



A Review on Mesoporous Chromium Silicate Material: Synthesis, Characterization and Applications

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Abstract:

This review introduced many kinds of mesoporous materials involving siliceous mesoporous material. These mesoporous materials called advance material because of with high surface area, large pore size, and large pore volume has attracted great interest for their potentially wide applications, such as in catalysis, adsorption, separation, and ion exchange. Up to now, a variety of highly ordered mesoporous silica materials have been successfully synthesized by number of researchers. Mesoporous materials are used as a catalyst which also one of the fundamental pillars of green chemistry, the design of chemical products and processes that reduce or eliminate the use and generation of hazardous substances. This review focus on mesoporous materials, their different forms, applications and advance characterization techniques of mesoporous material has discussed in brief. The green approach in the production of alkylaromatic ketones over hexagonally ordered mesoporous CrSBA-15 catalysts, which were used, in green routes, in the liquid-phase oxidation of alkylaromatics has discussed here. Mesoporous catalyst was found to be a highly active, recyclable and promising heterogeneous catalyst for selective synthesis of alkylaromatic ketones. The review presents systematically as uses of mesoporous materials, their characteristics techniques and applications in synthesis of aromatic ketones.

Keywords: Green chemistry, Mesoporous catalyst, Alkylaromatic ketones, Applications

I. INTRODUCTION

For almost 30 years, much attention has been paid to designing and obtaining new materials. The interest in such materials stems from the fact that they have at least one component of their structure on the nanoscale and thus show a number of unique properties and have a wide range of applications (Jarmolińska, S. *et al.* 2020). Meso, the Greek prefix, meaning—in between, has been adopted by IUPAC to define porous materials with pore sizes between 2.0 and 50.0 nm. The synthesis, characterization, and application of novel porous materials have been strongly encouraged due to their extensive range of applications in adsorption, separation, catalysis, and sensors. Out of them, catalysis is one of the fundamental pillars of green chemistry, the design of chemical products and processes that reduce or eliminate the use and generation of hazardous substances (Hudlicky, T. *et.al.* 1994). The design and application of new microporous material are simultaneously achieving the dual goals of environmental protection and economic benefits (Hudlicky, T. *et.al.* 1994). There are twelve principles of green

chemistry formulated by Paul Anastas and John Warner in the 1990's shown in Fig.1. These principles provide a roadmap for chemist to implement green chemistry.



Fig. 1: The principles of green chemistry

Mesopores material offers numerous green chemistry benefits including lower energy requirements, catalytic versus stoichiometric amounts of materials, increased selectivity, and decreased use of processing and separation agents, and allows for the use of less toxic materials. The first report on the synthesis of mesoporous materials was published in the beginning of the 1990's and it has been a milestone in materials chemistry. Heterogeneous catalysis, in particular, addresses the goals of green chemistry by providing the ease of separation of product and catalyst, thereby eliminating the need for separation through distillation or extraction. In addition, environmentally benign catalysts such as clays and zeolites, may replace more hazardous catalysts currently in use. This review highlights a variety of ways in which catalysis may be used in green chemistry point of view. The benefits to human health, environment, and the economic goals realized through the use of catalysis in manufacturing and processing are illustrated by focusing on the catalyst design and catalyst applications (Muzart, J. *et. al.*1992). There are a large number of studies of porous material trends in the material science. However, since the focus of this research is on mesoporous silicate material.

Mesoporous chromium silicate catalysts

Porous solids are universal due to their many versatile advantages such as a large surface area, an enhanced accessibility and the ability to anchor different chemical functionalities on their surface. The use of molecular and supramolecular templates, especially surfactants, has been one of the most successful strategies for the manufacture of materials with a controlled porosity. The history of porous materials started with discovery of natural zeolites, that are microporous aluminosilicates of crystal structure, having a developed system of micropores (Guo, C. *et.al.* 2003). Their use in the chemical and petrochemical industry has brought significant benefits both to economy and the natural environment. The absorbent surface property of the mesoporous catalysts makes them to absorb long-chain organic molecules. Mesoporous silica catalyst has

become apparent as a promising and novel vehicle due to their unique mesoporous structure that preserving a level of chemical stability, surface functionality and biocompatibility ensure the controlled release, and target drug delivery of a variety of drug molecules. Heterogeneous catalysis with mesoporous material in liquid phase is shown in fig.2.

