Thermal energy storage capacity on mineral Zeolite

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Abstract

This study analyses zeolite adsorption- systems based on the recovery of the residual heat generated in different urban processes. The quality and thermal storage capacity of natural zeolites has been experimentally evaluated focusing on the discharge process. This material is characterized by high porosity and cation exchange capacity, selective absorption and reversible hydration. Numerical models of natural zeolites define the geometry, boundary conditions and inlet variables. A specific prototype of zeolite reactor has been designed based on these numerical results. Different lab- experiments have been carried out, to evaluate natural zeolites performance under different conditions of drying (muffle, stove and desiccator) and operation. The sample is encapsulated in a cell and located in the middle position of a tube of about two meters length and outside air is blown through it. The empirical results show that the use of natural zeolites up to 8 cm size provides an increase in the temperature of the reactor due to heat conduction.

Keywords: Heat transfer, Building envelope, Thermal comfort, buildings, gaseous emissions, Physical-chemical characterization, Thermal, CAM

1. Introduction

Climate change mitigation needs to consider the design of sustainable urban areas, reducing the pollutant emissions and increasing the quality of life and the well-being of citizens (IPCC, 2022). To achieve these goals integrative energy systems should be implemented (UNEP 2015), connecting energy-efficient and energy-flexible buildings with global energy infrastructures, high renewable generation, low carbon emission systems and flexible management of the district's energy flows. Thus, European strategies are focused on the development of Positive Energy Districts and Neighborhoods, which aim to optimize the energy consumption through the active management and balance of demands, shaving peaks or/and shifting loads in mixed-use buildings and fostering sustainable energy sources (SET-Plan Working Group, 2018). In this framework, the storage systems act as energy buffers between energy demand and supply side, providing flexibility to the global energy system and increasing the resilience to the effects of climate change.

Storage systems are capable of effectively regulating energy from different urban sources (renewable energy, electricity grid, residual flows), through the modulation of its intensity in temporal and spatial terms. These elements store electrical and thermal energy according to the characteristics of the generation and consumption profiles (Kousksou et al., 2014), providing flexibility that allows urban flows to operate robustly and effectively. These solutions are especially valuable for covering peak energy demands or outages, which are becoming more frequent because of the extreme weather and disaster events as well as the energy market instability and uncertainties (Huang et al., 2021; De et al., 2020).

Even it is shown that thermal energy store it is cheaper than electrical storage (Hennessy et al. 2019), these systems must be deeply analyzed optimizing their dimensions, materials and operation, and taking advantage of the energy market prices to reduce end users costs (Telaretti et al., 2016).

Thermal storage systems play an important role balancing energy urban fluxes. These systems can be classified based on the state of the stored energy: sensible, latent and thermochemical (Guelpa and Vittorio, 2019). Currently, the use of thermochemical systems in cities are very innovative thanks to the high energy stored and low heat losses produced, nevertheless these technologies are mainly on the laboratory scale. The operating

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principle of these systems takes advantage of chemical transformations produced when absorbing heat to excite a reaction. Stored heat can be recovered by reversing the reaction, sometimes adding a catalyst (Kousksou et al., 2014).

There are many classifications for the thermochemical storage systems, but one of the most usual is based on the creation of new materials when a gas binds to the surface of a solid. Two typologies are identified: chemical reversible reactions / absorptions processes and adsorption processes (Guelpa and Vittorio, 2019). Adsorption processes highlight by the creation of different types of materials such as zeolites, aluminophosphates composites and metal-organic frameworks (Henninger et al., 2017). The performance of these materials will regulate the applicability, efficiency and profitability of these adsorption processes. The use zeolites as storage systems has a great potential due to their catalytic properties, the permeability as well as the adsorption properties thanks to its microporous structure (Feng et al., 2021). Currently it is easy to find natural zeolite on the marketplace because is a product that comes directly from the stone quarry and it is very common for building and construction uses. Therefore, its use as a thermal storage element has great economic and energy potential.

Previous studies reveal the high cationic exchange potential of zeolites (Seco et al. 2021). However, in order to deeply know the zeolites´ potential as an innovative thermal use storage, tailored experimental campaigns are needed. Thus, this study addresses a key issue in the development of sustainable cities quantifying the thermal storage capacity of thermochemical storage systems. Several configurations of natural zeolites have been evaluated with the aim of reducing the existing uncertainties in their operation. With this aim, numerical simulation models and laboratory experiments have been developed to quantify the thermal capacity of the studied zeolites.

