyrolysis	Biochars obtained by steam assisted slow pyrolysis of cellulose fibers [3]. The pyrolysis temperature was rise up to three different values: 530 °C, 650 °C and 700 °C	Char C 530 °C Char C 650 °C Char C 700 °C
	Biochars from populus nigra wood pyrolysis	Biochars obtained by steam assisted slow pyrolysis of populus nigra wood [4]. The pyrolysis temperature was rise up to two different values: 480 °C, and 600 °C	Char P 480 °C Char P 600 °C
	Carbonized rice husk	Rice husk carbonized in a auger furnace in the 500 - 800 °C temperature range for 3 hours	RH
iv	Biochar from populus nigra wood pyrolysis after base leaching	Biochar obtained by steam assisted slow pyrolysis of populus nigra wood at 600 °C after base leaching with a NaOH 5 M solution	Char P NaOH
	nFeM doped populus nigra biochar	Biochar obtained by steam assisted slow pyrolysis of populus nigra wood at 600 °C decorated with magnetite nanoparticle (15 wt%)	Char P nFeM
	Chemically modified biochar	Biochar obtained by steam assisted slow pyrolysis of populus nigra wood at 600 °C decorated with NH_2 groups introduced after a reaction with DETA on oxidized char sample [5]	Char P NH_2
	Carbonized rice husk after base leaching	Carbonized rice husk after base leaching with a NaOH 5 M solution	RH NaOH

Methods

The gas storage ability of the above reported materials was tested by using various probes (N_2 , CO_2 , CH_4) and various temperatures (from cryogenic to near ambient conditions) and from vacuum up to ambient pressures (760 mmHg), under equilibrium conditions.

High resolution N_2 adsorption/desorption isotherms were collected on a volumetric analyzer (Micrometrics Triflex) at 77 K, provided with three pressures transducers to allow high resolution in the low pressure range. The cryogenic temperature was maintained with liquid nitrogen bath. All the gas adsorption/desorption isotherms (i.e., CO_2 and CH_4 and N_2) at 273, 283, and 298 K were collected on a second volumetric analyzer

(Micrometrics Tristar 3020) provided with one pressure transducer, in the pressure range of 0.1–900 Torr. During the analysis, the temperature near ambient conditions (ca. between 273-298 K) was controlled and maintained using a water circulating- bath. Before the gas adsorption experiments, all the samples were outgassed under vacuum at 120 °C for 15-17 hours. Ultrapure gases were used for all the measurements. The reproducibility of the adsorption data was assessed by repeating twice selected measurements.

Data analysis

The adsorption data were analyzed and modeled in order to have a complete characterization of the textural properties of all the materials. The isosteric heats of adsorption and gas selectivity of a selection of them were also evaluated. The isotherm data were also used to predict the binary mixture adsorption from the experimental pure-gas isotherms of various gas mixtures of strategic impact (ca. CO₂/CH₄, and CO₂/N₂) by applying the Ideal Adsorbed Solution Theory (IAST) [6].

The isotherms were used to obtain various textural parameters such as: a) the specific surface area using the BET equation applied to the N₂ adsorption isotherms at 77 K; b) the total pore volume at $p/p^0=0.99$ from the N₂ adsorption isotherms at 77 K; c) the micropore volumes by using the Dubinin-Radushkevich (DR) equation applied to the N₂ and CO₂ adsorption isotherms at 77 K and 273 K, respectively; d) the distribution of pore sizes by applying the NLDFT model to the N₂ adsorption data at 77 K. The CO₂ adsorption isotherms at 273, 283 and 298 K of a selection of samples were used to evaluate the isosteric heats of adsorption. The CO₂, N₂ and CH₄ adsorption isotherms at 298 K of a selection of samples were used to evaluate the gas uptake and selectivity.

Timeline of activities carried out during STSM

The timeline of the activities performed during the STSM is reported in the Table 2.

