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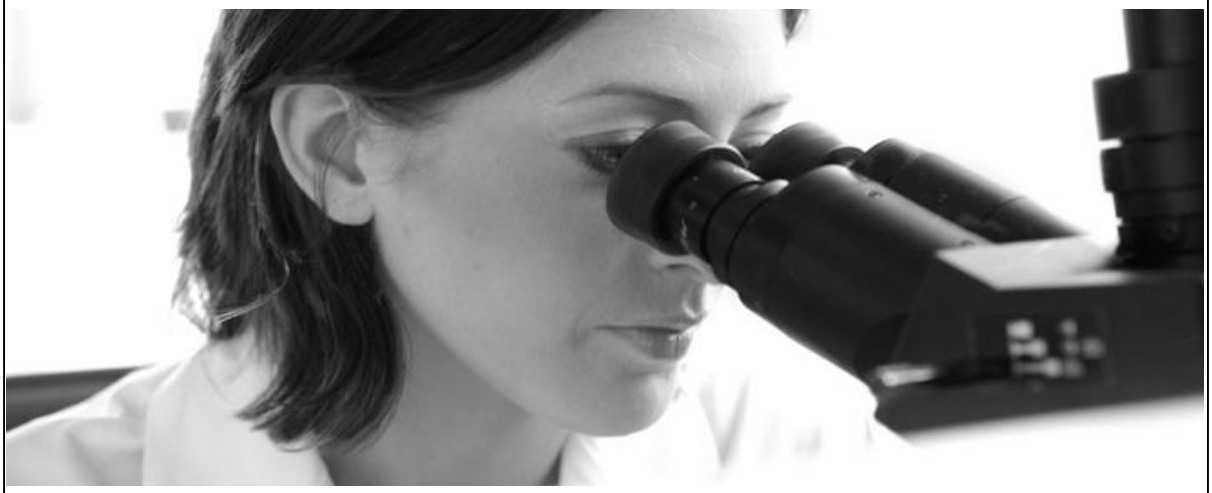

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RESEARCH ARTICLE

POROUS SIZE CONTROL ON SILICONE MATRIX

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ABSTRACT

An enormous development is experimented on the porous materials. The porous was considered a defect on solid materials some years ago, but right now, this defect is an advantage, based on the properties obtained from micro, meso and macroporous materials, and their possible applications on a medical field. The feasibility to use forming agents to control the density and size of the pores, and the possibility to improve the porous properties through adding functional groups or nanoparticles on the porous surface allows the application of porous materials in many areas, especially on medicine.

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INTRODUCTION

The porous material is a continuous and solid network material filled through and with voids, a material that is obviously porous when the voids are filled up with gas. However, the voids might be filled with a liquid or even a solid; some authors prefer to label nanostructured materials instead of porous material (Polarz, 2004).

Immense research during the last decade has been devoted to adjust the morphology (the size and shape) of solids at various length scales from the nanometer to the micrometer scale (Kuschel, and Polarz, 2008; Qin, Ren, Zhu, Zhang, Shang, and Wei, 2011). The porous materials research has been done with a variety of pore sizes, shapes and densities, but crucial feature is the increase in the material surface, which helps improve absorbent and adsorbent properties (Mitra, Vazquez- Vazquez, Lopez-Quintela, Bidyut, and Bhaumik, 2010; Park, Jung, Myung, Kim, Moon, Shin, and Seo, 2008).

Porous materials can be made with a variety of pore sizes, modifying the surface of materials that can obtain materials with micro-pores, surfaces with holes less than 2 nm, nanoporous, materials with pore size between 2 nm and 50 nm and macropores, materials with pore size greater than 50 nm, sizes and densities could be controlled by the formation conditions, but crucial feature is the increased surface that gives new properties to the materials (Yamauchi, Suzuki, Radhakrishnan, and Wang, 2009).

Porous materials can be manufactured using many substances such as carbon, silicates, ceramics, minerals and polymers (silicone). Attached to the advantage of the porous material, these micro, nano and macro channels can be coated with metallic nanoparticles to improve their properties. Most of the researches that have been designed to provide porous materials with surface functionality, where the structure can be functionalized with organic aliphatic or aromatic groups interact with metal (Ozin, and Arsenault, 2005).

Biocompatible implants allow the human body to restore biological and mechanical functions; therefore, to manage to increase the quality of life. Depending on the biomedical application, the implant has to withstand mechanical loads, while conducting a long term biological interaction with the surrounding biological tissue is promoted. The bulk properties of the implant are primarily responsible for the load bearing capacities, where the interaction with the surrounding tissue is governed by the implant surface. (Zhang, 2011).

