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Research article

The first sustainable material designed for air particulate matter capture: An introduction to Azure Chemistry

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A B S T R A C T

This work presents a new porous material (SUNSPACE) designed for air particulate matter (PM) capture. It was developed in answer to the European Commission request of an innovative, affordable, and sustainable solution, based on design-driven material, to reduce the concentration of air particulate matter in urban areas. SUNSPACE material was developed from by-products and low-cost materials, such as silica fume and sodium alginate. Its capability to catch ultrafine PM was evaluated by different ad-hoc tests, considering diesel exhaust fumes and incense smoke PM.

Despite the fact that procedures and materials can be designed for remediation, the high impact on the environment, for example in terms of natural resources consumption and emissions, are not usually considered. Instead, we believe that the technologies must be always evaluated in terms of material embodied energy (EE) and carbon footprint (CF). We define our approach to solve environment problems by a sustainable methodology "Azure Chemistry". For the SUNSPACE synthesis, the multi-criteria decision analysis was performed to select the best sustainable solution. The emissions and the energies involved in the synthesis of SUNSPACE material were evaluated with the Azure Chemistry approach, showing that this could be the best available technology to face the problem of capturing the PM in urban area.

1. Introduction

The European Environment Agency ("Air quality in Europe — 2016 report — European Environment Agency," 2016) estimated that in the 2012–2014 period 50–63% and 85–91% of the urban population in Europe were exposed to levels of PM_{10} and $PM_{2.5}$ (respectively) which exceeded the recommended World Health Organization (WHO) annual limits (PM_{10}: 20 $\mu g/m^3$ and $PM_{2.5}$: 10 $\mu g/m^3$). In the same report, it was reported that 467,000 premature deaths in Europe could be attributed to $PM_{2.5}$ in 2013. PM pollution due to fine particles (2.5 μg or less) is particularly harmful since fine PM can penetrate human bronchi and lungs owing to the small particle size (Harrison and Yin, 2000).

The number of publications per year devoted to "air pollution" problems and solutions is growing from 1960 (Fig. 1) and the trend is still growing, while the number of publications about "filter" and "air" is quite constant in the last ten years (the data were obtained by SCOPUS). This may suggest that the technologies for PM filtration is set and few researches are devoted to find new approaches to face the problem. Indeed, several techniques are already assessed for filtration (Liu et al., 2017a).

Air pollution control systems also concern anthropogenic emissions reduction (such as CO_2, SO_2, NO_x) such as gas capture (Zhang et al., 2018; Yan et al., 2018).

Possible, emerging nano-fibers materials, to reduce PM, may be produced in the future at accessible costs (Liu et al., 2015, 2017c; Singh et al., 2017). Since it is urgent to develop low-cost technologies to promote the air quality in urban areas, the European Commission encourages the research in the field of new materials, devoted to reducing the concentration of particulate matter in urban areas. The aim was to highlight innovative, affordable, and sustainable solutions, based on design-driven materials.

The research presented in this paper started in 2015 by using the multi-criteria decision analysis (Jahan et al., 2016) to support the most suitable synthesis strategy able to produce a new material to be used for the PM reduction in the urban area.

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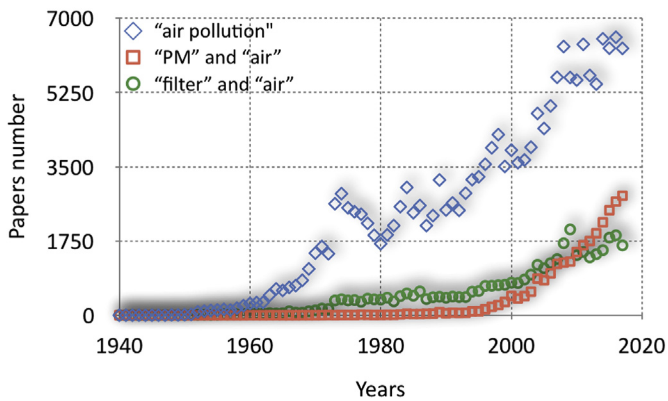

Fig. 1. Number of papers in SCOPUS containing to the following key-words: 1) "air pollution", 2) "PM" and "air", 3) "filter" and "air".

The new proposed material is designed to reduce the PM present in the urban air by using green and sustainable materials, process, and technologies (as formulated in the Green chemistry). We called this chemistry approach that want to link Green Chemistry and Remediation "Azure Chemistry": the goal is to restore or reconstruct the ecosystems by sustainable solutions in terms of energy, materials and emissions. Azure Chemistry concerns, for example, carbon dioxide sequestration, PM pollution reduction, waste minimisation, and energy neutrality. It requires low-energy paths, manufacturing and technologies reducing the use of non-renewable resources, and in which wastes and by-products are employed. Overall, Azure Chemistry approach must minimize the global impact of the remediation processes. Indeed, literature reports several examples of declared low-cost materials, synthesised for instance starting from wastes and by-products (Nayak and Pal, 2017; Silva et al., 2017; Wong et al., 2016). However, the declared sustainability of these new proposed materials is rarely demonstrated and quantified.