Table 2. Timeline of the activities performed during the STSM

Period	Experiments and data analysis carried out
1 th week	Adsorption tests with CO ₂ , and N ₂ at cryogenic temperatures.
2 ^d week	Adsorption tests with CO ₂ , and N ₂ at cryogenic temperatures. Analysis of the data.
3 ^d week	Adsorption tests with CO ₂ , and N ₂ at temperature near ambient conditions. Adsorption tests with N ₂ at cryogenic temperatures. Analysis of the data.
4 th week	Adsorption tests with CO ₂ , CH ₄ and N ₂ at temperature near ambient conditions. Adsorption tests with N ₂ at cryogenic temperatures. Analysis of the data, evaluation of isosteric heats of adsorption and CO ₂ /N ₂ , CO ₂ /CH ₄ selectivities.

High resolution nitrogen isotherms at 77 K were performed during the whole STSM period because they required very long acquisition times, being the samples under study microporous and showing constricted porosity. The first two weeks were devoted also to the complete characterization of the samples by acquiring CO₂ isotherms at 273 K. The analysis of these data then allowed the selection of six samples whose

adsorption capacities against CO₂, N₂ and CH₄ has been analyzed more in depth by acquiring, during the last two weeks, adsorption isotherms at different temperatures: 283 and 298 K. During the last week, the data acquired were then used for determining the isosteric heats of adsorption and CO₂/N₂, CO₂/CH₄ selectivities.

Description of the main results obtained

The N₂ adsorption/desorption isotherms collected at 77 K are reported in Figure 1. The shape of the N₂ isotherms of all the samples is different, pointing out to different porosity in all of them. For instance, all the chars, and the samples Ti-MIL125 and RH showed type I isotherms in the IUPAC classification, characteristic of microporous materials. At converse, sample CB and nFeM showed type IV(a) isotherms, characteristic of samples with a well-developed mesoporosity, as seen in the high uptake at relative pressures above 0.5 and the presence of marked hysteresis loops [7]. Sample RH-NaOH displayed also a type IV(a) isotherm, with a marked adsorption at low relative pressures, indicating a well-developed microporosity along with the mesoporosity.

