



# **The 6<sup>th</sup> International Conference on BIOFOAMS**

**September 25-28, 2018**

**Chengdu, China**

**Organized by**

**State Key Laboratory of Polymer Materials Engineering, China**

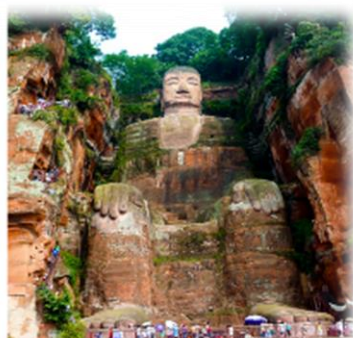
**College of Polymer Science and Engineering  
Sichuan University, China**

**Polymer Research Institute of Sichuan University, China**



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## INTRODUCTION

We extend a warm welcome to this international conference. This highly-regarded meeting covers a range of biofoam-related interests, and enjoys a strong support and wide-range inputs.

The 6<sup>th</sup> Biofoams Conference will be held in Chengdu, China. This conference will bring together researchers from around the world who are interested in exploring the link between processing, structures and properties of biofoams, and provide a broad context of scientific exchange in the aspects of characterization, modeling, and formulation including the use of additives, surfactants and blowing agents. The common features of the foamed structures, the common phenomena involved in processing and properties, the conference structure, the diversity of selected attendees from industrial and academic organizations are expected to contribute significantly to the objective of promoting scientific discussion and further collaborative research.

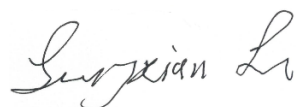
The conference covers the following themes, including (1) Bio-matrices: sustainable polymer, biodegradable polymer; (2) Biodegradable and Sustainable Foams: properties of foamable composite, foaming agents, foam equipment and technologies; (3) Bio-inspired Foams: biomimetic platforms, bio-hybrid systems; (4) Biomedical Foams: scaffolds for tissue engineering applications, controlled drug delivery; (5) Food Foams: materials and properties of food foams, foams in food technologies; (6) Foam of Advanced applications: energy, transport, packaging, environment; (7) Advances in Foaming: novel processes, advanced characterization, simulation and modeling.

It is a great honor for Chengdu to host Biofoams2018. As one of the most important economic, transportation, and communication centers in Western China, Chengdu is the capital city of Sichuan Province and hometown of giant pandas, having a long history of over 3000 years and a large population of about 14 million. As the World's Favored Tourism Destination City, there are many beautiful sceneries and historical sites within or around Chengdu, such as Wu Hou Shrine, Chengdu Research Base of Giant Panda Breeding, Jiuzhai Valley National Park, Emei Mountain, etc.

Biofoams2018 is organized by the State Key Laboratory of Polymer Materials Engineering, College of Polymer Science and Engineering of Sichuan University, Polymer Research Institute of Sichuan University. The scientific and organizing committees warmly welcome you to attend Biofoams2018, and we will strive to make your experience in Chengdu memorable and enjoyable as much as we can.

We look forward to working together with you, and to a very fruitful collaboration.

Yours Sincerely,



Professor Guangxian Li

Conference Chairman

## International Polymer Foaming Research Center of Sichuan University and University of Toronto

The International Polymer Foaming Research Center was founded with support from both Sichuan University and University of Toronto in 2013, which has become the important component of the International Joint Research Center for Polymer Materials and Engineering, the International Cooperation Base of the Chinese Ministry of Science and Technology. Prof. Guangxian Li and Prof. Qi Wang, the Academician of Chinese Academy of Engineering, from the State Key Laboratory of Polymer Materials Engineering of Sichuan University are the directors of the International Polymer Foaming Research Center, and Prof. Chul B. Park, the Fellow of the Royal Society of Canada and the Canadian Academy of Engineering, is the foreign director. The center has established the world-class platform for research, processing and characterization of polymer supercritical CO<sub>2</sub> foaming. Specifically, the supercritical CO<sub>2</sub> continuous extrusion foaming platform and the advanced polymer foam characterization instruments such as high-pressure DSC, high-pressure rheometer and magnetic suspension balance have been built. The center has made many achievements in the supercritical CO<sub>2</sub> foaming technology, avoided the highly toxic and the environmentally detrimental problems caused by traditional foaming technology, and promoted the level of supercritical foaming technology of China.





# BIOFOAMS2018 PROGRAMME



## Conference Programme

September 25-28 2018, Chengdu, China

Conference Venue: Multifunctional Meeting Room, Kehuayuan Hotel  
科华苑宾馆多功能会议室（宾馆后侧二楼）



25 Sept				
Registration (Cynn Hotel)			14:00-24:00	
26 Sept				
Registration (Kehuayuan Hotel)			7:30-8:30	
Conference Opening			8:30-8:50 Address by Professor Guangxian Li Address by Leader of Sichuan University Address by Leader of Department of S & T of Sichuan Province	
A	Chair: Guangxian Li, Luigi Ambrosio			
	Plenary	Chul B. Park	8:50-9:25	Advantage of Using Nanofibril Reinforcement for PLA Foaming
	Plenary	Qi Wang	9:25-10:00	Poly(vinyl alcohol) Based Functional Foams Prepared by Thermal Foaming
Coffee break & Photo			10:00-10:20	
	Chair: Chul B. Park, Qi Wang			
	Plenary	Luigi Ambrosio	10:20-10:55	Advanced Polymer Based Scaffolds for Tissue Engineering
	Plenary	Salvatore Iannace	10:55-11:30	Structural and Functional Properties of Multicomponent Biobased Foams

	<b>Plenary</b>	Costas Panayiotou	11:30-12:05	Polymer Foaming with Supercritical Fluids: Thermodynamic Aspects
	<i>Lunch</i>		12:05-13:20	Place: 7 <sup>th</sup> Floor, Kehuayuan Hotel
Biomedical foams	<i>Chair: Salvatore Iannace, Aleksander Prociak</i>			
	Keynote	Ling Zhao	13:30-13:55	Polyglycolic Acid and Its Composite Scaffolds Fabricated by Supercritical CO <sub>2</sub> Foaming for Tissue Engineering
	Keynote	Qian Li	13:55-14:20	Fabrication of Highly Interconnected Poly(caprolactone) Porous Scaffolds and Tissue Engineering Applications
	Keynote	Jie Weng	14:20-14:45	Angiogenesis and Osteogenesis of Bioceramic Scaffolds Regulated by Their Porous Structures
	Oral	Evgeni Ivanov	14:45-15:05	Electrical and Thermal Properties of Mono and Bi-filler PLA Composites Filled with Graphene and MWCNT
	Oral	Zhanhua Wang	15:05-15:25	Silicone Rubber Foam for Wound Dressing
	<i>Coffee break</i>		15:25-15:45	
Biodegradable and sustainable foams	<i>Chair: Ling Zhao, Jie Weng</i>			
	Keynote	Aleksander Prociak	15:45-16:10	Effect of Bio-polyols and Bio-fillers on Foaming Process and Properties of Polyurethane Foams
	Keynote	Wentao Zhai	16:10-16:35	Fabrication of PLA Foam with High Crystallinity and the Improved Heat Resistance
	Keynote	Maria Grazia Raucci	16:35-17:00	Composite Aerogel Microspheres with Enhanced Bioactivity

	Oral	Mandar Badve	17:00-17:20	Foam Flow Phenomena in Narrow Channels with Converging-Expanding Constrictions
	Oral	Xuetao Shi	17:20-17:40	Influence of PLA Stereocomplex Crystal on the PLA-based Blend and Its Microcellular Foam
<i>Banquet</i>			18:00	Place: Hexi Restaurant (和席酒家), 153 Kehua North Road
<b>27 Sept</b>				
Advance in foaming	<i>Chair: Hanxiong Huang, Long Wang</i>			
	Keynote	Wenge Zheng	8:30-8:55	Flame-retardant PP Foams Using CO <sub>2</sub> Extrusion Foaming
	Keynote	Guilong Wang	8:55-9:20	High-performance PLA and Its Foams Enabled by Carbon Dioxide Treatment and Foaming
	Oral	Maria Kurańska	9:20-9:40	Hydroxyl Derivatives Based on Used Cooking Oils as Component for Synthesis Open Cell Rigid Polyurethane Foams
	Oral	Zhonglei Ma	9:40-10:00	Lightweight, Compressible and Electrically Conductive Foams Coated with Synergistic Multiwalled Carbon Nanotubes and Graphene for Piezoresistive Sensors
<i>Coffee break</i>			10:00-10:20	
Biodegradable and sustainable foams	<i>Chair: Wenge Zheng, Guilong Wang</i>			
	Keynote	Hanxiong Huang	10:20-10:45	Biodegradable PLA-based Blend Foams for Oil-water Separation
	Keynote	Jianshu Li	10:45-11:10	Bioinspired Materials for Hard Tissue Repair: from Fundamental Research to Industrialization
	Oral	Martina Salzano de Luna	11:10-11:30	Optimization of the Performances of Chitosan/Graphene Oxide Aerogels for Water Purification



	Oral	Xiaofeng Wang	11:30-11:50	Preparation of Highly Porous Interconnected Poly( $\epsilon$ -caprolactone) Scaffolds Combined with Supercritical CO <sub>2</sub> Foaming and Polymer Leaching
	<i>Lunch</i>		12:05-13:20	Place: 7 <sup>th</sup> Floor, Kehuayuan Hotel
Bio-matrices and food	<i>Chair: Costas Panayiotou, Sheng Zhang</i>			
	Keynote	Marino Lavorgna	13:30-13:55	On the Embedding of RGO/Chitosan Aerogel in Open Cell Polyurethane Foams and Their Piezoresistive Properties
	Keynote	Long Wang	13:55-14:20	A Novel Low-pressure Foam Injection Molding Technology Using Different Type of Foaming Agents
	Oral	Tim Thysens	14:20-14:40	Bioproduction of Class II Hydrophobins Exhibiting Excellent Foam Stability
	Oral	Jeroen Vereman	14:40-15:00	Extraction and Spray Drying of HFBI Produced by Trichoderma Reesei
	Oral	Pengjian Gong	15:00-15:20	Nanocomposite Foams Thermal Insulation Performance, Nanoparticles' Connection and the Three-Dimensional Foam Structure
	<i>Coffee break</i>		15:20-15:40	
Bio-matrices	<i>Chair: Marino Lavorgna, Wentao Zhai</i>			
	Keynote	Sheng Zhang	15:40-16:05	Cyclodextrin-based Metal Organic Framework for Gaseous Messages Delivery
	Keynote	Xia Liao	16:05-16:30	Manipulation of Foam Structures Based on CO <sub>2</sub> -induced Changes in PLA Fundamental Properties
	Oral	Maria Grazia Raucci	16:30-16:50	Hyaluronic Acid-based Composite Hydrogels for Tissue Engineering

	Oral	Polya Angelova	16:50-17:10	Influence of Carbon Nanotubes and Graphene in PLA Nanocomposites on Thermal and Electromagnetic Properties
<b>Conference Closing</b>				
<i>28 Sept</i>				
<b>Social Event:</b> <i>Visit to National Technology Transfer Southwest Center and Tianfu New Area</i>			9:00-12:00	

## BIOFOAMS2018 ABSTRACTS

### ADVANTAGE OF USING NANOFIBRIL REINFORCEMENT FOR PLA FOAMING

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#### ABSTRACT

This plenary speech will explain the details of the modern foaming technologies that use crystals to control foam processing, especially in the context of foaming PLA. First, the effects of crystals on cell nucleation and cell growth of PLA will be discussed. Both growing and developed crystals can significantly affect the foaming process. The interface between the crystals and the amorphous sections produces heterogeneous nucleating sites. This is due to the crystals' expelled gas, the tensile stresses generated by their shrinkage, and the mismatched motion that occurs at the interface. After the cells have been nucleated, cellular growth is also affected by the crystals. This is caused by the increased viscosity (especially the extensional viscosity) and the increased melt strength that occurs from the molecules' connection to the crystals.

In this talk, I will also explain the PA nanofibrils' effect on the crystallization kinetics of the PLA/nanofibril composites, both with and without gas. The dissolved gas and the nanofibrils dispersed in the polymer matrix acted synergistically on the crystallization behavior of PLA materials with slow crystallization kinetics. Following this, I will show how crystallization is controlled during the bead foaming, extrusion foaming, and foam injection molding processes for PLA materials. I will then show how the growing crystals and the formed crystal structures affect the foaming behavior of PLA in each of these three processes.

1. Bead foaming of PLA: I will show how crystal development can be controlled before the foaming step in bead foaming, and the effect of those crystals on the cell morphology and on the bead-to-bead sintering behaviors. The intriguing phenomenon of the two-peak crystal formation and the bead foam properties it produces will also be presented.

2. Extrusion foaming of PLA: An in-situ visualization tool was used to demonstrate how crystals can form instantly in the extensional and shear field of an extrusion die, even at a temperature 13°C higher than the melting temperature of PLA. Such strain-induced crystallization is promoted before the foaming step, with a view to controlling the foaming behavior in extrusion. The strain-induced crystallization was quantified and correlated to the cell structure obtained at the extrusion die exit for PLA materials.