To this end, it is defined the following specific objectives: a) Design of a Zeolite prototype reactor based on Computational Fluid Dynamic (CFD) simulation models. b) Based on previous input and output values, execution of the physical reactor at lab conditions showing first empirical results of the zeolite thermal storage behaviour. Therefore, Section 2 and Section 3 show the method applied along the experimental camping and the study case, while Section 4 presents the simulated and empirical results. Finally, section 5 describes main conclusions of the work.

2. Methodology

To quantify the performance of some material as thermochemical storage systems, a step-by-step methodology has been developed, whose main phases are:

- *Selection of the studied samples: natural zeolites.*
- *Development of the initial Computational Fluid Dynamic Models to provide first results that allow defining the pre-design of the experiments with the proposed zeolites.*
- *Definition of the Zeolite Reactor built in the laboratory.*
- *Laboratory experiments necessary to quantify the thermal performance of the studied zeolite.*

The application of these phases produces the experimental characterization of the thermal properties of natural zeolites, allowing the development of a more detailed CFD model that, once validated, will enable the study of different conditions and modes of operation. Furthermore, the systematization in the application of these phases will allow characterizing the thermal performance of other types of materials such as synthetic zeolites, giving rise to new reactors and new simulation models.

3. Case Study

The methodology developed in this paper includes the CFD simulation to define a pre-design of the prototype, the construction of a reactor prototype and the execution of batteries of experiments, to quantify the thermal performance of natural zeolites. The study considers the quantification of their cationic exchanges in thermal storage analysis including different sieves at lab conditions.

3.1. Selection of materials

In this study, natural zeolites have been selected to be tested as thermal storage material in buildings (see Figure 1). The use of this type of zeolites in sectors such as agriculture or industry has been known for years, but these stones also have high potential as thermal storage materials for the use in buildings thanks to their catalytic and adsorption properties as well as their permeability (Feng et al., 2021). However, this last application is still in the research and development phase, and it is necessary to numerically quantify the thermal behavior of some of its properties under the operating conditions required in the thermal conditioning of buildings.

Natural Zeolite selected is a natural rock with high porosity and cation exchange capacity, selective absorption and reversible hydration. For this study, the application of zeolites cationic exchanges in thermal storage has been assessed including different sieves. The cationic exchange produced by the selected zeolites is characterized by two phases:

- *Charge.*
- *Discharge.*

The dry phase, named activated (charge phase), is the one used to extract the energy in heat form (Xu et al., 2019). For charging, high temperature heat is supplied to the adsorber desorbing all adsorbed water. To discharge the storage, outside air is blown through the zeolite and vapour molecules are adsorbed releasing the heat of adsorption at a high temperature level.

Fig. 1: Natural zeolite sample: a) laboratory prototype and b) material sample

This microporous material of volcanic origin presents a regular crystalline structure with unique characteristics. All types of zeolite have a very similar structure, as well as their physical and chemical properties. Table 1 shows the main properties provided by natural zeolites. Zeolites have specific densities in the range 1900-2800 Kg/m^3 and hardness between 3-6 on the Mohs scale. Its structure is based on a set of cuboctahedrons each made up of 24 tetrahedrons. Zeolites have an unusual cage-like, three-dimensional crystal structure and extraordinary ion-exchange capacity, both on the surface and within the mineral. This phenomenon is due to their size and the freedom of movement of the water molecules they contain.

Its ordered structure presents a large number of channels and cavities with molecular diameters between 2.6 Å and 7.4 Å. This property is called micro-porosity because the dimensions are slightly larger than those of water molecules. The pores are the same, and therefore so are their size, shape and volume, giving zeolites the property of molecular sieve with the capacity to retain cations and absorb water.