The Porous materials could be made with the use of organic compounds that can act as templates or pore-forming agents,

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through a versatile non-surfactant route in which non-surfactant organic compounds such D-glucose, D-maltose, dibenzoyl-L-tartaric acid, ascorbic acid, etc, were used and then remove by solvent extraction (Pang, Qiu, and Wei, 2000), see figure 1.

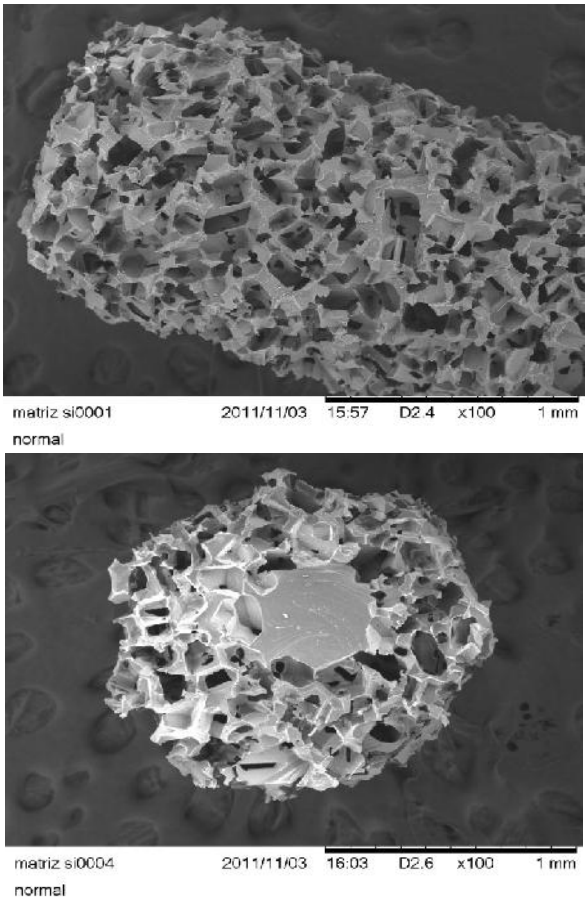


Figure 1 Porous silicon matrix made with the use of pore forming agent, SEM imaging 100X.

Porous materials with various composition and structures are subject of a new research and the porous matrix designed depends on the application. Size, shape, texture, compartmentalization, density and porosity impact the function of a biomaterial, once it is placed into a biological environment (Solano-Umaña, Vega-Baudrit, and González-Paz, 2015).

Porous materials on implants

The properties of micro, meso and macroporous bioactive materials, capable of inducing tissue regeneration, have been combined with their abilities to host and release specific biomolecules that promoted cell growing inside and over the porous surface. The possibility of locally release peptides and proteins is of great scientific importance because it opens new paths for design of implantable biomaterials than can promote bone formation, organs reconstruction, cell an tissue growth where needed (Balas, Manzano, Colilla, and Vallet-Regi, 2008).

Silicone based porous materials have been extensively proposed for a different biomedical applications, such as ceramics for bone regeneration, because these materials exhibit high surface areas and pore volumes, tunable and narrow pore size distributions, also this material is easily functional. These properties allow the materials to act as host matrices for active molecules (drugs, peptides, proteins, etc) (Izquierdo-Barba, Sánchez-Salcedo, Colilla, Feito, Ramírez-Santillán, Portolés, and Vallet-Regí, 2011). Recently, the applications of porous silica and silicon have been widely studied as prospective oral drug delivery vehicles to improve dissolution properties of hydrophobic drug molecules and this property is also used on implants the required some special drug treatment (Kinnari, Mäkila, Heikkilä, Salonen, Hirvonen, and Sanntos, 2011; Xia, Zhou, Ballell, and Garcia-Bennett, 2012).

The organic biomaterials with porous structure have flexible nature with multiple control function that made them attractive for many advanced applications such as sensing, medical uses, and selective material conversion. Immobilization of bio compounds into organized structures paid much attention. However, most of them have difficulty in mechanical stability, material diffusion and unsuitability to make contact with artificial devices. Organic-inorganic hybrids porous materials play an important role in the designed application and address these issues, for example, siloxane and organosiloxane have been used to prepare hybrid porous matrices with functional groups at the surfaces (Zhang, Lu, Xiu, Hua, Zhang, Robertson, Shi, Yan, and Holmes, 2004). Porous (micro, meso and macro) implants have the motility and bio integration, as well as low incidence of extrusion, displacement, and foreign body reaction. Silicone is a material being used in medical science for variety of prosthetic applications, because of its excellent biocompatibility and low cost, some limitations of silicone are limited ingrowths, but it can be improved with the surface morphology control, and the introduction of functional groups or nanoparticles in its surface (Son, Kim, and Yang, 2012), see figure 2.