The most promising new materials for air filters, e.g. polyethylene (PE), polypropylene (PP), polyamide (PA), and polystyrene (PS), are petroleum-based materials (Khalid et al., 2017; Liu et al., 2015). This makes the environmental costs in their production high. Indeed, the emissions associated with the production of these polymer materials can range from 30 to 150 kg-CO_2eq.

Even when biomaterials are used to make filters (as for example soy protein isolate and bacterial cellulose) (Liu et al., 2017b), the synthesis requires toxic chemicals, thermal treatments and refrigeration, that makes filters production not suitable to solve the PM problem of the urban areas. Moreover, the filter regeneration is usually not considered, making the material substitution the most common practice to replace exhaust filters, triggering a secondary environment pollution.

Several materials are reported in the literature and databases and they can be grouped in two categories (Jahan et al., 2016): traditional and, more recently developed, engineered materials (see Fig. 2). Engineered materials have been developed to guarantee functional and structural characteristics that cannot be provided by a single component alone. Two main distinct strategies design engineering materials:

1. conventional design strategy that aims to obtain materials with a uniform and homogeneous structure.
2. newly developed design strategy in which the goal is to disperse the constituents by a controlled and selectively heterogeneous way. By this approach, materials for niche applications have been obtained (Jahan et al., 2016).

By the second approach, it is possible to create porous materials that combine desirable properties of organic polymers (flexibility and elasticity) with those of inorganic solids (rigidity and chemical resistance) (Zou et al., 2008). Porous materials are used for trapping and encapsulation functional biomolecules, enzymes, drugs, and nutrients (Kim et al., 2015). Various methods have been used to obtain pore sizes control, as specific precursors for nano-casting or variable size surfactant templates (Suib, 2017).

The new material for air particulate trapping must have a wide range of pores sizes, from few nanometers (nm) to some microns (μm) to allow the capture of all sizes of PM. In particular, pores in the nanometers range are necessary for the trapping of the most dangerous smallest PM.

Indeed, these particles contain toxic substances such as heavy metals, PAHs, polychlorinated dibenzo-p-dioxins and dibenzofurans and polychlorinated biphenyls, making them more hazardous and carcinogenic (Dzierżanowski et al., 2011; Silverman et al., 2012).

For this reason, the newly developed material performances have been assessed in trapping of diesel exhaust and incense smoke PM. Indeed, diesel exhaust is enriched in ultrafine particles (<0.1 μm in diameter) (Williams, 2011) and incense burning emits particles with the mean particle diameter of about 100 nm (Ji et al., 2010; Tirler and Settimo, 2015).

2. Material design and synthesis strategies

To meet the Azure Chemistry requirements and reduce the global environmental impact of the new proposed material, we followed these principles:

- use low-energy materials by selecting nontoxic low-cost materials and by-products (material requiring low extraction energies);
  -use processes that need low energy. The selection of the material must be tightly coupled with the choice of a low-cost production process;
- make long-lasting and excellent products that can be easily regenerated;
- design products with a minimum amount of material. To be functional the new material must be in contact with the air and should be applied as a coating to whatever surface;
- quantify the sustainability of the new proposed material, in terms of materials, energy, and emissions.

A template process was developed to allow the formation of connected pores, which are mandatory to allow the PM-capturing and entrapment by a new synthesis of mesoporous materials involving the cross-linking of functional groups. No standard templates have been used and material decomposition process (made at low temperatures) generating gas was applied.

By this approach, a new porous material named SUNSPACE (SUStainable materials Synthesized from by-products and Alginates for Clean air and better Environment) has been obtained, based on sodium alginate, calcium iodate (or calcium chloride), and silica fume. Sodium bicarbonate is employed to generate CO_2 by thermal decomposition at low temperatures (60°–70 °C) and to produce the pores. Sodium alginate is a natural and low-cost polysaccharide, that can be extracted from various species of brown seaweeds (Ikeda et al., 2000). It has extensively been employed in different applications in view of its fascinating properties, such as gelling, film forming, emulsion capabilities, and low price (Augst et al., 2006). Its colloidal property makes possible the formation of an insoluble gel through gelation with calcium cations (Johnson et al., 1997; Yang et al., 2016). Silica fume, a by-product derived from