3. Foam injection molding of PLA: The most common foam injection molding technology is to use a pressure drop that occurs across the gate during filling to induce cell nucleation. But this process cannot make use of the crystals for cell nucleation. So, instead of this, a high-pressure foam injection molding with a full shot, to re-dissolve all the earlier nucleated cells, is utilized in the new foam injection molding technology. In this technology, foaming can be induced by mold opening, after promoting crystallization with packing first. The predicted temperature gradient across the thickness and the crystal-nucleating agent are utilized in crystallization of PLA with nanofibrils.

# **POLY(VINYL ALCOHOL) BASED FUNCTIONAL FOAMS PREPARED BY THERMAL FOAMING**

Qi Wang

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## **ABSTRACT**

One of the most important advantages of polymer materials is light-weight, maybe the lightest materials after foaming. The big challenge lies in making polymer foams with high performances and multi-functions. This paper reported the novel poly(vinyl alcohol) (PVA) based functional foams prepared by thermal foaming. PVA is an important polymer material having good comprehensive properties particularly biocompatibility and can be prepared in large industrial scale from non-petroleum routes. However, its melting point is very close to the decomposition temperature, making its thermal processing extremely difficult. Based on intermolecular complexation and plasticization, we have successfully realized the thermal processing of PVA. Furthermore, we have developed a series thermal foaming technologies to prepare novel PVA foams: using water as both plasticizer and blowing agent, which has the advantages of high efficiency, low cost and environmental-friendly; using inorganic particles, e.g. talc, calcium carbonate and graphene, as nucleating agents to adjust the nucleation process of bubbles in PVA matrix and optimize the cell structure and performance of PVA foams; using co-blowing agents, e.g. water/ethanol and water/supercritical carbon dioxide, to improve the cellular structure of PVA foams. Accordingly, a lot of novel PVA based functional foams with good comprehension performances applicable for biomedicine, waste water treatment, cold storage, flame retardant, etc., were successfully prepared. This is a big progress in polymer foam making and widens the application of PVA material.

**Keywords:** Poly(vinyl alcohol), Water, Thermal Foaming, Cell Structure, Functional Foams

**Acknowledgements:** The financial supports from the National Natural Science Foundation of China (No. 51720105012) are gratefully acknowledged.

# ADVANCED POLYMER BASED SCAFFOLDS FOR TISSUE ENGINEERING

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## ABSTRACT

Modern medicine is based on the implementation of a personalized approach together a less invasive surgery for the restoration of human tissues and organs. Nature-inspired material science can be considered as the last frontier in biomaterials research. Indeed, the design of complex structural architectures from nano to micro-macro dimensional scale allows geometrically and topologically mimicking the native state of extracellular matrix and its complex supramolecular assemblies. Biopolymers, biocomposites, nanofibrous & gel scaffolds could be used to mimic the complex structure of the extracellular matrix (ECM), and provide essential cues for cellular organization, survival and function.

In tissue repair/regeneration advanced routes and technologies are in progress to synthesize/process instructive biomaterials with biologically recognized functionalities. Among them, additive technologies such as 3D printing is an highly flexible process to design biomaterials able to mimic 3D ECM-like platforms and personalized structures for tissue repair/regeneration processes.

It has been shown that the use of minimally invasive surgery to treat bone defects has significant clinical potential. To achieve the ideal properties i.e. as bone filler, such as osteoinduction, osteoconduction and ease handling, specific compositions based on natural and synthetic polymers, ceramics, and composites have been developed. Here, advances in the synthesis, design and fabrication of 3-dimensional scaffolds and structures to guide regeneration processes in different tissues (i.e., bone, IVD) will be presented.

**Keywords:** Scaffolds, Tissue Engineering, Composites, Hydrogel, Additive Manufacturing.

## REFERENCES

- [1] A. Ronca, S. Ronca, G. Forte, S. Zeppetelli, A. Gloria, R. De Santis, L. Ambrosio. "Synthesis and characterization of divinyl-fumarate poly-ε-caprolactone for scaffolds with controlled architectures". *J Tissue Eng. Regen. Med.* 12, (1) 2018. DOI: 10.1002/term.2322. 12: e523–e531.
- [2] V. D'Antò, M.G. Raucci, V. Guarino, S. Martina, R. Valletta, L. Ambrosio. "Behaviour of human mesenchymal stem cells on chemically synthesized HA–PCL scaffolds for hard tissue regeneration". *J Tissue Eng. Regen. Med.* 2016;10: E147–E154. doi:10.1002/term.1768.
- [3] Demitri C., Raucci MG, Giuri A., De Benedictis VM, Giugliano D, Calcagnile P, Sannino A, Ambrosio L., "Cellulose based scaffolds for bone tissue engineering applications: Assessment of hMSCs proliferation and differentiation". *J Biomed Mat Res. Part A*, 2016, 104A, 726-733.

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# **STRUCTURAL AND FUNCTIONAL PROPERTIES OF MULTICOMPONENT BIOBASED FOAMS**

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## **ABSTRACT**

A great variety of porous cellular materials is present in nature. Their architectures as well as their unique properties have inspired researchers for the development of lightweight structures, energy-absorbing padding or thermal insulating panels.

Nature is able to produce a wide variety of materials or structures by combining few types of component materials. The control over shape and structure on many length scales through a hierarchical structuring allows materials to be grown in a self-organized manner, but also allows the structure to be adapted to needs at each of the different scales. Wood and bone are examples of multi-level cellular architectures found in nature whose structures and functions may lead to new design concepts for bio-inspired cellular materials. Nature has optimized the microstructure of these materials to perform a number of functions. For example, wood and cork have honeycomb-like cellular structures characterized by closed cells while trabecular bones are foam-like cellular materials with open-celled structures.

Even though biological materials are a source of inspiration, the development of hierarchical porous bio-inspired materials will be not easily applicable immediately to the design of new engineering materials. The reason arises from striking differences between the design strategies common in Engineering and those used by Nature. These differences are not only due to the different sets of elements, but also on the different approaches which are, basically bottom-up in Nature and top-down in man-made materials.

Several approaches employed to develop multifunctional cellular structures by means of gas foaming will be presented. Both synthetic and bio-based polymeric structures are used in combination with inorganic 1D and 2D reinforcing micro- and nano-particles to develop lightweight structures with controlled porosities and with tailored electronic properties.

## **POLYMER FOAMING WITH SUPERCRITICAL FLUIDS THERMODYNAMIC ASPECTS**

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### **ABSTRACT**

This lecture discusses key aspects of polymer foaming via green-chemistry processes, primarily, via supercritical fluids and ionic liquids. The production of three major types of porous materials is examined, namely, microporous scaffolds for tissue engineering applications, microcellular (microporous) polymers, and nanoporous aerogels. Rational design of the production process of these materials requires, among others, a thorough understanding of the foaming mechanism while appropriate modelling of the process requires robust models which would respond efficiently to a number of issues, primarily, of thermodynamic character. Such issues are the glass-to-rubber borderline, the polymer-solvent-antisolvent phase diagrams over a broad range of temperature and pressure, the interfacial properties and the nucleation barriers. Solvent and polymer screening studies requires in addition a robust predictive tool. The major focus, then, in the presentation will be on a recently developed thermodynamic model which is designed to respond to the above challenges. A key advantage of the model is that it may be used for the integral thermodynamic characterization of polymers, composites, and solid surfaces. Examples of applications in the design of polymer foaming will be discussed.

# POLYGLYCOLIC ACID AND ITS COMPOSITE SCAFFOLDS FABRICATED BY SUPERCRITICAL CO<sub>2</sub> FOAMING FOR TISSUE ENGINEERING

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## ABSTRACT

As a highly crystalline polymer, polyglycolic acid (PGA) has been applied to some clinical devices. Based on the study of thermal and rheological properties of PGA, an efficient technique, supercritical carbon dioxide (scCO<sub>2</sub>) foaming can be successfully applied for the controllable fabrication of PGA scaffolds with the porosity of 39–74%, average pore sizes ranging from 5 to 50  $\mu\text{m}$ , and interconnectivity over 90%.<sup>1</sup> These PGA foams also exhibited attractive compressive modulus of 68–116 MPa.

In order to improve the biocompatibility of PGA, rapid neutralization of the acidic degradation products of PGA foams was achieved by incorporating tripolyphosphate (TPP) micro-granules into it. It was shown that the acidic drop in the microenvironment of implants was significantly slowed as the pH values of their respective solutions were maintained in a well neutralized state during the degradation. A normalized microenvironment could thus be achieved for implants, and the expression of inflammatory cytokines were downregulated shown by immunofluorescence staining. A relieved inflammatory response from histological analysis could be observed in *in vivo* subcutaneous implantation in immunocompetent animals, pigs.

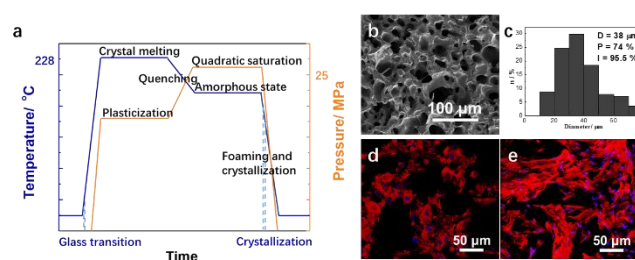


Fig. Foaming procedure developed in this work (a). The image of PGA foams (b) and the pore size distribution (c). The confocal images of fibroblasts seeded on PGA foams after one (d) and three days (e).

**Keywords:** PGA Foams, Properties, Composite Scaffolds, Immunocompetent Animal Model

## REFERENCES

Zhang, J.; Yang, S.; Yang, X.; Xi, Z.; Zhao, L.; Cen, L.; Lu, E.; Yang, Y., Novel fabricating process for porous polyglycolic acid scaffolds by melt-foaming using supercritical carbon dioxide. *ACS Biomaterials Science & Engineering*, 4 (2), 2017, pp. 694–706

# **FABRICATION OF HIGHLY INTERCONNECTED POLY(CAPROLACTONE) POROUS SCAFFOLDS AND TISSUE ENGINEERING APPLICATIONS**

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## **ABSTRACT**

Porous biomaterials have long been used to enhance implant fixation and more recently to engineer the formation of three-dimensional (3D) tissues in vitro and in vivo. The preparation of 3D highly interconnected porous scaffolds is a crucial segment in the tissue engineering. Porous biomaterials can supply this kind of “house” for cells. In order to fulfill the requirement of tissue engineering, the ideal physical topology of scaffold should satisfy the characteristics of three dimensional, highly porous structure with an interconnected channels and hierarchical in pore size. Supercritical CO<sub>2</sub> (scCO<sub>2</sub>) foaming is attracting more and more interests from researchers because of its advantages of organic solvent free and environment-friendly. This talk will go through the researches in National Center for International Research of scCO<sub>2</sub> foaming technology of biomaterials in the past three years including fabrication of porous biomaterials and application in tissue engineering vascular. The relationship among process, phase morphology, mechanical property and biocompatibility was investigated in this presentation. The results gathered in this report may provide a theoretical basis and data to support research into multi-layer tissue engineering vascular scaffold.

**Keywords:** Tissue Engineering, Porous Scaffold, Gas Foaming, Highly Interconnectivity, Property

# ANGIOGENESIS AND OSTEOGENESIS OF BIOCERAMIC SCAFFOLDS REGULATED BY THEIR POROUS STRUCTURES

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## ABSTRACT

The macro-pore sizes of porous scaffold play a key role for regulating ectopic osteogenesis and angiogenesis but many researches ignored the influence of interconnection between macro-pores with different sizes. In order to accurately reveal the relationship between ectopic osteogenesis and macro-pore sizes in dorsal muscle and abdominal cavities of dogs, hydroxyapatite (HA) scaffolds with three different macro-pore sizes of 500-650, 750-900 and 1100-1250  $\mu\text{m}$  were prepared via sugar spheres-leaching process, which also had similar interconnecting structure determined by keeping the d/s ratio of interconnecting window diameter to macro-pore size constant. The permeability test showed that the seepage flow of fluid through the porous scaffolds increased with the increase of macro-pore sizes. The cell growth in three scaffolds was not affected by the macro-pore sizes. The in vivo ectopic implantation results indicated that the macro-pore sizes of HA scaffolds with the similar interconnecting structure have impact not only the speed of osteogenesis and angiogenesis but also the space distribution of newly formed bone. The scaffold with macro-pore sizes of 750-900  $\mu\text{m}$  exhibited much faster angiogenesis and osteogenesis, and much more uniformly distribution of new bone than those with other macro-pore sizes. This work illustrates the importance of a suitable macro-pore sizes in HA scaffolds with the similar interconnecting structure which provides the environment for ectopic osteogenesis and angiogenesis.

