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Introductory Chapter: An Introduction to Nanoporous Materials

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1. Introduction

In the last few decades, research interests and efforts on the synthesis, characterization, functionalization, molecular modeling, and designing of new and novel nanoporous materials have exceedingly grown. In general, the materials having sizes smaller than 100 nm in at least one dimension are considered to be nanomaterials. The materials possessing porous morphology with porous features comparable to 100 nm are termed as nanoporous materials [1]. The properties of nanoporous materials are not only governed by the arrangement of atoms within the crystal but also by the porosity and specific surface area. These materials contain several voids with the controllable dimensions in atomic, molecular, or nanometer scales which enable them to interact more effectively with their environment. According to the International Union of Pure and Applied Chemistry (IUPAC) classification, nanoporous materials can be categorized on the basis of pore sizes as microporous materials (pore size <2 nm), mesoporous materials (pore size between 2 and 50 nm), and macroporous materials (pore size >50 nm) [2–6].

The main challenge in this research pertains to the tailor designing of nanoporous materials to obtain uniform particle/pore size and shape to suit a particular application. For nanoporous materials, it is also essential to achieve a precise composition in their chemical buildup which eventually becomes responsible for carrying out any chemical interaction with its surroundings. Moreover, based on the permeability of the pores to any fluid, these could be classified as closed pores and open pores. Materials properties especially the macroscopic properties like mechanical strength, density, thermal conductivity, etc. are associated to closed pores. On the other hand, the pores which are connected with the surfaces of the material through channels are called open pores. These are active in the processes like absorption and flow of fluids as well as adsorption of gases.

Nanoporous materials have potential applicability to obtain robust, miniaturized, and portable devices. Structural and morphological properties of these materials can be reliably characterized using X-ray diffraction technique (XRD), electron crystallography, field emission scanning electron microscopy (FESEM), and transmission electron microscopy (TEM). The oxidation state, coordination, and optical properties can be studied using X-ray absorption spectroscopy, UV-Vis spectroscopy, solid-state nuclear magnetic resonance (NMR), etc. For elemental analysis of nanoporous materials, energy-dispersive analysis of X-rays (EDAX), inductively coupled plasma mass spectrometry (ICP-MS),

Auger electron spectroscopy (AES), and X-ray photoelectron spectroscopy (XPS) are generally adopted, and the surface area and pore size are evaluated by N_2 adsorption-desorption isotherm.

The structure, pore size and hence the porosity, specific surface area, water insolubility, density, pH environment, hydrophilicity/hydrophobicity, charge distribution, conductivity, and catalytic activity of nanoporous materials could be tailored using different fabrication techniques such as precipitation, solid-state reaction (usually performed at high temperature), sol-gel, hydrothermal, and solvothermal routes.

Precipitation synthesis of nanoporous materials involves the creation of a solid from the solution. The synthesis occurs in a liquid solution, and the solid formed is called the “precipitate,” whereas the precursor that causes solid formation is called the “precipitant.” More importantly, without sufficient settling force to bring the solid particles together, the precipitate remains suspended in the solution and can be separated by centrifugation.

Unlike precipitation, sol-gel process is a colloidal route which is effectively used to synthesize nanoporous materials with an intermediate stage known as sol and/or a gel state. In this technique, a colloidal solution known as “sol” is formed which normally advances into a diphasic system called as gel featuring both a liquid phase and solid phase whose morphology could vary from discrete particles to continuous polymeric networks. During the colloidal synthesis, the volume fraction or density of the particles might be very low such that initially a desirable amount of fluid might need to be washed out to recognize its gel-like nature.

The hydrothermal synthesis is a solution-based reaction approach carried out in an autoclave for the synthesis of nanoporous materials. In a broader sense, this method involves the synthesis from the room temperature to a high-temperature in which the morphology of the as-synthesized nanoporous materials can be controlled. The hydrothermal process at the elevated temperatures provides stability for the as-synthesized nanoporous materials. Another variant of the solution-based synthesis is solvothermal route which is very similar to the hydrothermal with the only difference that the precursor solution is usually nonaqueous. The solvothermal route allows precise control over the size, shape, and crystallinity of nanoporous materials. These characteristics could be altered by changing certain experimental parameters which include temperature, time, type, and the concentration of solvent and the surfactant [7].

Another simple and rapid method of preparation of highly porous materials, namely, metal oxides, is the auto-combustion route wherein some precursors act as oxidizers, while others as fuel. In a typical experiment, a homogeneous aqueous solution containing the necessary reaction mixture of oxidizers and fuel is kept under constant stirring on a hot plate in air until drying. Subsequently, the fuel initiates the combustion reaction at specific temperature which results in porous and spongy nanocrystalline material in powder form. The particle and pore size can be controlled by optimizing the fuel concentration in the reaction mixture. The reaction herein is exothermic, and the heat evolved in the combustion process is sufficient for the proper phase formation of the as-synthesized material. Hence, it is noteworthy that the as-synthesized porous material does not require any high-temperature sintering/annealing [8].

In modern technology, porous thin films are attracting tremendous attention due to their applicability in gas sensors, chemical and energy conversion devices, catalysis, and so on. In view of this, spray pyrolysis is a simple and the effective technique to synthesize thin films possessing porous morphology. In this technique aqueous or alcoholic solutions of inorganic precursors are sprayed onto a hot substrate which then pyrolytically decompose into a desired compound in thin-film form featuring excellent adhesion. The thickness and morphology of the porous

films can be varied by maneuvering various process parameters such as molarity of the solution, substrate temperature, flow rate of the carrier gas, distance from spray nozzle to substrate, etc [9].

Research efforts in the field of nanoporous materials have been driven by the emerging and rapidly growing applications of nanoporous materials as adsorbents and ion exchangers. The nanomaterials with uniform pores and a high surface-area-to-volume ratio produce large amount of catalytic sites for efficient catalysis, guest-host interaction for immobilized homogeneous catalysts, enzymes, nonlinear optics, low dielectric constant mediate, etc. Nanoporous materials have shown excellent application in several bioanalyses such as in biomedical diagnosis and the monitoring of food, as decontaminants and environmental quality, antibacterial agents in drug release, enzyme mimetics, biosensors, and adjuvant in anticancer therapy [10]. The applicability of nanoporous material shows several exotic new and novel opportunities for the researchers and scientists to develop new methodologies, strategies, and techniques for synthesizing the nanoporous materials.

In this book, a series of systematic chapters based on the recent developments in nanoporous materials is presented. To facilitate the understanding and to highlight the essential concepts, the chapters are designed to explain the basics of nanoporous materials. The chapters are strongly interconnected and intermingled as well, with the fundamental principles forming a framework that permeates throughout the book. The important properties of the nanoporous materials are covered under the topics of nanoporous oxides and nanoporous composites, porous low dielectric constant material for semiconductor microelectronics, synthesis and application of kaolin-based ZSM-5 in petrochemical industry, nanoporous silicon materials fabricated by electrochemical method for bio-applications, etc. Our approach is to emphasize the essential concepts and to develop a basic textbook for the students and researchers of chemistry, physics, and materials science as well as engineers of chemical industry, environmental protection, automotive industry, refining technology, civil engineering, and electronics. The book covers a selected range of topics covering the most significant aspects involved in the understanding, properties, preparation methods, and emerging applications of nanoporous materials. Some of the topics have not been addressed in detail in a view to keep the length of the book manageable. Hopefully, this book fulfills its purpose of inspiring young brains and guiding them into the challenging realms of nanoporous materials.

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