Tab. 1: Characteristics of the 4-8cm rock mineral Zeolite

Property	**Units**	**Value**
Density	Kg/m^3	1900-2800
Porosity	--	0.4
Thermal conductivity	W/m·K	2.0
Specific heat capacity at constant pressure	J/kg·K	910-1180
Dynamic viscosity	Pa·s	50

3.2. Development of Computational Fluid Dynamic Models

Once the material is selected, 4-5 cm natural zeolite rocks, advanced fluid mechanics tools are used to support the design of the key laboratory experimental equipment: a Zeolite reactor. The characteristics of the selected natural zeolites are considered in order to ensure a sufficient ventilation flow providing proper conditions to enhance the cation exchange processes associated with thermal storage in the material.

The numerical study has been focused on the discharging processes of these zeolites adsorbents. The developed numerical model simulates the ventilation airflow through the material, the natural zeolites, placed in the reactor vessel. This was done using the commercial computational fluid dynamics (CFD) code called FLUENT. The characterization of the airflow is relevant because it affects the thermal storage capacity of the natural zeolites in the reactor.

The analysis of the fluid-dynamic problem has been done solving the Navier- Stokes equations with the finite volumes method. To this end, a three-dimensional CFD model has been developed. The geometry of the reactor model has been defined to facilitate the experimentation to be carried out in the laboratory, considering the physical properties of the natural zeolites.

The resulting geometry of the Thermal Energy storage prototype is simple. It basically consists of a hollow cylindrical pipe with a cylindrical reservoir in the central inner part of the pipe. Zeolite sieves are introduced into the storage cylinder. Outside air is blown through the pipe along its length but the presence of the zeolite produces a fluid volume blockage inside the tube that is not physically represented in the geometry design. Then, the flow through the packed beds have been simulated by using a porous media model.

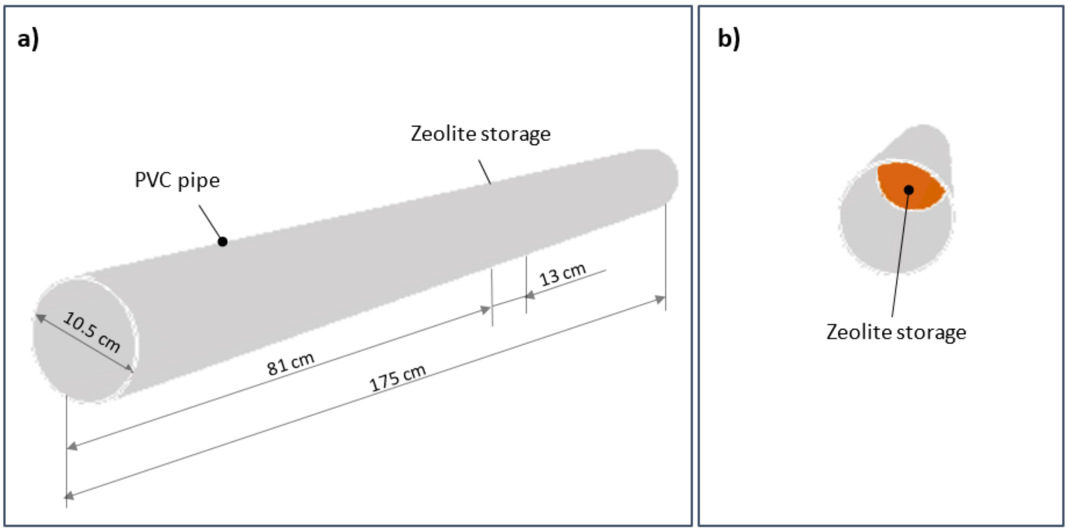

Fig. 2: Geometry of the natural zeolite reactor model: (a) Main dimensions, (b) zeolite storage chamber view

As shown in Figure 2, the geometrical model is a hollow cylindrical PVC pipe with an outside diameter of 11.0cm, an inside diameter of 10.5cm and a length of 175cm. A 13cm length storage cylinder is placed in the centre of the pipe. The reservoir is filled with 4-5 cm natural zeolite rocks.

