The properties of ion-exchange resin vary with the concentration of divinylbezene and the kinds of ion-exchange group. Treating with high concentration of sulfuric acid or sodium hydroxide solution attains regeneration of ion-exchange resin.

In addition to above cationic and anionic ion-exchange resin, chelating resin, which shows high affinity to certain metal is known. This resin is involved in cationic resin in wide interpretation. It has been reported that metal loaded chelating resin was used for anion remover.

1.4. Mesoporous and Mesostructured Materials

1.4.1. Mesoporous Silica

The mesoporous silica have received attention for its large surface area and well-defined pore size (adjustable in the range 2-50 nm) and uniform pore shape (Figure 1.3). The pore size of mesoporous materials is unlike those of the crystalline zeolite materials with maximum pore dimensions of < 2 nm. Therefore, their application to catalyst and catalyst support is attractive because a larger organic molecule, which cannot penetrate into zeolite’s micropore, is able to access into mesoporous channel.
Chapter 1.

Figure 1.3. Examples of micro-, meso-, and macroporous materials, showing pore size domains and typical pore size distributions (Sanchez et al., Chem. Rev., 102 (2002) 4093.).

Such new type mesoporous silicate was reported early in the 1990s. Yanagisawa and co-workers\(^9\) made the first discovery of mesoporous silicate. They pillered a layered silica of kanemite with surfactant cation and, after calcination, obtained a mesoporous aluminosilicate (FSM-16). Mobil researchers\(^10\) synthesized mesoporous silica (MCM-41) via different process from that of FSM-16. The synthetic mechanisms proposed by Mobil researchers are
Two possible synthetic mechanisms of mesoporous silica (MCM-41) were proposed by Mobil researchers. The first pathway is surfactant molecules organized liquid crystalline phase, which plays the role in a template. Inorganic silicate forms an inorganic structure around template arrays. In the second pathway, silicate influences the formation of this liquid crystal phase.

On the other hand, Stucky et al. proposed the so-called “cooperative templating mechanism”. Prior to silicate addition, the surfactant is in a dynamic equilibrium between spherical and cylindrical micelles and single molecules. Upon addition of a silica source, the multi charged silicate species displace the surfactant counterions to form organic-inorganic ion pairs that reorganize first into a silicatropic mesophase followed by silica cross-linking. This mechanism is based on the following experimental results, which cannot be explained by the Mobil researchers.
(i) it is possible to make three phases (hexagonal, cubic, lamellar) by changing the amount of silica source, everything else being the same.

(ii) mesoporous materials may be made in the presence of surfactant concentrations below CMC (Critical Micelle Concentration)

(iii) MCM–41 could be made from short-chain surfactants, which do not form rod-like micelle.

(iv) MCM-41 and MCM-48 may be formed at the temperatures above 70 °C where rod-like micelle are not stable.

In 1994, Huo et al.\textsuperscript{11,12} reported the first acid synthesis of mesoporous silica (SBA-1, SBA-2) unlikely MCM-41. They introduced four synthesis route \((S^+I^-), (S^-I^+), (S^-X^-I^+),\) and \((S^+M^+I^-)\) where \(S\) is surfactant, \(I\) is inorganic molecular species, \(X^-\) is Cl\textsuperscript{-} or Br\textsuperscript{-}, and \(M^+\) is Na\textsuperscript{+} or K\textsuperscript{+}. The synthesis mechanisms of various mesoporous transitional metal oxide, as well as silica, were explained by using these synthetic interactions.

In addition to Huo’s proposals, Tanev and Pinnavaia proposed a neutral templating synthesis mechanism based on hydrogen bonding between primary amines and neutral inorganic species. The interaction is different from Huo et al.’s proposal. The synthesis route is expressed as \(S^0I^0\). The superscript “0” means
neutral species. Because of weak interaction between the surfactant and inorganic species, elimination of the surfactant is relatively easier than in the case of using ionic surfactant. The synthesized mesoporous silica, which has wormhole structure, is called HMS.

Zhao et al. synthesized larger pore size mesoporous silica (SBA-15) by using triblock copolymer(poly(ethylene oxide)-poly(propylene oxide)-poly(ethylene oxide), PEO-PPO-PEO) instead of ionic surfactant. The pore size was uniform and could be controlled over a range from 46 to 300 Å. Various applications such as catalyst and adsorption are interesting because those materials also have uniform and large pore size.