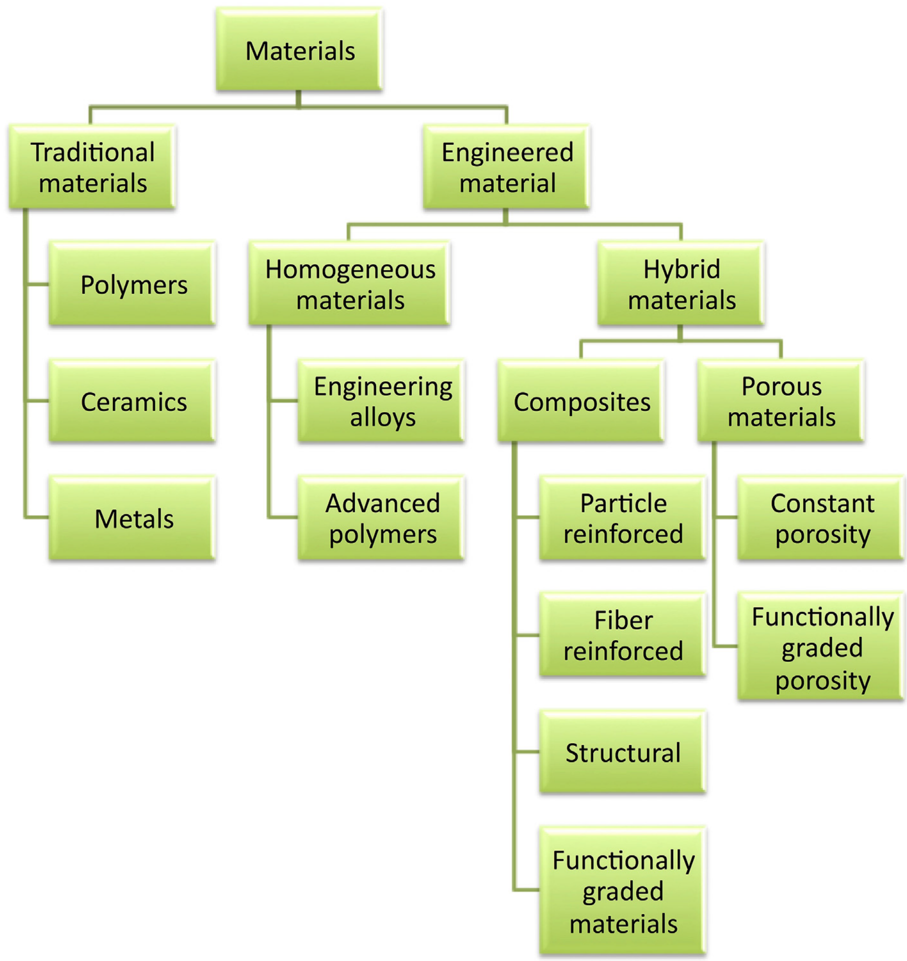

Fig. 2. Materials categories (Jahan et al., 2016).

ferrosilicon or silicon metal alloy processing (Rodella et al., 2017) was used in the preparation of the new material. To react with sodium alginate, calcium iodate is generally used. However, calcium chloride was also successfully tested.

To propose SUNSPACE as a coating (as a plaster on a wall or roof tiles) to reduce the PM in urban area, the sustainability of the material was assessed. Several factors must be evaluated: the material availability, the manufacturing process, the energy consumption, the emissions, and the recycling options (Elza Bontempi, 2017a).

In this context, Life Cycle Analysis (LCA) is one of the most used methods to evaluate the environmental pressures of the new product. However, because of the large variety of materials and processes, the complexity of the emissions and boundary conditions, LCA may require handling a large number of data and many calculations. A simple approach, accounting for example energies and emissions involved in the synthesis of a material (Elza Bontempi, 2017a), was recently proposed and can be applied based on the "embodied energy" (EE), defined as the energy required to produce 1 kg of the material, starting from ores and feedstock (Ashby, 2012). The material synthesis also involves the production of greenhouse gases (kg-CO_2eq) released into the atmosphere (Ashby, 2012), defined as the "CO_2 footprint" (CF). In the recently proposed approach, one of us (Elza Bontempi, 2017b) introduced a new simplified method to quantify the sustainability of the material in respect, for example, to a natural resource, only based on the comparison of embodied energy (EE) and carbon footprint (CF). It was also shown (E. Bontempi, 2017c) that the world materials production is correlated to the materials embodied energy (and CO_2 footprint), and a relationship of the material demand and the energies and emissions involved in its production was demonstrated.

