
P O L I M E R Y

Terminology relating to nano-, micro- and macroporous polymer materials – an overview

Piotr Szewczykowski^{1), *)} (ORCID ID: 0000-0003-2853-2132), Marek Bieliński¹⁾ (0000-0002-2867-511X)

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Abstract: In the article, based on literature data, an attempt was made to systematize polymer porous materials in terms of pore size and technological aspects.

Keywords: porous polymer materials, terminology, technological aspects.

Terminologia dotycząca nano-, mikro- i makroporowatych materiałów polimerowych – przegląd literatury

Streszczenie: W artykule, w oparciu o dane literaturowe podjęto próbę usystematyzowania polimerowych materiałów porowatych pod względem wielkości porów i aspektów technologicznych.

Słowa kluczowe: porowate materiały polimerowe, terminologia, zagadnienia technologiczne.

Porous materials have been known to humanity for years. The human body and many organisms created by nature are made of many materials and tissues that reveal a porous structure. It is known that the amazing properties of these natural products are often due to their porosity. Bones, which can also be considered a composite material, provide one of the best examples with a hard outer cover made of several layers, while the inside creates a light core. The presence of porous structures allows for reduced bone mass while maintaining the strength. From ancient times, civilizations used for medicine and water purification porous natural materials like charcoal [1]. Likewise, various porous materials like kaolinite, clay minerals, or mortars have been applied for years [2]. The development of different manufacturing techniques has led to creation of artificial porous materials, often without conscious intent, like ceramics

and cement. While people have made porous materials since ancient times, intentionally designed, and utilized porous materials represent a novel category of engineering materials. The valuable properties of these materials, such as lightweight and stiffness, found applications in different fields like membrane technology and filtration, catalysis, batteries and fuel cells, adsorption, and gas separation, among others [3]. Difficulties arise when trying to classify porous materials and establish unified definitions across different branches. Different criteria are used to classify porous materials, such as pore size, building frameworks or origin. The last-mentioned criteria comprise both natural and artificial materials, with porous metals, ceramics, and polymers being the main artificial classes [4]. Therefore, the aim of this paper is to propose the modification of the classification of porous polymeric materials, particularly considering pore size ranges.

¹⁾ Bydgoszcz University of Science and Technology, Faculty of Mechanical Engineering, Department of Manufacturing Techniques, Kaliskiego 7, 85-796 Bydgoszcz, Poland

*) Autor for correspondence: piotr.szewczykowski@pbs.edu.pl

DIFFERENCES BETWEEN PORES, VOIDS, CELLULAR STRUCTURES, AND FOAMS

Porous materials are composed of two phases, with tiny spaces or cavities known as pores in the continuous matrix phase, where the term “tiny” is crucial for classification, where the pores may be open and/or closed. Open pores enable valuable applications such as filtering, while closed pores minimize material mass and can be used for sound and thermal isolation. The word “pore” is frequently used interchangeably with “cell” or “void” even though it can have different meanings depending on the materials and specifications. Moreover, the literature may contain terms like “porosity of foams” [5] or “porous cellular foams” [6], adding to the potential confusion. In polymeric materials, the term “foam” is commonly associated with polyurethanes and is typically seen as a rigid or elastic porous material, although the concepts of “cell” and “pore” can have different meanings [7]. Polyurethane foam’s cellular structure is shown in Figure 1a), where the cell is designated by a white circle. As it may be seen, within one cell opened and closed pores may exist simultaneously [8]. Several authors used the term “pores” to describe “cell windows” within a foamed aluminum structure [9]. Figure 1b) highlights a porous polyamide 66 (PA66) that has been reinforced with glass fibers and produced using the MuCell® process. A “microcellular” structure description is used to depict pores when they have regular, circular shapes [10]. Hence, a pore within this structure can also be termed a cell. Authors recommend treating cells and pores as synonyms in polymeric materials to avoid confusion, meaning that microcellular can also refer to a microporous material.