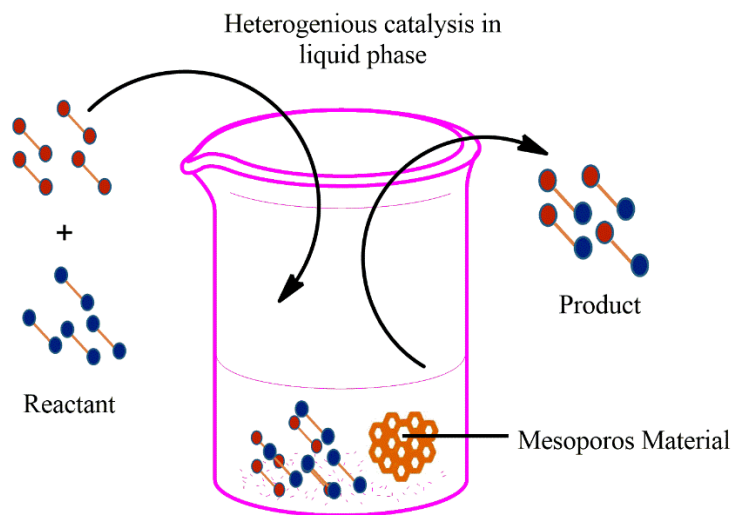


Fig.2: Heterogeneous catalysis with mesoporous material in liquid phase

II. APPLICATIONS OF MESOPOROUS MATERIAL

As mesoporous material has very good porosity and large surface area it opens up different application eras as shown in fig.4

1. As a catalyst in organic reactions: In number of organic reactions mesoporous material is use as a heterogenous catalyst some of them listed below:

a) Oxidation of alkylarenes:

Selective side chain oxidation of alkylarenes to aromatic ketones is one of the most important fundamental reactions. It is mainly based on the conventional homogeneous catalysis in chemical industries and is mostly carried out with acetic acid as the solvent as well as molecular oxygen as the consumable oxidant. In these catalytic oxidations the poisonous Cr (VI) reagents are also used as the homogeneous catalysts. However, the conditions of the conventional method are often harsh, the reagent mixture is corrosive and highly toxic. The reaction temperature is high and demands the use of autoclave reactors, which are made of expensive raw materials due to the corrosive medium. Therefore, there is a need for the development of new heterogeneous catalysts in the production of alkylaromatic ketones with higher selectivity's.

Oxidation of alkyl aromatic is largely used in the production of aromatic ketones, which are key products in the manufacture of pharmaceuticals, resins, alcohols and tear gas (chloroacetophenone) and are also used in perfumery industries. The aromatic ketones are produced by the liquid-phase alkylarenes oxidation using molecular oxygen or air as the oxidizing agent over homogeneous chromium containing inorganic/organic catalysts and organometallic catalysts (Alcántara, R.*et. al.* 2000). Although homogeneous catalysts exhibit excellent activity and selectivity, the technical problems encountered in their applications, such as the difficulty in product separation and self-aggregation of active sites, have slowed down their industrial applications. These catalytic systems also create other problems such as handling, catalyst recovery, recycling, environmentally unsuitable and highly produced tarry wastes. To overcome these problems, solid acid catalysts have been successfully used in the liquid phase oxidation of alkylarenes (Choudary, B. M., *et. al.* 1992, Lempers, H. E. B. *et. al.* 1998 & Das T. K. *et. al.* 1997). The heterogeneous chromium reagents are widely used as catalysts for the oxidation of alkylaromatic compounds in

organic chemistry (Sheldon, R. A., *et. al.* 2002). Therefore, several chromium containing solid materials have been utilized as the catalysts in liquid-phase oxidation of alkylaromatics with a variety of oxidizing agents (Corma, A., *et. al.* 2003). For example, the micro-structured Chromium-containing Aluminophosphate-5 (CrAPO-5) catalyst produced less conversion with higher aromatic ketone selectivity's under liquid phase alkylarenes oxidation because its small pore diameter ($\sim 0.73\text{\AA}$) restricts the diffusion of reactants/intermediate and products (Sakthivel, A., *et. al.* 2002).

Recent years, the mesoporous chromium containing Mobile Composition Matter-41 (CrMCM-41) catalyst was used in the alkylarenes oxidation to improve the conversion with higher aromatic selectivity because it has larger uniform pore size than CrAPO-5 (Sakthivel, A., *et. al.* 2002). In 2007, Selvaraj and co-workers reported the well hexagonally uniform pore structured (Chromium containing Santa Barbara Amorphous-15 (CrSBA-15) (Selvaraj, M., *et. al.* 2002 & Selvaraj, M., *et. al.* 2007). The mesoporous CrSBA-15 catalysts have been successfully used for the synthesis of very useful fine chemicals with higher selectivity, and have superior catalytic activities than CrMCM-41 due to the higher Cr species coordinated on the thick silica pore walls [12-14]. Even though the CrSBA-15 catalysts give the higher selectivity of aromatic ketones, the toxic chromium (VI) oxides are with the ketone products. Therefore, a green procedure needs to recover green mesoporous chromium silicate catalysts. Some of important mesoporous catalyst are shown in fig.3.

