

Industrial Applications of Silica-Based Gas Adsorbents

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Abstract

The emergence of silica-based gas adsorbents as nanoporous materials has created a new field of research subject in the chemistry world. This paper reviews the evolution of silica-based gas adsorbents from microporous region to mesoporous region with the main interest in the zeolites, M41S family and silica aerogel. An overview of the synthesis and modification of those adsorbents will be enumerated. Further reviews are focused on the industrial applications of the silica-based adsorbents both in current demands and future prospects with the aim to highlight the potential applications of those adsorbents in the near future.

Keywords: Nanoporous materials, gas adsorbents, zeolites, M41S, silica aerogel.

1. Introduction

1.1 General Background

The rapid development in nanotechnology over the last decade has led to the creation of nanoporous materials, which attracts much attention from the researchers in this field over other topics of research [1]. According to the definition of IUPAC, porous materials can be classified into three categories: microporous (< 2 nm), mesoporous (2-50 nm) and macroporous (> 50 nm). In conjunction with this definition, nanoporous materials are defined as those porous materials with pore diameter less than 100 nm [2]. Among the substances that have been employed to synthesize the porous materials, silica is ranked as one of the most prominent substances due to the possibility of developing variety of surface areas and pore volumes by an enormous amount of pores therein created per unit mass of silica [3]. Therefore, silica-based materials have been synthesized from microporous region to mesoporous region in tandem with the discovery of their potential applications in different areas such as catalysis, adsorption, nanostructure manufacture, environmental purification, host matrixes, ion exchange, chemical sensing technology, host for quantum structures and membrane separations [4-9], with gas adsorption as the main concern in this review paper.

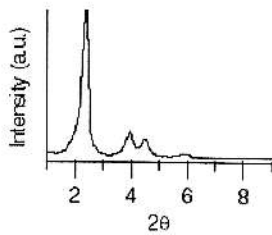
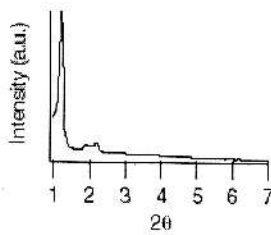
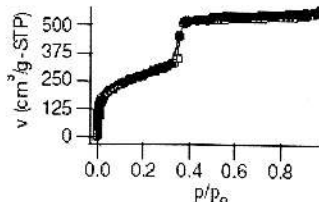
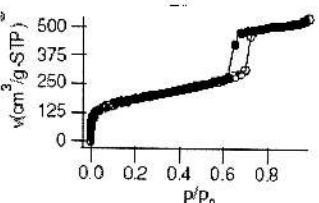
1.2 Silica Based Gas Adsorbents

Zeolites are crystalline, hydrated aluminosilicates of alkali or alkali earth elements such as sodium, potassium and calcium, which can be categorized in two forms, natural or synthetic [10]. Its history is started by the discovery of first zeolite mineral *Stilbite* by A. F. Cronstedt in 1756 [11]. The term zeolite derived from Greek words, "zeo" and "lithos" meaning "to boil" and "a stone" describes the zeolite behavior under fast heating conditions, when the zeolite minerals seem to boil because of the fast water loss [12]. Until today, the studies have been explored by the mineralogist for almost 250 years and the introduction of synthetic zeolites as a new class of adsorbent pioneered by Union Carbide in 1954 initiated the explosion of new

zeolites and new uses in the last few decades. The versatility of zeolites as good gas adsorbents is driven by their unique adsorptive properties: (i) selective adsorption of molecules based on molecular dimensions or molecular sieving effect; (ii) highly preferential adsorption of polar molecules; (iii) highly hydrophilic surface; and iv) variation of properties by ion exchange [13].

However, zeolites applications are rather limited by their relatively small pore openings [14]. Therefore, with the aforementioned limitation, a continuous interest in developing materials with constantly larger pores becomes another main aspect in both industrial and fundamental studies [15]. It was Exxon Mobil, which in 1992, discovered the M41S family of ordered mesoporous adsorbents with tailor-made properties, which showed a regularly ordered pore arrangement and a very narrow pore size distribution. Among the M41S family, the two most investigated materials are the hexagonal MCM-41 and the cubic MCM-48 [16-17]. The development was further expanded to the synthesis of mesoporous structures with uniform pore size up to approximately 300 Å, known as SBA-15 by Stucky *et al* in 1998 [18]. The basic overview of MCM-41 and SBA-15 is provided in Table 1 as a comparison of their properties.

