Postprint of: Giosuè C., Pierpaoli M., di Perna C., Citterio B., Mangiaterra G., Ruello M. L., Tittarelli F., Properties of an innovative multi-functional finish for the improvement of indoor air quality, BUILDING AND ENVIRONMENT (2023), 110091, DOI: 10.1016/j.buildenv.2023.110091 © 2023. This manuscript version is made available under the CC-BY-NC-ND 4.0 license https://creativecommons.org/licenses/by-nc-nd/4.0/

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2	Properties of an Innovative Multi-functional Finish for the Improvement of Indoor Air
3	Quality
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16	
17	Abstract
18	Due to lifestyle changes, people spend most of their time indoors at present; thus, Indoor Air Quality
19	(IAQ) is a matter of utmost importance. Multi-functional and innovative finishes can help to passively
20	improve the IAQ, benefitting the health and comfort of occupants. For this study, reference and pre-

21 mixed commercial mortars are compared to a new multi-functional hydraulic lime mortar for indoor
22 finishes, in which conventional aggregates are substituted by a highly porous adsorbent material and

biomass waste ashes. The up to 20% higher accessible porosity of the multi-functional finish led to 23 24 lower density (30%), higher thermal insulation properties (30%), higher water vapor permeability 25 (more than 40%), and improved moisture buffering capacity (three times higher), when compared to 26 the reference mortar. Different types of photocatalytic agents (TiO<sub>2</sub>) were also added into the new multi-functional hydraulic lime mortar, in order to investigate their effect on the de-polluting 27 28 properties of the finish. Even if the photocatalytic efficiency remained unexpressed under indoor 29 conditions, due to the predominance of the adsorption process, the de-polluting properties of the new 30 mix were more than 30% higher than that of the reference mortar. The obtained results confirm that 31 the developed innovative multi-functional finish-besides fulfilling the ordinary requirements-is better than commercial mortars, as it can improve the IAQ passively, thus benefitting the health and

33 comfort of occupants.

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### 35 Keywords

Indoor Air Quality (IAQ),) thermo-hygrometric behaviour, mould susceptibility, photocatalytic
oxidation (PCO), adsorption, de-polluting properties.

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### 39 1. Introduction

The sustainability of the building sector has recently received increased attention, as around 40% of global carbon dioxide (CO<sub>2</sub>) emissions are directly related to activities associated with the construction industry [1]. Consequently, several strategies have already been formulated to tackle their impact on the environment [2]. European Directives and laws have become stricter, in terms of energy efficiency; in particular, with more sealed buildings, if an appropriate air change rate is not guaranteed, the Indoor Air Quality (IAQ) may worsen as a result.

IAQ impacts the health, comfort, well-being, and cognitive performance of a building's occupants. 46 As up to 90% of the population's time is spent indoors, on average [3], the risks related to being 47 exposed to the different pollutants, such as Volatile Organic Compounds (VOCs), moulds, airborne 48 49 particles, and inadequate levels of Relative Humidity (RH) are growing. Indoor humidity affects 50 warm respiratory comfort, skin humidity, perceived IAQ, and well-being of occupants [4][5]. Moreover, high RH affects the durability of building materials and promotes biological attack of the 51 surfaces, thus increasing maintenance costs [6]. Fungi and micro-organisms are also unhealthy for 52 occupants, as they produce spores, allergens, toxins, and other metabolites that contribute to the 53 degradation of IAQ [7] with consequent allergies, irritations, and respiratory and skin diseases. These 54 55 negative effects can be short- or long-term, such as the well-known Sick Building Syndrome (SBS). SBS has been recognized by the U.S. National Institute for Occupational Safety and Health (NIOSH), 56 57 the major cause of which is poor ventilation quality.

To settle this problem, the main techniques used at present to improve IAQ are ventilation, source control, and air cleaning [8] by means of active systems. However, thanks to their wide surface of exposure, building materials such as finishes can positively interact in a passive way with the surrounding environment [9], so these materials can help active systems to be more effective, using a lower amount of energy [10]. It has been demonstrated that highly porous materials applied indoors can act as buffers for RH which affects the indoor concentrations of humidity and VOC such toluene, as reported in XX, both numerically and by means of laboratory studies [11] [12].

Adsorption is one of the most effective de-polluting processes, in which a gas or liquid is passively 65 removed from a fluid and transferred on the solid surface of the adsorbent, where the adsorbate covers 66 67 the surface of the adsorbent with a molecular layer. The capturing methods differ in terms of the 68 polarity (electrostatic forces), molecular weight (the higher the molecular weight, the higher the boiling temperature and the easier the deposition on the absorbent surface), and size (molecular 69 70 sieves) of the adsorbate and adsorbent. As it is focused on the surface of the solid, the adsorption process requires a high specific surface of the solid adsorbent and a high solid-fluid contact area. 71 Therefore, in order to obtain high removable efficiency of pollutants, very porous materials with a 72 73 wide specific surface area (of about  $300-3000 \text{ m}^2/\text{g}$ ) are required as adsorbents.

