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Preface

It was a pleasure for Prof. Luisa F. Cabeza and the GREiA research group full team at the University of Lleida (Spain) to organize the ***Eurotherm Seminar #116 "Innovative solutions for thermal energy storage deployment"*** from May 24th to 26th, 2023, in Lleida (Spain).

This seminar did bring together researchers and practitioners from academia and industry, who are working in the rapidly expanding field of thermal energy storage (TES).

The most recent trends in such areas as novel storage materials, advanced storage concepts and configurations, TES for renewable energy systems, TES in buildings and industry, use of advanced techniques such as artificial intelligence in TES, and social, environmental, and educational aspects.

Theoretical, numerical, and experimental approaches will be presented for the entire range of applications, from material characterization to complete storage systems.

We want to thank all participants to the conference.

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Innovative 3D adsorbent structures based on sapo-34/S-PEEK composite materials for energy storage

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Abstract

In this paper, the synthesis and characterization of an innovative three-dimensional lattice structure, based on a composite zeolite adsorbent material SAPO34 and sulphonated polyether ether ketone (S-PEEK), for thermal storage is evaluated. The structure was created with the aid of additive manufacturing technology which allowed to obtain a structure with a complex three-dimensional geometry, without a metal reinforcement support. The optimization of the technology has been developed in terms of sample geometry and composite material formulation. The three-dimensional structures thus obtained were morphologically and mechanically investigated in order to evaluate their structural integrity. To evaluate the adsorption/desorption capacity, the water vapor adsorption isobars at 11 mbar were measured at equilibrium in the temperature range 30-120°C, confirming that the matrix does not hinder the mass flow of water vapor and the zeolite filler participates in the adsorption/desorption process, indicating this material potentially suitable for thermochemical storage by sorption technology.

Keywords: zeolite, composite sorbent, sulfonated poly(ether ether ketone), additive manufacturing

1. Introduction

The growing need to propose more sustainable energy systems, based on a synergistic integration of different energy sources and the consequent development of technologies aimed at optimizing the recovery of waste heat has led to the investigation of innovative thermal energy storage technologies [1]. In this context, thermochemical heat storage represents a promising and viable technology, especially for long-term storage applications. However, its development on large-scale and realistic contexts has been partially limited by the need for more performing and suitable materials for fast and reliable storage design and tailoring.

Recent papers have shown that reactors consisting of coated heat exchangers (HEX) have multiple advantages in terms of heat transfer efficiency which make their applicability very promising [2]. However, limiting factors on the use of this technology are the low amount of absorbent material present and a non-optimal mass transfer (due to the low porosity and the presence of the binder in the composite material) in a coated HEX. This reduces the storage performance of the reactor.

In previous works [3,4], a composite material was proposed consisting of a high content of zeolite (up to 90% by weight of SAPO-34) and a sulphonated poly(ether-ether-ketone) (S-PEEK) binder. The S-PEEK matrix has a high water vapor permeability, thereby minimizing mass diffusion limits within the adsorbent composite bulk. Furthermore, this composite material has shown promising results in terms of mechanical, thermal and ad/desorption stability.

Based on these promising results, the present paper aims to investigate the use of this material for the realization of three-dimensional lattice structures entirely made of adsorbent composite material (without metal support) with the aid of the additive manufacturing technique.