Unlike pores, voids are unintentional cavities that are typically irregular in shape and much larger than regular pores. However, Figure 1b) illustrates the voids created when the glass fibers were extracted, resulting in a smaller-than-pore-size unintentional holes (void 1), as

well as an irregular-shaped void within a pore, caused by a surface crack after the sample broke (void 2).

NANO-, MICRO- AND MACRO- SCALE

In the context of this article, a “macro” is an object that can be observed and operated without special tools. By observing and referencing objects, it is evident to recognize a size of 1 mm (1000 μm). To compare, it may be stated that ants are typically just a few millimeters in size, while credit cards have a standard thickness of 1.5 mm and pencil graphite measures 0.5 mm in diameter. Not to mention that this is also the smallest unit on a standard ruler [11]. Similarly, as a reference point, helping in imaging sizes in micrometers (10^{-3} mm) can be the thickness of a printing paper of 80 g/m² which is 100 μm (0.1mm), or the thickness of a human hair, which is 50–100 μm (0.05–0.1 mm). Objects as small as 100 μm can be seen with the naked eye. Glass fibers, depending on its type, have a diameter in the range of 5–25 μm , but the most popular glass fibers of type E have a diameter between 6–9 μm (Figure 1b) [12]. Organelles like human cell nucleus, mitochondrion, or red blood cells are below 10 μm in size [13]. The size of bacteria can be assumed as 1–5 μm , but it is in a very wide range from 0.3 to even 750 μm [14]. For example, *Escherichia coli* bacteria is about 1–2 μm in length and 1 μm in diameter. Although viruses are smaller than bacteria, giant viruses like Mimivirus or Pithovirus can still grow up to 1.5 μm . Bacteriophages, such as ϕ 4 that target *Escherichia coli*, have a size of 200 nm (0.2 μm). The smallest objects that an optical microscope can detect are of this size. HIV and SARS-CoV-2 both have a size of about 100 nm (0.1 μm) [15–19]. Standard transistors are 20 nm (0.02 μm) in size, and 5 nm size is the smallest value [20, 21]. 1 nm (0.001 μm), which is a single piece after dividing 1 mm into 1 million pieces, is the diameter of a single wall carbon nanotube [22]. Covalent diameter of the largest atom of the periodic table cesium is half

