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Material design meta-protocols: a method for design-driven experiments in the Lab. The case of nature's porous intelligence as a strategy for materials innovation

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Abstract: This research explores a combination of design and materials science to develop a novel approach to materials' porosity. This paper examines how the intelligence of porous structures in nature can inspire the design of innovative materials and their applications by analyzing the biological references and their hierarchical and multi-scale principles. We propose and discuss a hybrid methodology that combines life science, chemistry, and design through creative, user-centered lab experiments. The process results in new materials formulations bridging science, society, and the market. We outline techniques to engage designers with scientific protocols called "Material Design Meta-protocols" and demonstrate their practical implementation through the results of the porosity design. These Meta-protocols help the designer familiarize themselves with the scientific research conducted in materials science laboratories and empower their role in shaping materials' properties.

Highlights

- A design-driven approach to materials innovation from the laboratory can stimulate new directions in scientific research.
- Design can help Materials Science to imagine and predict new scenarios and possibilities for scientific findings.
- Design connects objective paradigms of science with subjective perceptions of daily products.
- Interpretations and transfer of the intelligence of porous structures in nature can define new possibilities of tailored porosity for design.
- Science and Technology require a way to translate, interpret, and connect their progress and results with society.
- The process of synthesizing ceramic foams was improved by incorporating a creative method.
- Materials porosity was utilized to create *design metaprotocols* according to the scientific protocol.
- Product concepts and prototypes were utilized as scientific tools to advance new hypotheses during the experimental procedure.

Keywords: Design-driven Materials Innovation - Porous Intelligence - Interdisciplinary methods - Design Lab Experiments - Design Meta-Protocols

[Resúmenes en castellano y en portugués en las páginas 207-209]

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1. Introduction

In research and innovation for new materials, the design discipline faces a thrilling and promising challenge of actively contributing to developing these materials from the laboratory to their dissemination and implementation in the productive economic system (Camere & Karana, 2018; Kuijpers, 2019; Migliore, 2020; Tulu *et al.*, 2019; Wilkes *et al.*, 2014) This opportunity arises from the current need to manage the complexity that characterizes our rhizomatic knowledge, made of infinite connections, through cross and interdisciplinary research. In this work, the design links various branches of science, based on objective paradigms, with the world of user experience characterized by subjective and perceptive variables (Karana *et al.*, 2016; Wongsriruksa *et al.*, 2012).

Using advanced technological and scientific tools for characterization, modeling, and prototyping, we can now investigate and simulate the complex mechanisms governing nature at different levels of scale, ranging from the atomic to the macroscopic. As a result, we recognize the tremendous benefits that natural organisms and materials can provide as models. We are now developing systems that mimic them artificially, which can be applied in various fields, including product design (Service, 2020; Karana *et al.*, 2019; Ormondroyd & Morris, 2019; Peck *et al.*, 2015;Migliore, 2015; 2014), architecture (Migliore, 2015), transportation, medicine, biotechnology (Gordon *et al.*, 2013), and waste recycling (Liguori *et al.*, 2014; Verdolotti *et al.* 2014; Langella *et al.*, 2013). Nature is highly efficient in building different systems using only a few components. It creates adaptable and intelligent structures that serve various purposes and optimize features such as lightness, flexibility, mobility, etc. Sometimes, nature employs the principle of redundancy to enhance functionality.

Creatures or their parts possess unique characteristics and properties achieved through different ways of organizing their structures, including compact, fibrous, and porous arrangements. An extraordinary example is diatoms, photosynthetic microalgae, which use a hierarchical porous structure as a strategy for different functions, such as protection, mechanical strength, filtration of light rays and harmful micro-organisms, absorption of nutrients, and the efficient management of silica, the component for building their shells (Round *et al*, 1990). Material scientists, therefore, still have untapped sources of models and are now in full swing, trying to imitate the structures and logic of nature. In the case of porous media, there is an ongoing rich activity of synthesis, characterization, and structural-functional improvement of organic, inorganic, and hybrid foams (Verdolotti *et al.*, 2015; 2014; 2012; 2010). Design[1] can support research in materials science by envisioning and anticipating new scenarios and possibilities for the practical applications of scientific results. This involves translating the natural pattern of reference into strategies for designing products or systems that respond to the requirements and needs of everyday life. This approach is crucial to developing materials that can meet the diverse demands of modern society. (Langella, 2003).