Heterogeneous Photocatalytic Oxidation (PCO) also represents a promising option to ensure healthier 74 environments, exploring the possibilities of the use of nanotechnology in building materials [13][14]. 75 76 PCO is a superficial phenomenon [15], induced when a photocatalytic agent, such as titanium dioxide 77 (TiO<sub>2</sub>), is added to a finish—either in bulk or as coating—for the mineralization of adsorbed 78 pollutants into less harmful compounds. This process involves different steps [16][17]: PCO first requires UVA light wavelengths (320-400 nm) to activate the agent, generating a hole/pair. Then, 79 the pollutants are adsorbed onto the TiO<sub>2</sub>. PCO capacity is significantly affected by the presence of 80 macro/micro pores [18][19] and hydraulic lime mortars generally have higher de-polluting properties 81 82 than cement-based ones [16][20][19][21][22], due to the higher presence of micro-porosity; on the 83 other hand, the higher quantity of gel pores (nanopores) than in cementitious products may hide the 84 catalytic sites [18][19][23].

To improve the sustainability of finishes [24] [25], biomass by-products (e.g., ashes from the thermal 85 valorisation of biomasses) have been used as unconventional aggregates [26]. Biomass ashes are 86 considered carbon neutral as, in binder-based materials, they bind the same amount of CO<sub>2</sub> released 87 88 in the combustion process [27]. Ashes typically contain silicon, calcium, potassium, phosphorus, manganese, iron, zinc, sodium, and boron, in the form of oxides, and studies have demonstrated that 89 90 their use does not negatively influence the ecotoxicity of mortars [28]. Rather, they generally improve the mechanical and durability properties of the resulting concrete/mortar products, while also 91 92 lowering the release of hazardous elements [29]. "Green" building materials containing biomass ashes 93 able to decrease level of ozone [30] and indoor Volatile Organic Compound (VOC) de-pollution 94 properties due to their adsorption ability [31], have already been successfully developed [31]. 95 Biomass ashes also enhance the thermo-hygrometric performance of finishes/renders [32] [33][34]. As long as an RH level from 40-70% can be guaranteed, the primary requirements for indoor 96 97 materials are high transpirability to water vapour, avoiding the storage of humidity, and the ability to act as an hygroscopic buffer by absorbing and desorbing moisture [35]. The capacity of a finish to 98

absorb water vapor during exposure at high levels of RH and to release water vapour when the levelof RH decreases is expressed as the Moisture-Buffering Capacity (MBC) [36] [37].

101 This research is aimed at the development and testing of an innovative multi-functional mortar (Italian 102 Patent 102017000033750) for indoor finishes, which is capable of passively improving IAQ, thus benefitting the health and comfort of occupants, in terms of permeability, MBC, de-polluting activity 103 104 and inhibition of mould growth, besides fulfilling the ordinary requirements. In the developed mix, conventional sand is replaced by volume with biomass ashes and an unconventional aggregate, 105 106 characterized by high adsorption properties and currently not used in the building sector but, instead, 107 in water/air depuration processes [38]. The interaction with different  $TiO_2$  agents activable under UV 108 and VIS radiation [39], as declared in the product's datasheet, is also considered. If  $TiO_2$  is present, 109 the unconventional adsorbent aggregates give large contacting areas to the catalytic agent which, 110 thanks to their high specific surface, can potentially improve the de-pollution activity of the material. 111 In this way, saturation of the adsorbent material could also be avoided, providing the benefit of 112 maintaining the same efficiency over time [40]. As references, a traditional mortar and two different commercial pre-mixed products for the same indoor application were manufactured and tested. 113

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### 115 2. Experimental program

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### 117 2.1.Materials

A hydraulic lime, classified as LIC 3.0 according to UNI EN 15368:2010, with a density of 2650
 kg/m<sup>3</sup> (commercial product Plastocem, by Italcementi) was used as binder.

The commercial sand, CA 400 (provided by Cava Gola della Rossa, Italy), was chosen as conventional aggregate for the reference mortar, which is a calcareous sand with 98% purity, and water absorption to reach the saturated surface dry (ssd) condition of 5% in weight and ssd density of 2650 kg/m<sup>3</sup>.

The aggregates used in the patented mix are a porous adsorbent material and fly/bottom ash obtained from a biomass (corn cobs; USA) thermal plant. The porous adsorbent material is currently not used in the building sector, but in water/air depuration processes [38]. It was obtained in the form of granules about 10 mm in diameter, and its water absorption to reach the ssd condition is 86% with corresponding density of 1310 kg/m<sup>3</sup>. Before being added into the mix, it was ground and sieved at 300  $\mu$ m, in order to reach a grain size suitable for indoor finishes.

Fly and bottom biomass ashes are a mixture of inorganic and organic compounds, due to the presence
of unburnt components. The water absorption to reach the ssd condition is 49% and 20%, and the ssd
density is 1410 and 1940 kg/m<sup>3</sup> for fly and bottom ashes, respectively. The ashes were chemically

characterized by X-ray analysis, using an RX Philips PW 1730 X-ray diffractometer (operating
voltage/current 40 kV/30 mA; scan mode: continuous speed, 3°/min), and by thermogravimetric
differential (TG-DTA) analysis (STA 409 EP) up to a temperature of 1000 °C with a heating rate of
10 °C/min in an oxidizing environment. Ashes were crushed and sieved at 90 µm before the analysis,
in order to provide a homogeneous sample.