The specific cells zone of the domain corresponding to the sample storage chamber is defined as “porous” and the flow resistance is determined in this volume by adding a momentum sink in the governing momentum equations composed of a viscous loss term and an inertial loss term. This momentum sink contributes to the pressure gradient in the porous media creating a pressure drop. This pressure loss in the flow is determined by using the superficial velocity porous formulation. The superficial velocity inside the storage cylinder is calculated based on the volumetric flow rate inside it and it is assumed that Zeolite reservoir is homogeneous and isotropic with reference to porosity. Then the resistance coefficients in all directions are the same. For deriving the appropriate coefficients, the semi-empirical correlation of the Ergun equation is applied and viscous (Eq. 1) and inertial loss (Eq. 2) constants are calculated. Turbulence is computed in the porous region just as in the bulk fluid flow.

$$\frac{1}{\alpha} = \frac{150}{{D_p}^2}\frac{(1-\epsilon)^2}{\epsilon^3} \qquad \text{(eq. 1)}$$

$$C_2 = \frac{3.5}{D_p}\frac{(1-\epsilon)}{\epsilon^3} \qquad \text{(eq. 2)}$$

Where Dp is the mean particle diameter and ϵ is the void fraction.

It is important to highlight that a proper definition of the boundary conditions in the simulation model leads to the best possible solution of equations system characterizing the behaviour of the zeolite reactor. The physical geometry and the expected pattern of the flow field solution has mirror symmetry so symmetry boundaries are applied in order to reduce the extent of the computational model. In this case the domain is reduced to a quarter pipe and two symmetry planes are defined (Figure 3). The exterior border, flow output, has a constant pressure and temperature condition equal to the atmospheric and ambient values. The velocity inlet conditions at the entrance of the fluid are fixed by the experimental conditions.

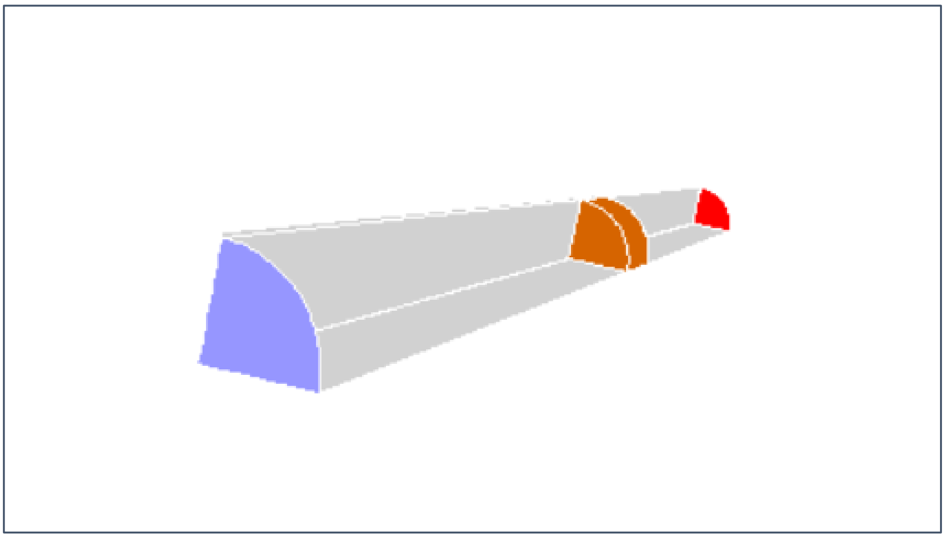

Fig. 3: Boundary conditions schema of the zeolite reactors

As regards the mesh, same type has been used in the domain: the unstructured one. The mesh has been refined in some parts of the geometry, such as the zones near the PVC pipe as shown in Figure 4. This increase in the cells density allows simulating accurately the airflow in the boundary layer close to the walls of the tube. A sensibility analysis of the mesh has been performed resulting in an optimized mesh with a total number of 0.4 million cells.

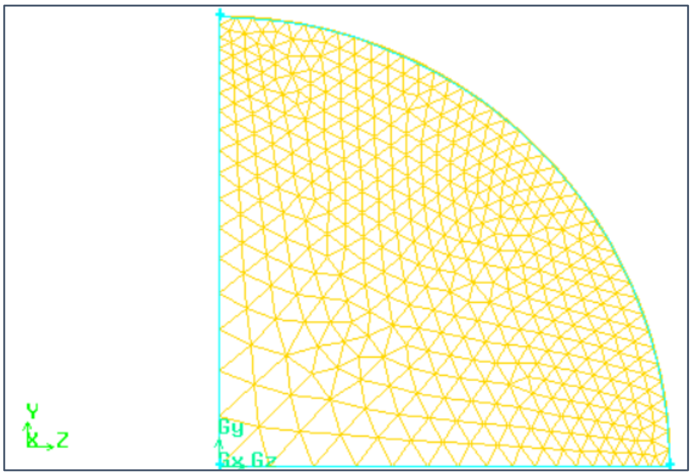

Fig. 4: Detail of the mesh in the inner volume of the tube

With respect to the solution methodology, the turbulence effects, as the ventilation flow inside the pipe is characterized by its turbulent regime, the k-ε RNG 3D RANS model of two equations has been tested and compared.