**Keywords:** Hydroxyapatite Scaffolds, Similar Interconnecting Structure, Osteogenesis, Angiogenesis



# **ELECTRICAL AND THERMAL PROPERTIES OF MONO AND BI-FILLER PLA COMPOSITES FILLED WITH GRAPHENE AND MWCNTS**

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## **ABSTRACT**

In this study, structure, electrical and thermal properties of ten polymer compositions based on poly(lactic acid), combining different proportions of graphene and multi-walled carbon nanotubes in mono and bi-filler systems with 1.5 to 6 wt.% filler content, were investigated. The obtained results have shown that at maximum filler content, for the mono-filler systems with MWCNT and graphene, the electrical conductivity increased almost 7-8 decades. On the other hand, some of the bi-filler composites have shown a synergetic effect on electrical conductivity with values higher than observed for the mono-filler systems. Thermal conductivity increases with higher filler content and this effect was more pronounced for the mono-filler composites based on PLA and graphene. The obtained composites are suitable for the production of multifunctional filament with improved electrical and thermal properties for different 3D printing applications. Acknowledgments: This work has received funding from the European Union's Horizon 2020-MSCA-RISE-734164 Graphene 3D Project.

**Keywords:** Biodegradable Polymers, Graphene, Carbon Nanotubes, Nanocomposites, Electrical and Thermal Properties

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## SILICONE RUBBER FOAM FOR WOUND DRESSING

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### ABSTRACT

Due to limited availability of autologous skin and inevitable rejection of allogenic skin [1], wound dressings used as temporary skin substitutes are believed to be essential during the treatment of extensive or chronic wounds [2]. To effectively accelerate wound healing, an ideal wound dressing should be a multifunctional device, which possesses appropriate porous structure, suitable mechanical property, satisfactory water vapor transmission rate (WVTR), excellent biocompatibility, effective antimicrobial activity, and so forth [3]. Silicone rubber, especially Polydimethylsiloxane (PDMS), as a commonly used polymer material, exhibits lots of extraordinary properties, including relatively low price, softness, transparency, outstanding physical properties, chemical stability, and high gas permeability [4].

The unique porous polymeric membranes with highly ordered micropores were fabricated using the phase separation method. In this way, we prepared porous silicone rubber membranes incorporated with reduced graphene oxide (rGO) sheets and novel bilayer wound dressing composed of porous silicone rubber membranes. The designed silicone rubber membrane with particular pore size and thickness achieved good physical property, adequate WVTR, good anti-infective property, good biocompatibility and capability for cell adhesion and proliferation, and was able to promote wound healing.

**Keywords:** Silicone Rubber, Polydimethylsiloxane (PDMS), Wound dressing

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# EFFECT OF BIO-POLYOLS AND BIO-FILLERS ON FOAMING PROCESS AND PROPERTIES OF POLYURETHANE FOAMS

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## ABSTRACT

The trends observed in industry, legislative requirements and limited crude oil resources, force the producers of polyurethane (PUR) materials to use raw materials derived from renewable sources. PUR foams are usually synthesized from two-component systems, (i) a polyol premix and (ii) an isocyanate. The polyol premix, apart from hydroxyl compounds, contains also catalysts, surfactants, blowing agents and other additives as fillers, flame retardants, etc. [1-5]. Among various ingredients of PUR systems, bio-polyols and bio-fillers are currently most often tested to increase the sustainability of foamed products [1].

Rigid PUR foams are widely used as heat insulating materials, because of their combination of low density, low thermal conductivity and good mechanical properties. Nowadays, rigid PUR foams are the most effective heat insulating materials among commercial products used in refrigerating and building industry. Their thermal conductivity mainly depends on the type of gas closed in cells, as well as on dimensions and shape of cells. Generally, bio-polyols can significantly influence on the cell structure and physical-mechanical properties of obtained foams. The use of natural and cheap fillers allows reducing prices and can improve cellular structure and selected properties of final foams [2].

This talk will be focused on the analysis of various effects of used bio-based components on the foaming process, as well as cell structure and selected properties such as apparent density, thermal and mechanical properties of modified PUR foams.

**Keywords:** Bio-polyols, Bio-fillers, Foaming Process, Rigid Polyurethane Foams,

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# **FABRICATION OF PLA FOAM WITH HIGH CRYSTALLINITY AND THE IMPROVED HEAT RESISTANCE**

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## **ABSTRACT**

In the past decades, polylactide (PLA), a kind of biodegradable aliphatic polyester, has been attracting significant attention, because it is synthesized on the basis of annually renewable resources. Unfortunately, PLA exhibits a low service temperature, due to its low  $T_g$  and low crystallization ratio. Therefore, it is critical to increase its heat resistance for broadening its applications. Microcellular foaming technology using supercritical or compressed CO<sub>2</sub> as the physical blowing agent has been verified as a novel approach to increase the crystallinity of PLA, where the as-obtained PLA foams possess high crystallinity of about 32%, well defined cell morphology, and high expansion ratio of 5-20 times. Unfortunately, once the induced crystallinity of PLA increases continually, the cell growth and the foam expansion during the foaming are restrained, resulting in poor cell morphology and low expansion ratio. In this presentation, I am going to presents our current research progress about the improved foaming behavior and the increased heat resistance of PLA foams with the strategies of crystallites adjustment, polymer blending, and nanoparticle compounding.

# COMPOSITE AEROGEL MICROSPHERES WITH ENHANCED BIOACTIVITY

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## ABSTRACT

Periodontitis is a chronic inflammatory dental disease leading to the destruction of both soft and hard tissue. Current treatments such as bone graft, barrier membranes and others have shown limitation in regenerating this complex tissue. Hydroxyapatite (HA) is a synthetic biomaterial and has been found to promote new bone formation when implanted in a bone defect site. However, its use is often limited due to its slow osteointegration rate and without antibacterial activity, particularly where HA has to be used for long term biomedical applications. The aims of this work is focused on the development of aerogel microspheres acting as bioactive systems to overcome all the complex events present in periodontitis. Here, it will describe the development of zinc substituted HA (ZnHA) as an alternative biomaterial to HA. Zn, as the most abundant trace metal in bone mineral, is an essential element that has stimulatory effects on bone formation *in vitro* and *in vivo* as well as inhibitory effects on osteoclastic bone resorption *in vivo* [1]. Furthermore, Zn shows good bioactivity and antibacterial properties. Here, we propose the development of aerogel microspheres based on Zn-modified hydroxyapatite/graphene oxide obtained by the combination of sol-gel approach [2] and electro spraying combined with fast-freezing technology. This combination allows to obtain porous microspheres which can be used as suspension or introduced as multifunctional signal in multifunctional biocomposites materials. The Zn-HA/GO microspheres show bioactive and biocompatible properties able to induce cell proliferation and osteogenic differentiation in basal medium. Furthermore, biocomposites microspheres reduce the expression of pro-inflammatory cytokine IL-1B and show antibacterial properties against Gram – positive and negative bacteria.

**Keywords:** Aerogel Microspheres, Graphene Oxide, Sol-Gel Method, Electrospray Technology, Antimicrobial

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# FOAM FLOW PHENOMENA IN NARROW CHANNELS WITH CONVERGING-EXPANDING CONSTRICTIONS

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## ABSTRACT

Gas-liquid foams enjoy numerous industrial applications including food, consumer goods, oil recovery, foamed concrete, firefighting and mineral processing. They are compressible, non-linear viscoelastic materials; their viscosity is much larger than the liquid constituent, and they often show an apparent yield stress (plasticity). Fundamental understanding of foam properties and flow dynamics is therefore limited and many issues remain unaddressed/unresolved. In particular, foam flow through narrow complex passages can have detrimental effects on foam structure and rheology. Some of the examples include flow of aerated confectionary in narrow channels and complex moulds, filling of cavities with insulation foam, flow of foamed cement slurries in narrow oil-well annuli, filling of hollow aerofoil sections with polyurethane foam to make aerodynamic tethers for communication and geoengineering applications, and production of pre-insulated pipes. These complex geometries can have significant effects on flow regime and may destabilize foam structure, giving important morphological transformations.

In this paper, we report on a fundamental study of the flow of dry gas-liquid foam through two-dimensional narrow channels containing sudden or gradual contractions and expansions. Foams with formulations of varying degrees of complexity (interfacial properties, liquid rheology) are studied. A full analysis of foam flow and rheology through the 2D channels with constrictions is obtained and related to foam structure, physical properties, and any instabilities or perturbations observed therein. Foam behavior is examined as a function of flowrate, initial bubble size and flow constriction geometry. The effects on the entire flow field including foam morphology, liquid holdup, pressure drop and velocity profile are analysed. We examine individual bubble motion and deformation using a “texture tensor”. We, thus, explore the parameter space to obtain the local strain and stress fields and determine transition from elastic to plastic deformation.

**Keywords:** Foam Flow, Protein Foams, Constrictions, PIV, Topology

# **INFLUENCE OF PLA STEREOCOMPLEX CRYSTAL ON THE PLA-BASED BLEND AND ITS MICROCELLULAR FOAM**

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## **ABSTRACT**

The introduction of PDLA on the rheological properties, crystallization behavior and dynamic mechanical properties of the PLLA matrix were investigated. The formed PLA stereocomplex between PLLA and PDLA enhanced the storage modulus and complex viscosity of PLLA/PBAT blends efficiently. However, the addition of PDLA in the PLLA/PBAT blends was unfavorable for the PLLA crystallization behavior. Both the enhanced melt strength and decreased crystallinity of the PLLA matrix are favorable for the cell nucleation and growth and the gas adsorption, respectively. The designed partially foaming of PLLA/PBAT with or without PDLA was carried out to investigate the foaming mechanism. The final cell morphology of PLLA/PBAT foams exhibited typical open-cell structure mainly attributed to the soft immiscible PBAT phase as separated domains. With further addition of PDLA in the PLLA/PBAT blends, the microcellular morphology exhibited decreased average cell size and increased cell density. The sc-PLA crystallites networks in the PLLA matrix acted as cell nucleating agents, which meanwhile resisted the force of cell growth and then prevented the cell collapse.

**Keywords:** PLA, Stereocomplexation, Supercritical Foaming

## **FLAME RETARDANT PP FOAMS USING SUPERCRITICAL CO<sub>2</sub> EXTRUSION FOAMING**

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### **ABSTRACT**

Polypropylene (PP) foams have been gradually used in electrical appliances, automobiles, buildings due to the good properties and the weight reduction. However, PP shows burning characteristics of easy flammability accompanied by severe dripping. Hence, enhancement of flame retardancy of PP foams is of great importance to meet the fire safety standards and thus to expand the applications. Intumescent flame retardant (IFR) has been extensively investigated on flame retardant PP due to its low smoke, low toxicity and anti-dripping, etc. However, the dispersion of IFR in PP is usually poor due to the bad compatibility, which results in a low efficiency of the flame retardant.

Incorporating synergistic agents is the common means to improve the efficiency of flame retardants and the flame retardant performance. In this study, without incorporating the synergistic agents, the efficiency of flame retardants was highly improved via scCO<sub>2</sub> continuous foaming to improve the dispersion of intumescent flame retardants (IFR) in PP matrix. A low loading of 23 wt% IFR allowed the PP composites to pass UL-94 V-0 rating, which usually requires no less than 30 wt% for the conventional approaches. Based on scCO<sub>2</sub>-assisted IFR dispersion, flame retardant PP foams were obtained that can pass HF-1 ratio in the horizontal burning test for foams. This application combines the characteristics of pilot-scale production of continuous extrusion and environmental friendliness, easy removal and low cost of scCO<sub>2</sub>. Therefore, it has demonstrated high potential to fabricate flame retardant PP foams via supercritical CO<sub>2</sub> extrusion foaming of PP foams.

# **HIGH-PERFORMANCE PLA AND ITS FOAMS ENNABLE BY CARBON DIOXIDE TREATMENT AND FOAMING**

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## **ABSTRACT**

Poly(lactic acid) (PLA) is a biodegradable and bioactive thermoplastic aliphatic polyester derived from renewable resources. As an environmentally friendly materials, it shows a promising future in replacing oil-based plastics such as polypropylene (PP), polystyrene (PS), and acrylonitrile-butadiene-styrene (ABS). Microcellular foaming can not only save materials and reduce weight, but also gives polymers new functionalities such as insulating and absorbing properties. However, PLA has poor crystallization ability, which leads to its poor mechanical properties and poor foaming ability. In this report, we presented some environmentally friendly methodologies for improving PLA's mechanical properties and foaming ability. For mechanical properties, CO<sub>2</sub> treatment at normal temperatures leads to simultaneously enhanced strength, rigidity and toughness of PLA. Notably, the toughness was enhanced by more than 10 times. For microcellular foaming, a pre-isothermal treatment was found to be very effective in improving the foaming ability, and low-density pure PLA foams with a 17.7-fold expansion ratio and an average cell size of less than 20  $\mu\text{m}$  can be fabricated. Finally, a sustainable foam injection molding-based method was introduced for efficiently fabricating renewable microcellular PLA/graphite nanocomposite foams, with improved mechanical and electrical properties for ultra-efficient EMI shielding applications. The microcellular PLA/graphite nanocomposite foam, with a density of 0.7 g/cm<sup>3</sup> and a thickness of 2.0 mm, exhibits an outstanding EMI shielding performance with a total electromagnetic interference shielding effectiveness (EMI SE) of up to 45 dB.

# HYDROXYL DERIVATIVES BASED ON USED COOKING OILS AS COMPONENT FOR SYNTHESIS OPEN CELL RIGID POLYURETHANE FOAMS

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## ABSTRACT

According to the 7th Environment Action Programme (EAP), which is guide for European Environment Policy until 2020, one of three key objectives is to turn the European Union into a resource-efficient, green, and competitive low-carbon economy.