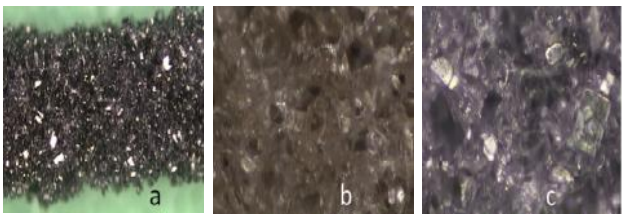


Figure 2 Porous silicone matrix functionalized with gold nanoparticles, a: 40X, b: 100X and c:100X magnification, SmartScope Flash 200, optic machine.

Experimental

Preparation

Chemicals

Poly-dimethylsiloxane (PDMS) product of Nusil Silicon technology (MED-4860), N-heptane puriss. p.a., Reag. ≥99% pure product of Sigma-Aldrich, Hydrochloric acid 37% ACS reagent product of Exaxol Chemical Corporation, Commercial fine sugar (SCS, brand), were procured and used as received. All preparations were carried out at 22±1 ◦C.

Procedure

Leaching

Leaching using solid fine crystals is one of the simplest to manufacture or production of matrices with controllable

porosity from polymers methods. The process for the manufacture of porous matrices or construction involves combining a low viscosity polymer or a dissolved polymer with a solid in a suitable mold, then the polymer is cured and the solid crystals are removed with a solvent. Different commercial sugar brand samples were sieve and the sample with a major crystal proportion between 100 and 300 micro meters was selected.

The selected sugar was divided into 15 samples and these samples are divided according to the following criteria: level 0 is not screened, level 1 pass through the sieve # 40, level 2 pass through the sieve # 50. Later the silicone was dissolved on heptane and the sugar sample was added under a mixing ratio of 28.6 % silicone, 14.3% heptane and 57.1% sugar; the produced paste was used to fill up a mold, the solvent was evaporated and the silicone was cured at 100 Celsius degrees. The silicone matrix was placed in di-ionize water at 50 degrees, stirring to remove the sugar, then it was placed in a chemical solution, sodium hydroxide solution 2.5% weigh, 50 °C for 1 hour and later in hydrochloride acid concentrate diluted 50% volume, at 50 °C for 5 hours. Finally the matrix rinses with water and dries at 50 °C.

Molding process and injection

Sodium chloride or sugar crystals treated at 95% moisture are ideal pore-forming agents and creating interconnected pores for a mold, several recent modifications of this method demonstrates the tremendous rate of improvement in the manufacture of porous matrices for medical applications (Dutta, Rinki, and Dutta, 2011).

Sugar samples were selected based on the following criteria. See Table 1.

Table 1 Sugar samples: criteria used to produce the silicone porous matrix through a molding process and injection.

Sieve	Blank	Sample				
		1	2	3	4	5
40	Accepted	Scrap	Scrap	Scrap	Scrap	Scrap
50	Accepted	Accepted	Scrap	Scrap	Scrap	Scrap
70	Accepted	Accepted	Accepted	Scrap	Scrap	Scrap
80	Accepted	Accepted	Accepted	Accepted	Scrap	Scrap
100	Accepted	Accepted	Accepted	Accepted	Accepted	Scrap
140	Accepted	Accepted	Accepted	Accepted	Accepted	Accepted
<140	Accepted	Accepted	Accepted	Accepted	Accepted	Accepted

The blank was the sugar sample without sieving process. All sugar samples (blank, 1,2,3,4 and 5) were mixed with 5% water by weight, and later the mold was filled up with each sample, one small channel was opened in the meddle of each mold, then the silicon was mixed with heptane in a proportion 80/20 and the mix was injected into the internal channel of each mold, the silicone was cured at 100 °C, the mold was placed in di-ionize water at 50 °C, then stirring to remove the sugar and release the silicone matrix, finally the matrix was dried on an oven at 50 °C.

All samples must be inspected under SmartScope Flash 200 and the samples must meet the establish criteria, average pore size between length and width, must be from 100 to 300 micrometers.