3. Experimental

3.1. Synthesis

Sodium alginate (CAS number: 9005-38-3, viscosity c = 1% water @ 25 °C 5.0–40.0 cps), calcium iodate ($Ca(IO_3)_2$, CAS number: 7789-80-2), sodium bicarbonate ($NaHCO_3$, CAS number 14455–8, ≥99.8% w/w), and calcium chloride ($CaCl_2$, CAS number 10043-52-4) were purchased from Sigma Aldrich. Conventional (grey) and white silica fume (industrial by-products derived from ferrosilicon and silicon metal alloy processing) were kindly provided by Metalleghe SPA, Brescia, Italy. Double deionised water (ddH_2O, Millipore DirectQ-5 purification system) was also used.

The synthesis was performed at room temperature (RT) by a process adapted from (Brandes et al., 2014): 0.6 g of gel-former (sodium alginate) were dissolved in 25 ml of solvent (ddH_2O) and mixed till complete dissolution. Then 1 g of cross-linker (calcium iodate or calcium chloride) was rapidly added to the sodium alginate, the solution under continuous stirring. In these conditions, a gel was formed. 17.88 g of silica fume (corresponding to 72% w/w of solid content) was added, and finally, 5 g of sodium bicarbonate was thoroughly mixed to the slurry.

The obtained slurry was then warmed on a heating plate at 60–70 °C for 1 h. The presence of Ca^{2+} ions (due to the cross-linker) and the annealing allowed the formation and consolidation of the hybrid material (Zanoletti et al., 2018). Also, thermal treatment promoted the sodium bicarbonate decomposition and the consequent release of CO_2 for pore formation.

Porous disks with a diameter of 2.3 cm and a thickness of 5 mm were obtained. They were rinsed with ddH2O and dried at ambient conditions and then used in the tests.

3.2. Characterization

X-Ray Diffraction (XRD) analysis was realised by means an Empyrean diffractometer (PANalytical, Netherlands). The XRD was performed in using Cu Ka ($\lambda = 1.5406$ A°) radiation, operated at 40 kV to 40 mA. Spectra were recorded by a parallel-plate collimated proportional Xe detector with a nickel large-β filter.

The morphology of porous samples was investigated using a field-emission scanning electron microscope (FE-SEM) LEO 1525, Gemini model; Carl Zeiss AG, Oberkochen, Germany. The electron beam was set at 3–5 keV energy and the samples were attached to metallic stub via carbon glue, to reduce charging effect due to the interaction of electron beam with the specimens.

Nitrogen (N_2) physisorption measurements were made by a Micromeritics ASAP 2020 analyser, at the liquid nitrogen temperature. Before the analysis, 500 mg of each sample was degassed at 100 °C overnight.

To verify the capability of SUNSPACE to trap air particulate matter, three ad-hoc tests were realised.

In the first test, SUNSPACE was exposed for 15 min to exhaust fume of a diesel car. It was placed at about 15 cm distance from the emission source. For this experiment, SUNSPACE was synthesised by using white silica fume. This allowed obtaining a white porous material, for better highlighting of the colour change due to the captured of the PM. After the test, SUNSPACE sample was washed by distilled water, to remove the trapped particles, and it was maintained at laboratory conditions (22 °C and 1 atm) for 1 day to dry.

In the second test, a SUNSPACE sample with a surface of about 20 cm^2 was exposed to the incense smoke emissions: incense stick was put on a working bench and the sample was placed on a holder at about 30 cm distance from the particle source.

In the third test, a SUNSPACE sample with a surface area of 4.15 ($\pm$0.01) cm^2 was exposed to the emissions of six incense sticks (burned in succession). The experimental conditions were the same of those reported in the second test.

The energy dispersive X-ray spectroscopy (EDXS) microprobe (Link Pentafet Oxford mod 7060) was used to investigate the elemental composition of SUNSPACE surface before and after the tests.

The colorimetric analysis was performed by a UV-VIS Spectroscopy - Color Measurement Minolta CM 2600 d. Colorimetry is a spectroscopic technique, developed to define a colour. To define a precise colour, a colour space is introduced by one channel for Luminance (lightness) (L) and two-colour channels (a and b). L ranges from 0 (black) to 100 (white). Measurements can be made with the specular component of light (SCI modality), or without a specular component of light (SCE modality).

METTLER TOLEDO XS3DU model balance operating at 12 V and 150 mA was employed to weight the samples. ASTM D6552 – 06 (Standard Practice for Controlling and Characterizing Errors in Weighing Collected Aerosols) was followed.

To reduce errors due to the humidity, SUNSPACE samples were conditioned, at 105 °C, for 6 h before the tests.

The temperature of the room was 22 °C and the relative humidity was 35% (it was constant within $\pm$5% RH, in accord to ASTM D6552 – 06 specifications).

The data for EE and CF of the materials used in the sample preparation were obtained from CES Selector 2016 (Granta, 2016).