Thus, many researchers have proposed synthetic mechanisms of mesoporous material. Surfactant molecules are used to form supramolecular assemblies in solution. These self-assembled structures act as templates for the formation of the porous matrix.

Three groups of researchers have independently synthesized examples of a new class of hybrid porous materials that incorporate organic components within a silica framework in 1999. The hybrid mesoporous silica, which consists of a silica framework with ethane bridging groups was first reported by Inagaki et al.\textsuperscript{13} Ozin and co-workers synthesized periodic mesoporous organosilicas (PMO) which have ethane,\textsuperscript{14} benzene,\textsuperscript{15} thiophene,\textsuperscript{15} and ferrocene\textsuperscript{15} bridging groups in silica.
frameworks. Stein and co-workers synthesized similar compounds, which is called them unified organically functionalized mesoporous network (UOFMN). Its channel have wormhole structure was differ from the other two compounds.\(^{16}\) Dai and co-workers synthesized new inorganic-organic ordered composite using organosilane with trialkylsilyl groups connected by linear, square, or tetrahedral bridges of coordination compounds and sodium dodecyl sulfate(SDS).\(^{17}\)

Recently, the application of such mesoporous materials has been developed in various fields. Transition metal oxide expected to be shown different property compared with silica. However, the synthesis of mesoporous transitional metal oxides are more difficult than that of mesoporous silica.

### 1.4.2. Mesostructured and Mesoporous Materials

As can be seen above, mesoporous silica is obtained by elimination of template molecules from mesostructured silica, which is the silica-surfactant micelle composite. Here, I designate inorganic component-surfactant micelle composites as "mesostructured material" and distinguish with mesoporous materials.

Many catalyses over metal (oxide) loaded mesoporous silica have been investigated. Investigation of inorganic wall component of mesoporous material is interesting for various application; catalysts, catalyst supports, and sensors. A major problem of nonsiliceous oxide composites is thermal stability. The
surfactant removal by calcinations, as used for preparing porous silicates, leads to collapse of its ordered structure. Incomplete condensation and redox process would result in the thermal instability.

The first synthesis of transitional metal oxide mesostructured materials\textsuperscript{18} was reported in 1995. Nowadays, many elements were used as inorganic components of mesoporous/mesostructured materials as follows.

\textsuperscript{Fe}\textsuperscript{19}, \textsuperscript{Sn}\textsuperscript{20,21,22}, \textsuperscript{Ge}\textsuperscript{23}, \textsuperscript{Ti}\textsuperscript{18,24,25,26}, \textsuperscript{Zr}\textsuperscript{27,28}, \textsuperscript{Nb}\textsuperscript{29}, \textsuperscript{Al}\textsuperscript{30}, \textsuperscript{V}\textsuperscript{31,32}, \textsuperscript{Y}\textsuperscript{33,34}, \textsuperscript{Mn}\textsuperscript{35}

Various transition metals were ingredient to mesostructured and mesoporous material. The synthetic mechanisms of these materials are explained by Huo’s route as mentioned above.

In addition to the mesoporous transitional metal oxide, a lot of mesoporous and mesostructured transitional metal phosphate and sulfate have been synthesized. Their synthesis methods could be divided into following four methods.

1. Using transitional metal sulfate as inorganic precursor
2. Using alkylsulfate and alkylphosphate as organic templates
3. Adding sulfuric acid and phosphoric acid
4. Phosphate treatment for mesostructured materials

Synthetic examples of non-siliceous mesoporous and mesostructured materials were well reviewed by Sanchez et al.\textsuperscript{36}

Additionally, there has been new approach for mesostructured material
synthesis. Many types of transitional metal mesostructured materials have been used phosphate or sulfate as inorganic wall constituent. Recently, novel Zr-carbonate mesoporous material has been reported.\(^{37}\)

### 1.5. Mesoporous/Mesostructured Materials as Adsorbents

#### 1.5.1. Surface Modified Mesoporous Materials

As mentioned above, mesoporous materials are expected to be effective adsorbents due to its high surface area reaching to 1000 m\(^2\) g\(^{-1}\). Therefore, many researchers reported application of mesoporous silica for adsorbents. In many cases, inside wall of the pore was modified with metal, metal oxide, or organic functional groups because the weak interaction between silica surface and adsorbates is usually weak and we need to enhance affinity for adsorbates. Organo-functionalized silicas have been reported since 1996.\(^{30}\) Representative examples of organo-functionalized silicas were listed in Table 1.4.