Table 1: Comparison of various properties of MCM-41 and SBA-15 [19].

Material	MCM-41	SBA-15
Template	Surfactant CTAB	Poly(alkylene oxide) triblock polymer Pluronic P123
Synthesis pH	Basic	Acid
Typical Pore Size	4 nm	8 nm
Accessible Pore Size	15-100 Å	46-300 Å
Pore Structure	Cylindrical	Cylindrical
Structural Ordering	Hexagonal	Hexagonal
Name of Corresponding Cubic Bicontinuous Phase	MCM-48	SBA-16
Powder XRD		
Nitrogen Adsorption Isotherm		

Apart from the above mentioned mesoporous materials, silica aerogel is another nanoporous material that appears as a fascinating solid material and has been ranked among the top ten scientific and technological developments in the recent report [20]. Its first discovery was published in 1931 by Kistler and his work was continuously being developed until present with the result of few commercial silica aerogels in the market such as Nanogel[®] by Hoechst [21] and Basogel[®] by BASF [22] as well as the Maerogel (Malaysia aerogel), a rice husk synthesized aerogels from UTM [23]. By virtue of its large surface area and high porosity (up to 98%) [24], it makes silica aerogels an attractive material for use in different field of science and industry including adsorption purpose. Table 2 shows some properties of silica aerogels produced under different trademark.

Table 2: Physical properties of silica aerogels [21, 23].

Properties		Nanogel	Maerogel
Density:	Particle	0.12 g/cm ³	-
	Bulk	0.08 g/cm ³	-
	Apparent	-	0.03 g/cm ³
Internal Surface Area		Up to 1000 m ² /g	800-900 m ² /g
Mean Pore Diameter		~ 20 nm	20.8 nm
Thermal Conductivity		~0.015 W/m K	0.099 W/m K

2. Synthesis and Modification of Silica-Based Gas Adsorbents

2.1 Microporous Materials

The systematic zeolites synthesis was first initiated by Barrer and continued by Milton, which involved the hydrothermal sol-gel synthesis in aqueous solution under low pressure and low temperatures, range between room temperature up to about 300 °C [12]. Basically, the synthesis mechanism involves the conversion of a mixture of different chemical sources into a microporous crystalline aluminosilicate as shown in Table 3. Aluminosilicate hydrogel is formed when the reactants are mixed. After the gel point, the hydrogel will undergo ion transportation steps until the final crystalline zeolite is obtained [25]. Until today, vast synthesis procedures are well established under different patents and publications. Therefore, only a brief overview will be given on the influence of different parameters on the synthesis of zeolites as given in Table 4.

Table 3: Chemical sources and their function in zeolite synthesis [25].

Sources	Functions
SiO ₂	Primary building units of the framework
AlO ₂ ⁻	Origin of framework charge
OH ⁻	Mineralizer, guest molecule
Alkali cation, template	Counterion of AlO ₂ ⁻ , guest molecule
Water	Solvent, guest molecule

Table 4: Key parameters and their influence on the zeolite synthesis [25].

Key parameters	Influence on the synthesis
Hydrogel composition	The nucleation and the crystallization kinetics
	The nature of the crystalline obtained
	The framework composition
	The Si and Al distribution in the lattice
	The crystal size and morphology
Alkalinity	The Si/Al ratio of the crystalline product
	The crystal morphology
	The crystal aspect ratio
Temperature	The crystallization rate
Time	The crystallinity of the zeolites
Structure-directing agents	The gelation, nucleation and crystal growth process
	The chemical potential of the zeolite

Another alternative synthesis method for zeolite apart from hydrothermal sol-gel synthesis is the high pressure synthesis process in the high pressure autoclave, which was the simulation of natural formation of zeolites minerals by Schäfer and coworkers in 1999. From the experiment, it seems that the process of complete dissolution-recrystallization under high pressure conditions will be a much more powerful process for zeolite synthesis. Therefore, this method is recommended by the researcher for the application in the industrial-sized process with technical and economical optimization [12].

Since the diameters of micropores of zeolites are around the same size as small molecules and their geometric and energetic parameters can be fine-tuned, therefore, it is possible to modify and tailor the materials to suit their application as gas adsorbents. Generally, there are two ways to modify the zeolites, which include structure and framework modification, where it involves a change of pore size distribution, pore opening, pore volume or formation of mesoporous structure in the zeolites and surface interaction modification, where polar molecules or other components are introduced to the surface of the adsorbents in order to enhance its sorption capability towards molecules with a permanent dipole moment. Surface and framework modification can be achieved by various processes such as cation exchange [26], dealumination [27], desilication [28], impregnation [29] and silanation [30] while the dispersion of metal oxides on this microporous material is commonly used in the surface modification by virtue of its specific adsorptive properties [31].