Characterization method

Optic measure

Pores size were measured using a SmartScope Flash 200, model CNC200, serial SVW2003849, it is an optic machine.

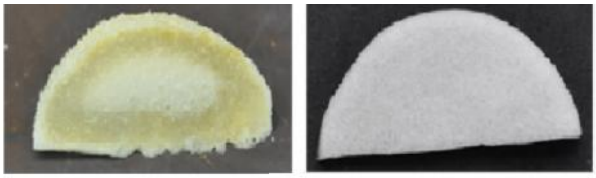
Scanning electron microscopy (SEM)

SEM imagines and measures were obtained, using JEOL JSM-6390LV Scanning Electron Microscope, also pore sizes measure were confirmed with this equipment, too.

RESULTS AND DISCUSSION

Leaching

On the leaching process, the sugar removal after curing the silicone was difficult since some sugar crystals were enclosed by the silicone rubber and these crystals cannot be removed with hot water, this is confirmed by performing a cross-section of the porous silicone matrix.
See Figure 3.



Silicon porous matrix after the rinse with water

Silicon porous matrix after etching process and the rinse with water

Figure 3 Comparison of a porous PDMS surface after wet treatment taxed.

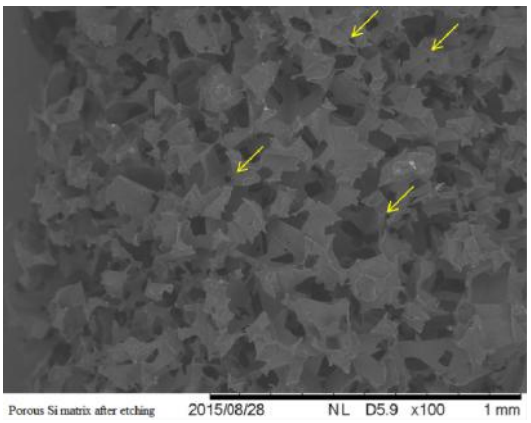


Figure 4 Porous silicone matrix after etching process.

An etching process was required to remove the enclosed sugar crystals, during the etching process the matrix was placed in sodium hydroxide solution 2.5%, 50 °C for 1 hour and later in hydrochloride acid concentrate diluted 50% volume, at 50 °C for 5 hours, finally the matrix was rinsed with DI water and dried at 50 °C in an oven.

After the etching process, the enclosed sugar was removed (see figure 3), the porous from the silicone matrix were freed and interconnected. This can be proved from the appearance, soft surface and the inspection under visual microscope and SEM image of the cross section from the matrix, also in figure 4 you can see small holes not present before the etching process.

Five samples per each proposed level (level 0: no sieved, level 1: pass through sieve # 40, level 2: pass through sieve # 50) were prepared with the leaching method. Each sample was placed in the optical equipment, SmartScope Flash 200 (digital measuring device designed for measurements of stationary parts), five pores were randomly chosen over the cross section surface and width and length were measured and recorded for each sample. Later an average measure from the width and length were obtained and analyzed with Minitab 16 statistic program to compare average pore size, obtained for the levels 0, 1 and 2.

When each sample was cleaned, a distribution graph for each level was made (see Figure 5), the variability in levels 0 and 1 is greater than the level 2, and the results are consistent with what would be expected.

The distribution from levels 0 and 1 is shifted to the right on the graph because the pores of the silicon matrix have a larger size, while the sample 2 is shifted to the left, because these matrices used the sugar which passed thru sieve # 50 (300 microns, level 2, Figure 5C). The control of the sugar crystal size distribution is directly related with the matrix porous size.

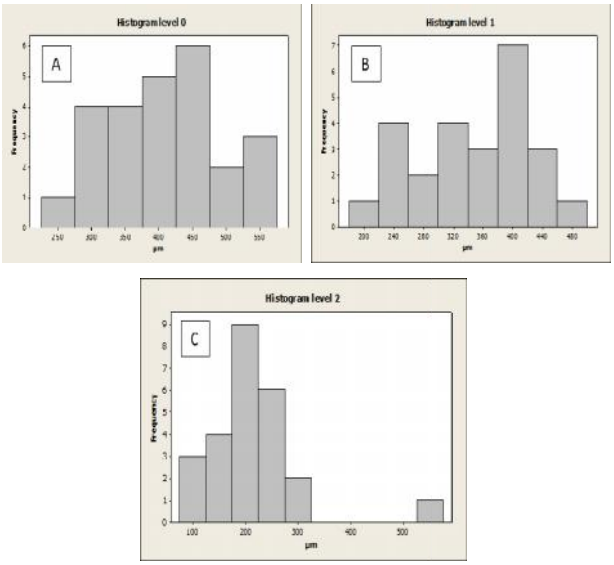


Figure 5 Distribution of pore size in the sample obtained from the three selected levels, A: level 0, B: level 1 and C: level 2.