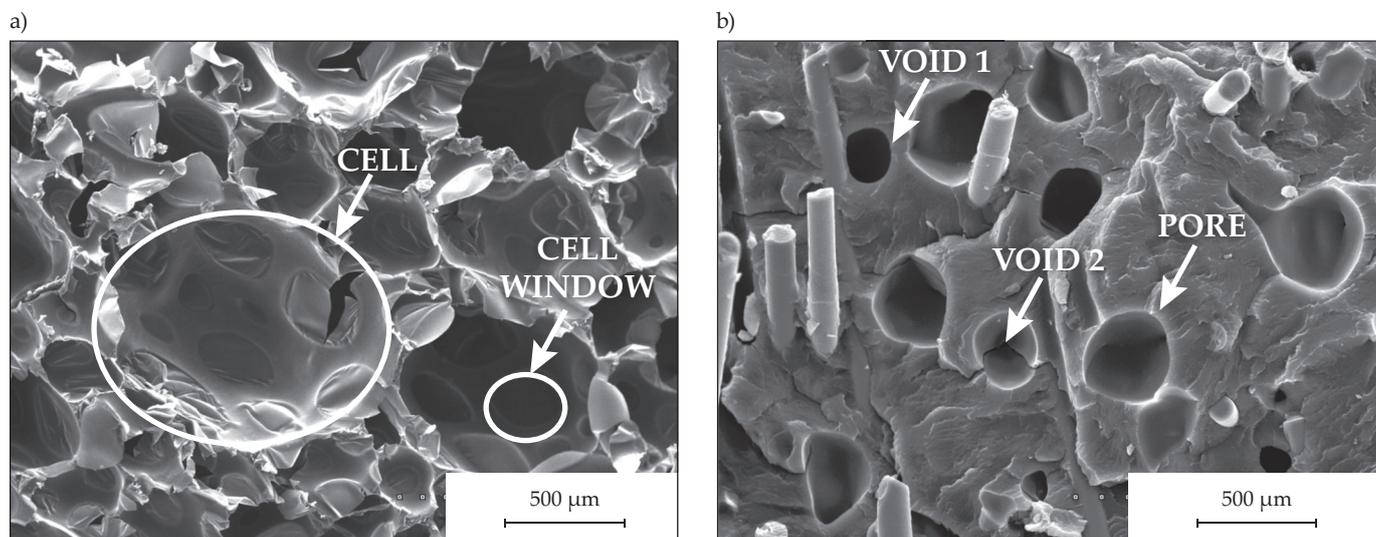


Fig. 1. SEM microphotographs: a) cellular structure of polyurethane foam, b) microcellular structure of polyamide 66 reinforced with glass fiber

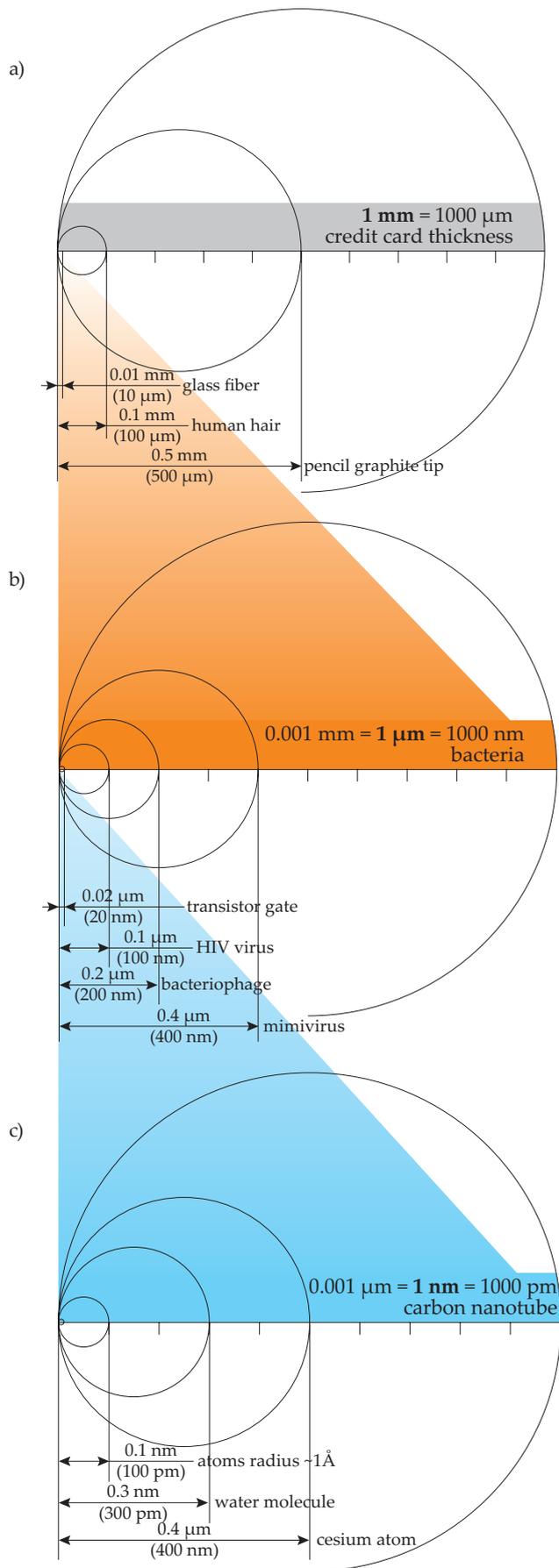


Fig. 2. Comparison between objects sizes: a) 1 mm, b) 1 μm, c) 1 nm

a nanometer (500 pm) while the water molecule is 0.27 nm measuring the distance between hydrogens [23, 24]. Theoretically in the diameter of a carbon nanotube, one can place two Cesium atoms or three water molecules. Graphene thickness is 0.3 – 0.4 nm [25]. Finally, the smallest objects we can observe with electron microscopes are single atoms, at the level of 0.1 nm (1 Å) or even smaller [26, 27]. In Figure 2 the relative dimensions of objects with a size of 1 mm, 1 μm, and 1 nm are presented.

