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Silica-based aerogels as aggregates for cement-based thermal renders

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ABSTRACT

Subcritically dried silica-based aerogels were synthesized by design to be used as aggregates for lightweight cement-based thermal renders. The molecular and pore structure of the aerogels and of the corresponding renders were correlated with their thermal conductivities. A subcritical hybrid aerogel proved to have advantages over a supercritical commercial one, since the particle size distribution may be controlled, it is more hydrophobic, and imparts higher specific surface areas and total pore volumes to the renders. Good stabilization of the hybrid aerogels within the aqueous cement paste, without affecting the final renders' structure, was accomplished by using an anionic surfactant. The efficient range of aerogel contents for thermal insulation purposes (above 60 vol% of total aggregate) was optimized using an inorganic subcritical aerogel. Thermal conductivities as low as ~0.085 $W.m^{-1}.K^{-1}$ and densities of 410 $kg.m^{-3}$ were achieved by total replacement of silica sand with a designed hybrid aerogel.

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1. Introduction

The building sector plays a remarkable role on the global energy consumption, with a larger share than that of the entire mobility sector [1]. With the increasing emphasis on energy-conscious strategy and the broader environmental impact of buildings, greater attention is necessarily being drawn to the appropriate use of thermal and acoustic insulation solutions, for both new and existing buildings. In fact, European contemporary construction industry must comply with demanding energy and environment-oriented directives (2002/91/CE, 2010/31/UE and 2012/27/UE) [2,3]. Developing new high-performance thermal insulation materials and solutions is crucial to accomplish those requirements, replacing or improving traditional ones. The key strategy is to achieve as low thermal conductivity as possible, enabling the application of relatively thin building envelopes with high thermal resistance. Thermal mortars are among the most promising materials used in energy saving, due to thermal conductivities lower than 0.1 or 0.2 $W.m^{-1}.K^{-1}$, categorized respectively as T1 or T2 [4]. In this regard, studies in the field have brought-in advanced materials as mortars' aggregates, which improve thermal performance, while maintaining acceptable mechanical and physical properties. Classical examples are expanded polystyrene (EPS) [5], cork [6,7] and hollow ceramic microspheres (HCM) [8]. EPS is a rigid and resistant thermoplastic polymer, with closed cells [9], whereas cork is a cellular, natural and renewable material with slow combustion and good mechanical properties [10], and HCM are porous materials with closed cells and high stiffness [11]. The common property between these aggregates is the very low thermal conductivity (in the range 0.033–0.057, 0.035–0.070 and 0.050–0.200 $W.m^{-1}.K^{-1}$, respectively for EPS, cork and HCM).

Recently, silica-based aerogels have proven their relevance as aggregates in high-performance plasters [12], mortars [13] and concrete [14]. In fact, silica aerogels are among the best known thermal insulating materials (with thermal conductivity around 0.015 $W.m^{-1}.K^{-1}$ at ambient temperature and pressure), due to the combination of low density (typically in the 3–500 $kg.m^{-3}$ range), porosity above 90% and small mesopores (between 4 and 20 nm) [15–17]. Additionally, they present good fire and acoustic resistance and, when broken down, aerogel particles do not lose their insulating properties, contrarily to hollow ceramic microspheres, which

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degrade. Since the structure and properties of silica-based aerogels may be fine-tuned, they may offer further advantages such as resistance to moisture, waterproofing and self-cleaning properties, corrosion protection, UV reflection, durability, fire retardation and low contents in volatile organic compounds (VOCs) [15,18].

The studies on the effects of incorporating silica aerogels in non-Portland cement-based insulation materials are very recent, and most are still being published as patents [19,20]. Stahl et al. developed a new high-performance thermal render (thermal conductivity as low as 0.025 $W.m^{-1}.K^{-1}$) by incorporating hydrophobic silica aerogel granules (60–90 vol% relative to the total mixture), which is already commercialized in Switzerland [13,20]. Higher contents of hydrophobic aerogel (96–99 vol%) lead to even lower values of thermal conductivities (0.014–0.016 $W.m^{-1}.K^{-1}$) [12]. The thermal insulation plasters developed by Achard et al., which reach thermal conductivities between 0.095 and 0.034 $W.m^{-1}.K^{-1}$ by varying the hydrophobic aerogel content from 17% to 54 wt% (relative to the hydraulic binder) [19,21,22], also have very high performance. Notwithstanding the spectacular thermal conductivities of these aerogel-based renders, there are some associated drawbacks, such as difficulties in the successful mixing of the aerogels due to their hydrophobicity, and decrease in the mechanical strength of the plasters because of the high porosity of the aerogels [12,13]. The low density of the hardened mortars mixes can condition their application field.