2.2 Mesoporous Materials

In the synthesis of M41S family members, the four main components involved are structure-directing surfactants, a source of silica, a solvent and a catalyst (an acid or a base). The original recipe of the M41S synthesis was carried out in water under alkaline conditions, where alkyltrimethyl ammonium halides, cationic surfactant was used as the structure-directing surfactants, a combination of sodium silicate, tetraethoxy silicate (TEOS), fumed silica and Ludox as the silica source, and sodium hydroxide or tetraethyl ammonium hydroxide as basic additives. In order to synthesize aluminosilicate materials, an aluminium source can be added as well. After the solutions preparation, they were kept at temperatures ranging from 100 to 150 °C for 24-144 h. Then, the solid products were filtered, washed and dried. Finally, the calcination process was carried out at 540 °C under a gas-flow of alternately nitrogen and air to remove the templates. Basically, the synthesis of MCM-48 and MCM-41 are quite similar. One can differentiate the synthesis by varying the surfactant to silica ratio in the synthesis solutions, where MCM-41 is usually produced when the ratio is less than one and vice versa [32].

Similar to zeolites synthesis, MCM-41 and MCM-48 also undergo modifications to improve the structure properties. Many modifications have been developed for catalytic application, which is considered as one of the first areas studied in the M41S application. It is believed that the catalytic activity of the siliceous framework can be enhanced by incorporation of other elements (often metals) through three methods: direct addition of the extra element precursors to the synthesis solution, post-treatment of the synthesized materials and incipient wetness impregnation technique. Aluminium modified M41S materials is the most studied metal because it can form acid sites in the framework which enables them to be used in acid catalysis. Other metals that have been incorporated into mesoporous materials are, for instance, copper, cobalt, vanadium, chromium, iron and gallium [32]. Besides catalytic application, the M41S family also find themselves a place in the adsorption field since it exhibits both hydrophobic and hydrophilic character depending upon the exact composition and/or post-modification [15]. Recently, a novel polyethylenimine (PEI)-modified MCM-41 has been prepared and tested as a CO₂ adsorbent. This adsorbent behaves as a "molecular basket" and shows good adsorption capacity over unmodified MCM-41 [33-35]. Huang and Yang (2003) also studied the ability of MCM-48 as gas adsorbent by modifying the surface using amine functional groups. The adsorption capacity was again enhanced by this modified adsorbent [36].

Basically, SBA-15 has the same synthesis route as the M41S and the major difference resides in the use of surfactant and synthesis conditions. The SBA-15 materials are prepared at low temperatures (35 to 80 °C), using amphiphilic poly (alkylene oxide) triblock copolymers as surfactant in acidic media and by the addition of cosolvent organic molecules [18]. The latest research is still concentrate on the improvement of synthesis condition towards short-time synthesis [37]. Not much modification has been reported on the SBA-15 and the recent modification is done by Gray *et. al* (2005) which grafting the SBA-15 with γ -aminopropyltriethoxysilane (APTS) to form aminated-SBA-15 as CO₂ capture sorbents [38]. From catalytic application aspect, aluminium is once again being incorporated into the SBA-15 silica framework via direct synthesis or post synthesis procedures. The FTIR

spectroscopic study is performed to investigate the nature of the acidic sites in the SBA-15 in the recent report [39].

Silica aerogel as aforementioned is another mesoporous material that presents some properties, which are quite remarkable in its application as adsorbent. The synthesis of silica aerogels consists of three steps, which are: i) the preparation of gel through sol-gel process; ii) the aging process, and iii) the drying process. These three steps are involved in all production of silica aerogels. Additional procedures can be implied in order to influence the final product [40]. For further reading, the chronological development of methods for aerogel synthesis is well reviewed by I. Smirnova (2002).

3. Industrial Applications

3.1 Current Applications

From the report of the *American Ceramic Society Bulletin* (Apr 2005), the commercial applications of global market for inorganic nanoporous and microporous adsorbents is estimated at > \$ 1.77 billion. This market is expected to rise at an average annual growth rate of 3.7% to reach \$ 2.12 billion by 2009. Zeolites account for ~ 61% of the total market and it proves the versatility of zeolites as adsorbents. Table 5 shows a list of representative industrial applications of zeolite as gas adsorbent in the early of 1980's. Most of these processes employ pressure swing adsorption cycles over other conventional separation processes such as distillation and cryogenic separation [10].