POROUS MATERIALS CLASSIFICATION

The International Union of Pure and Applied Chemistry (IUPAC) classifies porous materials into three groups (Table 1), depending on pore size. Microporous materials have pores smaller than 2 nm, while mesoporous materials have pores between 2 and 50 nm, and macroporous materials have pores larger than 50 nm [28]. This classification is valuable in nanotechnology, membrane technology, and similar disciplines dealing with nanoscale materials. The classification system groups all materials and products with pores larger than 50 nm as macro-porous. Both 100 nm and 100 μm pores belong to the same class, despite being three orders of magnitude apart, which does not have a critical meaning in nanotechnology. Such classification does not consider macroscale polymeric products used in polymer processing technologies.

T.J Mays introduced another classification of porous materials in 2007, categorizing them into nano-, micro-, and milliporous. This classification considers technical aspects and divides each group into sub-groups [29]. Thus, the author suggests using the classification boundaries outlined in Table 1. According to the author, a drawback of the IUPAC system is the absence of SI units and prefixes in the definitions of micro-, meso-, and macroporous materials.

Four papers have been found on the Web of Science platform regarding milliporous materials. Harini Sri, K. *et al.* reported in their paper that milliporous polylactide revealed pores in the range of 100–300 μm which covers the above definition [30]. Obviously, there are over 7000 records when searching for articles on macroporous materials, which aligns with the IUPAC definition and covers a vast range of pore sizes. Conversely, the development of technology and polymer processing leads to a reclassification of porous materials that do not fit within the IUPAC ranges. The Massachusetts Institute of Technology (MIT) in the 80s developed a method for injection molding polymers by adding gas in a supercritical state, which enabled getting moldings with regular and evenly distributed pores [31]. The Trexel, Inc. (USA) company registered the technology under the trademark MuCell® and described it as a microcellular injection molding process, with the pore size within the range of 2–10 μm in ideal cases and as regular as possible, which is characteristic of the “cellular” materials [32]. According to the literature, pore diameters vary depending on the material and gas used,

Table 1. Classification of porous materials according to T.J. Mays [29]

Nanoporous 0.1–100 nm		Microporous 0.1–100 μm		Milliporous 0.1–100 mm	
Sub-	0.1–1 nm	Sub-	0.1–1 μm	Sub-	0.1–1 mm
Inter-	1–10 nm	Inter-	1–10 μm	Inter-	1–10 mm
Super-	10–100 nm	Super-	10–100 μm	Super-	10–100 mm

with 100 μm being a critical size, above which unexpected voids may be detected [31]. Thus, it can be accepted, that microporous materials should have pores up to 100 μm , while macroporous materials should have larger ones. The lowest range of microcellular polymeric materials is questionable. In addition to microcellular materials, also nanocellular polymeric materials are described in the literature. According to other researchers, nanocellular materials may have cell size below 1 μm [33, 34]. Others establish the border to lower values as 300 nm, 200 nm, or 100 nm [35–37]. In the authors' opinion, from a polymer processing point of view, 500 nm (0.5 μm) is a proper limit between nanoporous and microporous polymeric materials, where this range is quite broad, around \pm 400 nm. Figure 3 presents schematically the classifications of porous materials according to IUPAC, T.J. Mays, and the authors of the article.

IUPAC classification leaves the lowest and the upper border for porous materials opened, which is below 2 nm and above 50 nm, respectively. According to the authors, a pore size of 0.1 nm (1 Å) as the lowest for nanoporous materials is sufficient, if not excessive, given the submolecular distances. On the other side, the size of pores in macroporous polymeric materials should not exceed 10 mm, as they would then be better described as undesirable voids than pores. Polymeric macroporous foams like polyurethanes, are described and compared by their density rather than pore size and classified as high foaming (< 0.1 g/cm³), moderate (medium) foaming (0.1–0.4 g/cm³) and low foaming (>0.4 g/cm³) [38].

