" Maturing the production standards of ultraporous structures for high density hydrogen storage bank operating on swinging temperatures and low compression" – MAST3RBoost

D1.2. Database kick-started and fed with preexisting data

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PROJECT INFORMATION

Project full title: Maturing the production standards of ultraporous structures for high density hydrogen storage bank operating on swinging temperatures and low compression

Acronym: MAST3RBoost

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Topic: HORIZON-CL4-2021-RESILIENCE-01-17

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List of participants:

Number	Name of beneficiary	Acronym of beneficiary	Country
1	ENVIROHEMP	ENV	Spain
2	CONTACTICA	CTA	SPAIN
3	Consejo Superior de Investigaciones Científicas	CSIC	Spain
4	Spike Renewables Srl	SPIKE	Italy
5	EDAG Engineering GmbH	EDAG	Germany
6	Nanolayers	NANO	Estonia
7	FUNDACIÓN CIDETEC	CIDETEC	Spain
8	Leichtmetallkompetenzzentrum Ranshofen GmbH	LKR	Austria
9	University of Pretoria	UP	South Africa
10	Council for Scientific and Industrial Research	CSIR	South Africa
11	PSA	PSA	Portugal
12	TWI Ltd	TWI	UK
13	University of Nottingham	UoN	UK

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DELIVERABLE DETAILS

Document Number:	D1.2
Document Title:	Database kick-started and fed with preexisting data
Dissemination level	PU – Public
Period:	PR1
WP:	1
Task:	T1.2
Author:	Nanolayers OÜ
Abstract:	In collaboration with University of Nottingham, we have compiled a database of pre-existing nanoporous samples including information about their synthesis process and characterisation measurements of the resulting materials. The database is stored in a LabCore digital notebook, along with a description of the experimental methods involved, and the relevant bibliography. Thanks to LabCore data management protocol, the data can be easily accessed and used in machine-learning applications with the LabCore python API.

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1 Database of Pre-existing Experiments

The materials database was generated from pre-existing experimental data from University of Nottingham. The information was taken from a set of publications (section 0) and organised into tables using the LabCore digital notebook platform (Figure 1). The notebook can be found here:

https://labcore.nanolayers.com/notebookfluid/63761cd8e05f2a53fab2fdb7

however, access is currently restricted to the notebook author and Nanolayers.

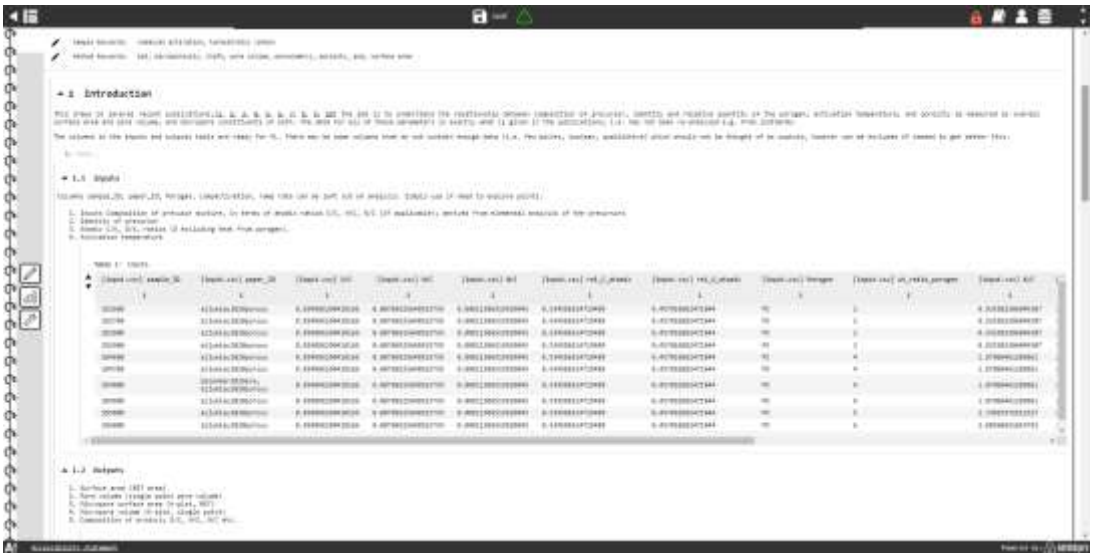

Figure 1: snapshot of the LabCore digital notebook with pre-existing data.

There are two main tables in the notebook, listing the input and output quantities of each sample. Input quantities are the control parameters that characterise the sample manufacturing process and are listed in Table 1:

Table 1: input quantities describing the samples.