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Figure 1. X-ray diffraction patterns of biomass fly and bottom ashes.

For both ashes, the most present crystalline phase was quartz, followed by calcium carbonate (CaCO<sub>3</sub>)
(Figure 1). In the case of fly ash, the presence of alite was also detected, as well as traces of calcium
hydroxide due to hydration of CaO under environmental humidity. In case of bottom ashes, trace
albite was observed. As the presence of amorphous phases gives a curve baseline in the range of 30°
[41], no amorphous phases were detected in the current ashes.



149 Figure 2 TG/DTA of fly (a) and bottom ashes (b)

- Calcium carbonate was detected also in TG, as an endothermic peak at T ~ 700 °C (step 4 in Figure 151 152 2a; step 3 in Figure 2b), and was quantified as 3.71% and 4.91% of the total weight in fly and bottom ashes, respectively. Moreover, the endothermic peak at T = 80-130 °C (step 1 in Figure 2a, b) was 153 154 related to the loss of the unbound water. Step 2 in Figure 2a, b, related to an exothermic reaction at T ~ 400 °C, was due to the loss of unburnt organic carbon, quantified as 0.32% and 0.27% of the total 155 weight of fly and bottom ashes, respectively. In fly ash, the presence of Ca(OH)<sub>2</sub> was confirmed by 156 the endothermic reaction (labelled step 3 in Figure 2a) from 470 to 650 °C, and quantified as 2.98% 157 158 of the total weight. Before addition into the proposed mix, the bottom ash was milled and sieved at  $d_{max} = 500 \ \mu m$ , providing a grain size suitable for indoor finishes. 159
- The effects of three different types of nano- $TiO_2$  on the de-polluting properties of the new finishes 160 was also considered. The selected products were P-25 Aeroxide® by Evonik (TiO<sub>2</sub>A), KRONOClean 161 162 7000 (TiO<sub>2</sub>K), and KRONOClean 7404 (TiO<sub>2</sub>J) by KRONOS International Inc., which are activable 163 by VIS radiation. According to the technical data sheet, P-25 is a mixture of anatase-rutile-164 amorphous phases (78, 14, and 8% in weight, respectively) [42]. The particles had a nano-size of about 20–50 nm. The specific surface, measured by BET, was 35–65 m<sup>2</sup>/g. The pH of a 4% water 165 dispersion was 3.5–4.5, and the density was reported as 3.1 g/cm<sup>3</sup>. KRONOClean® is a TiO<sub>2</sub> 166 photocatalyst that degrades pollutants both under VIS and UV radiation. As reported in the data sheet, 167 the content of  $TiO_2$  is higher than 97.5%, with a prevalence of anatase phase. The particle size is 168 approximately 15 nm. The specific surface area, measured by BET, was higher than 225 m<sup>2</sup>/g, the 169 density was 2.9 g/cm<sup>3</sup>, and the pH range of 4–9 in water dispersion, as declared in the technical 170 datasheet. The main difference between the last two photocatalytic products is the carbon content 171 which, for  $TiO_2J$ , was up to 6.1% when measured by EDAX analysis, as shown in [43] and in TiO2K 172 was 0.7%, measured by means of TGA, evaluated as organic carbon. 173

Boron salts (from Durga) were added as a biocide admixture, to prevent possible biological attack insome specimens of the new mixes.

Two different commercial inorganic finish pre-mixes (Commercial 1 and Commercial 2), sold for thesame indoor application, were selected and tested for further comparison.

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179 2.1.1 Mix design

According to a previous work [44], the optimum water to binder ratio to reach the best consistency was evaluated as 0.58. Table 1 provides the mix design parameters of the tested mortars. Aggregate was used in ssd condition; in this state, the aggregate does not absorb or release water in the mix, without any changes to the water to binder ratio (w/b).

	Water	Hv-lime	Sand	Adsorbent	Bottom	Elv ash	TiO <sub>2</sub>		Premixed
Mix		, -			ash	,	P-25	KronoClean	mortar
	g/l	g/l	g/l	g/l	g/l	g/l	g/l	g/l	g/l
Reference (REF)	256	437	1535						
Reference TiO2 (REF TiO2)	256	437	1535				26		
Commercial 1 (COM1)	286								1360
Commercial 2 (COM2)	408								1360
MIX1	256	437	-	379	281	204			
MIX1 TiO2A	256	437	-	379	281	204	26		
MIX1 TiO2K	256	437	-	379	281	204		26*	
MIX1 TiO2J	256	437	-	379	281	204		26**	

185	Table 1. Mix design (g/l) of the tested mortars.	* KRONOClean 7000 (TiO2K),	and **KRONOClean 7404 (TiO2J)
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187 The two commercial pre-mixes (Commercial 1 and Commercial 2) were prepared by adding the188 amount of water suggested in the technical data sheets.

In presence of biomass ashes, boron salt was added in MIX1, MIX1  $TiO_2A$ , and MIX1  $TiO_2J$ specimens as a water-based solution (active principle of 16%), at the dosage indicated in the technical data sheet (0.06 g for each litre of cast).