4. Results and discussion

4.1. Material characterisation

N_2 physisorption measurements at the liquid nitrogen temperature have been made to investigate the textural properties of SUNSPACE and the pores dimensions less than 200 nm. Large mesopores and macropores are observed (Zanoletti et al., 2018). Notably, pore size distributions calculated from the desorption branch of the isotherms show relative maxima (at 15 and 30 nm) smaller with respect to those calculated from the correspondent adsorption branch (100–150 nm). This suggests that ink-bottle shaped pores are present, as confirmed by the hysteresis loops observed in the N_2 physisorption isotherms (Zanoletti et al., 2018). PM capture can be obtained by adsorption of the particles in the pores. Indeed, ink-bottle shaped pores are the most suitable pores to act as a PM capture for ultra-fine and fine particles, since when the PM enters in the pores are trapped, due to pore shape and dimensions.

The XRD pattern of SUNSPACE sample is shown in Fig. 3: The spectra were collected on the porous materials synthesised with the two different cross-linkers: calcium iodate or calcium chloride. A large halo in both spectra is due to the amorphous silica phase present in the SUNSPACE. Crystalline peaks can be attributed to calcite, cristobalite, and sodium chloride. The two peaks, at about 32.4° and 18.2° may be attributed respectively to a calcium silicate phase and to nahcolite phase.

SEM images of SUNSPACE material at different magnifications are shown in Fig. 4. Samples synthesised with the two different cross-linkers, i.e. calcium iodate (a, b) or calcium chloride (c, d) are shown. In both cases, it appears that the porous material is formed by agglomerated spherical particles. The spherical particles dimensions are different, generally lower than 300 nm (Fig. 4b). EDXS results (not reported) revealed that these samples are mainly composed by oxygen (about 50%) and silicon (about 33–39%). Trace

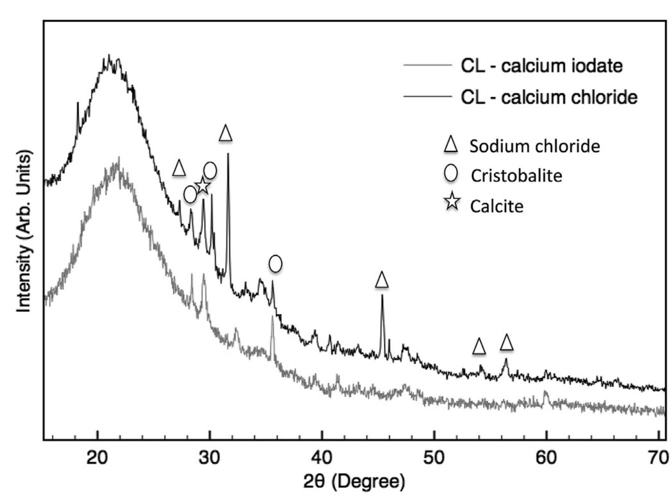

Fig. 3. XRD pattern of the SUNSPACE materials, synthesised with the two different cross-linkers (CL) (calcium iodate or calcium chloride).