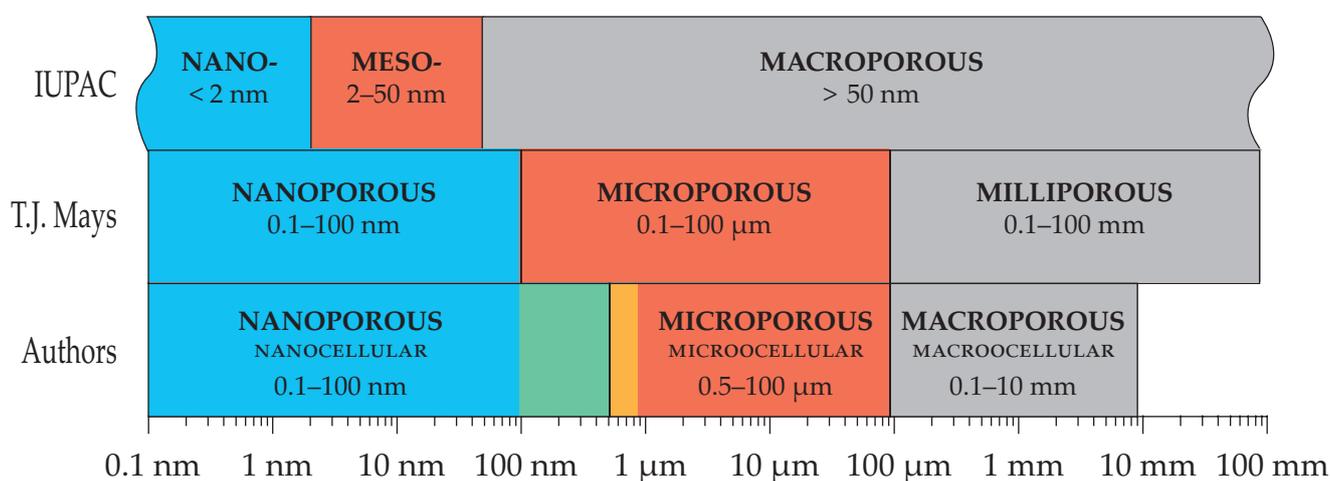
CHARACTERIZATION, MANUFACTURING, AND APPLICATIONS

The presented classifications of porous polymeric materials cover a range of ten orders of magnitude in the pore size diameters. Numerous manufacturing and synthesis techniques allow for a wide range of materials, which require different methods to determine pore size and distribution. The article presents only a few selected examples, intending to develop the topic in a separate article.

Nanoporous polymeric materials can be produced by employing the technique of self-assembly of block copolymers into symmetrical structures on the nanoscale [39]. The synthesis of block-copolymers is a critical step that involves ionic polymerization or atom transfer radical polymerization [40]. Nanoporous materials can be obtained by removing one block through chemical etching or in nanolithography process [41, 42]. The pore sizes obtained in this manner fall within the IUPAC definition of mesoporous materials (2–50 nm) and can be categorized as nanotechnology-based manufacturing.

However, additive manufacturing technologies enable the production of materials with pore sizes within the range of nanoporous range, according to the proposed classification [43].

The main methods of investigation of nanoporous materials include transmission electron microscopy (TEM), scanning electron microscopy (SEM), atomic force microscopy (AFM), x-ray diffraction (XRD), while x-ray