### 193 2.1 Methods

## 194 2.1.1 Fresh, mechanical, and micro-structural properties

The fresh properties of mortars were evaluated in terms of workability class. The slump was measuredwith a truncated conical mould and jolting table, according to the standard UNI EN 1015-3:2007.

After 28 days of curing at Temperature (T) of  $20 \pm 2$  °C and Relative Humidity (RH) at  $95 \pm 5\%$  for the first 7 days and  $65 \pm 5\%$  for the following 21 days, the hardened properties of mortars were

investigated. The compressive strength was evaluated, according to the standard UNI EN 1015-

11:2007, in 4 × 4 × 16 cm specimens, by means of a Galdabini hydraulic press with a precision of
1%.

The density of hardened mortars ( $\rho$ , in kg/m<sup>3</sup>) was evaluated by weighing and measuring (with a calliper) the dimensions of the different dried specimens. Specimens were considered dry when, after exposure in an oven at T = 50 °C, two different weights in a time interval of at least 24 hours did not exceed 0.1%.

In hydraulic lime-based materials (as well as cementitious ones), the porosity can be divided as 206 follows: i) gel pores—nanopores from about 0.5 to 10 nm, inside the hydration products; ii) capillary 207 208 pores—micropores from 10 nm to 5 µm, mainly affected by the degree of hydration and the w/b ratio; 209 iii) macropores—higher than 5 µm, due to the entrainment of microbubbles; and iv) the porosity 210 within the aggregate, which is related to the type of aggregate [34]. The pore size distribution of 211 mortars was studied in the pressure range of 0.01-200 MPa by means of Mercury Intrusion 212 Porosimetry (MIP) using a PASCAL 240 (Thermo Fisher Scientific, Waltham, MA, USA). For each type of mortar, three fragments with dimensions of about 1 cm<sup>3</sup> were sampled and tested, and the 213 214 average results are reported.

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### 216 2.1.2 Thermo-hygrometric properties

As reported in a previous study [18], small amounts of  $TiO_2$  do not affect the thermo-hygrometric behaviour of mortars. In this case,  $TiO_2$  was added at low percentages (2% by solid weight); thus, the thermo-hygrometric properties, in terms of water vapor permeability, Moisture Buffering Capacity (MBC), and thermal conductivity, were tested only for mixes without  $TiO_2$ .

The water vapor permeability was tested according to UNI EN 1015-19:2007, and data were processed according to UNI EN ISO 12572:2007, which evaluates the water vapor resistance factor  $(\mu)$  in cylindrical specimens (h = 3 cm, d = 13 cm).

The MBC of finishes was assessed on cylindrical specimens (h = 3 cm, d = 10 cm), by means of a simplified version of the NORDTEST methods [44].

The thermal properties were measured in terms of thermal conductivity, according to UNI EN 12664:2002, at T =  $20 \pm 2$  °C and RH =  $50 \pm 5\%$ . Equation (1) was used to evaluate the data:

228 
$$\lambda = \frac{Jd}{(T_2 - T_1)^2}$$
 (1)

where *J* is the heat flux (W/m<sup>2</sup>), *d* is the distance between the thermocouples (m), and  $T_1$  and  $T_2$  are the temperatures at the two different sides of the sample (K). For each mix, three specimens were tested and the average values are reported.

### 233 2.1.3 Inhibition of mould growth test

234 Evaluation of the mould growth inhibition effect was performed according to UNI EN 15457:2014 235 using Aspergillus niger (DMS 126) which generally grows in humid indoor environments such as 236 bathrooms, and which can cause health problems including allergies and asthma, especially during 237 prolonged exposure. The methodology for quantification of the inoculum was performed as described in previous works [45][46]. The specimens ( $5 \times 5 \times 0.5$  cm prisms) were casted and cured for 28 238 239 days, as described in Section 2.2.1. After the curing period, the pH of the mortars was tested, in order to ensure that the initial basicity of the mortar was lost, as alkalinity generally inhibits mould growth 240 241 [46]. To guarantee sterile conditions for the inoculum, the specimens were sterilized in an oven at T 242 = 150 °C for 2 hours. Then, the inoculum was provided on an agar substrate under a laminar flow 243 hood and the specimens were inserted into petri boxes, in order to maintain sterility outside the hood, 244 and incubated at 24°C.

245 Mould growth was monitored for 28 days under two different conditions: without any irradiance (dark 246 condition) and under visible irradiation for at least 8 hours of exposure per day. As blank tests, three 247 samples of Aspergillus grown on sterile filter paper were provided for each condition of inoculum, to 248 verify the vitality of Aspergillus. Quantification of the inoculum was conducted once a week for 28 days, and the average results of three inocula are reported. To quantify the percentage of colonized 249 250 area on the surface of the specimens, photos of the specimens were taken and elaborated by means of 251 the ImageJ and GIMP2 software. The results obtained from the inoculum after 28 days are reported 252 and discussed below.