In our research we focus on the analysis of chemical structure of various used cooking oils as potential resource of bio-components, which could be transform to hydroxyl derivatives. Due to the high content of unsaturated fatty acids confirmed by iodine value ca. 100 gI<sub>2</sub>/100g, it is possible to functionalize such oils to hydroxyl derivatives using different methods. In our work, cooking oils were epoxidized and hydroxylated using diethylene glycol.

The obtained hydroxyl derivatives were used to synthesize open cell rigid polyurethane foams. In the modified formulation, a petrochemical polyol was replaced by the oil based hydroxyl derivatives up to 100%. The effect of more beneficial cellular structure (Fig.1) as well as better thermomechanical properties for the foams modified with bio-polyols was observed. The detailed characteristic of open cell polyurethane foams and hydroxyl derivatives obtained will be presented.

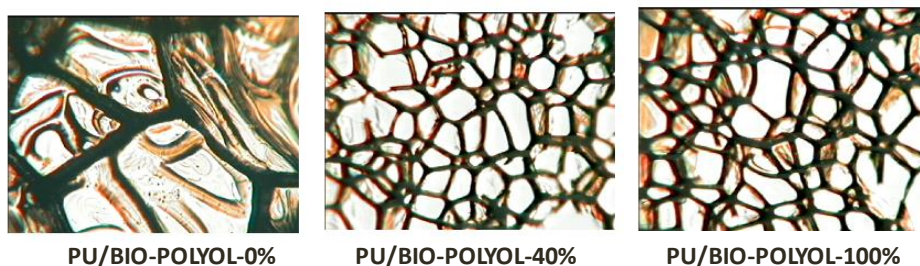


Fig. Cellular structure of reference foams (PU/BIO-POLYOL-0%) and foams modified with 40 and 100% of bio-polyol (PU/BIO-POLYOL-40% and PU/BIO-POLYOL-100%, respectively).

**Keywords:** Used Cooking Oils, Polyols, Epoxydation, Open Cell Rigid Polyurethane Foams

**Acknowledgments:** The research leading to these results has received funding from the National Centre for Research and Development in Poland in the frame project no. LIDER/28/0167/L-8/16/NCBR/2017 „Development of technologies of feedstock recycling of used cooking oils and use of hydroxyl bio-components derived for production of innovative high-efficiency polyurethane insulating materials“



# LIGHTWEIGHT, COMPRESSIBLE AND ELECTRICALLY CONDUCTIVE FOAMS COATED WITH SYNERGISTIC MULTIWALLED CARBON NANOTUBES AND GRAPHENE FOR PIEZORESISTIVE SENSORS

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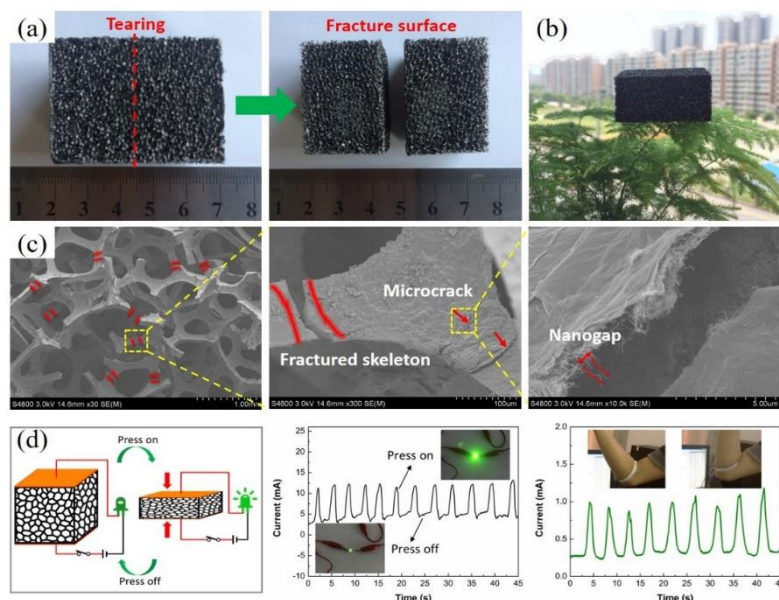
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## ABSTRACT

Lightweight, compressible and high sensitive pressure/strain sensing materials are highly desirable for the development of health monitoring, wearable devices and artificial intelligence. Herein, a very simple, low-cost and solution-based layer-by-layer (LBL) electrostatic assembly approach followed by in-situ reduction is presented to fabricate versatile piezoresistive sensors based on conductive polyurethane (PU) foams coated with synergistic multiwalled carbon nanotubes (MWCNTs) and graphene. The resultant conductive MWCNT/RGO@PU foams exhibit very low densities ( $0.027\text{--}0.064\text{ g/cm}^3$ ), outstanding compressibility (up to 75%) and high electrical conductivity benefiting from the porous PU foams and synergistic conductive MWCNT/RGO structures. The MWCNT/RGO@PU foams present larger relative resistance changes and superior sensing performances under the external applied pressures ( $0\text{--}5.6\text{ kPa}$ ) and large-range strains ( $0\text{--}75\%$ ). Fully functional applications of the MWCNT/RGO@PU foam based piezoresistive sensors in lighting of LED lamp and detecting human body's movements indicate the excellent potential for emerging applications such as health monitoring, wearable devices and artificial intelligence.



**Fig.** Lightweight, compressible and electrically conductive MWCNT/RGO@PU foams for piezoresistive sensors

# BIODEGRADABLE PLA-BASED BLEND FOAMS FOR OIL-WATER SEPARATION

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## ABSTRACT

Effective removal of oils from water is of global significance for environmental protection. In this work, a new strategy was proposed for preparing biodegradable PLA-based blend foams used for oil-water separation. Biodegradable PLA/TPU (90/10, 70/30 and 50/50 w/w) and PBS/PLA (93/7, 85/15 and 80/20 w/w) blend samples were prepared using a single-screw extruder. The samples were then foamed using supercritical carbon dioxide as physical foaming agent. The cellular structure, hydrophobic-oleophilic characteristics and oil sorption capacity of the foamed samples were investigated. It was demonstrated that the PLA/TPU and PBS/PLA blend foams exhibit higher porosities for oil storage as well as good hydrophobic-oleophilic characteristics and oil-water separation property. The foamed PLA/TPU (90/10) blend sample has higher oil-sorption capacities [29.9 g/g (cyclohexane)], which is attributed to its higher porosity and more obvious effects of swelling. The foamed PLA/TPU (50/50) sample has better reusability, which is attributed to the better resilience of the blend. The foamed PBS/PLA samples also exhibit higher oil sorption capacities.

# BIOINSPIRED MATERIALS FOR HARD TISSUE REPAIR: FROM FUNDAMENTAL RESEARCH TO INDUSTRIALIZATION

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## ABSTRACT

It is very important to manipulate the surface properties of biomaterials for their biomedical applications. In recent years, we report a series of rapid and universal methods for the preparation of multifunctional coatings on biomaterials using a salivary acquired pellicle (SAP) inspired strategy. For example, we prepared a DDDEEK peptide conjugated dendrimer. It mimics the adsorption function of statherin, which is one of the main components of SAP, to endow the coating with a universal capability for adhesion on various biominerals such as hydroxyapatite, tertiary calcium phosphate, calcium carbonate, pearls, enamel, dentin, and bone. The strategy can also be applied to manufacture a mineralization coating on various surfaces. The amyloid protein (CsgA) is the main proteinaceous component in the Escherichia coli (E. coli) biofilm, which can withstand detergents in the harsh environment. The bioinspired coating was successfully secreted by the engineered E. coli, which was transformed with recombinant PET-22b-CsgA-DDDEEK plasmid. The uniform coating could bear shear force and stay on virtually any type of material surface for at least one month. The animal experiment results suggested that the coated Ti6Al4V screws with induced HA presented better osteogenicity and osseointegration than HA-sprayed screws after 12 weeks, as well as no extra immunogenicity.

**Keywords:** Salivary Acquired Pellicle, Hard Tissue, Multifunctional, Mineralization, Anti-bacterial.

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# OPTIMIZATION OF THE PERFORMANCES OF CHITOSAN/GRAPHENE OXIDE AEROGELS FOR WATER PURIFICATION

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## ABSTRACT

The removal of synthetic dyes from wastewater streams is an important challenge for the scientific community. Adsorption of pollutants is considered one of the most feasible and low-cost approaches for water purification. In this context, chitosan (CS) is largely investigated as environmentally friendly material for adsorption of anionic dyes, thanks to the favourable electrostatic interactions occurring among its amine and hydroxyl groups and negatively charged molecules [1]. On the other hand, its effectiveness towards cationic dyes is rather scarce. Different approaches are currently pursued for the realization of CS-based adsorbents that are able to simultaneously remove anionic and cationic pollutants from wastewater. In particular, the use of graphene oxide (GO) in combination with chitosan is greatly promising. The present work addresses the optimization of the preparation procedure of CS/GO aerogels for dye removal. The material performances have been tailored by tuning the mixing method, the crosslinking strategy, and the freezing conditions in order to obtain highly adsorbing and mechanically resistant nanocomposite aerogels.

**Keywords:** Chitosan, Graphene Oxide, Aerogel, Dye Removal; Mechanical Properties

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# **PREPARATION OF HIGHLY POROUS INTERCONNECTED POLY( $\epsilon$ -CAPROLACTONE) SCAFFOLDS COMBINED WITH SUPERCRITICAL CO<sub>2</sub> FOAMING AND POLYMER LEACHING**

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## **ABSTRACT**

In recent years, tissue engineering, as one new treatment, has been used to improve the recovery of various types of tissues. The application of vascular tissue engineering technology is considered to be great promise for improving outcomes in patients with peripheral vascular diseases and cardiac ischemia. At present, there is still a challenge to generate vascular grafts with small diameter ( $\leq 6\text{mm}$ ) since risk of infection, thrombus and calcification when implanted. In order to fulfill the requirements of tissue engineering, an ideal tissue scaffold should contain suitable pore sizes, homogeneous pore distribution and highly interconnected porous structures. These structures can not only facilitate cell adhesion, migration and growth into the interior of scaffold, but they can also provide mechanical support and transportation of cell nutrients and waste. Highly porous interconnected poly( $\epsilon$ -caprolactone) (PCL) scaffolds combined with supercritical carbon dioxide foaming and a polymer leaching process were fabricated by blending PCL with water-soluble poly(ethyleneoxide) (PEO) as a sacrificial material in this report. The effects of foaming conditions and the phase morphology of blend on foaming behavior and pore morphology were investigated. Outer porosity and mechanical properties of porous tubular scaffolds have also been taken into account. This allows the extension of their fields of application to vascular tissue engineering.

**Keywords:** Poly( $\epsilon$ -caprolactone), Supercritical Foaming, Leaching, Pore Morphology, Porous Scaffold

# ON THE EMBEDDING OF RGO/CHITOSAN AEROGEL IN OPEN CELL POLYURETHANE FOAMS AND THEIR PIEZORESISTIVE PROPERTIES

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## ABSTRACT

Graphene-based aerogels exhibit a plenty of outstanding properties, such as low density, high specific surface area, unique thermal, electrical, and adsorptive properties.<sup>[1-3]</sup> However, their intrinsic poor mechanical properties limit the diffusion of these materials in practical applications. In order to overcome this limitation, in this work it is proposed the embedding of the reduced graphene oxide (rGO)-based chitosan (CS) aerogel into open cell polyurethane foam (PUF) through an in-situ bidirectional freeze-drying process. The rGO/CS aerogel with conductive and parallel flat lamellas enables outstanding electrical properties, while the PUF provides excellent mechanical properties. Thus, the resulting aerogel/foam composites exhibited simultaneously excellent compression-resilience performance and stable piezo-resistive properties.

The anisotropic morphology of rGO/CS-based aerogel embedded in PU foams is shown in Figure 1 (a, b, c). In the *z*- and *y*-axis directions, the aligned rGO-based lamellas run throughout the whole cross section of the foams and provide an effective pathway for the electron conduction. While, in the *x*-axis direction, the electron conductivity is hindered by the interlayer air-gaps between the lamellas. When the aerogel/PUF composite is compressed in *x*-axis direction, new effective conductive paths form leading to a decrease in the electrical resistance. When the load/strain is gradually removed, the distance between the adjacent lamella recovers to its original value, which brings about the recovery of the initial resistance for the graphene-based aerogel/PUF composite sample, as shown in Figure 1(d, e). The aerogel/PUF composites exhibit a high sensitivity and reliable performance over a wide range strain. For instance, when a 60% strain was applied on the aerogel/PUF composite, the resistance decreases of about 85.6% as compared to the initial resistance. The unique anisotropic aerogel-foam composites are promising candidates for the production of wearable sensors and healthcare monitoring devices.