**Fig. 3. Classification of porous materials concerning their pore sizes**

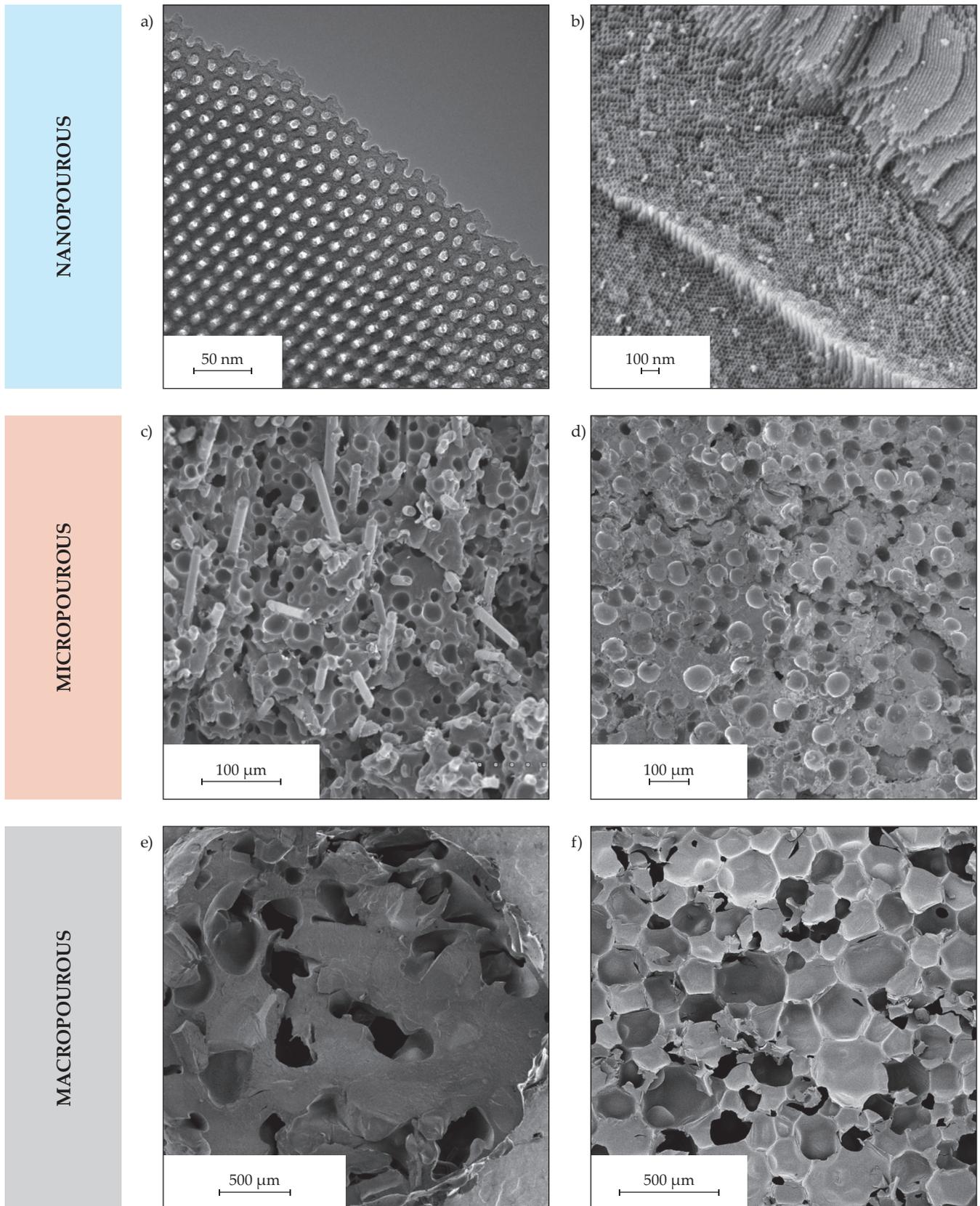


Fig. 4. Images of nano-, micro- and macroporous materials: a) TEM of diblock-copolymers with hexagonally distributed pores, b) SEM of diblock-copolymers with hexagonally distributed pores, c) SEM of microporous polyamide with glass fiber, d) SEM of polylactide with chemical blown agent made pores, e) SEM of 3D printed filament with chemical blown agent made pores, f) SEM of polyurethane with closed cells

Table 2. Examples of characterization methods, manufacturing techniques and applications of polymeric porous materials