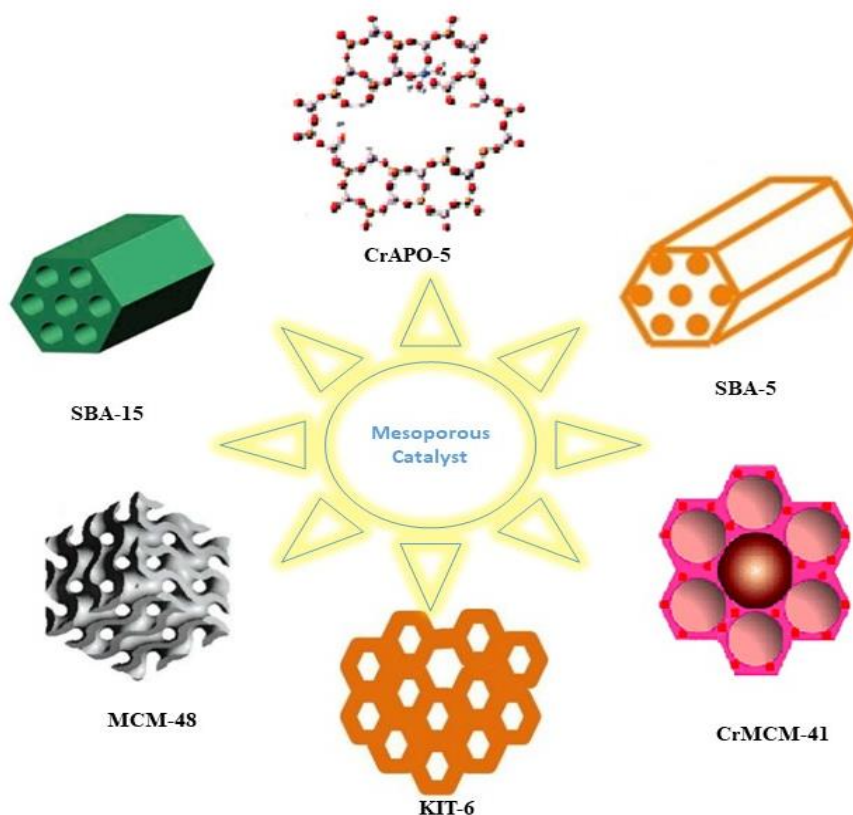


Fig.3: Different Mesoporous catalyst

b) Synthesis of mesoporous chromium silicate catalysts

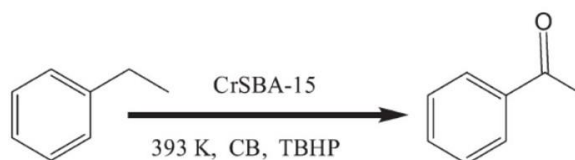
As per the M. Selvaraj and S. Kawi, Mesoporous CrSBA-15 molecular sieve materials were synthesized using a Pluronic P123 as a structure-directing agent. In a typical synthesis, 4 g of Pluronic P123 was added to 25 mL of water to get a clear solution (Selvaraj, M., *et. al.* 2011). Thereafter, a required amount of dilute HCl solution was added, and the solution was again stirred for another 1 h for the hydronium ions to be associated with the alkylene oxide units. Next, 9 g of tetraethyl orthosilicate and a required amount of the desired Cr source were added, and the resulting mixture was stirred for 24 h at 313 K and finally get the product (Selvaraj, M., *et. al.* 2008). Finally, the sample were calcined at 813 K in air for 6 h for complete removal of the template. The calcined as CrSBA-15 catalysts (Selvaraj, M., *et. al.* 2012).

c) Synthesis of green mesoporous chromium silicate catalysts

The original CrSBA-15 catalyst treated with ammonium acetate in order to remove the extra-framework chromium species was reported by Selvaraj et al. In a typical procedure, about 0.5 g of the calcined CrSBA-15 catalyst was washed with 1 M ammonium acetate (150 ml) solution under constant stirring for 12 h at an ambient temperature. Finally, the CrSBA-15 catalyst was filtered, calcined at 773 K for 6 h in air to remove the adsorbed species. This treatment of catalyst is denoted as green CrSBA-15 catalyst (Selvaraj, M., *et. al.* 2010).