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### 2.1.4 **De-polluting properties**

The photocatalytic activity of mortars was assessed according to UNI 11247:2009, which measures 255 the activity of inorganic photocatalytic materials in terms of the degradation rate of nitrogen oxides 256 257  $(NO_x)$  in plug flow condition. The photocatalytic activity (PC) was tested without irradiation (dark) and under both UVA and VIS irradiation and was expressed in terms of the ratio of mineralized NO 258 over total NO<sub>x</sub> flux directed in a plug flow reactor. The NO<sub>x</sub> flux was provided by a tank at 500 ppb 259  $\pm$  5 NO (SAPIO S.r.l., Monza, Italy), kept constant by mixing with air at T = 25 °C and RH 50  $\pm$  10% 260 261 using a dilution system (Calibrator 8188, Rancon Instruments S.p.A., Milan, Italy). In the outlet flux, 262 the concentrations of NO and  $NO_2$  were continuously monitored using a chemiluminescence  $NO_x$ analyser (nitrogen oxide analyzer model 8841; Monitor Labs, Englewood, CO, United States). 263 264 According to the standard, the borosilicate glass photoreactor had a volume of 3 litres and the specimen (cylinder with d = 8 cm and h = 0.8 cm) was placed in the centre. Then, 20 W/m<sup>2</sup> UVA 265 266 irradiance, measured by a photo-radiometer (Delta Ohm, HD2102.2, Padua, Italy, equipped with 267 LP471 probe), was provided using a UV 300 W metal halogen lamp, placed 25 cm from the surface of the specimen outside the reactor. VIS light was provided by placing an UV screen protector
between the lamp and the reactor. Irradiation of the sample was guaranteed until stable conditions
were reached (usually 30 min).

271 The de-polluting property regarding VOCs adsorption was evaluated by means of batch tests, where the VOCs concentration decay was measured over time using a Gas Chromatographer (GC-Carlo 272 273 Erba Gas 8000 Top). Tests were conducted in dark conditions, and under UV and VIS radiation, in order to evaluate the possible photocatalytic activity. Under UVA radiation, an irradiance of 10 274 W/m<sup>2</sup>—as measured by a photo-radiometer (Delta Ohm, HD2102.2, Padua, Italy, equipped with 275 LP471 probe)—was provided by a 9 W UV-A lamp (Philips); whereas, for VIS radiation, a 9 W lamp 276 was used (Osram). The de-pollution test was performed on cylindrical specimens (h = 0.8 cm, d = 8277 cm) following the procedure described in [46], for a total exposed surface of 50 cm<sup>2</sup>. Methyl ethyl 278 279 ketone (MEK) was used as a model pollutant, injected into a borosilicate glass box where the 280 specimen was inserted before. MEK concentration was assessed each 10 minutes for 120 minutes. A 281 pseudo-second-order (PSO) [47] adsorption kinetic model was used to describe the adsorption kinetics of MEK into the specimen, as described by the following equation: 282

283 
$$q_t = q_e (1 - e^{-\kappa_1 t})$$
 (2)

where  $q_t$  and  $q_e$  (mg/cm<sup>2</sup>) are the adsorptive removal capacity at equilibrium and a specific time (t), respectively, normalized on the sample surface; and  $k_l$  is the adsorption rate constant. The difference between the initial MEK concentration without specimen (C0, equal to 2402 mg/m<sup>3</sup>) and the concentration monitored over the time (Ci) was used to determine  $q_t$ . The test was repeated at least three times, and was directly fitted to Equation 2.

# 289

### 290 **3 Results and Discussion**

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### 292 3.1 Fresh, mechanical, and micro-structural properties

All the prepared mortars were classified as stiff, according to UNI EN 1015-6:2007.

294 The compressive strength, density, and accessible porosity results, evaluated after 28 days of curing, 295 are reported in Figure 3. Both commercial and new mortars had lower compressive strength than the reference mortar. In the case of COM1 and COM2, the compressive strength values were 5% and 296 297 55% lower than reference mortars, respectively (Figure 3a). MIX1 (with and without  $TiO_2$ ) showed a decrease in compressive strength of about 25% (when compared to reference mortar) or 20% (when 298 compared to COM2). The reduction of compressive strength in the proposed mix was due to the 299 300 addition of the very porous adsorbent aggregate [44], even if the addition of biomass ashes facilitated 301 partial recovery of the mechanical resistance loss, thanks to their pozzolanic activity [48][49][50,51].

302 The presence of alite in the ashes, as detected by XRD (Figure 1), would favour the formation of 303 calcium silicate hydrates in the binding phase, thus enhancing the mechanical performance. As 304 expected, the mechanical behaviour was not significantly changed with the addition of  $TiO_2$ .

According to UNI EN 998-1:2010, the maximum value of density for indoor mortar to be classified 305 as lightweight is 1300 kg/m<sup>3</sup>. As the highly porous adsorbent aggregate and biomass ashes have lower 306 307 density than conventional sand, the obtained mortars were lighter than the reference and both 308 commercial mortars. As reported in Figure 3b, MIX1 and COM1, classified as lightweight mortars, 309 and the new mortar had lower density than the commercial products and the reference mortars, by 310 about 15% and 35%, respectively, regardless of TiO<sub>2</sub> presence.