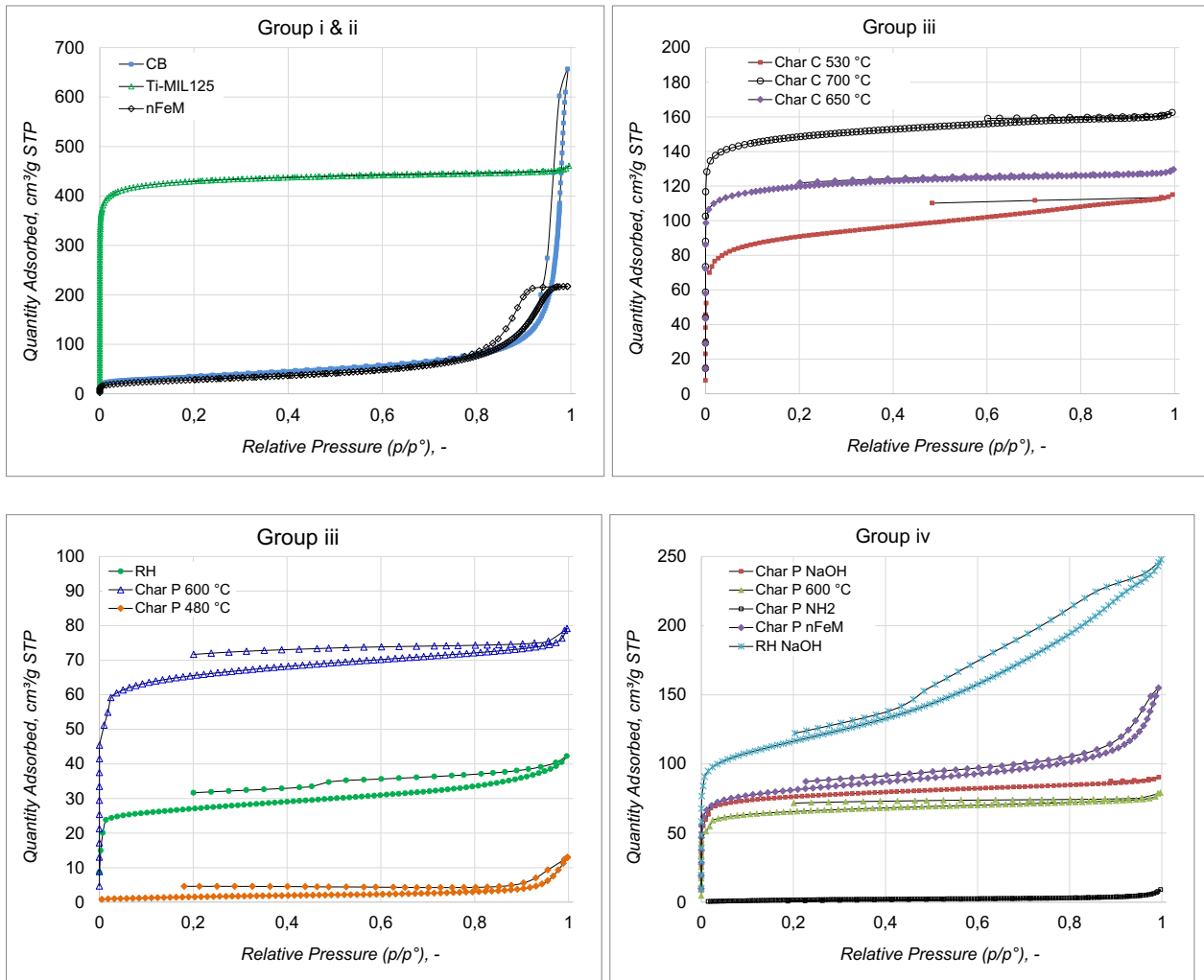


Figure 1. High resolution N₂ adsorption/desorption isotherms at 77 K of the studied samples.

In contrast, samples char P 480°C and char P-NH₂ showed almost no porosity, as seen in the very low N₂ adsorption in the whole range of relative pressures, revealing the absence of pores accessible to nitrogen

probe at this temperature. It is interesting to point out that the char samples are characterized by irreversible nitrogen isotherms even at low relative pressures (the desorption branch does not overlap the adsorption branch); this is a typical feature of char samples [8] and it is related to the presence of microporous samples with constricted porosity.

The main textural features of the samples were calculated analyzing the N₂ adsorption isotherms, and are reported in Table 3. Data indicates that CB, selected as reference material, has quite a high surface area compared to other carbon black reported in literature [9], as well as a low volume of micropores, as expected from the isotherm depicted in Figure 1. On the other hand, the MOF sample presented a high surface area and a very high total pore volume, corroborating that it is a microporous material. This is in accordance with previous literature findings for this material [1]. As for sample nFeM, the contribution of microporosity is also low, as in the case of the carbon black.

Table 3. Main textural properties calculated from the N₂ adsorption isotherms at 77 K

Group	Sample	BET area	V _{tot} (p/p ⁰ =0.99)	V _{micro} (DR)
		m ² /g	cm ³ _{liq} /g	cm ³ /g
i	CB	121	0.115	0.047
	nFeM	97	0.100	0.033
ii	Ti-MIL125	2144	62.6	8.03
	Char C 530	351	0.177	0.141
iii	Char C 650	473	0.199	0.183
	Char C 700	593	0.250	0.225
	Char P 480	6	0.018	0.002
	Char P 600	217	0.121	0.093
	RH	105	0.064	0.041
	Char P NaOH	315	0.139	0.118
iv	Char P nFeM	313	0.235	0.118
	Char P NH ₂	7	0.011	0.002
	RH NaOH	431	0.375	0.170

The char samples obtained by steam assisted slow pyrolysis of cellulose fibers at different temperatures are characterized by moderate surface areas, with increasing values as the pyrolysis temperature is raised. Accordingly, the volume of micropores grows with the increase of temperature, and in all the three samples the total pore volume roughly corresponds to that of micropores, which indicates a low contribution of mesoporosity to the overall porous structure of these samples.