Name	Units	Notes
O/C	adim.	precursor mixture atomic ratios O/C
H/C	adim.	precursor mixture atomic ratios H/C
N/C	adim.	precursor mixture atomic ratios N/C
rel_C_atomic	adim.	relative concentration of carbon
rel_O_atomic	adim.	relative concentration of oxygen
K/C	adim.	precursor mixture atomic ratios K/C
K/O	adim.	precursor mixture atomic ratios K/O
Activation_temperature	°C	temperature of pyrolysis
wt_ratio_porogen	adim.	porogen weight ratio
porogen	adim.	type of porogen, 0: $KHCO_3$, 1: KOH

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Most quantities describe the substance that is used to create the sample in terms of atomic ratios or relative concentration of various elements. Precursor mixture ratios and relative concentrations are determined via CHN elemental analysis. Values of K/O and K/C are derived from the mass ratio of precursor to activating agent, also known as porogen, which for our samples is either KOH or $KHCO_3$. Activation temperature is the temperature onset for pyrolysis, monitored by the furnace during the sample synthesis. There are other tables in the notebook that contain more details of the synthesis conditions and precursor molecules, which were used in the calculation of the input quantities.

The output quantities, listed in Table 2, are the result of the characterisation measurements of the synthesis products.

Table 2: output quantities describing the performance of the samples.

Name	Units	Notes
ABET	m² / g	surface area (BET method)
Amicro	m² / g	micropore surface area (t-plot method)
Vsp	cm³ / g	pore volume (single point method)
Vmicro	cm³ / g	micropore volume (t-plot method)
rel_C_atomic	adim.	relative concentration of carbon
rel_O_atomic	adim.	relative concentration of oxygen
O/C	adim.	sample composition, atomic ratios O/C
H/C	adim.	sample composition, atomic ratios H/C
N/C	adim.	sample composition, atomic ratios N/C

The first four are measures of porosity: (micro)pore surface area and volume characterisation, obtained with different methods. The last five quantities are relative concentration and atomic ratios from the elemental analysis of the samples. All porosity data is calculated from N_2 isotherms measured at 77 K. BET method relies on a transform of the isotherm, yielding a linear plot. From the intercept and slope of the fitting line it is possible to determine the amount of N_2 adsorbed in a single molecular layer on the surface of the material; combining this information with the known cross-sectional area of N_2 we finally obtain the pore surface area (ABET). The single point method is performed by selecting the volume of a single point close to saturation of the isotherm - this is taken to be the total pore volume (Vsp). The t-plot method simply converts the isotherm to a plot relating amount of N_2 adsorbed to the thickness of the adsorbed layer(s) by use of a thickness model. Identifying inflexions in the plot allows the quantification of surface area (Amicro) and volume (Vmicro) within micropores (<2 nm in width).

There is a total of 90 samples in the database, however all input and output quantities are available for 80 of them. The database will be expanded during the course of project, as new samples are made and characterised. We also hope to complement it with additional input and output quantities, such as the full adsorption isotherms and other raw data that could be used directly in machine-learning to produce a more reliable and predictive model.

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2 LIMITATIONS OF PRE-EXISTING DATABASE AND FUTURE WORK

The preliminary database has a number of identified limitations. First, some samples do not have all the output quantities, and this effectively reduces the size of the dataset usable by machine-learning methods. Additionally, the output quantities do not include the measurements of H_2 adsorption tests at the relevant pressure range, but only quantities derived from the N_2 adsorption isotherm up to 1 atm.

Regarding the N_2 adsorption isotherm the underlying assumption is that isotherm analysis has been performed consistently across all samples, thus it is not necessary to include the raw data source. However, the assumption is likely not fully correct since the analysis is not fully automated and requires the operator to set certain parameters from visual inspection and experience, and not all measurements were performed and analysed by the same person. The process of sample densification was also not consistently recorded across the existing data.

The porogen descriptor consists of its chemical formula, which is understandable by humans, but not by machine-learning methods. The chemical formula hides a great deal of physics and chemistry well-known to scientists, and it is not reasonable to expect machine-learning methods to infer them from data. If a wider variety of porogen molecules will be used in new experiments, it will become necessary to provide a more computer-friendly descriptor, for example including molecular topology and physical properties, to improve performance of machine-learning models.

When compiling an equivalent database of materials in the MOF family it will also be important to decide which is the information that needs to be used. It can be in relation to one or more of the several aspects: the effect of the purification of recycled streams on the final quality of the MOF crystals, the effect of the post-synthetic strategies such as supercritical activation and production of MOF-carbon composite, and/or the effect of using metal and/or ligand mixtures, to model how these affect the final properties of the MOF.

To discuss all these issues, a specific Workshop (or Working session) will be arranged in the context of the 1st General Assembly of the project.

3 BIBLIOGRAPHY

All data was taken from the following publications:

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