**Keywords:** Graphene-based Aerogel, Bidirectional Freeze-drying Process, Anisotropic Composites, Piezo-resistive Properties

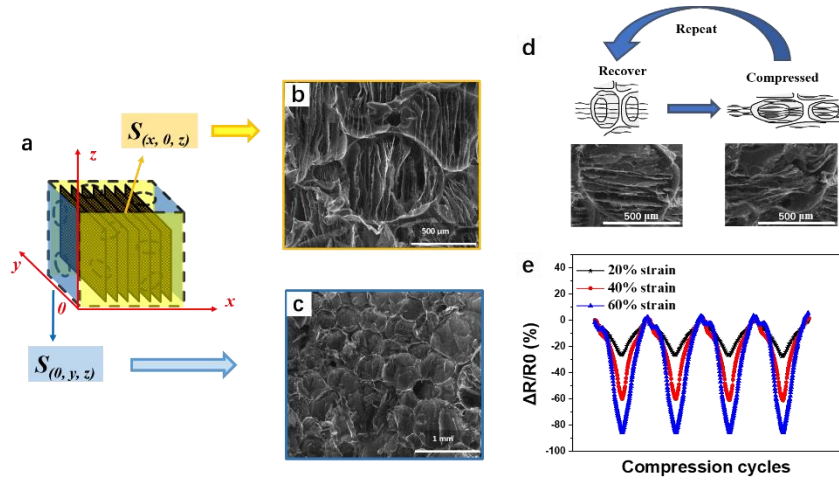


Figure 1: (a) Schematic illustration of the graphene-based aerogel/PUF composite. Aligned lamellas and flat wall structure of the graphene-based aerogel/PUF composites observed in (b)  $S_{(x, 0, z)}$  and in (c)  $S_{(0, y, z)}$ . (d) Schematic illustration to show the changes of graphene-based aerogel structures when the aerogel/PUF composite was compressed in  $x$ -axis direction. (e) Electrical resistance variations recorded in four loading/unloading cycles compressed in  $x$ -axis direction under 20%, 40%, and 60% strain.

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# A NOVEL LOW-PRESSURE FOAM INJECTION MOLDING TECHNOLOGY USING DIFFERENT TYPE OF FOAMING AGENTS

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## ABSTRACT

Recently, we have developed a resilient and innovative cellular foam injection molding (RIC-FIM) technology, which demonstrated that pressurization of physical blowing agent (PBA), such as nitrogen (N<sub>2</sub>) and carbon dioxide (CO<sub>2</sub>), was unnecessary for preparing the microcellular foams [1,2]. To completely differentiate the new foaming process from the Mucell technology, our RIC-FIM technology was further simplified by using the previously auxiliary venting unit as the delivery unit [3,4]. Herein, high void fraction plastic foams were successfully obtained using this novel RIC-FIM machine. In addition to the regular PBA including N<sub>2</sub> and CO<sub>2</sub>, synthetic air, argon and helium are also used as the novel foaming agents for producing the microcellular foams. The effect of different types of gas on the cellular structures and mechanical properties of the final foams were studied.

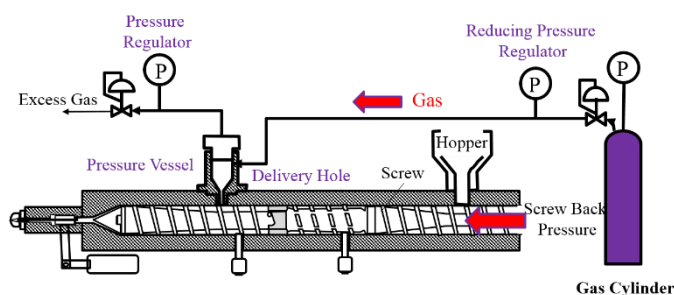


Fig. Schematic diagram of the resilient & innovative cellular foam injection molding machine.

**Keywords:** Low-pressure Gas, Foam Injection Molding, Microcellular Foams

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## BIOPRODUCTION OF CLASS II HYDROPHOBINS EXHIBITING EXCELLENT FOAM STABILITY

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### ABSTRACT

Hydrophobins (HFBs), produced by filamentous fungi, are amphiphilic cysteine-rich proteins of relatively low molecular weight showing unique physicochemical properties [1]. HFBs are considered the strongest surface active proteins from microbial origin. By self-assembling at hydrophobic-hydrophilic interfaces, HFBs form an amphiphilic membrane and consequently reduce water surface tension and stabilize foams and emulsions at low concentrations. HFBs are also highly appreciated due to their ability to change the polarity of surfaces [2]. HFBs are divided in two Classes, namely Class I and Class II HFBs, based on differences in solubility and morphology of the membranes [3]. Class II HFBs show beneficial features (elasticity) over Class I HFBs making them of significant economic value for personal care, pharmaceutical, medical and food applications [4]. However, industrial applications currently remain lacking despite their great potential. Therefore, a fed-batch design for the bioproduction of the mycelium bound HFBI, produced by *Trichoderma reesei*, was developed. A yield of 0.14 g/g mycelium bound proteins per gram dry biomass was obtained using a concentrated feed solution. Foam stabilization tests were effectuated on extracts originating from the mycelium of the fungal strain and showed promising results with R5 factors up to 97 %.

**Keywords:** Bioproduction, Hydrophobin, HFBI, *Trichoderma Reesei*

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## EXTRACTION AND SPRAY DRYING OF HFBI PRODUCED BY TRICHODERMA REESEI

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### ABSTRACT

Hydrophobins are considered as the strongest surface-active proteins from microbial origin. The low molecular weight, amphiphilic proteins are produced by filamentous fungi and exhibit unique physicochemical properties [1]. Self-assembly into membranes at hydrophobic-hydrophilic interfaces and excellent foam stability are some examples of the unique properties of hydrophobins, leading to a broad range of applications, from foam and emulsion stabilization to biosensors and protein immobilization [2]. Hydrophobins are divided into two Classes, namely Class I and Class II hydrophobins. Class II hydrophobins exhibit strong elasticity in addition to their surface-active properties and have been proposed as foam stabilizing agents for food applications [3]. HFBI is a Class II hydrophobin produced by *Trichoderma reesei*. HFBI has a prominent role in the development of fungal hyphae during growth and is known to be bound to the fungal biomass [4]. Downstream processing of HFBI requires extraction of the protein from the fungal biomass. Therefore, the protein extraction capacity of several extraction solvents was studied. Extracts of HFBI showed excellent foam stability with an R5 factor up to 97 %. HFBI extracts were also further purified using reversed-phase fast protein liquid chromatography (RP-FPLC) and subsequently, purified fractions containing HFBI were spray dried using a Buchi B-90 nano spray dryer. Addition of trehalose to stabilize HFBI during spray drying resulted in a residual activity of  $93.6 \pm 5.1$  %.

**Keywords:** Hydrophobins, HFBI, Foam stability, Spray drying

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**Acknowledgments:** The authors gratefully acknowledge Impulse Fund KU Leuven (IMP/16/030) for financial support.

# NANOCOMPOSITE FOAMS THERMAL INSULATION PERFORMANCE, NANOPARTICLES' CONNECTION AND THE THREE-DIMENSIONAL FOAM STRUCTURE

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## ABSTRACT

In polystyrene (PS)/multi-walled carbon nanotube (MWCNT) nanocomposite foam, the MWCNTs significantly decreased the radiative thermal conductivity of the foams with the high infrared (IR) absorption capability and increased the optimal expansion ratio of the foams to minimize the total thermal conductivity. A PLA foam with a thermal conductivity as low as 30 mW/m-K was also developed. The PLA foam's larger optimal expansion ratio and IR block ability greatly helped to achieve this outcome. The PLA foams' intrinsic IR-absorbing characteristic, which acted via the ester group in the PLA molecular chain, further enhanced its environmental impact.

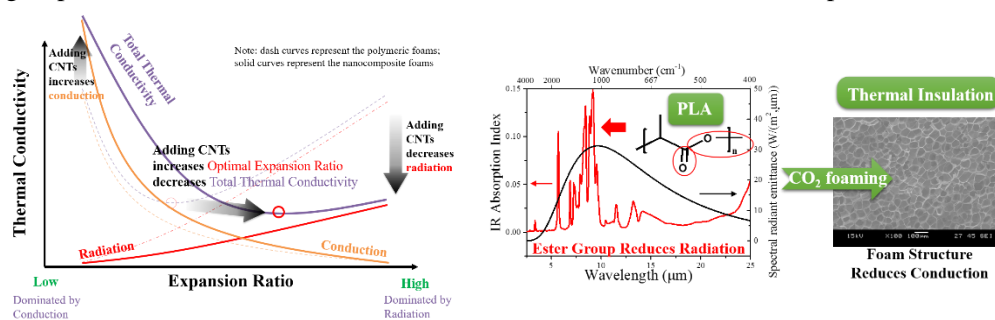


Fig. Thermal conductivity with expansion ratio, and radiation behavior

**Keywords:** MWCNTs, IR Absorption Index, optimal expansion ratio, bimodal nanocomposite foam, thermal insulation

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# CYCLODEXTRIN-BASED METAL ORGANIC FRAMEWORKS FOR DRUG DELIVERY

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## ABSTRACT

Gasotransmitters are essential signaling molecule that are endogenously produced in humans and play an important role in physiology as well as show great potential in pharmaceutical applications. For instance, Sulfur dioxide (SO<sub>2</sub>) is an important signalling molecule, performing functions in anti-oxidation, anti-mycobacteria, anti-atherosclerosis and anti-hypertension etc.<sup>1,2</sup> In this study, we developed nanometer-sized cyclodextrin metal-organic frameworks (CD-MOFs) as carriers for the delivery and control release of SO<sub>2</sub>. The results showed that CD-MOFs had good SO<sub>2</sub> adsorption capacity and were able to release SO<sub>2</sub> spontaneously in physiological environment. The release rate could be tuned by modified CD-MOFs with pluronic.

**Keywords:** Cyclodextrin, Metal Organic Framework, Drug Delivery

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# MANIPULATION OF PLA FOAM STRUCTURES BASED ON CO<sub>2</sub>-INDUCED CHANGES IN PLA FUNDAMENTAL PROPERTIES

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## ABSTRACT

Regulation of the poly(L-lactic acid) (PLLA) foam through nanofillers probably sacrificed its biodegradable and biomedicine application and simultaneously encountered the ticklish loose interface bonding. In order to avoid these problems and meantime increase the heat resistance and mechanical properties, the spherulites were in situ created under CO<sub>2</sub> to control the morphology and structure of PLLA foam. It was found that cells could nucleate and grow in the interlamellar amorphous regions of the spherulites. At 70-80 oC, a large number of nanocells were developed due to both the heterogeneous nucleating effect and the strong constraint imposed by the neighboring lamellae. By increasing the foaming temperature to 90 oC and above, the viscosity of the amorphous regions was considered to decrease, leading to the further growth of nanocells or the cell coalescence. However, nanocells actually grew along the radial direction of spherulites instead of freely due to the lamellar constraining. With further increasing the foaming temperature to 115 oC, the crystallinity of PLLA decreased a lot. Cells nucleated in the large amorphous regions could grow into microcells with less restriction, whereas those in the rest crystalline regions still evolved into nanocells with much limitation.

**Keywords:** PLA, CO<sub>2</sub>, Crystallization, Foaming

# HYALURONIC ACID-BASED COMPOSITE HYDROGELS FOR TISSUE ENGINEERING

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## ABSTRACT

Hyaluronic acid is widely used for numerous medical applications, such as viscosupplementation, eye surgery and drug delivery [1]. In this study, hyaluronic acid sodium salt (HAs) was chemically modified in order to obtain a photocrosslinkable hydrogel with tailored mechanical properties. Composite materials based on modified hyaluronic acid (i.e. methacrylated, maleated) and hydroxyapatite nanoparticles were developed by in situ sol-gel approach. The process is a versatile procedure which ensures a more controlled and homogenous distribution of nanofillers in the polymer matrix and a suitable approach for incorporating biologically active compounds [2]. Furthermore, to obtain anti-inflammatory properties, composite materials were loaded with a non-steroidal anti-inflammatory drug such as Diclofenac Sodium (DS). Biological analyses on macrophage cell line were performed to investigate the effect of composite materials, with and without DS, on expression of pro, anti-inflammatory and chemotactic cytokines such as tumor necrosis factors and interleukins at different time points of cell culture. The results demonstrated that the chemical modification of HA showed a strong effect on drug release and the presence of DS in composite hydrogels reduced TNF- $\alpha$  release, stimulating the production of an important anti-inflammatory cytokine such as IL-10. Moreover, the chemical modifications allow hydrogels to be 3D printed, through stereolithography technology.

**Keywords:** Hyaluronic Acid, Composite Materials, Sol-gel Method, Cellular Behaviour, Tissue Engineering

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# INFLUENCE OF CARBON NANOTUBES AND GRAPHENE ON THERMAL AND ELECTROMAGNETIC PROPERTIES OF PLA NANOCOMPOSITES

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## ABSTRACT

Nanocomposites with graphene (GNP) and MWCNTs (supplied by Times Nano, China) in polylactic acid (PLA), from Nature Works, USA were prepared by melt extrusion (ME), at IPCB-CNR, Pozzuoli. Various formulations of monofiller composites (GNPs/PLA and MWCNT/PLA) and bi-filler composites GNP/MWCNT/PLA at filler contents 0-6 wt% were produced based on Robust Design pre-planning. Electromagnetic properties in GHz and THz wave range were measured by TA Instruments, and the thermal properties DSC and TGA were measured.