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Accessible open porosity was evaluated by MIP (Figure 3c), and both the relative and cumulative 315 pore volume distribution curves are shown in Figure 4. The REF mortar had the lowest total porosity. 316 317 COM1 and COM2 had 10% and 15% higher total porosity than REF, respectively, whereas the new 318 mortar showed the highest value of total porosity (again, regardless of TiO<sub>2</sub> addition). Furthermore, 319 the pore size distribution was not significantly affected by the type or the presence of  $TiO_2$ , as shown 320 in Figure 4, in which the pore distribution curves of reference, commercial, and multi-functional 321 mortars are compared. REF and commercial mortars present unimodal distributions, whereas the new 322 mortar shows a bi-modal distribution. The main factor usually affecting the porosity of the matrix is the type of binder. In hydraulic lime mortar, the pore diameter threshold—which is the first inflection 323 324 point of the curve—generally ranges from 1 to 3  $\mu$ m; that is, hydraulic lime introduces a higher medium-pore size porosity into the matrix than cement [20]. In this case, the REF mortar had a modal 325 326 pore diameter of 0.93 µm and a threshold pore diameter of 1.54 µm; while COM1 and COM2 had 327 modal diameters of 1.60  $\mu$ m and 0.90  $\mu$ m and threshold pore diameters of 3.27  $\mu$ m and 1.20  $\mu$ m, respectively. Regardless of the type of TiO<sub>2</sub>, the unconventional mortars all had the first peak at 1.2 328 329  $\mu$ m, except for M1 TiO<sub>2</sub>A, which was at 0.93  $\mu$ m. The second peak, at a lower pore diameter, was at 330  $0.04 \,\mu\text{m}$  for M1 and M1 TiO<sub>2</sub>A, and  $0.04 \,\mu\text{m}$  and  $0.021 \,\mu\text{m}$  for the mortars with titanium dioxides.



333 Figure 4 Relative (a) and cumulative (b) pore volume distribution of different mortars

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335 3.2 Thermo-hygrometric properties

As the influence of  $TiO_2$  on hygro-thermal behaviour of mortars at low percentages has been reported to be negligible [18], only mortars without  $TiO_2$  were tested for thermo-hygrometric properties. The obtained results, with relative standard deviations, are reported in Table 2.

339

Mix μ s.d.\* MBV s.d.\* λ s.d.\* (g/m<sup>2</sup>\*RH%) (-) (-) (-) (W/mK) (-) REF 19.8 1.2 0.15 0.01 0.124 0.016 COM1 14.0 0.2 0.30 0.05 0.096 0.001 15.8 0.9 0.25 0.01 0.101 0.004 COM2 MIX1 11.4 0.2 0.61 0.18 0.083 0.001

Table 2. Thermo-hygrometric properties of mortars: water vapor resistance factor, moisture buffering value, and
 thermal conductivity.

s.d. standard

deviation

Low  $\mu$  factor values indicate high permeability. REF mortar showed the highest  $\mu$  values and, therefore, lower water vapor permeability. Commercial mortars had a  $\mu$  value lower than that of the REF mortar: of about 20% for COM1 and 30% for COM2. M1 had the lowest value of  $\mu$ —40% lower than REF and about 20 and 25% lower than COM1 and COM2 mortar—and, therefore, the highest water vapor permeability. Taking into account that the same hydraulic binder was used for all the tested mortars, the differences were mainly due to the different porosities of aggregates. In particular,

the results follow the well-known principle in which a higher pore threshold radius (Figure 4) leads to higher transpirability [34] [52]. The total amount of porosity is also very influential on permeability: in the case of M1, the higher volume of pores, in terms of total porosity (Figure 3c), led to a lower hygroscopic resistance factor, as reported in Figure 5a.

The interaction between mortars and the indoor RH was also studied, by measuring the change in 353 354 moisture content of specimens ( $\Delta m$ ) exposed at different RHs. Table 2 shows  $\Delta m$  normalized on the surface of specimens, representing the MBV values. The REF mortar had the lowest ability to 355 356 exchange water vapor and, consequently, the lowest MBV. Both commercial mortars had a higher capacity to exchange water vapor than the REF mortar, about 100% and 75% for COM1 and COM2, 357 358 respectively. The unconventional mortar had up to three times higher MBV, compared to the 359 reference mortar. Furthermore, in this case, the result was strictly related to the porosity of the mortar, as a higher total volume of pores implies a higher available mortar surface with large pore volume, 360 361 providing sufficient space for the adsorbate (in this case, water) to be trapped in [53]. The obtained 362 results confirmed that the higher the accessible porosity, the higher the MBV value (Figure 5b); unconventional mortars had the highest total porosity (Figure 3c) and, therefore, the highest MBV 363 364 values (Table 2). Moreover, higher permeability usually indicates higher water vapor-exchanging 365 ability [54] [55].

Concerning the ability of the materials to contribute to wall system insulation [53], the REF mortar had the highest thermal conductivity, while commercial mortars both had about 20% lower thermal conductivity values than the reference mortar. MIX1 had the lowest value: 35% lower than the REF and about 20% lower than COM 1 and COM 2. Furthermore, in the case of thermal conductivity, the higher the porosity, the lower the thermal conductivity; with a good linear correlation, as shown in Figure 5c.