The design discipline has always supported materials science in finding applications and generating social and economic values for new materials. Today, there is the rise of a new paradigm of multidisciplinary collaborations, where design is increasingly integrated into the evolution of materials research (Langella, 2012).

Science and technology pervade our daily lives and require a mediator to communicate their progress and results to society and build awareness. Society comprises citizens, individuals, and users with unique cultural backgrounds and daily needs. As Karana *et al.* (Karana *et al.*, 2015) explain: "Functional aptness is taken for granted at the first commercial launch of a new material—meaning that the 'material' should make sense from the perspective of a performance or utilitarian advantage. Nonetheless, this alone may not be enough for its commercial success and widespread use". The material should also be socially and culturally accepted - or acceptable (Manzini, 1986); thus, the material should also make sense. This is one of the reasons why adopting a new material requires a long gestation period - typically 20 years and above - between technical innovation, first commercial application, and widespread uptake of the material (Maine *et al.*, 2005). For example, the market diffusion of nitinol shape memory alloys took about three decades from their introduction in 1962 to the first commercial applications in the medical field in the 1990s (Jani *et al.*, 2014). Likewise, the production of early bioplastics, such as PLA, discovered around 1890, started to be successful among packaging industries only in the 1960s (Stevens, 2001).

A bottom-up approach, which is not only based on purely functional and performance needs or economic urgencies of the society but also end users' desires and experiential needs, proposes the novelty as a non-invasive response to their latent daily needs. It is a powerful strategy to shorten the gestation time of a materials innovation (Ashby *et al.*, 2009).

The relationship between design and materials science has become more frequent and significant in the past twenty years. Nowadays, designers and materials scientists work together from the beginning of the research process to plan and define the formulation of new multifunctional, lightweight, and greener materials (Rubino *et al* 2024, Verdolotti *et al* 2015, Verdolotti *et al* 2017, Pascarella 2024).

Below, we present the outcomes of an interdisciplinary process involving design-driven and user-centered experimentation on a geopolymeric matrix (Lirer *et al.*, 2006, Ferone *et al.* 2015) and hybrid organic-inorganic foams (Verdolotti *et al.*, 2015; Verdolotti *et al.*, 2014). Our approach demonstrates the potential for predicting new scenarios and research directions based on interpretation and experiments with the natural porosity reference as a design strategy.

2. Experimental

2.1. Materials

The Sodium Silicate (SiO_2/ Na_2O = 3.2, namely SS) was provided by Prochin Italia Srl; the silico-aluminate powder (MK) was supplied by BASF with the following composition (% by weight): Al_2O_3 42 %; SiO_2 53 %; K_2O 0.60 %; Fe_2O_3 1.70 %; TiO_2 1.83 %; MgO 0.50 %; CaO 0.37 %. Merck and Sigma-Aldrich purchased Si metal powder and Na_2SiF_6 catalyst, respectively. Vegetable foaming agent in water solution (pH=7) was kindly provided by Isoltech s.r.l. Italia. Diatomite (Celite 545 C) flux calcined diatomaceous earth was provided by Sigma Aldrich. Plexiglass, latex, and borosilicate glass were used as a mold to produce samples and products.

2.2. Preparation of hybrid, design-driven foams

Starting from the combination of two scientific set-up protocols developed by the authors in previous papers (Capasso *et al.*, 2020; Verdolotti *et al.*, 2015; Verdolotti *et al.*, 2014; Lirer *et al.*, 2006;), based on geopolymer and diatomite-based formulations, new sustainable hybrid diatomite-based foam samples with tailored porosity for size, characterization, and functionality were formulated.