It was found that in the microwave range, 32-33 GHz, the EMI absorption is ~ 33% for 6wt% MWCNT/PLA (ME) and ~ 38% for the 6wt% GNP/PLA (SB), with electromagnetic shielding efficiency of 87%. and 76% respectively. In the THz wave range (0.3 THz), the GNP/PLA composites demonstrate much better absorption ability, with electromagnetic shielding efficiency 92%, then the MWCNT/PLA (62%). The higher aspect ratio of GNP and MWCNTs, the higher absorption is achieved for the nanocomposites. Interestingly, in the THz wave range the bi-filler composites of 6wt% GNP/MWCNT/PLA show very high EMI shielding efficiency (97-100%), with absorption ~ 49%, compared to the monofiller composites at 6wt% filler content [1].

The DSC analysis of the 'as received' samples (1<sup>st</sup> heating run) shows the glass transition (T<sub>g</sub>) around 66-68°C, followed by cold crystallization (T<sub>cc</sub>) with significant enthalpy ( $\Delta H_{cc}$ ), than second cold crystallization (T<sub>cc2</sub>) and melting (T<sub>m</sub> = 178°C), due to  $\alpha'$  –  $\alpha$  phase transition. The 6% MWCNT/PLA has low % crystallinity (around 10%), similar to the neat PLA.

During the second heating, the cold crystallization peak was not observed for all Nanocomposites. Only neat PLA show cold crystallization peak at T<sub>cc</sub>=110°C. Sharp endothermic peak appear for melting around T<sub>m</sub> = 178°C. The results confirm that, the reprocessing of Nanocomposites at 200°C increases strongly the %crystallinity (to 40-43%), compared to the neat PLA (10%).

The TGA analysis show, that the thermal stability of PLA polymer is improved by addition of 6% MWCNTs and GNP. Thus, the peak of degradation increases with ~ 7°C from pure PLA to 6 wt.% MWCNT. The T<sub>onset</sub> (initial degradation) also increases with ~ 8°C from pure PLA to 6wt%GNP. Residue ash increases with 7% from pure PLA to 3wt%GNP/3wt%MWCNT.

**Keywords:** Biodegradable Poly(lactic acid) Nanocomposites, Graphene, Carbon Nanotubes, Electromagnetic Properties, Thermal Stability

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## Organic Solvent Free Preparation of Porous Scaffolds Based on the Phase Morphology Control in Supercritical CO<sub>2</sub>

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### Introduction



Fig.1 Coarsening behavior of co-continuous polymer blends

scaffold, acting as a substrate for cell growth, is especially essential to tissue engineering. At present, most preparation methods have their own shortcomings such as poor interconnectivity and residual organic solvent. In this work, a combination of supercritical CO<sub>2</sub> treatment and polymer blending was proposed to solve mentioned problems.

### Results and discussion

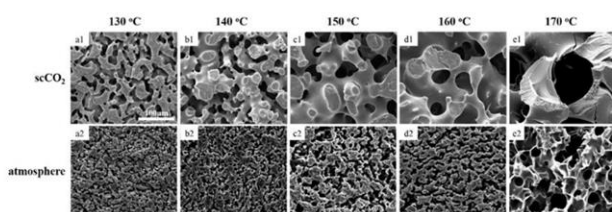


Fig.2 The effect of temperature on morphology.

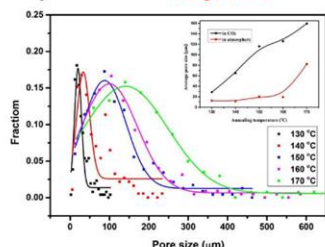


Fig.3 Distribution and average pore size at different CO<sub>2</sub> temperatures.

The pore size and distribution of porous PLA scaffolds increase with CO<sub>2</sub> temperature.

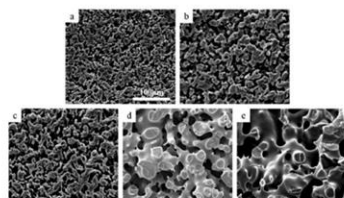


Fig.4 The effect of pressure on morphology.

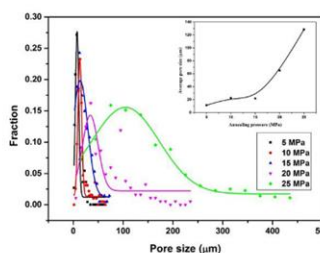


Fig.5 Distribution and average pore size under different CO<sub>2</sub> pressures.

The pore size and distribution of porous PLA scaffolds increase with CO<sub>2</sub> pressure.

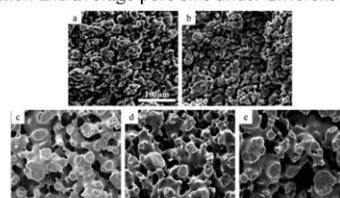


Fig.6 The effect of annealing time on morphology.

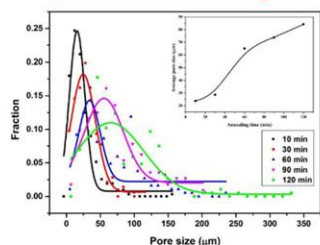


Fig.7 Distribution and average pore size at different annealing time.

The pore size and distribution of porous PLA scaffolds increase with annealing time.

### Conclusion

- Coarsening behavior occurred in the phase area of PLA/PVP blends after scCO<sub>2</sub> annealing treatment.
- The adjustment of scCO<sub>2</sub> condition could be used as an effective tool for controlling the morphology of the polymer phase.

### Acknowledgements

This work is supported by the National Natural Science Foundation of China (51373103 and 51721091) and the Science and Technology Department of Sichuan Province, China (2015HH0026).



## Polydimethylsiloxane incorporated with reduced graphene oxide (rGO) sheets for wound dressing application: Preparation and Characterization

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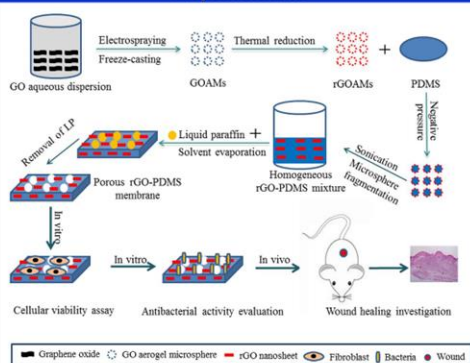
## Introduction

Graphene has been displaying a huge application prospects in the field of biomedical<sup>[1]</sup> because of its good biocompatibility, non-toxic and antimicrobial properties. Silicone rubber, as a widely used biomedical material<sup>[2,3]</sup>, has good thermal stability, chemical stability and biocompatibility. Toward fabricating a novel multifunctional wound dressing material, we prepare the porous PDMS membranes incorporated with reduced graphene oxide (rGO) sheets. We filled the PDMS into the graphene aerogel microspheres using the negative pressure method<sup>[4]</sup>. The coating of the graphene sheets by the PDMS molecules prevented the agglomeration of the graphene sheets during the ultrasonic dispersion process.

The rGO-PDMS composite membrane exhibited bionic performance (ordered pore structure and suitable WYTR), improved mechanical properties, good compatibility and effective antibacterial activity. *In vivo* experiment indicated that the rGO-PDMS composite membrane could accelerate wound healing via enhancement of the re-epithelialization and granulation tissue formation. These findings suggest that rGO doping PDMS uniquely resulted in a multifunctional material for potential use in wound dressing.

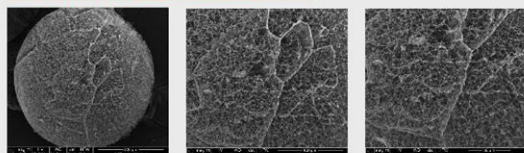


## Experimental

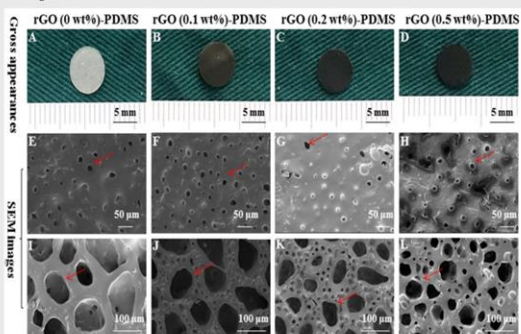


## Results and discussion

### 1. Morphology characterization



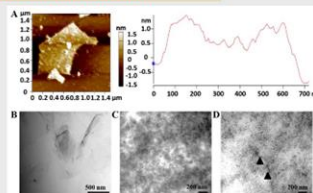
**Fig 1.** SEM images of graphene aerogel microspheres made from a GO concentration of  $6.5 \text{ mg mL}^{-1}$ .



**Fig 2.** Representative gross appearances (A-D) and SEM images (E-I) of the rGO-PDMS composite membranes. E-H: Top surface, with an average pore size of 18.64  $\mu\text{m}$ , 17.26  $\mu\text{m}$ , 15.19  $\mu\text{m}$  and 15.10  $\mu\text{m}$ , respectively; scale bar, 50  $\mu\text{m}$ . I-L: Bottom surface, the main pores with an average pore size of 78.56  $\mu\text{m}$ , 72.60  $\mu\text{m}$ , 68.74  $\mu\text{m}$  and 61.73  $\mu\text{m}$ , respectively; scale bar, 100  $\mu\text{m}$ .

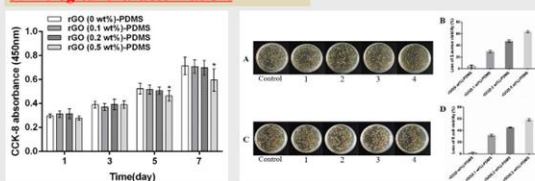
With increasing content of rGO, the average pore size of both the bottom and the top surfaces is decreased. Meanwhile, the pore sizes of the top surfaces are significantly smaller than those of the bottom surfaces. The pores had an independently cellular or spherical structure instead of a continuous structure.

## 2. Dispersion characterization.



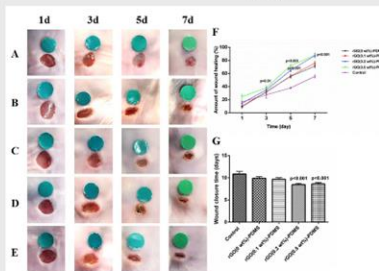
**Fig. 3.** Representative AFM image of the rGO sheets (A), TEM images of the rGO sheets (B), rGO (0 wt%)-PDMS (pure PDMS) (C) and rGO (0.5 wt%)-PDMS composite membranes (D). Triangles indicate well-dispersed rGO sheets; scale bar, 500 nm and 200 nm.

### 3. Biological characterization.

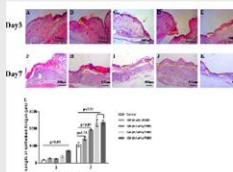


**Fig 4.** The cellular viability detected by CCK-8 assay (OD<sub>450</sub>) at each set time point. \*  $p < 0.05$ . The values are shown as the means  $\pm$  SD (n=3).

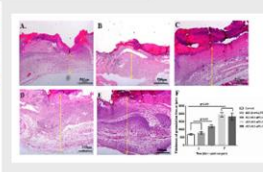
**Fig 5.** Antibacterial activity of PDMS incorporated with different concentrations of rGO (A) Photographs of bacterial colonies formed by *S. aureus* cells; (B) Inactivation of *S. aureus* bacteria; (C) Photographs of bacterial colonies formed by *E. coli* cells (D) Viability loss of *E. coli* bacteria.



**Fig 6.** Representative macroscopic appearance of the wounds: (A) Control group; (B) rGO (0 wt%)-PDMS group; (C) rGO (0.1 wt%)-PDMS group; (D) rGO (0.2wt%)-PDMS group; (E) rGO (0.5 wt%)-PDMS group; (F) Wound healing curves; (G) Wound closure time. The values are shown as the means  $\pm$  SD (n=5).



**Fig 7.** Representative histological images of the length of the newly formed epithelium tongue at days 3 and day 7 post-surgery in the (A, I) control, (B, H) rGO (0 wt%)-PDMS, (C, J) rGO (0.1 wt%)-PDMS, (D, J) rGO (0.2 wt%)-PDMS and (E, K) rGO (0.5 wt%)-PDMS groups.



**Fig 8.** Representative histological images of the granulation tissue thickness at day 7 post-surgery in the (A) control, (B) rGO (0 wt%)-PDMS, (C) rGO (0.1 wt%)-PDMS, (D) rGO (0.2 wt%)-PDMS and (E) rGO (0.5 wt%)-PDMS groups.

## Conclusion

- The rGO-PDMS composite membrane had an ordered porous structure. Small pores on the top surface could prevent wounds from excessive water loss. Meanwhile, large pores on the bottom surface were beneficial for cell adhesion and proliferation.
- The rGO-PDMS composite membrane showed significant inhibition of the growth of the *S. aureus* and *E. coli* bacteria and the membrane with lower content of rGO (0.1 and 0.2 wt%) was biocompatible *in vitro*.
- The rGO (0.2 and 0.5 wt%) PDMS composite membrane remarkably accelerated wound healing via enhancement of the re-epithelialization and granulation tissue formation.