Table 5: Representative industrial gas separations of zeolite [10].

Separation	Adsorbent
I. Gas bulk separations	Zeolite
Normal paraffins/iso-paraffins, aromatics	Zeolite
N ₂ /O ₂	Zeolite
CO, CH ₄ , CO ₂ , N ₂ , NH ₃ /H ₂	Zeolite, activated carbon
II. Gas purification	
H ₂ O/olefin-containing cracked gas, natural gas, air, and synthesis gas	Silica, alumina, zeolite
CO ₂ /C ₂ H ₄ and natural gas	Zeolite
Sulfur compounds/natural gas, hydrogen, and liquefied petroleum gas (LPG)	Zeolite
NO _x /N ₂	Zeolite
SO ₂ /vent streams	Zeolite
Hg/chlor-alkali cell gas effluent	Zeolite

Contrary to catalytic applications, zeolite adsorbents are mostly applied in a fixed-bed, cyclic operation. Some major industrial adsorption processes using zeolite gas adsorbents are classified as follows: i) hydrocarbon separation processes; ii) drying gases and odours; iii) gas purification and separation; iv) pollution control applications, and v) non-regenerative applications [13].

i) Hydrocarbon Separation Processes

In the petrochemicals industry, hydrocarbon separation is widely studied using zeolites gas adsorbents. Among the processes that have been applied, just to name few are the normal (*n*)-paraffin separation, para (*p*)-xylene separation from mixed C₈ aromatics mixture and olefin separation. Zeolite CaA molecular sieve, which has an effective pore diameter of about 5Å is utilized in the *n*-paraffin separation from a mixture of paraffins. Its pore size possesses sieving effect by adsorbs only *n*-paraffin, and excluding other branched paraffins and a small amount of aromatics existing in the feed stream. *n*-paraffins are very useful hydrocarbon and

their applications include octane value enhancement of gasoline, solvents and raw materials for biodegradable detergents, lube oil additives and alcohol [13].

Revolution of fabrics industry towards polyester fibres has boosted the demand of *p*-xylene as raw material in the last two decades. New adsorption process has been developed by using ion-exchanged forms of synthetic faujasite zeolite to separate *p*-xylene from a C₈ aromatics mixture. Apart from the polyester product, the manufacture of polyethylene terephthalate for bottles recyclable and environmental benign has diversified the use of *p*-xylene and these demands have necessitated the continuous development of this new adsorption process [13, 41]. Other hydrocarbon process, which is available in industrial applications, is the olefin separation from a feedstock containing olefins and paraffins. The UOP's Olex process is well known in this separation process, which uses a zeolite adsorbent to produce light olefins product with improved purity [41].

ii) Drying Gases and Odours Removal

Zeolites are known as very efficient desiccants due to its highly hydrophilic surface. The 3A molecular sieve adsorbent is in particular suits for the drying purpose (selective adsorption of water) because of its small pore size and it is very useful in drying other polar gases as well. For instance, in petrochemical industry, zeolites are used in cracked gas drying, where 3A molecular sieve adsorbents have an overall economic advantage over other commonly used desiccant. Apart from that, zeolites adsorbents are used to remove impurity, including odours in the specialty and fine chemicals as well as pharmaceuticals industry [13, 41].

iii) Gas Purification and Separation

Natural gas treating is one of the major parts in this application. Usually, zeolites are used to purify or sweeten natural gases through the removal of impurities such as carbon dioxide, sulphur dioxide, hydrogen sulphide and water thereby producing a higher value natural gas which might otherwise be useless due to unsuitable quality caused by high concentrations of impurities. Besides, purification helps to protect transmission pipelines from corrosion and freezing problem. 4A and 5A zeolites are generally used to dry and desulfurize natural gas. However, 13X can be used when sulphur content is high in the natural gas [41].

In addition, oxygen enrichment of air is another application of gas separation that benefits the society. Nowadays, the pressure swing adsorption and vacuum-swing adsorption processes by using zeolites as adsorbents able to produce O₂ up to 95% purity. They are definitely advantageous over the cryogenic air separation because zeolites provide benefits in energy efficiency, process efficiency, improved processing rates and product quality as well as good environmental impact [41].