The hybrid diatomite-based foam (namely HCFs) prepared (Verdolotti *et al.*, 2015; Verdolotti *et al.*, 2014) was modified using a design-driven approach. The hybrid foams were manufactured using the above-mentioned compounds, SS, Na2SiF6, MK and/or Diatomite, metal Si powder, and a “mousse” type foam at different amounts and ratios. The mousse was obtained from a water solution containing a mixture of vegetable surfactants using an UltraTurrax disperser at 12000 rpm up to obtain a volume 8 times the initial. The slurry was cast in several molds different in shape and/or dimensions and cured at various temperatures (ranging from 25°C to 40°C) at room humidity.

For all the samples, the pore-forming agent, the shape and size of molds, the process parameters (stirring speed, curing temperature, etc.), and the chemical composition (with or without MK or the addition of diatomite) were modulated to control and tune the porous size, the open and closed type of cells and the chemical interaction with the glass container to test application properties.

This laboratory experiment was conducted using an unconventional process that involved various design tools and methods. Specifically, we used a user-centered design approach to identify new research directions and modify protocol based on people's daily needs. We also utilized research through design (RTD) to produce scientific knowledge through future-oriented and project-based design research. Additionally, we used modeling and prototyping to test the effect of porosity crafted in the lab on products, geometries, and manufacturing techniques.

2.3. Experimental characterization

The following experimental characterization was carried out to investigate the correlation between the designed foams' structure and properties. Chemical analysis by infrared spectroscopy-FTIR was performed to highlight the chemical interaction between the produced hybrid ceramic foams and a glass container. The study was conducted at room temperature using a Perkin Elmer apparatus and selecting a wavenumber resolution of 4 cm^{-1} for 64 scans from 4000 to 600 cm^{-1}. The spectra were collected in reflectance mode (ATR) on the solid samples.

The cellular morphology of hybrid ceramic foams was examined using optical microscopy using Olympus mod. BX51 and scanning electron microscopy (SEM) operating at 20 kV, mod. S440 from Leica Microsystems GMbH (Germany).

3. Results and discussion: Experimental by envisioning functions

The study aimed to control the porosity of ceramic foams for product design by combining a chemical protocol with a design-driven approach.

The chemical synthesis procedure was hybridized with a more intuitive and creative approach, foreshadowing functions and needs (Langella, 2007). The experiments started with a preliminary phase, in which a series of samples, shown in *Figure 1*, defined *Probing Experiments*, were obtained by following the protocol closely [25-27] so that the designer could master the process and study the conditions of replicability.

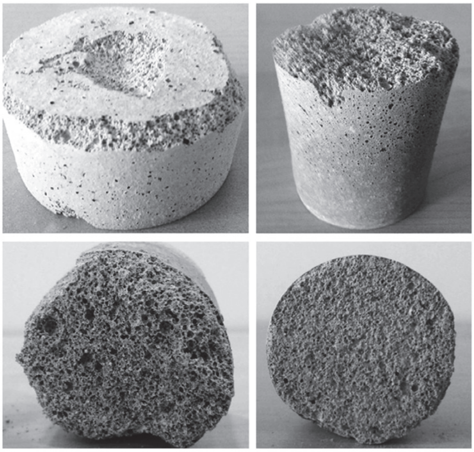

Figure 1. "Probing Experiments": samples of hybrid ceramic foams from protocol.

Subsequently, a "tinkering" (Karana *et al.*, 2015) step of *Jumping Experiments* was conducted, in which the components' process and proportions were dramatically and substantially altered to obtain a rich range of results and errors to reflect and open possibilities, as in *Figure 2*. This is based on the Learning by Doing, Research Through Design (Durrant *et al.*, 2017; Wakkary *et al.*, 2015; Woodward, 2020; Anzai *et al.*, 1979) and Reflection in Action (Schon, 1983) approaches, foundational to design practice and research.

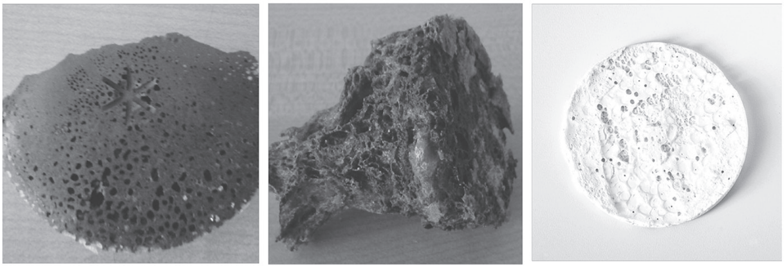

Figure 2. "Jumping Experiments": samples of tinkering with the protocol.