The porous zone is assessed by applying the Relative Velocity Resistance Formulation. A cylindrical coordinate system is used to define the loss coefficients. Porous model constants ie. viscous and inertial resistance coefficients, the Direction Vectors and the porosity, are calculated for the natural zeolite case of study. Viscous resistance coefficients (Direction X, Y, Z): $2.4*10^6$, Inertial Resistance coefficients (Direction X, Y, Z): $2.7*10^3$, and Direction Vectors (X,Y,Z): (1,0,0) and (0,1,0) are specified. Porosity of the porous medium is defined as the volume fraction of air within the zeolite region.

The turbulence equations have been solved using a second order discretization scheme, and the momentum equations with a second order - pressure staggered one. The pressure-velocity coupling employs the SIMPLE algorithm. And regarding the convergence criterion, a sufficient number of iterations has been computed to ensure all the residuals were lower than 10^{-5}.

3.3. *Definition of the Zeolite Reactor*

To check the thermal storage capacity of different types of zeolites, different reactor prototypes are needed. Given that natural zeolites have been selected in this study and based on the results obtained with the initial CDF models, a new Zeolite Reactor is designed and constructed in the laboratory scale at the CIEMAT facilities in Madrid (Spain). This reactor will allow testing the thermal storage capacity of the proposed natural zeolites under controlled laboratory conditions.

The final design of this reactor includes three sections (Figure 5):

- *Hollow cylindrical pipe with a bazooka at the beginning.*
- *Sample storage chamber.*
- *Hollow cylindrical pipe.*

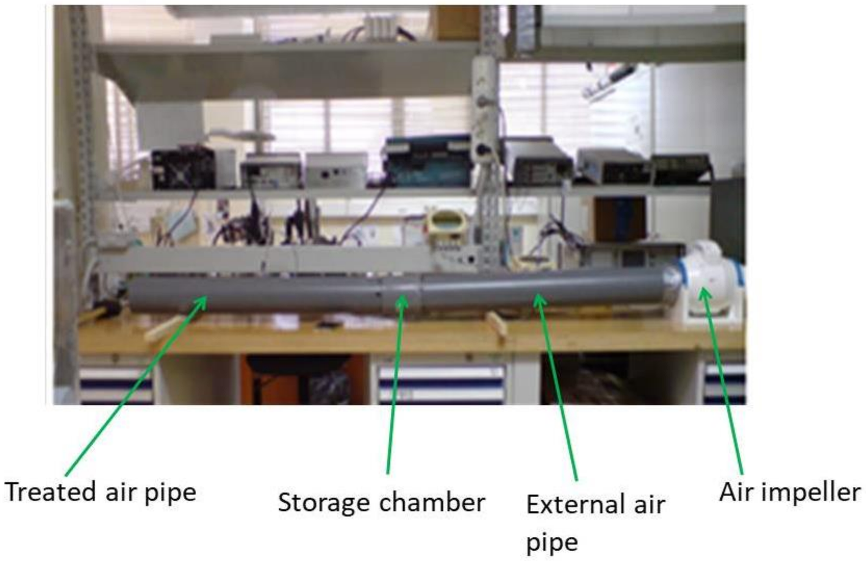

Fig. 5: Prototype built Natural zeolite reactor on the laboratory prototype

The hollow cylindrical pipe is made of PVC and has a global length of 175 cm divided in two sections: before and after the storage chamber. The pipe has an outside diameter of 11 cm and inside diameter of 10.5 cm. The storage chamber is a 13 cm long cylinder located in the center of the pipe. The interior of this chamber is used as a tank that is filled with 4-5 cm natural zeolite rocks. The air impeller located at the beginning of the tube allows controlled the entry of airflow into the reactor (see Figure 6). To regulate the flow of air circulating through the tube, a ventilation system is defined based on the diameter of the reactor.