d) Synthesis of aromatic ketones

Selvaraj research group developed a green method in the production of alkylaromatic ketones over hexagonally ordered mesoporous CrSBA-15 catalysts as shown in Scheme 1, which were used, in green routes, in the liquid-phase oxidation of alkylaromatics. A promising chemical treatment method was used with ammonium acetate solution to remove the toxic nature of non-framework chromium oxides deposited on the surface of calcined CrSBA-15, and the obtained green mesoporous CrSBA-15 catalyst was used to find its catalytic activity while the recyclability of mesoporous CrSBA-15 catalysts was also studied (Selvaraj, M., *et. al.* 2005). On the basis of all catalytic results, the mesoporous CrSBA-15 catalyst produced a higher selectivity of alkylaromatic ketones (76–100%) as compared to other CrMCM-41 catalysts and was found to be a highly active, recyclable and promising heterogeneous catalyst for selective synthesis of alkylaromatic ketones, as shown in Table 1 (Borsari, C., *et. al.* 2020).



Scheme 1: Oxidation of ethylbenzene over CrSBA-15
(CB: Chlorobenzene and TBTP: t-butylhydrogen-peroxide)

Sr.no	Substrate	Ketone product	Conversion (%)	Selective Ketone (%)
1	1-ethyl-4-methoxy benzene	1-(4-methoxyphenyl) ethanone	96	99
2	1-chloro-4-ethyl benzene	1-(4-chlorophenyl) ethanone	70	90
3	1-ethyl-4-nitrobenzene	1-(4-nitrophenyl) ethanone	70	80
4	1-ethyl-2-methoxy benzene	1-(2-methoxyphenyl) ethanone	85	90
5	1-ethyl phenol	1-(4-hydroxyphenyl) ethanone	80	92
6	1-chloro-2-ethyl benzene	1-(2-chlorophenyl) ethanone	65	84
7	1-ethyl-2-nitrobenzene	1-(2-nitrophenyl) ethanone	60	80
8	2-ethyl phenol	1-(2-hydroxyphenyl) ethanone	62	75

Reaction condition: 0.1 g of CrSBA-15 catalyst, reaction time= 10 h, temperature=373 k and 1:3 ratio of substrate.

Table: Selective oxidation of ethyl benzene compounds over CrSBA-15 (Selvaraj, M., *et. al.* 2010).

2. Water purification:

The world is suffering problem of lack of water and unclean water so to overcome this global issue of water supply, treatment of water and reuse of waste water is crucial. Mesoporous materials have some unique features, porosity and high surface area, with this it also has good thermal and mechanical stability which can be employed it in water treatment plant (Lu M., *et. al.* 2013). Some of researchers are working on carbon bounded mesoporous material and these types of material are utilizes for absorption of heavy metal ions from waste water. With this also silica supported mesoporous material are employed in waste water treatment for because silica increase the porosity of native material and absorption rate is increases (Al-Shehri *et. al.* 2019).

3. Senser:

Mesoporous materials are finding increasing utility in sensing applications. Sensing property is depending on porosity and surface of material. As the surface area of mesoporous material increases the diffusion of analyte is increases. Pyrylium-containing mesoporous materials were used for the chromo-fluorogenic sensing of biogenic amines in aqueous environment (Almeida, M. G., *et. al.* 2010). Another example of the practice of mesoporous materials as sensors was designated for the determination of methylmercury in real samples using organically capped mesoporous inorganic materials capable of signal amplification (Climent, E., *et. al.* 2009). Alternative effort using mesoporous materials as sensors was established by Zhaoa *et al.* (2012). In this paper, a novel chemiluminescence-molecular imprinting (CL-MI) sensor for the determination of fenprothrin (used to control the range of insects, especially mites, in fruits and vegetables) in foodstuff was developed (Zhao P. *et. al.* 2012).

4. Chromatography:

The principal of chromatography is based on surface area and mesoporous silica compounds have large surface area, pore volume and narrow pore-size, makes it a best choice for size exclusion chromatography. Due to this property, these materials have been application High Pressure Liquid Chromatography (HPLC) as a stationary phase. It may also be employed in size exclusion chromatography, proteomics separations and enantioselective HPLC (Hou L. *et. al.* 2018).

5. In electrochemistry:

The relation between electrochemical science and the chemistry of porous silica materials is the capability of electrochemical techniques to employ redox-active species to diffuse into porous cavities. Numerous electrochemical properties can be detected. The properties of the porous materials developed the main driving force and have been broadly exploited in electrochemistry via numerous innovative applications (Walcarius, A. 2013). For example, porous silica can be used in electroanalysis, where the incorporation of mesoporous materials to an electrochemical interface shows selectivity on the basis of solute size, shape and charge. As defined by Etienne and co-workers. The electrochemical response of porous silica is intensely influenced by the charge and the size of the molecular probe (Zhu W. *et. al.* 2007).