**Thiol functionalized mesoporous silica**

Several groups have reported the application of thiol groups functionalized mesoporous silica. It is known that sulfur forms hardly soluble compounds. Liu and co-worker\(^{38,39}\) showed that the first example of toxic metal removal with the functional group introduced mesoporous silica (MPTS). They modified
mesoporous silica with thiol group and removed Hg$^{2+}$ ion. The thiol group on mesoporous silica formed monolayer and showed mercury removal property. The thiol-functionalized mesoporous silica retaining 5.2 mmol of S per one gram showed 210 mg-Hg/g (1.05 mmol-Hg/g) of removal capacity. The distribution coefficient, $K_d$, has been measured to be as high as $10^8$, ($K_d = M_t/M_s$, where $M_t$ is the amount of adsorbed metal ($\mu$g) on 1 g of adsorbing material and $M_s$ is metal concentration ($\mu$g/mL) remaining in the treatment waste stream.)

Pinnavaia and co-workers\textsuperscript{40} compared the removal of Hg$^{2+}$ with two types of mesoporous silicate; MCM-41 with an anionic silicate framework and HMS with an electrically neutral framework. The thiol grafting process of MCM-41 proceeded more effectively than that of HMS. The reason is that the concentration of surface hydroxyl groups of MCM-41 was higher than that of HMS. Thiol-functionalized HMS derivative was able to bind quantitatively more Hg$^{2+}$ ions from aqueous solution compared to MCM-41. They also found that thiol functionalized mesoporous silica (HMS) prefer Hg$^{2+}$ ion over Cd$^{2+}$, Pb$^{2+}$, Zn$^{2+}$, Co$^{3+}$, Fe$^{3+}$, Cu$^{2+}$ and Ni$^{2+}$ ions.\textsuperscript{41} The modified HMS showed extreme lack of affinity toward d$^{10}$ ions (Cd$^{2+}$, Zn$^{2+}$, and Pb$^{2+}$). Approximately one half of the incorporated thiol groups in each adsorbent was accessible for Hg$^{2+}$ binding. The reasons of high S/Hg value were explained by (1) microporous channel prevent access of Hg$^{2+}$, (2) the existence of multibinding -SH site.
$H_2S + M^{2+} \rightarrow MS + 2H^+$

They postulate that the restricted volume of the mesopore channels reduce $S_0$ values of the above reactions, resulting in positive $\Delta G_0$ values for most of the intraframework metal-ligand reactions.\textsuperscript{41}

The high selectivity of thiol for Hg$^{2+}$ was also explained by the hard-soft acid base (HSAB) theory\textsuperscript{42}. Sulfur has been regarded as a softer donor atom, and therefore it is understandable that the thiolated adsorbent exhibited higher complexation affinity for Hg$^{2+}$ ion, the soft acceptor.

Liu et al. used SBA-15, which has thicker pore wall and higher stability than other mesoporous silica support. The effectiveness of removal for heavy metals was comparable to that of MCM-41. The stability of the functionalized SBA-15 was higher than that of MCM-41\textsuperscript{42}.

Antochshuk et al.\textsuperscript{43} measured Hg$^{2+}$ ion adsorption capacity on 1-allyl-3-propylthiourea modified MCM-41. The modified MCM-41 was synthesized via reaction of primary amine (mesoporous aminopropyl silica) with allyl isothiocyanate. Maximum Hg$^{2+}$ adsorption capacity of the material was 1.5 mmol g$^{-1}$, which was 40% higher than that of the first reported by Liu and co-worker. After washing of Hg$^{2+}$ with 10% thiourea solution, the sorption capacity of the second was about 50% of the initial capacity, which was also higher than the result of Liu and co-worker.
The high capacities of these materials are thought to be due to the large surface area of mesoporous materials and the ability of MPTS to form a “close-packed " monolayer on the surface at a high density. These particles are quite difficult to handle and must be bound to an inert material such as kaolin clay for use of in package bed. This lead to increased bed volumes and can cause a reduction in mass transfer rates.44

It is noteworthy that thiol-functionalized layered silicates such as magadite and kenyaite little adsorbed heavy metal ion binding, despite a similar thiol group content to that of functionalized montmorillonite.