The char obtained by the pyrolysis of populus nigra wood is characterized by lower surface area and micropore volume compared to those of cellulose chars. This might be likely due to the presence of the other

two main wood components (lignin and hemicellulose) that during the pyrolysis process entrapped the cellulose fraction in a more compact matrix.

When the Char P is treated with NaOH, an increase in the surface area was detected (ca. 30%), which is reasonable if one considers that the data is normalized per gram of sample and the treatment in NaOH removes the ash content (typically non porous matter). The doping of this char with magnetite nanoparticles was not followed by a decrease of the surface area, which we attribute to the low amount of magnetite added (~15 wt.%) and the fact that magnetite itself shows some porosity. In contrast, the impregnation with amine caused a dramatic decrease in the textural properties of the char P, leading to an almost complete blockage of the pores by the amine molecules, as seen in the surface area near to zero.

RH exhibited a low surface area and a quite low total pore volume; after the treatment with NaOH 5 M to remove most of the inorganic component (sample RH NaOH), the surface area grew considerably as well as both the total pore volume and the volume of micropores.

The textural characterization of the samples was completed by the CO₂ adsorption isotherms at 273 K, that in carbon-based samples allow to evaluate the narrow microporosity [8]. The corresponding isotherms are reported in Figure 2, and the data about narrow microporosity, calculated from the DR equation applied to the CO₂ adsorption isotherms at 273 K are reported in Table 4.

As seen, CB and nFeM are the samples with the lowest amount of adsorbed CO₂ at a relative pressures of $p/p^0=0.035$, while the char obtained after the pyrolysis of cellulose fibers at 700 °C is the sample with the highest amount of adsorbed CO₂. At converse, Ti-MIL125 exhibited the highest amount of CO₂ adsorbed at $p/p^0=0.035$, but a different isotherm shape (almost a straight line) compared to that of char isotherms (concave curves); overall this behavior agreed with previous literature findings [1], and indicates a lower interaction of the framework with CO₂ molecules, compared to the carbon-based materials.

The comparison between the CO₂ isotherms of the chars from cellulose and populus nigra wood collected at different pyrolysis temperatures revealed that the CO₂ uptake of the chars increased with pyrolysis temperature, indicating that a higher pyrolysis temperature allowed the development of a narrower microporosity.

The chemical modification of char from populus nigra wood, as expected, affected the CO₂ sorption capacity; as already observed in the case of nitrogen adsorption, the treatment with NaOH increased the gas uptake due to the effect of ash removal in the mass of the sample. The doping with magnetite nanoparticles did not bring about an increase in the CO₂ uptake, whereas the impregnation with amine resulted in a decrease of the CO₂ uptake. Also in the case of RH, the treatment with NaOH leads to an increase of the gas uptake.