Regarding Portland cement-based materials, lightweight concrete has been recently reported by Gao et al. by incorporating hydrophobic aerogel granules into a concrete matrix and attaining a thermal conductivity of 0.26 $W.m^{-1}.K^{-1}$, at an aerogel content of 60 vol% (of the concrete sample) [14]. Kim et al. obtained a decrease of 75% of the thermal conductivity of cement paste (from 0.533 to 0.135 $W.m^{-1}.K^{-1}$) with the incorporation of a superhydrophobic aerogel powder in 2.0 wt% of cement [23], but not addressing the effect of different particle sizes and the influence of a coarse aggregate. In fact, the aerogel was not added as such, but as gel-typed by methanol, which should be avoided in construction sites due to its toxic effects. Other authors concluded that the addition of hydrophobic silica aerogel powder as filler in the cement paste reduces the thermal conductivity at the expense of a reduction of the compressive strength and an increase in water permeability [24]. Nevertheless, the thermal conductivity values indicated are of no use, since regrettably the authors do not refer the aerogel contents.

So far, the suppliers of the existing cement-based thermal renders tend to use aerogels from the few well-established manufacturers in the world, instead of developing functionalized aerogels appropriate for each mortar. Besides, most of these aerogels are supercritical (dried at temperatures and pressures above the supercritical point of the solvent), making their application undesirable in terms of energy, safety and costs [15,25,26]. By using as aggregates subcritical aerogels, the drying costs are reduced and the safety is increased, partially overcoming those drawbacks.

This work is focused on silica-based aerogels, prepared under atmospheric conditions, intentionally for incorporation as aggregates in cement-based thermal renders. Emphasis is given to the structural and morphological characterization of different types of aerogel, ranging from hydrophilic (purely inorganic) to hydrophobic (organically modified by post-synthesis silylation), as well as to their influence on the chemical and physical properties of the renders. The influence of using a surfactant to promote the paste mixture is analyzed, and the aerogel type and content are tuned in order to optimize the renders' thermal conductivity.

The results obtained encourage further work on cement-based thermal renders using silica aerogels as aggregates, with improved mechanical resistance and broader application fields.

2. Materials and methods

2.1. Preparation of the silica-based aerogels

Two silica-based aerogels, an inorganic (IA) and a hybrid one (HA), were developed in the present work to be used as aggregates in cement-based renders (Fig. 1).

Both silica-based aerogels were synthesized by a two-step sol-gel process. The first step consists of the acid-catalyzed hydrolysis of tetraethoxysilane (TEOS) and the second one of the poly-condensation of the resulting silanol groups, induced by addition of ammonia. TEOS (from Aldrich, 98%) was previously diluted in 2-propanol (*i*-PrOH, p. a. from Sigma-Aldrich), and distilled water was added, dropwise, while stirring. The reaction mixture was acidified with HCl (p.a. from Carlo Erba) to initiate the hydrolysis process. The acidic colloidal solution was placed in a sealed container, heated at 60 °C, and stirred (at 120 rpm) for 60 min. The required amount of ammonia (NH_4OH, from Merck, 33%) was then added, and the resulting homogeneous sol was left to gel, with no further stirring.

For inorganic aerogels, the alcogels were aged for 24 h in the residual liquid and for a further 24 h in an equal volume of ageing solution, containing TEOS, *i*-PrOH and H_2O in the same proportions as used for gelation, to strengthen the silica network. The pore liquid was then exchanged with *i*-PrOH to completely remove any residual water or TEOS, and the gels subcritically dried under ambient pressure, in a solvent-saturated atmosphere, until their weight loss became negligible (aerogels IA). After 24 h in the residual liquid, some of the aged alcogels were further hydrophobized by chemical surface modification with an ageing solution of hexamethyldisilazane (HMDZ, 99.9%, Aldrich) in *i*-PrOH (20% volume), and left to dry until their weight loss became negligible (aerogels HA).

A hybrid commercial supercritical aerogel from a well-known industrial manufacturer (aerogel CA) was also used, in order to compare its incorporation and the performance of the mortars.

2.2. Preparation of the aerogel-based renders

Cement-based mortars were produced with a constant binder/aggregate volumetric ratio of 1/4. Portland cement CEM II/B-32.5 N was used as binder and the aggregate varied from 100% of silica sand (from Areipor) to 100% of aerogel, as indicated in Table 1. The reference render has 100% of silica sand with the particle size distribution from Table 2. The water/cement ratio was optimized in each case, in view of the render paste's workability. Since the reference render used a ratio equal to 1 [27], it was adjusted to obtain the same flow value of 175 ± 10 mm, according to EN 1015-2 [28].

Since the aerogels are inherently fragile materials, due to their open pore structure, the production of the mortars in terms of mixing was adapted, in order to preserve them. The aerogels synthetized in this work were previously grinded and passed through a set of sieves with progressively decreasing mesh size. The particle size distribution of the inorganic aerogel (IA) was the same as in Table 2, whereas for the hybrid aerogel (HA) only the finer grades (below 0.250 mm) were used. The commercial aerogel (CA) was supplied with different grain size particles, below 2 mm. Because of its greater fragility (they crush by simple finger pressure), this aerogel was added to the mortar mix without any previous particle size selection. Two methods were followed for the incorporation of the aerogels: i) due to their lightweight and hydrophobic nature, the hybrid aerogels were previously mixed with a commercial anionic surfactant (*Hostapur OSB*); then, water was added slowly to obtain a uniform mixture; when applicable, the other aggregate

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