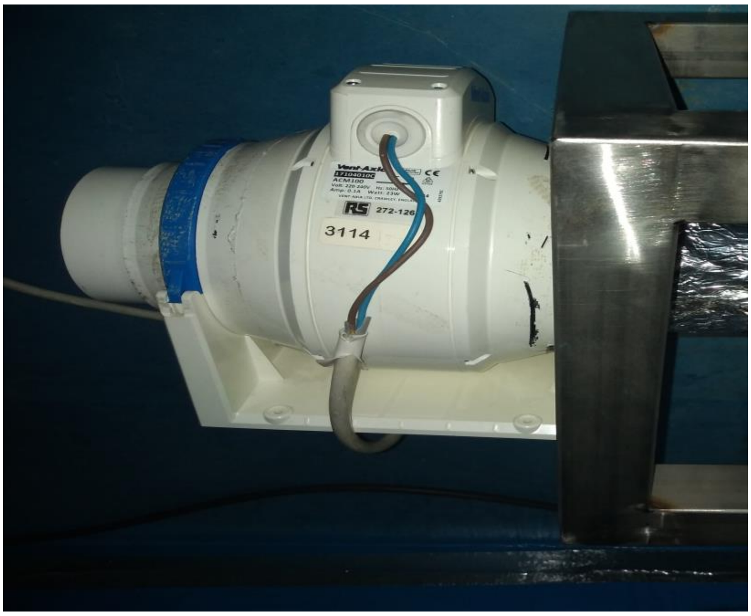

Fig. 6: Detail of the industrial air impeller located at the beginning of the tube

To characterize the thermal performance of natural zeolites under different ambient conditions of outside temperature, pressure and humidity rates, it is necessary to install anemometers and temperature sensors, both at the inlet and outlet of the tube. This arrangement will allow the evolution of the air flow inside the tube to be quantified, measuring temperature and air speed values in both positions. With this aim, Kimo transmitters have been installed to measure air velocity and airflows, as well as multi-variable measurement Kester and Fluke IR thermometers have been used to monitor temperature and air velocities. In addition, a thermographic camera ThermaCAM has been used to control the temporal evolution inside the tube and take pictures of the inside of the tube. These records will allow detailed CFD models to be better adjusted to real conditions. Table 2 shows the main characteristics of the measuring instruments used in this reactor:

Tab. 2: Characteristics of the measuring instruments used in the new Zeolite reactor constructed

Equipment	Variables	Accuracy
"ThermaCAM SC660 Wes (TC)"	Temperature and photos	±1°C or ±1% of reading for limited temperature range; ±2°C or ±2% of reading with Thermal sensitivity NETD <30 mK @ +30°C
Kimo instrument CTV 200	Air velocity and airflows	Accuracy air velocity from 0 to 3 m/s : ±3% of reading ±0,03 m/s; Accuracy temperature ±0,5% of reading ±0,3°C
Kestrel 3000 Environmental meter and Fluke IR thermometer	Temperature and velocity	±3% of reading or ±0.1 m/s / ±1ºC

3.4. Laboratory experiments

Before running the experiments with natural zeolites in the new reactor, it is necessary to prepare these materials to operate in good conditions. The laboratory process to prepare the material has been done in four steps:

- *Sample heating* and *dehydration*
- *Storage in a desiccator*
- *Encapsulation*
- *Sample hydration*

The first step consists on using the powerful laboratory muffle furnaces to dehydrate the sample (see Figure 7). This process has been carried out with muffle furnaces available for temperatures up to 500°C in a small cell. The second step involves placing the sample in an oven at 110°C for one day in order to heat the material. Based on the numerical results obtained in the simulations, the natural zeolite cell reactor requires an internal capacity to storage 1 kg. The third step is to store the heated sample in a vacuum desiccator for further analysis. The most important consideration in this process is the absence of air or moisture in the room. At this time, the sample is ready for thermal heat capacity assessments in the reactor prototype. Finally to test the thermal performance of these natural zeolites in the reactor, it is necessary to encapsulate the prepared material in a cell located in the middle position of the tube. In this way, the samples are ready to be hydrated in the development of the experiment.