### **377 3.3 Inhibition of mould growth test**

- REF mortar had the highest resistance to the biological attack under both test conditions [46], as shown in Figure 6, probably due to the absence of any nutrient on the mortar surface (e.g., organic carbon or potassium). P-25 was chosen as benchmark  $TiO_2$  (TiO2A) and KRONOClean 7404 (TiO2J) was tested, due to the high presence of organic carbon [43], which creates favourable environmental conditions, in terms of mould growth. Commercial mortars were not tested, as their datasheets have already reported their mould growth inhibition ability.
- All M1 mortars (without and with TiO<sub>2</sub>) were unable to completely inhibit the growth of microorganisms on their surfaces. The main reasons for this could be the presence of biomass ash, which favours mould growth, as well as the roughness of the surface [56]. The high accessible porosity of the M1, as reported in Figure 3c, also assists in the capillary uptake of the agar nutrient in the mortar, providing a better substrate for mould growth, when compared to REF.
- The presence of inactivated  $TiO_2$  (under dark condition) increased the susceptibility of mortars to biological attack, with an enhancement in the colonized area up to 6 and 10 times for  $TiO_2A$  and  $TiO_2J$ , respectively, when compared to M1 without  $TiO_2$ . This was probably due to the hydrophilicity of  $TiO_2$ : the test was conducted in dark, but the inoculum was provided under VIS radiation, which could be enough to activate the photo-induced hydrophilicity [57][58][44]. In the case of  $TiO_2J$ , the higher biological attack could be due to the higher content of the organic carbon [43], which provides additional nutrients for the growth of mould.
- 396 Therefore, surface treatment of the materials should be considered necessary to prevent biological 397 attack [59] when the control of environmental factors (e.g., irradiation, composition of the material, 398 and humidity) cannot be guaranteed [60]. In this case, the addition of boron salt (SB) relevantly decreased mould growth under dark conditions. In the case of M1 TiO<sub>2</sub>A, the colonized area 399 400 decreased from 33 to 7% and, in the case of M1 TiO<sub>2</sub>J, it decreased from 54 to 15%. When specimens were prepared with both  $TiO_2$  and boron salt and tested under VIS conditions, the colonized area 401 402 became negligible, showing that the well-known antibacterial ability of activated TiO<sub>2</sub> under UV assists the biocidal action of the boron salts [61][62] [63]. 403



#### 405 Figure 6. Percentage of colonized area.

406

### 407 3.4 **De-polluting properties**

As expected, NO decomposed only in the presence of TiO<sub>2</sub>, as shown by the NOx abatement factor
and the NO<sub>2</sub>/NO removal results reported in Figure 7. The values lower than 5% detected in REF,
COM1, and MIX 1 under UV radiation were considered negligible.

The removal of up to 6% of NO in the reference sample was ascribed to photolysis of the pollutant,and to the sorption and conversion of NO into nitrous acid [64][65].

413 In the case of commercial mortars, the presence of  $TiO_2$  was declared for COM2, and 18% NOx 414 removal was assessed under UV radiation. The abatement was detected also under VIS radiation, but 415 only at a negligible percentage (8%).

Both types of TiO<sub>2</sub> enhanced the photocatalytic degradation of M1 mortar: up to 30% under UV irradiation. The photocatalytic efficiency was also demonstrated under VIS radiation, but with lower values (of about 18% and 7% for TiO<sub>2</sub>A and TiO<sub>2</sub>K, respectively).

419 The ratio between  $NO_2$  and NO was also considered: the higher the ratio, the higher the production of unwanted NO<sub>2</sub> during the reaction. Figure 7b shows that the production of unwanted NO<sub>2</sub> was 420 negligible for all the mortars. For M1 mortars, with adsorbent materials as unconventional aggregate, 421 422 this can be ascribed to the synergistic effect between adsorption and photocatalysis, as has already 423 been detailed in previous studies [66][67]. The enhanced reactivity with Ti-OH by NO<sub>2</sub> disproportion is due to the higher amount of water adsorbed in the proximity of  $TiO_2$  [68] and the consequent 424 reaction of  $NO_x$  with alkaline products [53]. Additionally, the pore structure of MIX 1 with the 425 addition of  $TiO_2$  enhanced the photocatalytic effect [69]. These pores act as a booster for 426 427 photocatalytic activity, as has been stated in [70], in cement mortars with  $TiO_2$ . These authors 428 concluded that the formation of capillary pores in the range of  $0.01-0.05 \ \mu m$  was critical for the

enhancement of the photocatalytic activity (NO abatement), which has also been demonstrated in
[71], where higher NO degradation was detected in specimens with outstanding increase in capillary
pores.

432



Figure 7. Percentage of NO<sub>x</sub> removed (a), and ratio between NO<sub>2</sub> produced and NO removed (b) during plug-flow
test.

436

437 During the VOCs de-polluting capacity test, the specimen was sealed inside a box, into which a
438 known quantity of the model pollutant MEK was injected. Then, the MEK residual concentration was
439 monitored for 120 mins. The MEK concentrations at the end of the tests are shown in Figure 8.