After the designer had gained familiarity with the scientific formulation and comprehended the role played by each ingredient and condition, *Functional Experiments*, as we named them, were carried out to design porous features for specific functions of daily-use products through the chemical-physical process. In particular, the material's porosity was tuned to respond to light filtration as one of the most challenging and fascinating material properties for designers. Light filtration needs a rough porosity. One of the samples (obtained through *Jumping Experiments*) characterized by meso and macro porosity was selected as the starting point for this experiment *(See Figure 3)*.

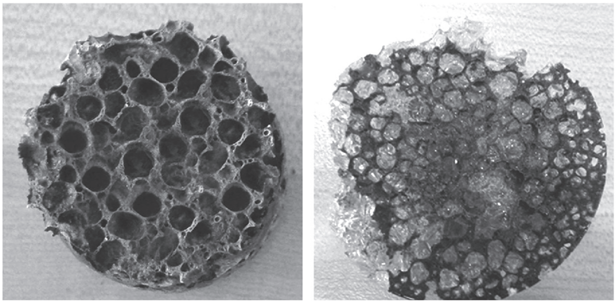

Figure 3. "Functional Experiments": Samples with rough porosity were formulated for lighting function.

The different pores' morphology (closed/open cells and interconnection) and transparency (the glassy effect induced by silicate solution altered in the new formulation), observed under optical microscopy, affect the interaction of the microstructure with the light at the macro scale, as can be seen at different magnifications *(See Figure 4)*.

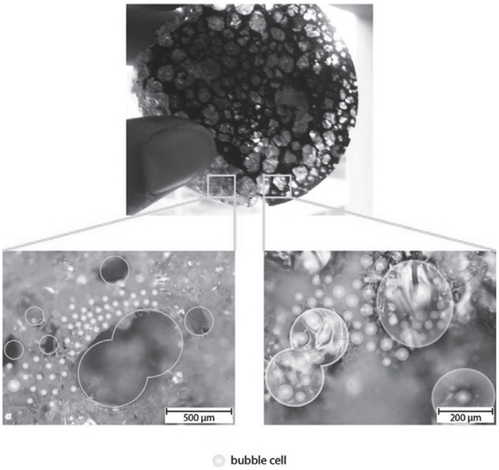

Figure 4. Relation between macro scale light interaction properties and the microstructure observed in optical images.

Once a certain number of promising and replicable samples were achieved, the phase of *Intrusions Experiments* was conducted: new components, selected among natural porous references, were added to the formulation. A successful result of this approach was the introduction of diatomite, a siliceous sedimentary rock composed mainly of the fossilized skeletal remains of diatoms, single-celled organisms related to algae *(See Figure 5)*.

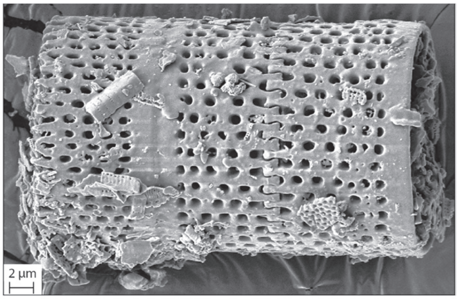

Figure 5.
SEM of the diatomite powder.

This diatom's hierarchical and nano-porous structure influenced the formulation of the final product. It led to promising gradient porous properties to be applied in product design, such as filtering material for domestic use, e.g., cleaning harmful microorganisms for food storage or as part of kitchen tops and tools. Our samples showed interesting hierarchical and porous features from macro to nanoscale porosity *(See Figure 6)*. In particular, the foam also presented a nano-porous grid in its struts.