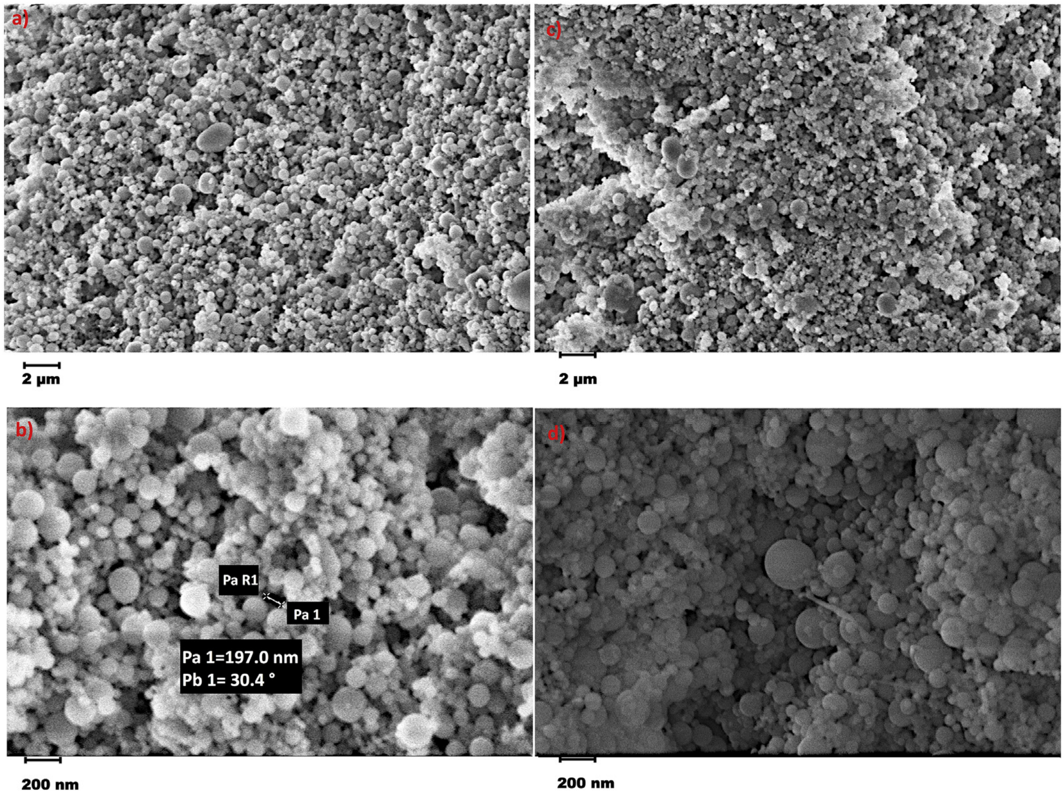

Fig. 4. SEM images of SUNSPACE materials, synthesised with the two different cross-linkers: calcium iodate (a, b) or calcium chloride (c, d). One pore was measured and it resulted large less than 200 nm.

concentration of the following elements: Cl, Na, K, I, and Mg are detected.

4.2. Evaluation of material capability to trap air particulate matter

As described in the experimental section, three ad-hoc tests were realised to evaluate the SUNSPACE capacity in PM trapping. Fig. 5 reports the images of SUNSPACE material before (a) and after 15 min the exposition (b) to exhaust fume of diesel car emissions and Table 1 reports results of EDXS analysis of the two samples. It is shown that the amount of C is much higher after the

Table 1
EDXS analysis made on the two SUNSPACE samples, before and 15 min after the exposition to exhaust fume of a diesel car.

Element (%)	C	O	Na	Si	Ca	Ti	I
Before exposition (a)		39.44	1.44	51.27	1.01	4.98	1.87
After exposition (b)	23.35	39.69	22.97	11.63		0.76	1.6

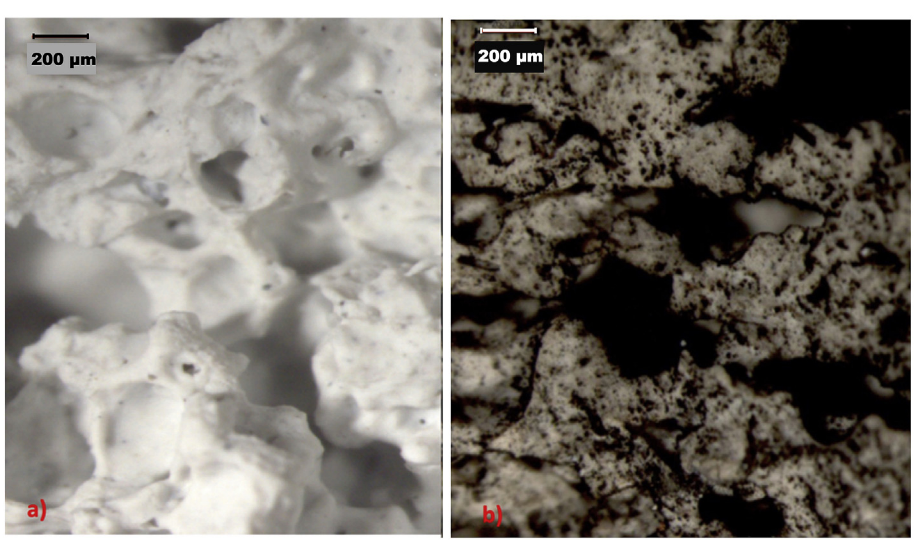

Fig. 5. Optical microscopy images of SUNSPACE material before and 15 min after the exposition to exhaust fume of a diesel car.

Table 2
Results of colorimetric analysis made on SUNSPACE sample exposed to diesel exhaust. Data were also collected on SUNSPACE sample before the exposition and on the exposed sample, after washing with distilled water.

	L	a	b
Porous material before exposition			
SCl	77.54	−0.52	−0.1
SCE	77.47	−0.47	−0.13
Porous material after exposition			
SCl	43.84	1.11	5.22
SCE	43.64	1.16	5.22
Porous material after washing			
SCl	76.57	−0.5	0.31
SCE	76.41	−0.45	0.27

SUNSPACE exposition to diesel fume. EDXS analysis, made on the sample before and after the exposition, revealed that exposed sample contains a significant amount (more than 23% in weight) of carbon in its surface. Indeed, diesel exhaust particles contain a carbon core, which is generally coated by polyaromatic hydrocarbons, quinones and metals (Williams, 2011).