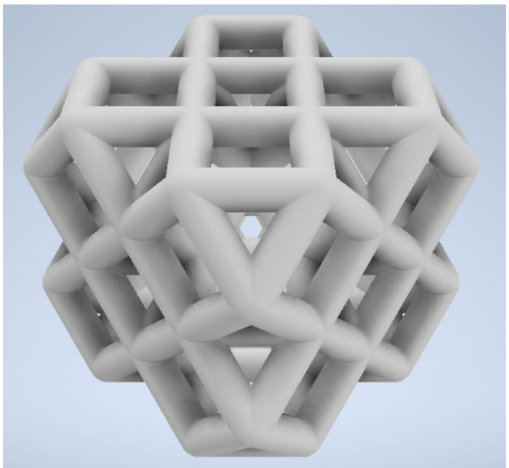
2. Materials and method

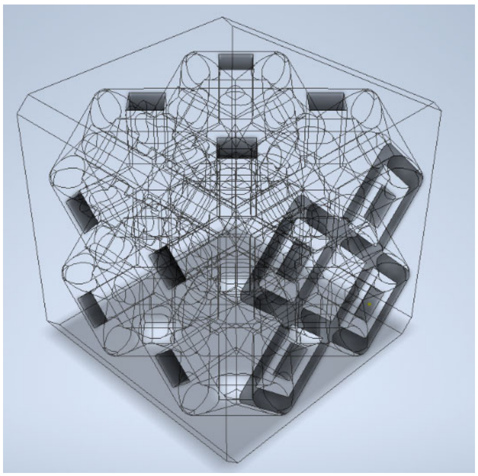
The sulfonation PEEK, used as matrix was prepared according to [5]. A SAPO 34 zeolite (AQSOA Z02) produced by Mitsubishi Plastics Inc. was used as an absorbent filler with main grain size of about 5-10 µm. For the composite adsorbent, S-PEEK polymer was dissolved in N, N-Dimethylformamide (DMF) under magnetic stirring for 5 min. Then SAPO-34 powder was progressively added to the solution, under agitation at room temperature until a homogenous slurry is obtained. In parallel, a PVA negative shape of the attended 3D structure is obtained by additive manufacturing. The composite slurry was gently poured into the channels of the PVA mold until it is completely filled. Then the filled PVA mold has been dried at 60 °C for 12 h in a vacuum oven. Afterward, the negative PVA mold is dissolved in water by ultrasound until only the now solidified three-dimensional S-PEEK/zeolite composite structure is obtained.

3. Results and discussion

Figure 48 shows the proposed steps for the manufacturing of the 3D printed lattice sorbent structure. In particular:

- *CAD Design*: Parametric design of the unitary lattice cell. Spatial replica 2x2x2 (Figure 48a).
- *Molding*: Design of the negative molding of the lattice structure (Figure 48b) and subsequent additive manufacturing by using PVA support.
- *Composite filling*: Preparation of the composite SAPO-34/S-PEEK slurry. The slurry is filled in the channel of the negative mold by using a micro-syringe. The slurry was added until the mold was fully filled (Figure 48c). The drying is performed in oven at 40°C for 1 day.
- *Mold dissolution*. To remove the negative PVA mold a dissolution procedure in water was applied. In particular, the materials were placed un an ultrasonic bath at 70°C for 5h until the complete dissolution of PVA support occurred. The residual material is the solid zeolite-S-PEEK sorbent material with the shape of the chosen lattice structure (Figure 48d).


a)


b)


c)


d)

Figure 48. Manufacturing of the 3D printed lattice sorbent structure: a) parametric definition of the 3D cell; b) negative molding of the cell c) negative molding (PVA polymer) filled with composite SAPO-34/S-PEEK slurry; d) demolded lattice sorbent structure.

The 3D lattice structure thus obtained was morphologically and structurally assessed with the purpose to well define the synthesis characteristic, structural homogeneity and integrity. The adsorption/desorption capacity was performed by water vapor adsorption isobars at 11 mbar measured at equilibrium in the temperature range 30-120°C (by using a DVS equipment). The results reported in Figure 49 , confirm that the matrix does not hinder the mass flow of water vapor and the zeolite filler participates in the adsorption/desorption process, indicating this material potentially suitable for thermochemical storage by sorption technology.

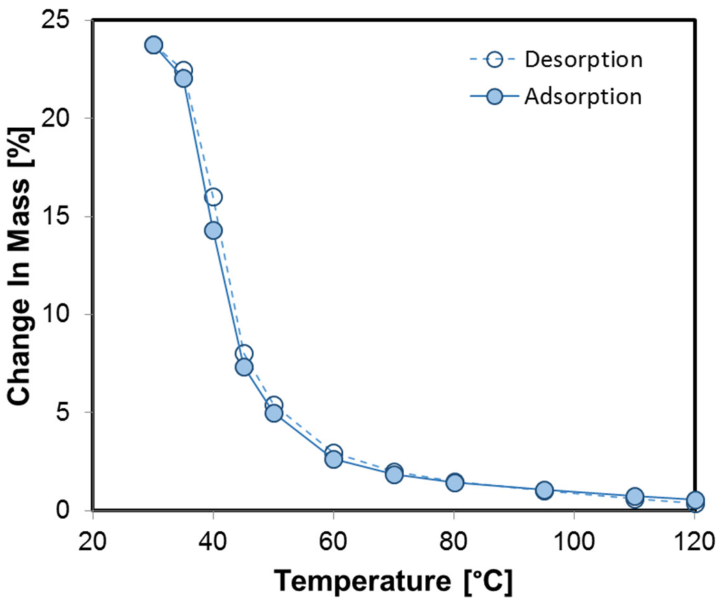


Figure 49. Water adsorption (filled marker) and desorption (empty marker) isobars at 11 mbar for 80% SAPO-34 zeolite filled in S-PEEK polymer material

4. Conclusions

The present work investigated the synthesis and characterization of an innovative three-dimensional lattice structure, exclusively constituted by an adsorbent composite material (SAPO34 zeolite and S-PEEK as filler and matrix, respectively), for thermal storage. The results highlight that:

- The composite has an effective adsorption/desorption capacity, indicating that the polymeric matrix does not hinder the diffusion of water vapor towards the zeolite.
- The additive manufacturing, by using a negative PVA mold, is a suitable technology in order to procedure adsorbent tridimensional lattice structures.
- The 3D structure is structurally stable, regular, homogeneous and with a high surface area, indicating it as a promising solution for the construction of reactors for energy storage.

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