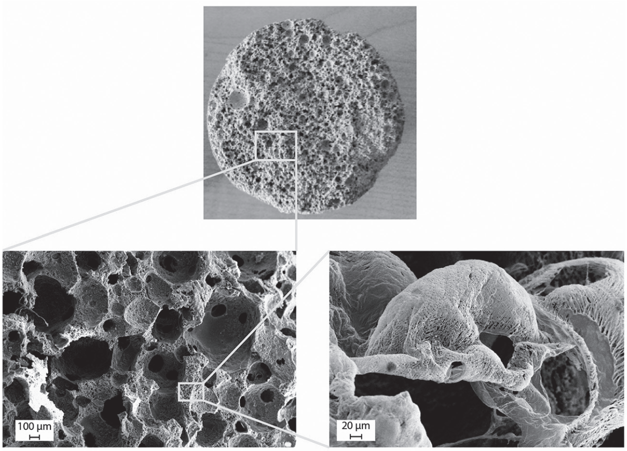

Figure 6.
"Intrusions Experiments": sample of ceramic foam with diatomite powder showing nanoporous and hierarchical structure.

Different variations unrelated to chemical properties were tested to achieve the desired results. In this case, the shape of the container was used to manipulate the gas flow during the expansion process. The results of this experimental stage, which we defined as *Shaping Experiments*, are illustrated in *Figure 7*.

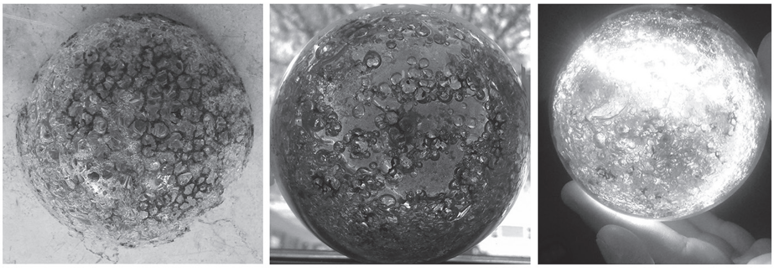

Figure 7. "Shaping Experiments" were conducted using molds different from the usual lab tools. These samples exhibit unique lighting properties, which are promising for design applications.

During the last phase of the experimental program, the designer explored an approach based on interference in the procedure. We tested an intuitive process that deviated from the protocol's standard mixing and reaction time. We modified the process parameters, such as stirring speed and the sequence for adding the solid components. These experiments, called *Process Experiments*, produced promising results for design applications. Different layers of cellular porosity at the macroscopic level were visible, indicating regular, controllable, and repeatable stratification *(See Figure 8)*. The gradient porosity related to the ingredients deposition was determined by a faster and shorter duration of the stirring speed and was reflected into different colors.

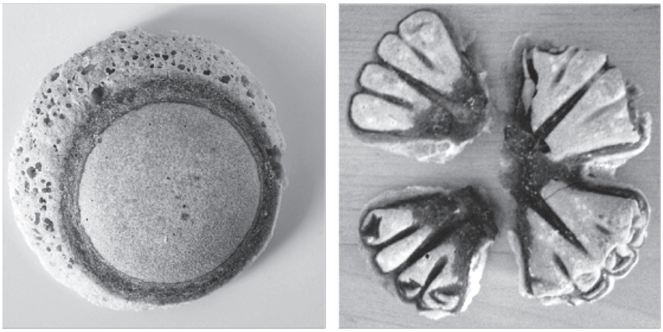

Figure 8.
Process Experiments: samples showing macroscopic hierarchical and layered porosity.

A crucial design method introduced in the lab was visualizing and testing product concepts through prototypes during the experimental procedure. This helped assess, share, and make tangible the value of the results. We realized several prototypes and objects, such as Diaphanea, a system of lamps in which the foams formulated for light filtration were reproduced and cast into designed borosilicate glass to become materials for LED-light diffusion and refraction, shown in *Figure 9*.

Figure 9.
Diaphanea, a system of lamps, borosilicate, and experimental ceramic foam.

Besides the visible lighting qualities, the objects showed a better glass resistance to fracture due to the excellent chemical interaction between the hybrid foams and the glass covering, as observed in the FTIR investigation *(See Figure 10)*.