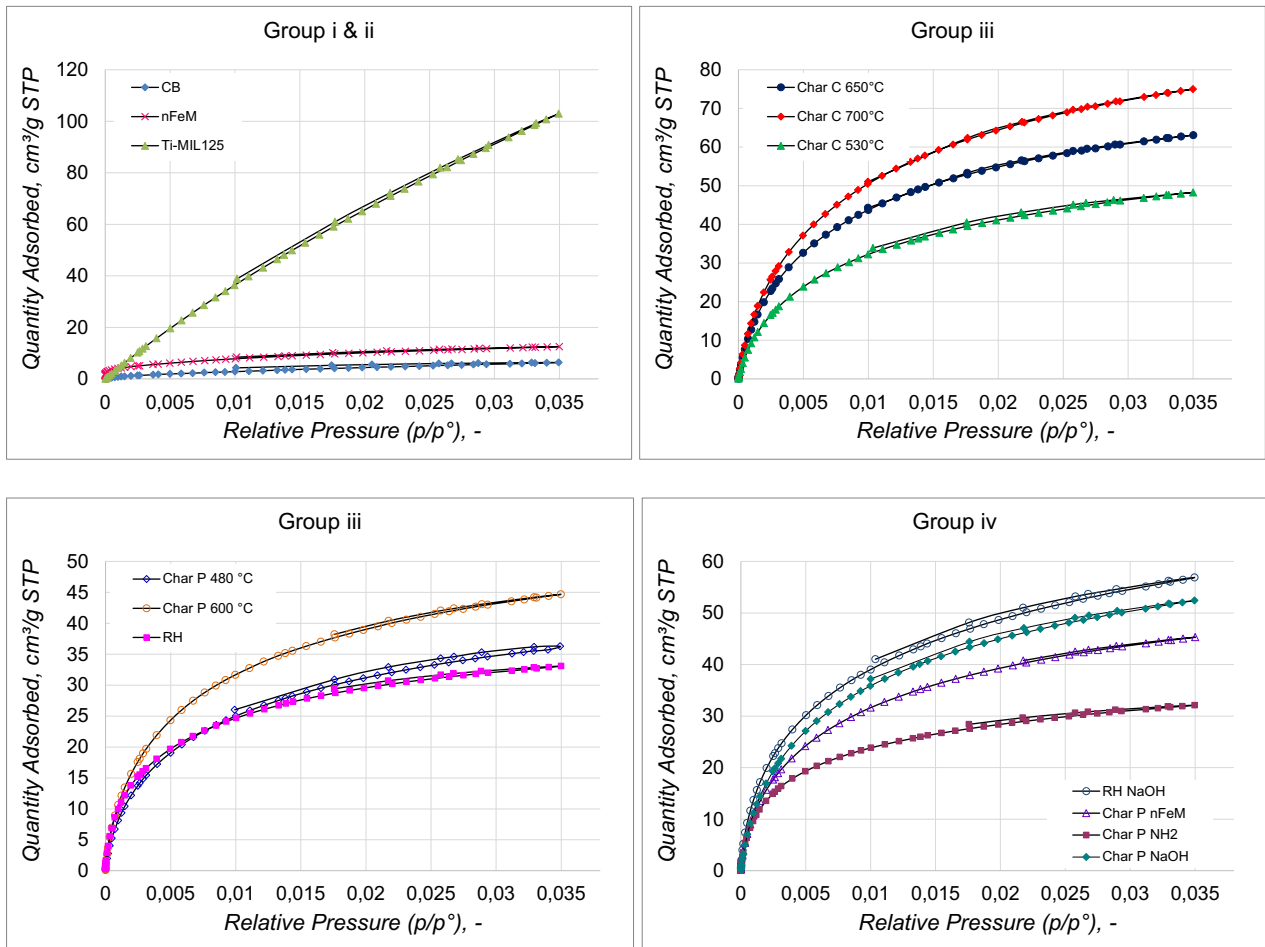


Figure 2. CO₂ isotherms at 273 K

The CO₂ isotherms at 273 K were analyzed to evaluate also the narrow micropore volume (W_o) using the Dubinin-Radushkevich (DR) equation. Also, an average micropore diameter (L) was obtained by the application of Stoeckli–Ballerini equation [10]. Data are reported in Table 4. It is important to remark that for some samples, the shape of the CO₂ adsorption isotherms – a marked knee in the isotherm at low p/p^0 -, suggested the presence of specific interactions between CO₂ and the samples (for instance in the case of Char P NH₂), for which the use of Dubinin-Radushkevich (DR) equation to obtain the micropore volume is not advisable.