In petroleum refining and petrochemical processes, zeolites adsorbents are capable to remove impurities from a variety of streams down to low level to protect the downstream processing by virtue of its affinity and strong binding to polar compounds, including hydrogen sulphide, organic chlorides, CO and mercury. Therefore, it is clear that the impact of zeolites in the petrochemical field is undeniable and it is worthwhile to continue the development in the zeolites adsorption towards higher performance and capability.

iv) Pollution Control Applications

Industry often encounters with pollution problems from its effluent streams. Several pollution controls have been implemented to preserve the environment from detrimental effect. One of the strategies to treat the industrial off-gas streams is the gas adsorption process. There is a keen interest for industrial people to develop efficient adsorbents and it has been found out that zeolites manifest good adsorption capability that make them a better choice for many cost effective air pollution control, for example, volatile organic compounds and the hazardous air pollutants such as SO₂, H₂S and NO_x can be effectively removed by zeolite adsorbents with high selectivity compared to other adsorbent media namely activated carbon. Several commercial processes have been developed by the Union Carbide corresponding to the pollution issues such as mercury vapour removal, NO_x removal from nitric acid plant off-gas and SO₂ removal from sulphuric acid plant-off gas [13]. Until today, many promising zeolites for air purification and odour controlling are being developed to meet the need for industrial applications.

v) Non-regenerative Applications

Apart from the aforementioned fixed-bed, cyclic operation, zeolites gas adsorbents can be applied in process, which involve no regeneration and therefore no cyclic operation. Drying Freon-type refrigerants and manufacture of insulated glass windows are some important non-regenerative applications. In the refrigerants application, zeolites can function as desiccant to keep the refrigerants super dry thus protecting the system from freeze-up and corrosion while in most insulated glass produced worldwide, zeolite is primarily used as desiccant contained in channels that separate the window panes to prevent fogging, mists or formation of dew between the window panes especially in winter months for seasonal countries [13, 41].

3.2 Future Prospects

Although the past four decades have seen a dramatic growth in adsorption separation process, only the microporous materials such as zeolites have found extensive use in the industrial applications as described earlier. Indeed, these zeolites adsorbents can be further developed in another interesting field apart from the existing industrial applications, which is the natural gas storage application. The inherent properties of zeolites, which provide large surface area, high packing density and volume plus its energy efficiency enables them to be potentially used as an alternative gas storage medium known as adsorbed natural gas (ANG) process over conventional compressed natural gas (CNG) method. However, when compared to activated carbons, which have been known as very good adsorbents for ANG storage, zeolites have relatively lower micropore volumes than activated carbon. In addition, zeolites are extremely hydrophilic and this character can lose their adsorption capacity for methane with time due to preferential moisture adsorption [42]. Therefore, some modifications and improvements have to be performed prior to the usage of zeolites in natural gas storage.

Continuous exploration of zeolites potential applications due to its sorption property has led to the usage of zeolites in gas sensing technology. Since most of the current sensors are suffering from the problem of poor selectivity, a novel method has been proposed to enhance the selectivity of existing sensors by placing ahead of them a filtering cell used to physically separate the interfering molecules from the interesting ones. For instance, Hugon and his co-workers had associated the SnO_2 gas sensor with zeolite filter to have a selective detection of alkanes in the presence of ethanol by means of zeolites adsorptive property, where they can strongly adsorb polar molecules than non-polar molecules. The results verified the selective adsorption of ethanol over the mixture of methane or *n*-hexane [43]. In short, by utilizing the adsorptive property of zeolites, gas sensing technology can breakthrough its bottleneck into a wider applications in domestic and industrial field.

When we extend the applications of zeolites to the fuel cell technology, it seems that they can also be used in both the hydrogen purification and hydrogen storage. Since the fuel cells, for instance proton exchange membrane fuel cells (PEMFC) have good perspectives of a significantly higher efficiency and of almost no emission of pollutants, they have been extensively studied in the last two decades for many applications especially for low emissions vehicles. Although pure hydrogen is the ideal fuel for the fuel cell, most researchers generate hydrogen from methanol reforming or other liquid hydrocarbon fuel in fuel processor due to practical and economical limitation for pure hydrogen usage [44]. In tandem with the generation method used, hydrogen purification, where the removal of CO ca. 1% remaining in the reformates to a trace level is essential to avoid a serious poisoning of anode catalysts in the cells. So far, a preferential oxidation of CO is used to purify the hydrogen by developing zeolites-supported noble-metal catalysts through the zeolites chemical and/or physical molecular sieve effect to make CO react with O_2 . Different zeolites supported Pt catalyst have been tested and their selectivity was found to be affected by the types of support used, in the order, A-type zeolite > mordenite > X-type zeolite > Al_2O_3 [45].