Range	Characterization techniques	Manufacturing processes	Applications
Nanoporous 0.1–500 nm	transmission electron microscopy (TEM), scanning electron microscopy (SEM), atomic force microscopy (AFM), X-ray diffraction (XRD), X-ray nano-computed tomography (nano-CT), gas porosimetry, mercury porosimetry	block-copolymer self-assembly, nano-lithography, foaming with CO ₂ , 3D printing, injection molding	membranes, catalysts, drug delivery, thermal isolators
Microporous 0.5–100 μm	scanning electron microscopy (SEM), optical microscopy, X-ray micro-computed tomography (micro-CT), mercury porosimetry	microcellular injection molding, extrusion, chemical blowing agents, 3D printing	automotive, scaffolds, acoustic absorbents, packaging, footwear industry
Macroporous 0.1–10 mm	scanning electron microscopy (SEM), optical microscopy, X-ray micro-computed tomography (micro-CT)	polyurethanes, chemical blowing agents, 3D printing	automotive, foams, furniture industry, building isolators, packaging

computer nanotomography (nano-CT) is getting more popular method in resolving 3D structures of nanoporous materials [44, 45]. Gas porosimetry and mercury porosimetry is applied to characterize porous materials, while gas adsorption can be used to pores of size up to 50 nm, and mercury porosimetry covers the range of materials from 4 nm to 950 μm diameter [46]. Materials of this pore size range can be applied in separation and membrane technologies, catalysis, or drug delivery [47, 48].

The range of nanoporous materials up to 500 nm pore diameter according to the proposed classification, can be obtained for example in the foaming process with adsorption of carbon dioxide or injection molding with gas in the supercritical state [49–51]. Examples of diblock-copolymers nanoporous material with hexagonally distributed pores observed using TEM and SEM are presented in Figure 4a) and 4b), respectively [52].

Microporous polymeric materials can be produced by the different techniques of microcellular injection molding with gas like MuCell[®], Ergocell or IQFoam[®] or in extrusion process [53, 54]. Chemical blowing agents are applied as well to obtain evenly distributed microcellular structure; however, it is more difficult to control the pores [55, 56]. Microcellular thermoplastic polyurethanes can be obtained as well in the process of injection molding with supercritical gas [10, 57]. Just like nanoporous materials, microporous polymers can also be made by using 3D printing methods and observed by using both SEM and optical microscopy due to the range of pores being 0.5–100 μm [58, 59].

X-ray microcomputed tomography (micro-CT) is a very useful, nondestructive method to reveal the internal structure of porous materials, but revealing the structure can be a challenge, especially in the case of three phases in the system (e.g. polymeric matrix, glass fiber, pores) and pores size close to the apparatus resolution limit [10]. Microcellular polymeric materials find applications in automotive, scaffolds, acoustic absorbents, packaging, footwear industry etc. [60–62]. Examples of microporous structure of polyamide reinforced with glass fibers

obtained by MuCell[®] and the structure of porous polyacetaldehyde with pores resulting from chemical blowing agent applications are shown in Figure 4c) and 4d), respectively.

Macroporous polymeric materials are related to polyurethane foams, thermoplastic polymeric materials produced by adding chemical blowing agents, and 3D printed materials. Like microporous polymers, materials of this kind are used in various industries including automotive, furniture, building isolators, packaging, and more [28]. Examples of macroporous 3D printing filament modified with chemical blowing agents and polyurethane with closed cells are presented in Figure 4e) and 4f), respectively. Discussed methods of characterization together with some examples of nano-, micro- and macroporous materials applications are summarized in Table 2.

CONCLUSIONS

The classification of porous materials into nano-, meso- and macroporous materials adopted by the International Union of Pure and Applied Chemistry IUPAC does not consider the diversity of materials in the macroporous regime and as noted by T.J. Mays in his work, it does not consider compartments according to the SI system. Although Mays proposed a division according to the SI system, literature data show that the adopted category of milliporous materials is not reflected in practice. The authors of this article have made an effort to sort out certain terminological issues related to porous polymeric materials and adopted the names of porous materials to their ranges, reflecting the results of scientific research and technological conditions.

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