The CO₂ data collected at 273 K confirmed the trend highlighted by the analysis of N₂ isotherms at 77 K: the CB and the nFeM resulted the samples with the lowest narrow micropore volume -in agreement with their classification as mesoporous materials made on the basis of N₂ isotherms at 77 K-, while the Ti-MIL125 and the chars from cellulose fibers were the samples with the highest narrow micropore volumes. For char samples prepared from the same precursor, the volume of narrow micropores grows with the pyrolysis temperature. The NaOH treatment of Char P resulted in a higher narrow micropore volume while, as expected, the impregnation with amine caused a decrease. Also in the case of RH a higher narrow micropore volume was evaluated for RH NaOH compared to RH.

Table 4. Main textural parameters of the studied samples calculated from the CO₂ adsorption isotherms at 273 K

Group	Sample	Wo micro cm ³ _{liq} /g	L nm
i	CB	0.02	0.72
ii	nFeM	0.03	0.35
	Ti-MIL125	0.50	---
iii	Char C 530	0.16	0.61
	Char C 650	0.22	0.60
	Char C 700	0.25	0.60
	Char P 480	0.12	0.56
	Char P 600	0.14	0.54
	RH	0.10	0.47
iv	Char P NaOH	0.17	0.58
	Char P nFeM	0.14	0.53
	Char P NH ₂	-	--
	RH NaOH	0.17	0.52

On the basis of CO₂ sorption capacities at 273 K, six samples were selected to perform a deeper characterization of the sorption capacities: Char C 650 °C, Char C 700 °C, Char P 600 °C, Char P NaOH, CharP NH₂ and RH NaOH. The gas uptake and the CO₂/N₂ and CO₂/CH₄ selectivities of these six samples were evaluated by comparing the adsorption isotherms of CO₂, N₂ and CH₄ at 298 K.

As seen in the isotherms, in all the cases the adsorbents exhibited a good affinity toward the adsorption of CO₂ and low affinities toward the adsorption of CH₄ and N₂. The CO₂, CH₄ and N₂ adsorption isotherms at 298 K of Char C 700 °C and Char P 600 °C are shown in Figure 3, as an example of this behavior.

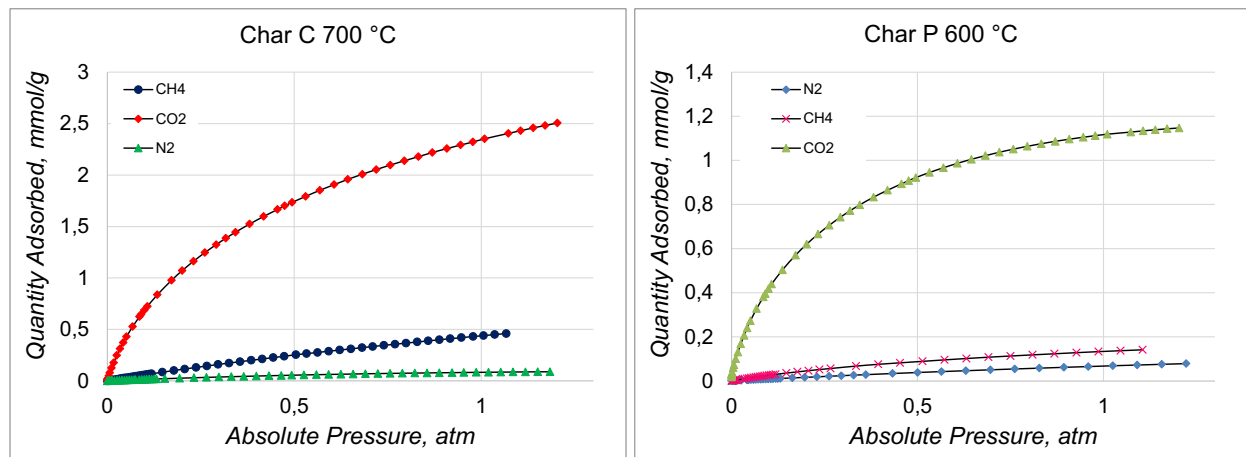


Figure 3. Comparison of the CO₂, N₂ and CH₄ isotherms at 298 K for char C 700 °C (left) and Char P 600°C (right)