6. Adsorption:

The mesoporous material has high surface area which facilitates their use as adsorbents for different liquids, gases and toxic heavy metals (Chiang, W. S., *et. al.* 2016). The adsorption capacity of this the mesoporous silica materials is majorly depending on its surface properties such as hydrophilicity, hydrophobicity, or functionality. With

this this material has multiple applications like waste water treatment, xylene separation using adsorption, storage of gases (e.g., CO₂, H₂, O₂, CH₄, H₂S) and in separation of biological and pharmaceutical compounds, have been addressed by employing mesoporous materials as adsorbents (Trouvé, A., *et. al.* 2012).

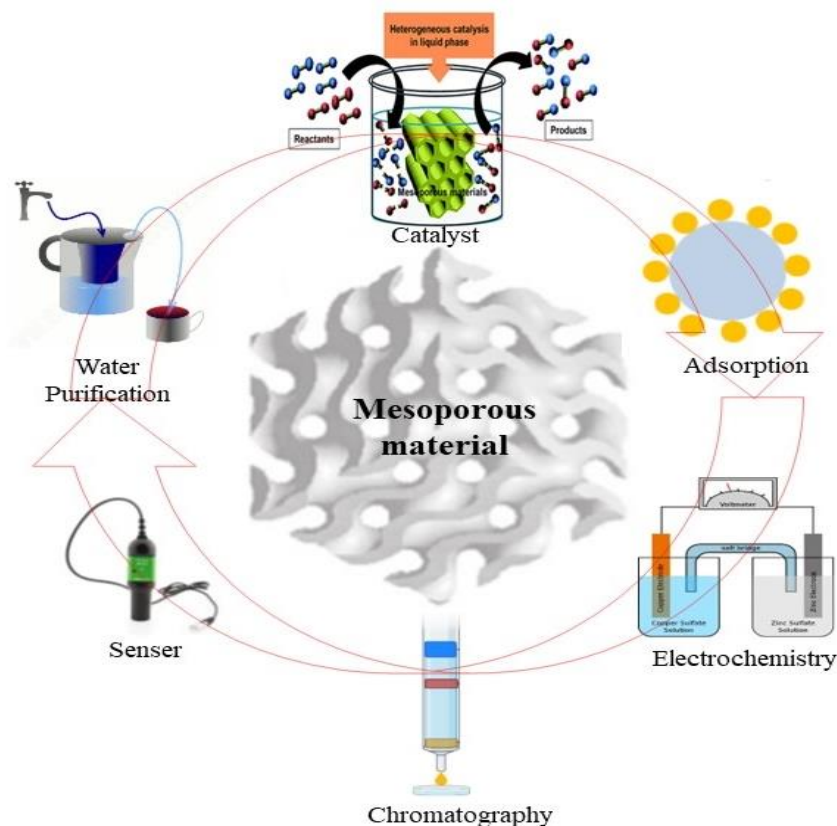


Fig.4 Applications of mesoporous material in different fields

III. DISCUSSION OF CHARACTERISTIC RESULTS OF CATALYSTS

The catalysts synthesized by method have been characterized by Fourier- Transform Infrared (FT-IR), UV-vis spectroscopy, inductively coupled plasma atomic emission spectroscopy (ICP-AES), X-ray powder Diffraction (XRD), N₂ (Nitrogen isotherm), ESR (Electron Spin Resonance Spectroscopy), FE-SEM (Field Emission Electron microscope), and TEM (Transmission Electron Microscope) according to the published procedure (Gómez-Cazalilla, M., *et. al.* 2007, Shirsath N. B. *et. al.* 2020 & Shirsath N. B. *et. al.* 2022).

IV. CONCLUSION

The literature reviewed revealed modern approach of mesoporous materials over the last few decades. The review also discusses the systematic synthesis, characterization and applications of silicate mesoporous material. Majorly this type of porous material is used as green catalyst and it is important according to green chemistry principals such as Mild conditions, good to excellent yields, reusable catalysts and short reaction time are the advantages of the mesoporous catalyst in organic synthesis. These modified materials can be used in a variety of applications such as catalysis, adsorption, sensor and separation as chromatographic column packing. These types of mesoporous material have such advance applications due to its high surface area and porosity which will open new areas of research.

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Conflicts of interest

There are no conflicts to declare.

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