Using statistical tools analysis of variance Minitab 16 program and specifically ANOVA analysis, average pore size obtained for the levels 0, 1 and 2 were compared, and the value of P was equal to 0.000. This value indicated that the null hypothesis, which established that all mean pore size from levels 0, 1 and 2 were not significantly different is rejected with a confidence level of 95%, the level 2 is significantly different from the levels 0 and 1.

The individual value plots graph show how the level 2 samples have a smaller dispersion regarding to the levels 0 and 1, also level 2 has a lower pore size mean value (see Figure 6). These results confirm that sugar crystals size or (grain size distribution) is directly related to the pore size obtained in uncoated porous silicone matrix.

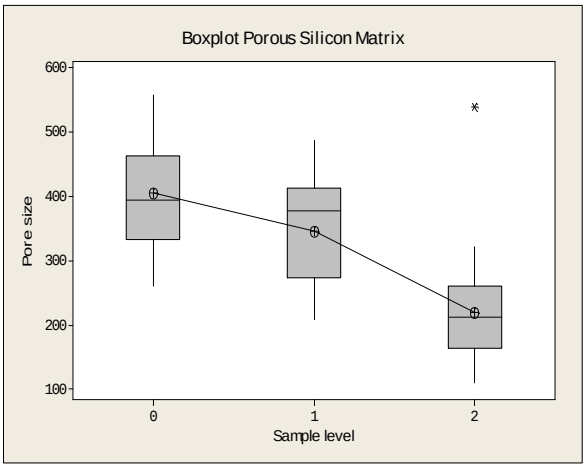


Figure 6 Variability of pore size in the samples obtained from the three selected levels 0, 1 and 2.

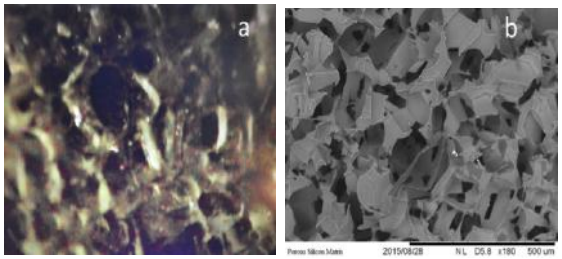


Figure 7 Uncoated porosity silicone matrix obtained using the technique of leaching, a: 125X magnification, SmartScope Flash 200, optic machine, b: SEM 180X.

Injection molding

The selected sugar sample was prepared adding 5% of water by weight and mixed, then it is compacted in a mold without breaking the sugar crystals and the silicone/heptane mix was injected into the open channel, allowed to evaporate the heptane and cure the silicone on the oven, finally the mold was washed in deionized water at 50 ^{o}C to remove the sugar and release the silicon porous matrix. The injection molding process has an advantage over the method of leaching, because the sugar crystals could be removed faster. The injecting silicone into the mold sugar fills up the spaces between the sugar crystals; so that there are not occluded sugar crystals and the pores are interconnected, too.

The silicone matrices pore size measuring (width and length), average value from each sample, prepared based on table 1, ratify the results obtained by leaching process, the porous matrix silicone obtained used the sugar crystals passed through the sieve 50 (300 micrometers) gave 100% productivity, all matrices met the established criteria, the pores size was between 100 and 300 micrometers (see Table 2), the same results were obtained on the samples 3, 4 and 5. The measurements were made using SmartScope (digital measuring device, designed for measurements of stationary parts). On the other hand, a scanning electron microscope image was used to check the cross-sectional and side view from the silicone matrix, and the pore size. See Figure 8.

Table 2 Results of the pore size of the silicone matrices made with different grain sugar.