The prediction of the CO₂/N₂ and CO₂/CH₄ selectivities of selected samples were calculated from the single component experimental adsorption data, using the Ideal Adsorbed Solution Theory (IAST) [6]. The selectivity of a gas X over Y (S_{x/y}) was calculated as the ratio:

$$S_{x/y} = \frac{q_x/q_y}{p_x/p_y}$$

where q_x and q_y are IAST predicted uptakes (cm³/g) and p_x and p_y are the partial pressures of gas X and Y respectively. Data for various gas mixtures (i.e., CO₂:N₂ 15:85 for a typical post-combustion application and CO₂:CH₄ 30:70 for natural gas upgrading) at 1 bar are reported in Table 5.

Table 5. CO₂/N₂ and CO₂/CH₄ selectivities at 1 bar for binary mixtures, evaluated from the single component experimental adsorption isotherms applying the IAST approach.

	CO ₂ /N ₂	CO ₂ /CH ₄
	15:85	30:70
Char C 650	34	32
Char C 700	119	14
Char P 600	105	34
Char P NaOH	41	16
Char P NH₂	211	31
RH NaOH	31	14

All the selected samples exhibited good CO₂/N₂ and CO₂/CH₄ selectivities; for the cellulose derived chars, the sample prepared at 700 °C has a higher selectivity CO₂/N₂ in a mixture with a typical post-combustion fuel gas composition (ca. 15:85 of CO₂:N₂), than the one obtained at 650 °C. In the case of the PN-derived chars, the sample modified with amine moieties exhibited the largest selectivity. When a typical natural gas composition is considered (ca. 30:70 of CO₂:CH₄) the selectivity CO₂/CH₄ is good for all the samples as well, but the values are slightly lower than those over nitrogen. Among all, Char C 650°C, Char P and Char P NH₂ are the samples with the highest selectivity CO₂/CH₄.

To understand the results about gas selectivity the main parameters governing both the CO₂ uptake capacity and selectivity (micropore volume and size and chemical functionalization of the pores) must be taken into account. In general, high CO₂ uptakes correspond to samples with high micropore volumes. On the other hand, selectivity depends on the presence specific interactions between the adsorbent surface and the CO₂ molecules, which explains the higher values for the samples decorated with the amine groups [11].

The CO₂ adsorption isotherms of the six samples listed above acquired at 273, 283 and 298 K were used to calculate the CO₂ isosteric heats of adsorption using the Clausius–Clapeyron equation. The adsorption isotherms of Char C 700 °C and Char P 600 °C at 273, 283 and 298 K are shown in Figure 4, as an example.