## Reference

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2. Rui, X., et al., *layer-by-layer induced assembly of silicone rubber with particulate piezoelectric enhanced axial anisotropic piezoelectric and piezoresistive*. *Monomers*, 2015, 4(1): p. 1.
3. Zhao, J., et al., *Self-Loading Poly(hexamethylenetriamine) Fluoropolymer with Remotability Shape Memory and Piezoelectricity*. *Polymer Chemistry*, 2016, 7(47): p. 7278-7286.
4. Luo, S., J. Zhu, and H. Xia, *Highly adhesive graphene aerogel piezocomposites with center-diverging microchannel structures*. *Journal of Materials Chemistry*, 2016, 4(3): p. 1068-1077.

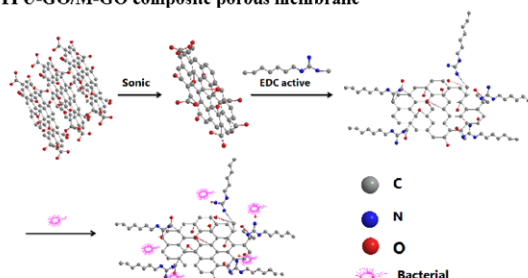
## Introduction

The skin is the largest organ on the body, protecting the body from pathogens<sup>[1]</sup>. However, when the human body loses this barrier (for example, large-area burns), this condition can lead to severe fluid losses or infections depending on the area and depth of the wound, which seriously endangers the patient's life<sup>[2]</sup>. Therefore, developing a new kind of wound dressing with sustainable antibacterial properties and good biocompatibility properties is very essential.

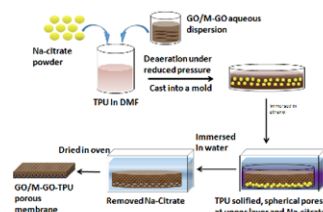
Here, firstly, we use the guanidine polymer (PHMG) modified GO (M-GO), and then combined immersion precipitation and particle filter leach prepared TPU-GO / M-GO porous dressing having a bilayer skin bionic. The IR, UV, XPS, XRD, TGA characterization of M-GO and the mechanical properties, WVTR, SEM, bioexperiment characterization of the TPU-GO/M-GO Compound porous dressing were tested. It was confirmed that PHMG was successfully grafted onto the GO surface and the introduction of GO and M-GO improved the mechanical strength of the material. At the same time, the biocompatibility, antibacterial property of the material were tested, which indicated that the materials have good biocompatibility and effective antibacterial properties.

## Experimental

### TPU-GO/M-GO composite porous membrane



Scheme 1. Illustration of the chemical modification routes of M-GO and the antibacterial mechanism of M-GO.



Scheme 2. Schematic Preparation of TPU-GO/M-GO composite porous membrane.

## Results and discussion

### Structural characterization of M-GO

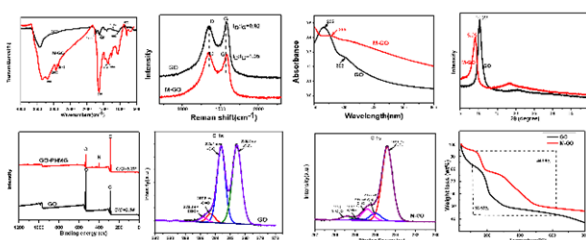


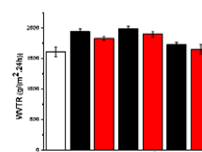
Figure 1 (a) IR spectra, (b) Raman spectra, (c) UV-vis absorption, (d) XRD spectra, (e) XPS, (f) TGA.

The structural characterization of M-GO proved that PHMG was successfully grafted onto the GO surface while partially reducing GO.

### Mechanical properties

	拉伸应力 (MPa)	断裂伸长率 (%)	弹性模量 (MPa)
TPU-pure	2.04 ± 0.71	1000 ± 88	0.79 ± 0.26
TPU-0.1% M-GO	2.07 ± 0.25	1152 ± 108	0.87 ± 0.10
TPU-0.1% GO	2.09 ± 0.24	1250 ± 202	0.94 ± 0.08
TPU-0.5% M-GO	2.32 ± 0.15	1399 ± 94	0.82 ± 0.05
TPU-0.5% GO	2.29 ± 0.06	1131 ± 208	0.81 ± 0.15
TPU-1.0% M-GO	2.10 ± 0.05	1530 ± 200	0.78 ± 0.14
TPU-0.1% GO	2.20 ± 0.06	1429 ± 105	0.75 ± 0.05

### Water vapor transmission rate



### SEM: pore structure

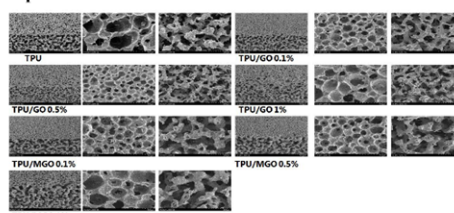


Figure 2. SEM images of cross section of different content of TPU-GO/M-GO composite porous membrane.

The SEM results showed that different content of TPU-GO/M-GO composite porous dressing showed obvious double-layer structure.

### Biological experiments

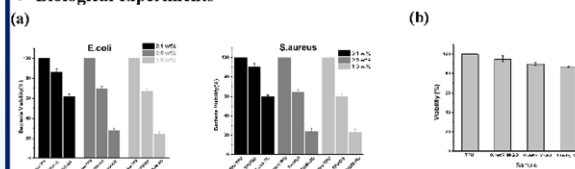


Figure 3 (a) Antibacterial effect of different content of TPU-GO/M-GO, (b) Biocompatibility of different contents of M-GO.

The antibacterial experiment results of the materials show that M-GO has better antibacterial property than GO, and the antibacterial effect is similar to GO 1.0wt% when the M-GO content is 0.1 wt%. Biocompatibility experiments show that TPU-MGO has good biocompatibility.

## Conclusion

- A two-layer structure with bionic skin is prepared by immersion precipitation method and particle filtration method, and the lower layer absorbs the upper layer of the permeate to maintain the wound moist environment.
- The introduction of GO and M-GO significantly improved the mechanical properties and water vapor transmission rate of TPU-GO/M-GO, and achieved the best effect at 0.5%.
- The addition of M-GO and GO significantly improved the antibacterial properties of the TPU-GO/M-GO porous membrane. In contrast, M-GO has a better antibacterial effect, and the antibacterial effect of M-GO is 0.1%. GO addition amount is similar to 1.0%.

## References

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- [2] Kai H, Yamauchi T, Ogawa Y, et al. Accelerated Wound Healing on Skin by Electrical Stimulation with a Bioelectric Plaster[J]. *Advanced Healthcare Materials*, 2017, 6.





# Three Dimensional Construction for Polymeric Foams via scCO<sub>2</sub> Foaming and its Connection with Two Dimensional Foam Characterization

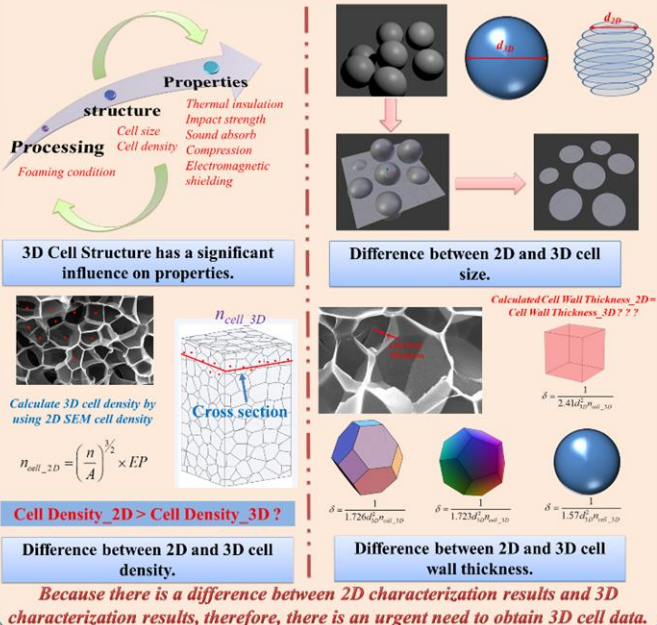
Haoyu Ma<sup>1</sup>, Pengjian Gong<sup>1,2\*</sup>, Chul B. Park<sup>2</sup>, Guangxian Li<sup>1</sup>

<sup>1</sup> College of Polymer Science and Engineering, State Key Laboratory of Polymer Materials Engineering, Sichuan University

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## General Introduction



## Objectives of This Work

- ✓ To enhance the fluorescence effect of TPU foams by using fluorescent probe;
- ✓ To construct the 3D cell structure of TPU foams by confocal laser scanning microscopy (CLSM);
- ✓ To study the relationship between 2D and 3D cell size, cell density and cell wall thickness.

## 3D construction of TPU foams by CLSM

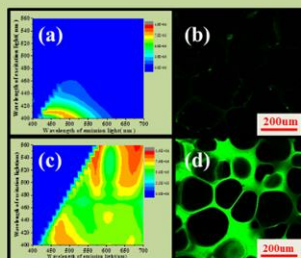
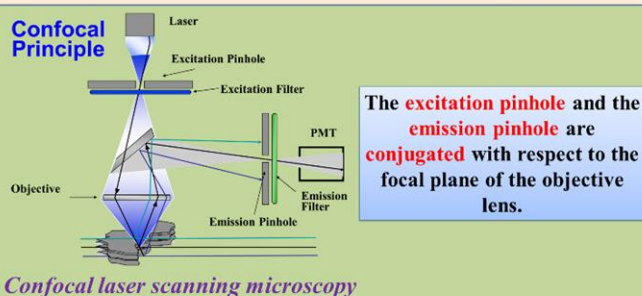


Figure 1. (a) The fluorescence of unstained TPU, (b) CLSM image of unstained TPU foam (Fluorescence mode, Excitation light wavelength is 405nm), (c) The fluorescence of stained TPU, (d) CLSM image of stained TPU foam (Fluorescence mode, Excitation light wavelength is 488 nm)

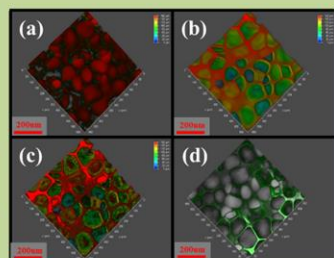


Fig.2 CLSM image of TPU foam, sample 4. (a) transmission mode, (b) fluorescence mode, (c) reflection mode, (d) overlay channels.

(1) Nile Blue A was used as fluorescent probe to enhance the fluorescence of TPU foams, then, CLSM was used to construct 3D cell structure of TPU foams.

## Morphology characterization of cell structure

### SEM Characterization

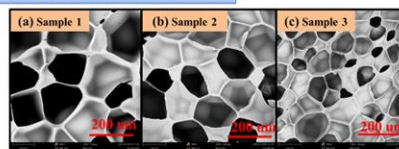


Figure 3. SEM images of TPU foams. (a) sample 1, (b) sample 2, (c) sample 3. Scale bar is 200um.

### POM Characterization

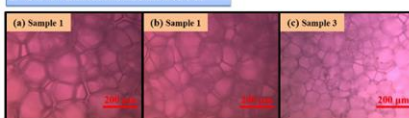


Figure 4. POM images of TPU foams. (a) sample 1, (b) sample 2, (c) sample 3. Scale bar is 200um.

### CLSM Characterization

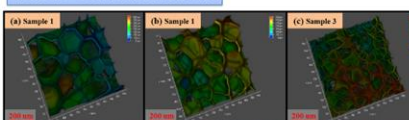


Figure 5. 3D construction of TPU foams. (a) sample 1, (b) sample 2, (c) sample 3. Scale bar is 100um. Depth coding is used.

(2) Different characterization methods (SEM, POM and CLSM) were used to obtain 2D and 3D cell data. By comparing of 2D and 3D cell data, the relationship was obtained.

## Voronoi modeling

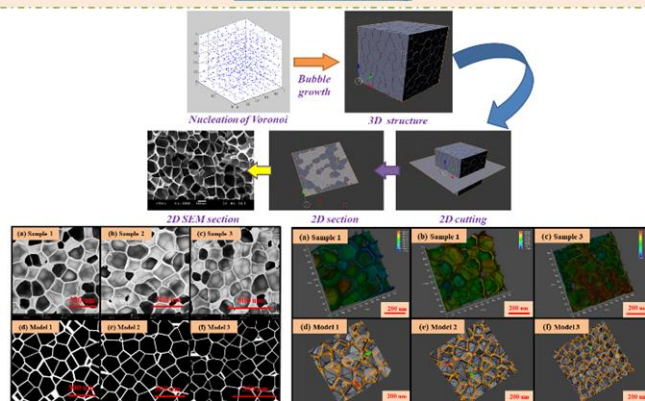


Figure 6. 2D section of samples and models. (a) sample 1, (b) sample 2, (c) sample 3, (d) model 1, (e) model 2, (f) model 3.

(3) Voronoi modeling method was used to construct 3D cell structure, by analyzing the 2D and 3D cell data of models, the relationship between 2D and 3D cell data was further verified.