Sieve	Blank	Sample				
		1	2	3	4	5
40	Accepted	Scrap	Scrap	Scrap	Scrap	Scrap
50	Accepted	Accepted	Scrap	Scrap	Scrap	Scrap
70	Accepted	Accepted	Accepted	Scrap	Scrap	Scrap
80	Accepted	Accepted	Accepted	Accepted	Scrap	Scrap
100	Accepted	Accepted	Accepted	Accepted	Accepted	Scrap
140	Accepted	Accepted	Accepted	Accepted	Accepted	Accepted
<140	Accepted	Accepted	Accepted	Accepted	Accepted	Accepted

Pore size	Blank	Sample				
		1	2	3	4	5
Qty from 100 -300 µm	17	52	35	40	52	52
Qty < 100 or > 300 µm	35	25	0	0	0	0
Total	52	77	35	40	52	52
Yield/%	32.7	67.5	100.0	100.0	100.0	100.0

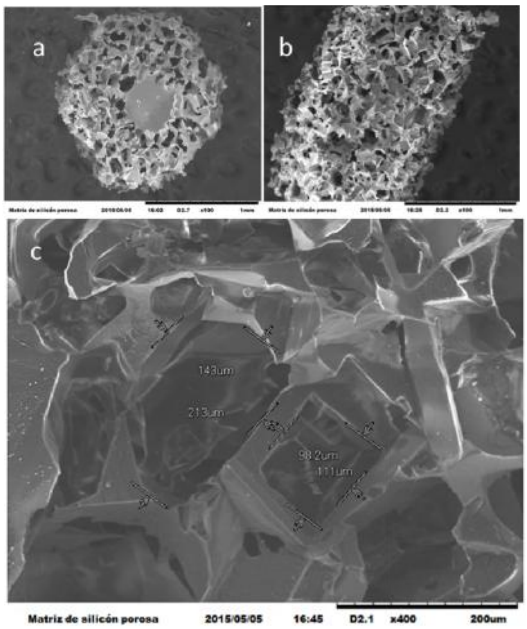


Figure 8 Scanning electron microscope images from uncoated silicone matrix, a- cross-sectional view b: side view, c- pore size measure.

The diversity of different framework compositions and various methods to modify porous solids with guest species allow the materials parameters of porous solids to be tuned over a very wide range, and correspondingly, functional solids based on ordered porous materials have been suggested for many different fields of applications (Schüth and Schmidt, 2002). The surface modifications for polymers can increase the biocompatibility properties, favor interaction of living cells with the biomaterial, improved the effect on cell adhesion and proliferation, and favoring many medical applications (Solano-Umaña, and Vega-Baudrit, 2015)

CONCLUSION

The use of micro, meso and macroporous materials can be a very practical approach to reaching several goals in current medical applications like surgical implants, drug delivery, medical imaging, biological sensors, and others. Their performance under ambient conditions, mechanically and thermally strong under harsh conditions is the key.

The supporting materials with a porous structure control have been proposed to attain both biological and mechanical requirements, on this subject the pore-forming agents place an important role, when pore-forming agent is a crystal that could be removed with a solvent and release the porous matrix, the size of the pore is directly related with the pore-forming crystal size, also pore density and pore connections are related to the pore-forming crystal quantity, see figure 9.

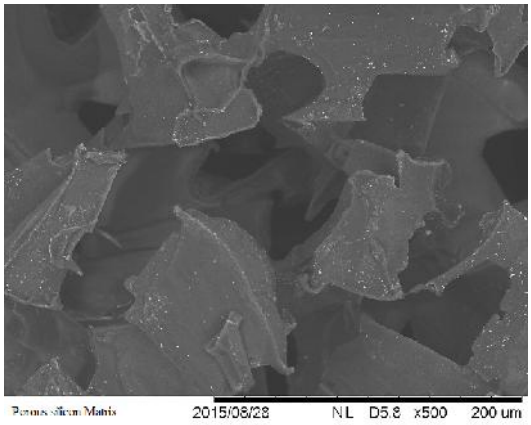


Figure 9 Scanning electron microscope image from porous silicon matrix, cross-section the shows pore connections and density.

Great progresses in structure control of micro and meso and macroporous materials have been achieved for biotechnological and biomedical applications through the size and quantity control of the pore-forming agent. Silicone porous matrix control through the sugar or salt control represent a cheaper and easy way to achieve medical requirements for implants, scaffolds for the in-vitro cultivation of tissues and organs, drug delivery, medical imaging, biological sensors, and others. Scaffold materials with superior properties must have good water absorption, if the scaffold material have a control porosity it could absorb sufficient cell culture medium, and the cells adhered to the walls can obtain ample nutrition and proliferate inside and over the scaffold surface (Zeng, Liu, Shi, Qiu, Fang, Rong, Guo, and Gao, 2015). The control over the interior and exterior surface from a porous material will have a broad impact on the study and future medical applications of these nanostructures where high surface area, large pore volume and controllable pore size give these materials especial properties for amazing applications (Kilian, Böcking, Gaus, and Gooding, 2008).

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