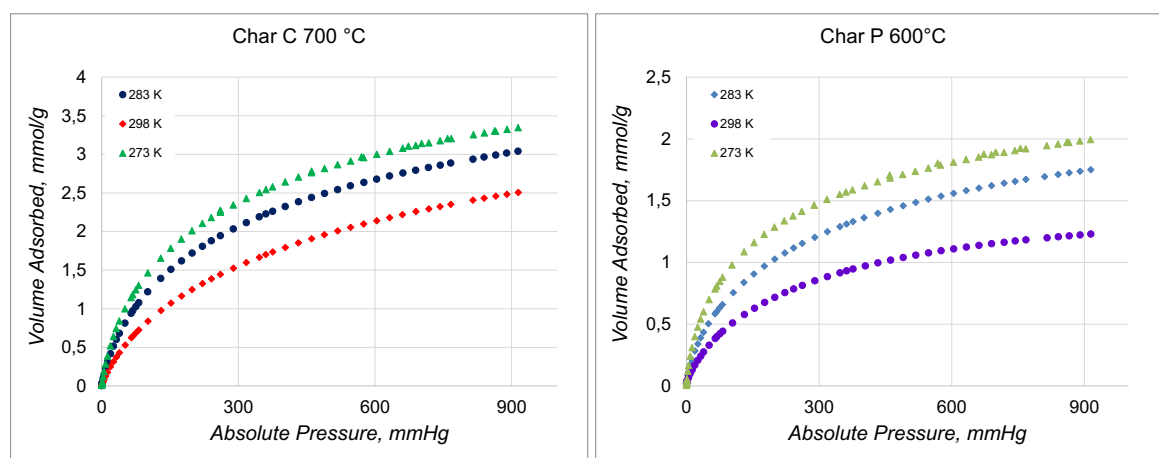


Figure 4. Comparison among CO₂ isotherms at 273, 283 298 K for char C 700 °C (left) and Char P 600 °C (right)

The calculated values of the isosteric heat of adsorption ranged between 25 and 35 kJ/mol for all six investigated samples. The highest value was found for Char P NH₂ (ca. 35 kJ/mol), while the lowest one was obtained for Char C 700 (ca. 28 kJ/mol). Overall, the CO₂ isosteric heats of adsorption estimated are in line with those evaluated for other biochars obtained by slow pyrolysis experiments [12] and indicated, for the most of the samples, a non-specific interactions with the gas. Furthermore, the trend of the heats of adsorption agrees with the gas selectivity evaluation: the higher heat of adsorption corresponds to Char P-NH₂, namely the only material bearing specific functional groups selective to carbon dioxide molecules.

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Confirmation of the Host Institution of the successful execution of the STSM

See attached documents.

How the STSM has contributed to the action's aim

The STSM of Dr Gargiulo within the COST action Smartcats was devoted to the collection of new data and the development of diagnostic protocols and methods. The funding of this STSM has contributed to strengthen the competences of the young researcher involved in monitoring pollutants emissions in SECs combustion and also in the fields of engineering and material science. A fundamental value-added was the knowledge transfer between ADPOR Group in the host institution CEMHTI and the young researcher involved: competences in the measurements of gas adsorption isotherms with high resolution instruments, data elaboration and modelling to identify possible applications of materials with tailored chemical and textural as gas storage systems. Moreover, the funding of this STSM has contributed to start a new fruitful collaboration between CNR and CNRS.

Future collaboration with host institution

A new fruitful collaboration between CNR and CNRS on the study of carbonaceous materials for environmental applications stemmed from the funding of this STSM. The first outcome of this new collaboration will be in the next future the publication of some joint papers aimed to disseminate the results of the STSM research activity. Both parts are intentioned to keep alive this collaboration through continuous discussion and exchange about the data, and overall the possibility to submit joint proposals to national and European founding agencies will be considered surely.

Foreseen publications/articles resulting from the STSM

The dissemination of the results as communications at national and international meetings will be achieved in the next future as follows:

Presentation of oral and WIP contributes at:

- Tenth Mediterranean Combustion Symposium, 17-21 September 2017, Napoli, Italy
- 3rd General Meeting and Workshop on SECs in Industry of SMARTCATs Action , Prague, October 25-27, 2017

Submission of one or more papers to international peer reviewed journals will be accomplished in the next future.

Other comment

Dr. Valentina Gargiulo sincerely acknowledges the COST action CM1404 and the chair Dr. Mara de Joannon for the opportunity to join the ADPOR group activities at the CEMHTI institute at CNRS campus of Orleans, and to start a fruitful collaboration between CNR and CNRS. VG thanks go to Dr. Conchi O. Ania and all the members of ADPOR groups, Dr. Alicia Gomis Berenguer, Miss. María Cocina Fernández de Córdoba and Miss. Ana Casanova Martinez for their availability and support, for the experimental training and for the fruitful discussions. VG acknowledges also Dr. Michela Alfè for her constant support and Dr. Paola Giudicianni for the help in the production of char samples.