Fig. 7: Laboratory muffle furnace to dehydrate the Natural zeolite sample

Once both the samples and the measurement equipment have been prepared, the experimental batteries are carried out in the Zeolite Reactor. These campaigns allow different experimental conditions to be tested by controlling, in real time, the variables of ambient temperature and air humidity. This is achieved by regulating the entry of airflow through the first section of the reactor. The thermographic camera has been used to regulate the boundary conditions and the thermal evolution of the ambient variables.

4. Results

The adsorption process has been modeled considering a hollow cylindrical tube, a cylindrical storage chamber located in the centre of the pipe, natural zeolites as storage elements and an air impeller that defines the boundary conditions of the study volume. The initial characteristics of this geometrical model are defined in table 3.

Tab. 3: Geometric and initial conditions defined for the natural zeolite reactor

Element	Characteristics and values
Cylindrical pipe	Outside diameter of 11 cm, inside diameter of 10.5cm and total length of 175cm
Air impeller	Inlet air velocity of 2 m/s
Storage cylinder	Length of 13cm and porosity value of 0.4
Reservoir	Filled with 4-5 cm natural zeolite rocks

Computational fluid dynamics simulations have been executed with Fluent in order to characterize the thermal performance of natural zeolites inside the pipe. Three-dimensional velocity (m/s) is obtained and showed in pipe section (Figure 8). With these conditions, the fluid flow is accelerated at the exit of the pipe up to a value of 2.5m/s. The velocity profile shows a maximum value at the center of the pipe.

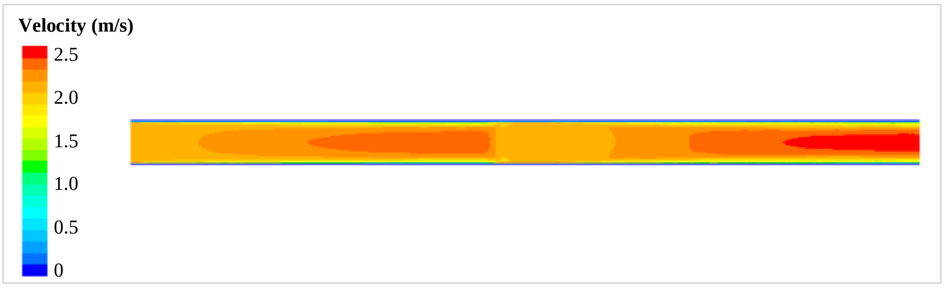

Fig. 8: Numerical results: air velocity inside the Zeolite reactor

To characterize the cationic capacity of the zeolite reactor, the thermographic camera TC has been used during the first experiments (Figure 9). These photos reveal high cationic exchange capacity for all types of zeolites (Seco et al., 2020)., where drying and cooling conditions in the absence of humidity have been seen to influence results in a high extent, as well as the adequate design of the reactor, to provide effective air friction with the zeolite sample.

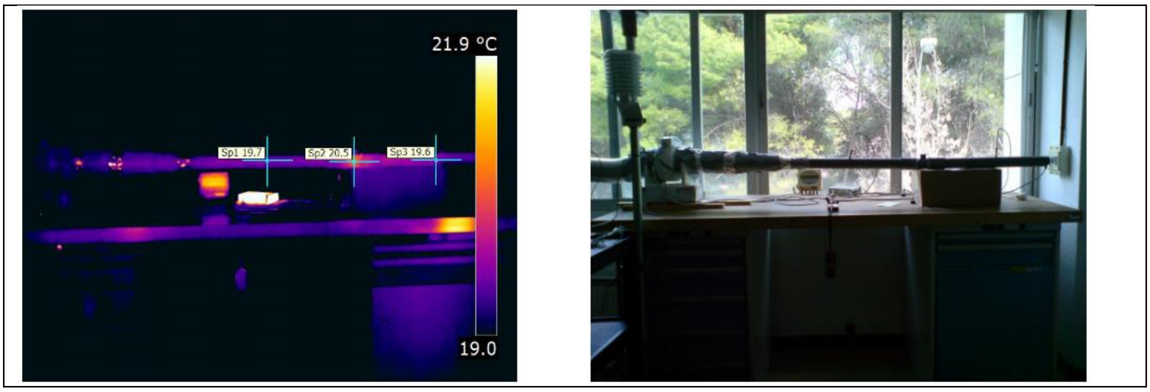

Fig. 9: Thermography done to characterize the cationic exchange of natural zeolites during the experiments