The SUNSPACE exposed sample was washed by distilled water to remove entrapped particles, and it was maintained at laboratory conditions (22 °C and 1 atm) for 1 day to dry. The colorimetric analysis was performed on the porous sample before and after the exposition. The same analysis was performed after washing the sample.

The results of colorimetric analysis made on SUNSPACE materials are reported in Table 2. These data show that SUNSPACE characteristic color becomes darker (L changes from about 77 to about 44) after the test and it was regenerated just by washing (only b coordinate resulted slightly different in respect to the raw material). This test demonstrated that SUNSPACE can trap air PM, and the material regeneration can occur just by rainfall washing.

The second and the third tests were realized to quantify the amount of ultra-fine PM that can be adsorbed by SUNSPACE. On this purpose, PM was generated by burning incense sticks.

The smoke incense PM particles have a broad size distribution from <300 nm to >10 nm, with the majority of particles being <1 μm (Liu et al., 2015).

A porous sample with a surface of 20 cm^2 was used. After the incense stick burning, the mass of porous material increment of 0.028 (±0.002) g corresponds to the amount of air particulate matters produced by a single incense stick (W. Tirler and Settimo, 2015). As a consequence, we concluded that SUNSPACE sample trapped about all the ultra-fine emitted particle.

For the third experiment, a SUNSPACE sample with a surface area of 4.15 (±0.01) cm^2 was used. This test was carried out to evaluate the maximum amount of ultra-fine PM (with the diameter less than 1 μm) that can be trapped by a fix SUNSPACE surface area.

The experiments were realised by burning six incense stick in sequence. After burning each stick, the sample was weighted. In accord with ASTM D6552 − 06 specifications, the tests were disregarded when the humidity changed more than 5%.

The surfaces of the material before and after the test are very different (see Fig. 6). Indeed, the surface of the sample after the test appeared coated by a black layer, even if the macro-pores morphology is similar.

Fig. 7 shows the SEM images of the SUNSPACE sample after the test: the surface is covered by a homogeneous coating layer, as already highlighted in Fig. 6. However, the pores present in the raw sample (Fig. 4) cannot be seen and the morphology of the material is different. The lateral surfaces that were not directly exposed results not entirely covered by a continuous layer and the agglomerated spherical particles can still be seen.

The behaviour of incense smoke PM followed by particles aggregation onto nano-fibres of a filter was already reported in the literature (Liu et al., 2015). However, this is the first time that a similar aggregation level (a continuous layer was formed) of incense smoke is reported. Besides, the change in the SUNSPACE color, after the incense smoke capture, is not clear, and by washing a yellow solution is obtained. As for the previous test, we concluded that SUNSPACE, if used as a plaster in urban area, can be regenerate by rainfall.

Table 3 reports the EDXS analysis performed on the surface covered by the incense smoke: it results that the main detected elements are Ca, O, and N (>than 6%). On the contrary, Si contribution is not detectable. This means that the probe does not reach the porous material. Indeed, these results are in accord with the composition of the incense smoke reported in ref (Liu et al., 2015).

Table 4 reports the mass of SUNSPACE material (initial value 0.843 ± 0.001 g), evaluated after each single incense stick burning. It results that this material adsorbed till to 2407 (±581) μg/cm^2 of air particulate matter particles, with dimensions lower than 1 μm. This value corresponds to about 24 (±6) g/m^2 of air particulate matter. This means that employing SUNSPACE to cover for example roof tiles, for a city like New York, where roofs globally occupy a surface of 92 millions of m^2, about 2200 tons of air particulate matter may be trapped only by the roofs. Also, the material regeneration by rainfall can allow increasing the amount of PM

Fig. 6. Optical images of as synthesised disks of SUNSPACE material and corresponding disks, after the capturing of particles emitted from 6 incense sticks burning. The disks diameter is about 2.3 cm.