## Conversion method between 2D and 3D characterization results

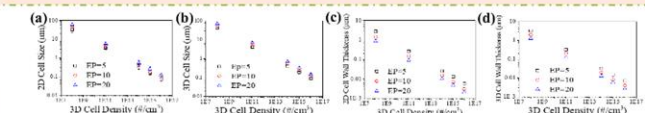


Figure 8. (a) 2D average diameter of cells, (b) 2D average cell wall thickness, (c) 3D average diameter of cells, (d) 3D cell wall thickness;

Table 1. 2D/3D cell data

Models	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
2D/3D cell size	0.82	0.80	0.82	0.78	0.81	0.79	0.82	0.82	0.80	0.80	0.77	0.80	0.81	0.84	0.81
2D/3D cell wall thickness	0.80	0.87	0.79	0.85	0.82	0.77	0.84	0.76	0.72	0.73	0.82	0.83	0.78	0.71	0.72
2D/3D cell density	1.13	1.04	1.08	1.13	1.10	1.16	1.28	1.16	1.20	1.18	1.12	1.04	1.04	1.09	1.15

(4) Models with a larger range of cell density and expansion ratio were constructed to verify the relationship between 2D and 3D cell data.

## Conclusion

- ✓ For the first time, 3D cell structure of TPU foams was constructed by CLSM and the fluorescence method was proved to be most powerful technique for 3D construction of porous materials;
- ✓ By comparing the characterization results of POM, SEM and CLSM, we found the ratio of the 2D and 3D diameters of the cells is between 0.78-0.80;
- ✓ The ratio of the 2D and 3D cell density of is between 1.1-1.2;
- ✓ The ratio of the 2D and 3D cell wall thickness is close to 0.787;



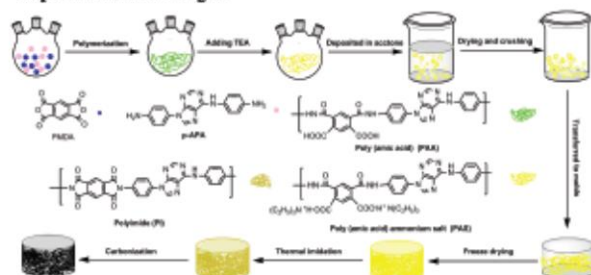


## Abstract

Nitrogen doping can introduce many excellent comprehensive properties (e.g. electrical properties and catalytic properties) to carbon materials. As an important part of DNA, adenine contains a large amount of nitrogen atoms in its heterocyclic structure, which made adenine becomes an ideal nitrogen-doped structure of carbon materials. In this study, adenine was introduced into the polyimide backbone, and the corresponding polyimide aerogel (API) was prepared by freeze-drying method. Then, the APIs were used as precursor to prepare corresponding carbon aerogel (ACG) by carbonization. The carbon aerogel (PCG) prepared by adenine-free PI (PMDA ODA PI) was used as a comparison. The differences in microstructure, carbonization behavior, nitrogen content and electromagnetic shielding properties of API and adenine-free PI were carefully studied. The results show that both ACG and PCG have three-dimensional porous structures. The nitrogen content of ACG after carbonized at 900°C (API-900) was as high as 5.15%, which was about twice higher than PI-900 (2.82%). The electromagnetic shielding effectiveness value of API-900 was 39.0dB, which much higher than PI-900 (22.6dB). This work presents a new route of nitrogen doped carbon materials for electromagnetic shielding materials.

## Experimental

### Preparation of carbon aerogels



Scheme 1 carbon aerogel preparation flow chart

\* The carbonized samples were recorded as API-700, API-800, API-900, PI-700, PI-800, PI-900, API, and PI represented adenine-containing polyimide and Kapton-type polyimide. 700, 800, 900 represent the carbonization temperature.

## Results and Discussion

### Micromorphology before carbonization

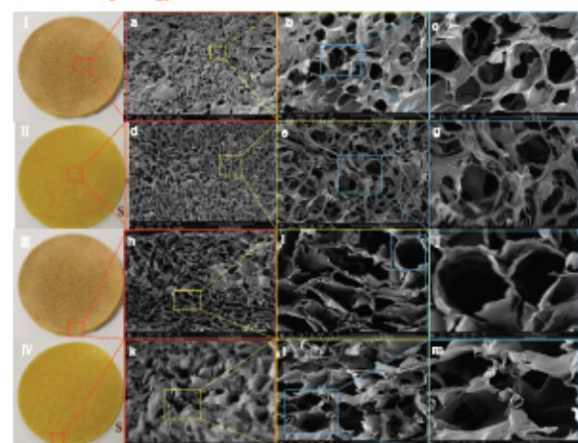


Figure 1 SEM image of PI and API aerogel before carbonization (a, b, c are API aerogel surface topography; d, e, f are PI aerogel surface topography; g, h, i, j are API aerogel section topography; k, l, m are PI aerogel section topography. I, II, III, IV are PI aerogels)

As shown in Figure 1, the API and PI aerogel surfaces exhibit random porous structures and the cross section exhibit honeycomb-like three-dimensional porous structures, which demonstrates that the API and PI aerogels were successfully prepared by freeze drying.

### Morphology and nitrogen content after carbonization



Figure 2 optical images and nitrogen content of API and PI aerogel after carbonization at different temperatures

As shown in Figure 2, as the carbonization temperature increases, the color of API and PI aerogels change from yellow to black, the volume gradually decreases, the degree of carbonization increases, and the nitrogen content gradually decreases. After been carbonized at 900 °C for 1 h, the nitrogen content of API-900 is still as high as 5.15%, which is 1.83 times higher than PI-900. This indicates that adenine can be used as a bulk nitrogen doping building block for carbon materials.

### Electromagnetic shielding performance

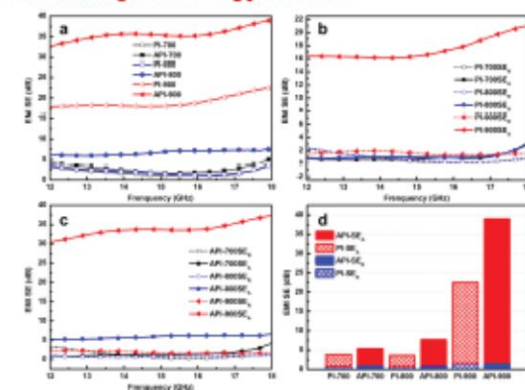


Figure 3 (a) electromagnetic shielding efficiency of API based carbon aerogel and PI based carbon aerogel (b) absorption efficiency and reflection efficiency of PI aerogel (c) absorption efficiency and reflection efficiency of API aerogel (d) absorption efficiency (SEA) and reflection efficiency (SER) of API and PI aerogel at 8 GHz.

The electromagnetic shielding properties of carbon materials prepared by different carbonization processes were studied. As shown in Figure 3a, the electromagnetic shielding efficiency of ACG and PCG increases with the increase of carbonization temperature. Among them, the total electromagnetic shielding performance of API-900 at 18GHz reaches 39.0dB, which means that 99.99% of incident electromagnetic waves have been shielded. In contrast, the total electromagnetic shielding efficiency of the PI-900 is 22.6dB, which means that 99.45% of incident electromagnetic waves have been shielded. The total electromagnetic shielding efficiency of the API-900 was 72.57% higher than PI-900. From Figure 3b, c, one can see that with the increase of carbonization temperature of ACG and PCG, the absorption efficiency of electromagnetic shielding increases greatly, but the reflection efficiency increases slightly. As shown in Figure 3d, the electromagnetic shielding effectiveness of ACG and PCG is dominated by absorption, which is consistent with the reported works, namely electromagnetic shielding mechanism of carbon materials are mainly dominated by absorption. In addition, at the same carbonization temperature, the electromagnetic shielding efficiency of ACG is always greater than PCG. The data show that introducing adenine structure into polymer system can improve the electromagnetic shielding performance of carbonized products.

## Conclusions

This work reports a feasible route to prepare N-doped carbon aerogels using adenine-containing build block. The microstructure, carbonization behavior, nitrogen content and electromagnetic shielding properties of API aerogels were carefully studied. API-derived carbon aerogels exhibit superior electromagnetic shielding behavior compared to adenine-free PI carbon aerogels, and are primarily based on absorption performance. This may be induced by the introduction of adenine building block. This work presents a new route to prepare N-doped carbon materials for electromagnetic shielding materials.



# **MACROPOROUS ADSORBENTS BASED ON POLY(VINYL FORMAL)-CHITOSAN (PVF-CS) FOAM FOR WATER PURIFICATION**

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## **ABSTRACT**

In polyvinyl alcohol solution, suitable amount chitosan was added to form a homogenous solution, with the introduction of formaldehyde and sulfuric acid, the solution was quick stirring with surfactant for certain time to make sure to get macroporous structure, then the solution was transferred to a sealed bottle for another 5 h at 65 °C, finally a macroporous foam PVF-CS was obtained. Because of the existence of amino groups in chitosan, the foam can be used to absorb heavy metal ions such as  $\text{Cu}^{2+}$ ,  $\text{Pb}^{2+}$  in waste water. The foam was studied by the IR, elemental analysis (EA), scanning electron microscope (SEM). The influence of pH, contact time, initial metal ions concentration on absorption was investigated. The foam can achieve absorption equilibrium within an hour. The pseudo-first-order, pseudo-second-order model were used to learn the absorption kinetics, finding that the adsorption kinetics would be compatible with the mechanism of pseudo-second-order model. The absorption equilibrium data of PVF-CS fitted well with the Langmuir model. According to Langmuir model the maximum adsorption capacities of PVF-CS foam for  $\text{Pb}^{2+}$ ,  $\text{Cu}^{2+}$  are 340.28 mg/g, 96.568 mg/g, respectively. PVF-CS foam shows better adsorption capacity for  $\text{Pb}^{2+}$  in all pH ranges. The specimen also exhibits rapid desorption and wonderful reusability at least five cycles.

# Study on the Crystallization and Foaming Behavior of LCB-PLA in Supercritical CO<sub>2</sub>

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## Introduction

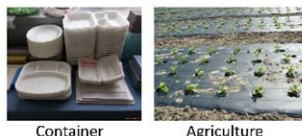
**Poly(lactide) (PLA)**

### Advantages

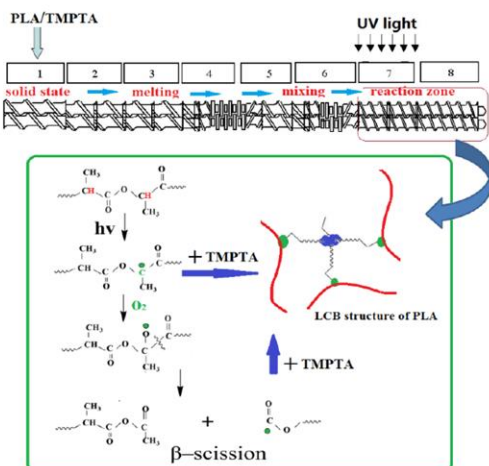
- Biodegradable
- Biocompatible
- Renewable resource

### Disadvantages

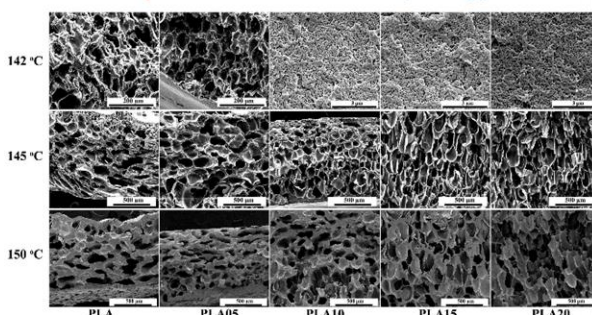
- Poor melt strength
- Shear-insensitive melt viscosity



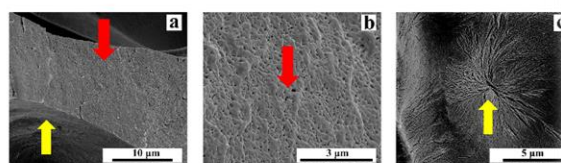
## How to solve?



## Effect of temperature on the cell morphology of LCB-PLA



different saturation temperature, 12 MPa, 4 h

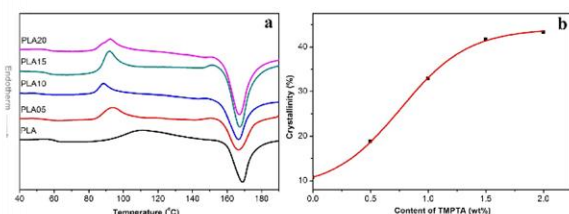


PLA05, 150 °C, 16 MPa, 4 h

- The higher matrix strength and crystal nucleation potential of LCB-PLA benefited its foaming behavior.
- Crystals formed during foaming and cooling process at high temperature.
- The spherulites on the interface of cell and cell wall restricted the coalescence of cells.

## Results and discussion

### Crystallization behavior of LCB-PLA



Secondary heating curves of LCB-PLA experienced isothermal crystallization at 150 °C under 12 MPa CO<sub>2</sub>

## Conclusion

- ❑ LCB structure could enhance the matrix strength of PLA and benefit its crystal nucleation.
- ❑ LCB-PLA possesses better cell morphology (less coalescence, none collapse and uniform cell distribution) than linear PLA.
- ❑ UV-induced reactive extrusion provides us with a new method to prepare high melt strength and pollution-free PLA, which could widen its foaming processing window.

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