Under the internal storage capacity of 1 kg no thermal storage is achieved because outside air has almost the same temperature than the incoming ambient air. Process to dry the material uses laboratory muffle furnaces with temperatures up to 1.000°C in small cell. After different tests to check the necessary strength to apply in any zeolites tested, it has been observed that in any case with 1 kg of sample, 60 minutes at 465°C is enough energy to dehydrate completely the zeolite. At this moment, the sample is ready for thermal heat capacity analysis in the reactor prototype.

Figure 10 represent the evolution between incoming ambient air (blue lines) and the outlet air (green lines) once it passed through zeolite chamber. Two variables are represented: temperature (left side) and air velocity (right side) along the time. The outlet temperature presents higher value than ambient air, assuring the thermal store capacity of the zeolites.

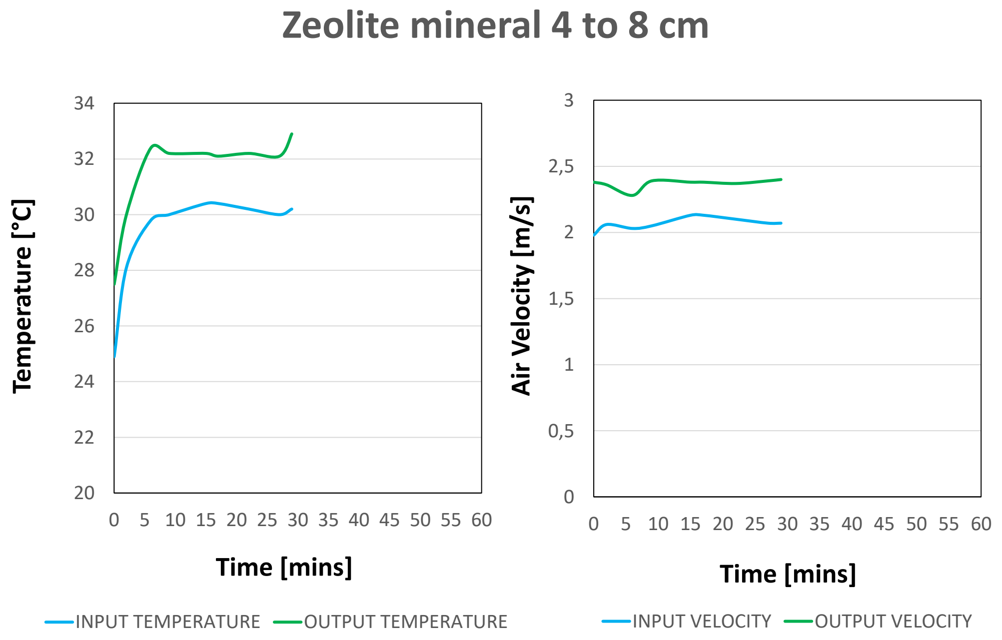

Fig. 10: Experimental results obtained using mineral zeolites: a) air temperature and b) velocity inside the Zeolite reactor

5. Conclusions

Thermal storage systems allow matching district energy consumption with energy generation depending on the urban requirements. There are several types of thermal storage available, but one of the most promising technologies uses natural zeolites. These storage systems take advantage of chemical processes by absorbing heat to excite a reaction and recovering heat reversing the reaction. These processes have been assessed through the use of fluid dynamic simulation models, which results made it possible to define and build a laboratory-scale zeolite reactor. The numerical results identify the main characteristics that are experimentally implemented in the zeolite reactor for the analysis under laboratory conditions. The experimental results indicate that the mineral zeolite does not obtain a regular cooling process throughout the study time. This fact highlights the importance of knowing how the zeolite gives the energy, since their characteristics define the type of storage application that can be applied.

Zeolite storage shows a very potential technology thanks to high energy storage and low heat losses. Nevertheless, it needs further development on a laboratory scale to be able to analyze different compositions, configurations and structures. With this aim, new models and new experimental developments will be made where different samples (synthetic zeolites) will be included for the new prototypes.

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