440 The commercial products COM1 and COM2 showed 20% higher and 20% lower de-polluting 441 properties than REF mortar, respectively. M1 mortar showed a de-polluting capacity about 40% higher than the REF mortar and both COM mortars. After 2 hours of the test, the residual MEK in 442 the box was only 20% when a M1 finish was used [38]. The addition, both types of TiO<sub>2</sub> did not 443 444 significantly influence the de-polluting capacity under dark conditions. Under VIS radiation, the 445 effect of TiO<sub>2</sub>K was not observed; whereas, under UV radiation, the percentage of residual MEK 446 inside the box ranged from 11–22%. TiO<sub>2</sub>A provided negligible (about 4%) enhancements in de-447 polluting capacity under both visible and UV radiation.



449 Figure 8. Residual concentration inside the box after 120 min of monitoring.



Figure 9. Adsorption rate constant  $k_1$  a) and the adsorption rate constant and specific MEK removal capacity at equilibrium  $q_e$  b).

454 The unconventional aggregates added in M1 cause the adsorption process to be predominant over the 455 PCO, even under visible and UV radiation, as has been shown in [72]. In fact, when the monitored 456 residual concentration data were represented in terms of adsorption kinetics, in terms of constant 457 adsorption rate, the results could be classified in two groups with similar values (Figure 9a): one 458 related to the M1 mortars with adsorbent unconventional aggregate, and the other to the REF and 459 COM mortars. High errors bars occurred during the elaboration of the data for REF, COM1, and 460 COM2, as well as when the specific MEK removal capacity at equilibrium  $q_e$  was calculated (Figure 461 9b), highlighting that no significant adsorption mechanism was present in those specimens [73]. On 462 the other hand, MIX1, MIX1 TiO<sub>2</sub>A, and MIX1 TiO<sub>2</sub>K had higher MEK removal capacities (approx. 463 30%) than REF and COM mortars, and the test conditions (UV, VIS, or dark) did not affect the 464 adsorbent properties.

The adsorption efficiency of the finish was extremely enhanced when the adsorbent aggregate was used and, consequently, a high specific surface interacted with the environment. Moreover, with a higher pore size reduction, a higher de-polluting capacity was observed [71], with good linear correlation, as reported in Figure 10.

4

450



# 471 Figure 10. De-polluting properties, in terms of specific MEK removal capacity at equilibrium q<sub>e</sub> under dark 472 conditions.

473

The de-polluting property was higher in M1 mortars than REF and COM mortars, due to the combination of the adsorbent process and PCO. The photocatalyst activity can be enhanced with a high presence of micro–nano-pores [74]: the larger the volume of pores with diameter higher than 80 nm, the higher the PCO efficiency [19]. Therefore, the high porosity of M1 mortars allowed for the absorption of pollutants into their internal structure [22]. Moreover, during the test, STD and COM mortars were close to reaching saturation conditions, while the new mortars continued to adsorb and decompose the loaded MEK, as has been detailed in [46].

481

### 482 4 Conclusions

An unconventional mortar for use as an indoor finish was designed, characterized, and compared to a traditional reference mortar and two commercial pre-mixes for the same application. In the new finish mortar, an unconventional aggregate—based on an adsorbent material which is generally not used in the building sector, but in air/water purification processes—was adopted, instead of traditional sand. The patented new finish has potentially high sustainability, thanks to the use of hydraulic lime and biomass wastes, instead of cement and conventional aggregates.

The innovative mix was able to fulfil all traditional requirements for an indoor finish. The new mortar can be classified as a lightweight mortar ( $\rho < 1300 \text{ kg/m}^3$ ), with density 30% lower than the REF mortar and around 10% lower than COM mortars. This low density was due to the use of unconventional lightweight adsorbent aggregates, which also led to 20%, 10%, and 5% higher porosity, when compared to the REF, COM1, and COM2 mortars, respectively.

494 Regardless of the high porosity and low density, only a 5% decrease in compressive strength, with respect to the REF mortar, was detected; as such, the proposed mortar can be considered acceptable 495 496 for the proposed application. Compared to commercial products, the compressive strength was 5% 497 lower or around double, according to the type of mortar. The new formulation had thermal conductivity 35% lower than that of the REF mortar and about 20% lower than those of commercial 498 499 mortars; as well as the lowest water vapor resistance factor (about 40% lower than REF and about 20 500 and 25% lower than the COM mortars). This ensures the highest transpirability, preventing the 501 stagnation of water vapour.

502 In terms of moisture buffering capacity, the best performance was also detected in the new mortar, 503 with three times higher MBV than the reference mortar and the double and 75% higher, when 504 compared to the respective COM mortars.

505	Conc	cerning the de-polluting ability, the patented finish reached the NOx abatement of 35%, 75%						
506	highe	er than COM mortar with the addition of $\mathrm{TiO}_2$ activated under UV radiation, whereas the						
507	adsorbent properties were enhanced by 50% with respect to REF and both commercial mortars.							
508	Finally, the behaviour of the innovative mortar, in terms of biological attack, was improved when a							
509	bioci	de was added into the mix.						
510								
511	Pate	nt						
512	Italia	an Patent 102017000033750						
513								
514	Ackı	nowledgements						
515	The authors wish to thank EVONIK and KRONOS, which kindly supplied titanium dioxides. The							
516	co-grant for research contract was provided thanks to the program 'FSE-REACT-EU, PON Ricerca							
517	e Inn	ovazione 2014-2020 DM 1062/2021'.						
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