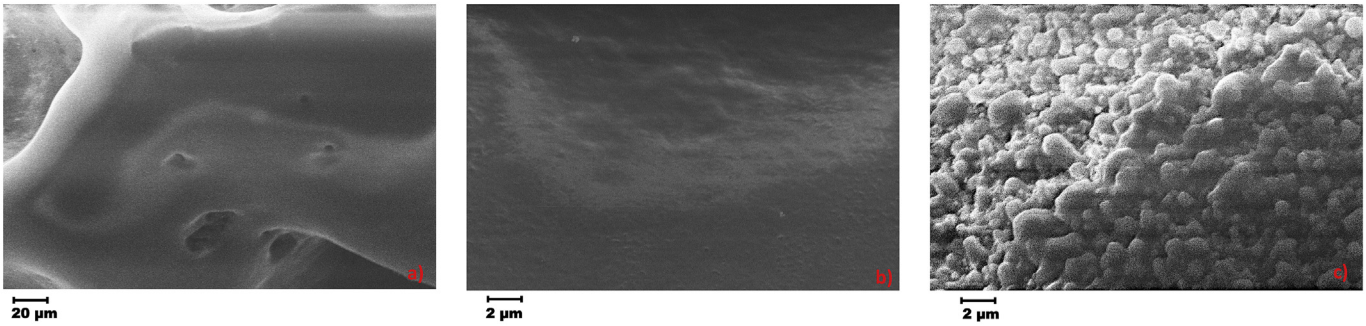

Fig. 7. SEM images of SUNSPACE material after smoke incense capture.

Table 3
EDXS analysis made on the SUNSPACE sample after exposition to incense smoke.

Element	Weight (%)	Atomic (%)
C	53.61	60.22
N	6.34	6.11
O	39.67	33.45
Na	0.38	0.22

Table 4
Mass of the SUNSPACE material (initial value 0.843 ± 0.001 g), evaluated after the exposition at single incense stick burning.

Incense stick burning	Mass (g) + S.D
Blank	0.843 ± 0.001
1^{st} stick	0.845 ± 0.001
2^{nd} stick	0.847 ± 0.001
3^{rd} stick	0.850 ± 0.001
4^{th} stick	0.851 ± 0.001
5^{th} stick	0.852 ± 0.001
6^{th} stick	0.853 ± 0.001

removed from the air.

4.3. Consideration about material sustainability

Fig. 8 shows the EE and CF of SUNSPACE. The data for some natural materials, ceramic, and composites are indicated. The energies and emissions involved in the production of materials used for air filters, e.g. polyethylene (PE), polypropylene (PP), polyamide (PA), polystyrene (PS), and glass are also displayed.

The energies and emissions involved in the SUNSPACE synthesis are less than 3 MJ/kg and 1 kg/kg respectively, making this material the best choice in accord with the Azure Chemistry approach. Indeed, because of the main material used to obtain SUNSPACE is a by-product, the energies involved in its synthesis are comparable to energies required to produce natural materials. In particular, it results that SUNSPACE can be considered similar to cement regarding material sustainability, making this material very promising for building applications.

SUNSPACE is designed to be regenerated, after PM capture, only by rainfall. The discharge water can be collected by the urban wastewater collection system (for example with wastewater deriving from street washing) to be conveyed at the urban wastewater treatment plant, with no additional impacts.

5. Conclusions

This work reports the characteristics of the first material expressly designed for air PM capture in urban area. SUNSPACE material was realised following the basic principles of the green sustainable chemistry, that we defined “Azure Chemistry”: In this approach, the strategies for remediation must evaluate their environmental impact by means of quantitative parameters, as the “embodied energy” (EE), and the “CO_2 footprint” (CF). Therefore, the key of this vision is to evaluate the global environmental impact of the remediation approach, though, for example, materials selection.

By this approach, we demonstrated that SUNSPACE material is effective in ultra-fine PM trapping, and its capability to be naturally

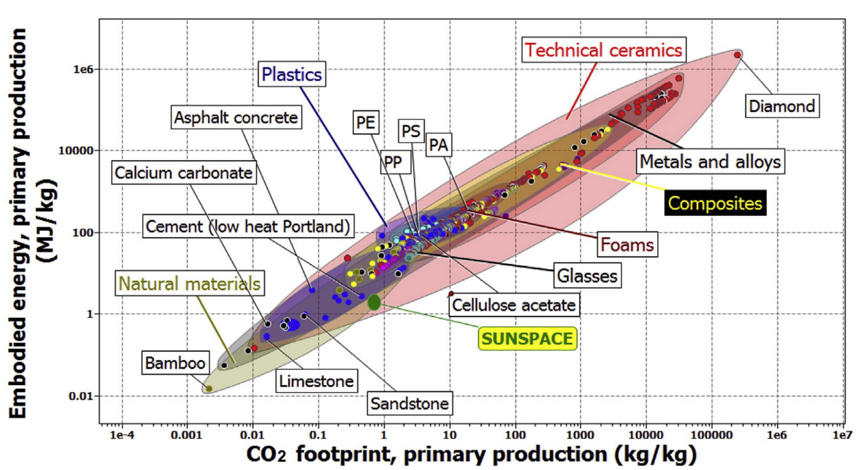

Fig. 8. EE and CF of SUNSPACE. As a comparison also EE and CF of other materials are reported.

regenerated by rainfall makes it the most sustainable materials to be used for PM capture in urban areas.

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