At the same time, hydrogen storage is currently another popular topic in the research of fuel cell technology. One of the most common candidate methods is the physisorption [46]. Zeolites A, X and Y and their ion-exchanged forms have been chosen for study and the results showed a maximum storage capacity of 1.81 wt% (at -196 °C and 15 bar) for NaY zeolite. As a conclusion, these materials show great potential as low cost media for

stationary hydrogen storage applications [47]. In addition, zeolites have also been used as host to confine the semiconductors such as Se, RbSe and CsSe in the quantum confinement study. This study was successfully demonstrated and these semiconductors are known to preserve their semiconducting behavior upon melting [48].

Up to date, there is still no obvious industrial applications for mesoporous materials even though the development of mesoporous materials in adsorption have aroused quite recently due to the increasing demands in both industrial and fundamental studies, for example, separation and synthesizing large molecules and treating heavy feeds. Most attentions are still concentrating on the rationalization of their synthesis, the formation mechanism, together with a rapid study on the implications of changes of the synthesis parameters on the final characteristics of the structure rather than their industrial applications. It is an unavoidable phenomenon since they are relative novelty materials (just over few decades) compared to zeolites. However, a few trends for potential applications already emerge from those studies with most applications devoted to the catalytic application instead of adsorption application. Several excellent reviews, which give a reasonably complete overview of their application in catalysis for the different mesoporous materials have been published [32, 49]. Indeed, the earlier exploitation of the mesoporous materials was examined in the adsorption field due to their large pore volumes. Such applications include the removal of volatile organic compound (VOC) from the industrial setting. To achieve such goals, the behaviour of a variety of adsorbates has been gathered and the data agree that mesoporous materials have large accessible internal pore volumes, which can be filled at pressures commensurate with the pore size.

Early studies and characterizations such as adsorption on argon, nitrogen, oxygen, water and hydrocarbons are mostly devoting on the MCM-41, the hexagonal member of the M41S family because of its earlier discovery compared to other members. The results show that MCM-41 has a narrow pore size distribution and exhibits extraordinary pore volume compared to classical microporous materials, which will enhance the adsorption capacity. However, there is a limitation of their type IV isotherm behaviour in the adsorption capacities because most industrial applications required adsorption at low partial pressures (Type I isotherms) i.e. for VOC uptake rather than high partial pressure [50]. Besides, to be an effective adsorbent, many properties such as adsorptive capacity and selectivity, adsorption-desorption rate, physical strength and attrition resistance, low catalytic activity, thermal-hydrothermal stability, chemical stability and particle size and shape have to be clarified [13]. It therefore complicates the studies.

Despite the obstacles, the most recent research on the adsorption using novel molecular basket adsorbent, which is the MCM-41 modified with polymer has come to a good conclusion on the application of mesoporous materials as gas adsorbents. The new developed adsorbent is capable to adsorb a small amount of N_2 , O_2 and CO together with a selective CO_2 separation from the flue gas of a natural gas fired boiler [35]. Undoubtedly, with this successful case study, it is believed that the future prospects of these mesoporous materials in the adsorption field is foreseen with the condition that their adsorbent-adsorbate interaction is thoroughly investigated and their modification towards stable chemical and physical properties is continuously being explored.

For silica aerogel, its application as gas adsorbent is still not clear. Its most promising application is in the field of thermal insulation due to its low thermal conductivity. However, its potential application as gas adsorbents is not limited with the scarce information since it has a high specific surface area and porosity that can be utilized in the adsorption process [21]. Hence, further studies are required to verify its application in this field.

4. Conclusion

Silica based gas adsorbents have gained much attention in the research studies due to their unique property as nanoporous materials. The effort to enable them to be used as industrial gas adsorbents requires an exploratory research on structure, property and adsorption phenomenon. Zeolite has found itself as successful gas adsorbent after unceasing research

activities for decades. Therefore, the continuous studies and systematic investigations of the structure and adsorption properties of the mesoporous materials will certainly aid in the future research and bring opportunity to the utilization of mesoporous materials in industrial gas adsorption process.

Acknowledgements

The authors acknowledge the Ministry of Science, Technology and Environment Malaysia for the financial support through IRPA grant No. 74512.

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