Amino-functionalized mesoporous silica

The amino functionalized mesoporous silica was firstly reported at 1996.45 Cu\(^{2+}\) is often employed as adsorbates, probably because Cu\(^{2+}\) has highest affinity to amino ligands. The affinity sequence is as follows.

\[
\text{Ba}^{2+} < \text{Sr}^{2+} < \text{Ca}^{2+} < \text{Mg}^{2+} < \text{Mn}^{2+} < \text{Fe}^{2+} < \text{Co}^{2+} < \text{Ni}^{2+} < \text{Cu}^{2+} > \text{Zn}^{2+}
\]

this series (Irving-Williams series) is insensitive to the kind of ligands and mainly reflects electrostatic effects.46

Dai and coworker47 pointed out that the drawbacks of associated with direct coating method (involves hydrolysis of siloxane group) are; (1) the requirement of time consuming (>24 h) synthesis under reflux and (2) low loading of functional
silane ligands. They employed ion exchange method for introducing amino functional group. The concept of ion exchange was follows;

\[ m(S-O^-)M_1^+ + M_2^{m^+} \rightarrow (S-O^-)mM_2^{m^+} + M_1^+ \]

where \((S-O^-)\) is surface \(\text{SiO}^-\) groups, \(M_1^+\) is a cationic surfactant ion, \(M_2^{2+}\) is cationic complex.

Copper chelating complex loaded mesoporous silica have been prepared by two groups. Liu and co-worker\(^{48}\) modified mesoporous silica with \([1-(2-aminoethyl)-3-aminopropyl]\) trimethyilsilane, followed by loading \(\text{Cu}^{2+}\) as coordinative metal center. Dai’s group\(^{49}\) directly introduced copper-ethylenediamine complex onto mesoporous surface.

Liu et al.\(^{48}\) immobilized metal cheleted ligands on mesoporous silica, and investigated removal of arsenate and chromate ion. The mesoporous silica was functionalized with an ethylenediamine (EDA) terminated silane. The maximum sorption capacities of the mesoporous silica for chromate and arsenate are about 1.12 mmol-Cr g\(^{-1}\), and 1.00 mmol-As g\(^{-1}\), respectively. The binding mechanism was suggested by computer modeling. Three EDA molecules form the \(\text{Cu-(EDA)}_3\) complex on mesoporous silica. The complex has \(C_3\) symmetry and works as electrophilic basket for tetrahedral anion (\(\text{HAsO}_4^-\) or \(\text{HCrO}_4^-\)). The anion comes close to the complex and displace EDA ligand on Cu metal center. The EDA modified mesoporous silica could effectively remove radioactive Cesium from
aqueous solution.\textsuperscript{50}

Dai and coworker\textsuperscript{49} accomplished high selectivity for Cu\textsuperscript{2+} by using imprint coating method. Cu adsorption selectivity of imprint coated mesoporous silicas were much higher than that of blank-coated mesoporous silicas. Similar results were obtained by using hybrid mesoporous silicas\textsuperscript{51} and other mesoporous silicas, which was synthesized with anionic and neutral surfactants.\textsuperscript{52}

Yoshitake and co-workers prepared various amino-functionalized mesoporous silica and investigated removal of toxic anions; arsenate and chromate ion\textsuperscript{53,54}. Fe(III) loaded amino-functionalized mesoporous silica also showed high capacity for As(V), Se(VI), Cr(VI), Mo(VI) oxyanions.\textsuperscript{55} In the case of Se(VI) sorption, Se(VI) oxyanion would form outer sphere complex with Fe.\textsuperscript{56} The affinity was not depend on the number of nitrogen atoms in functional groups of aminosilane. Arsenate and chromate sorption capacity was increased with increasing the number of amino groups in the adsorbent.\textsuperscript{54} Similar results were observed for Co, Cd, and Cr sorption.\textsuperscript{57} The thorium ion uptake capacity and selectivity of amino-functionalized mesoporous silica was significantly larger than that of quaternary ammonium anion-exchange resin, BioRad MP1.\textsuperscript{58}

Descalzo et al.\textsuperscript{59} modified surface of MCM-41 with aminoanthracene and demonstrate the potential use of functionalized MCM-41 for anion sensing systems.

All of the reports in this area have focused on the grafting or incorporation of
ligand groups onto the surface of the pore channels, using ligand-functionalized organosilanes. Hossain synthesized new type of ligand functionalized mesoporous material in which the 1,4-bis-[3-trimethoxysilyl]propyl]ethylenediamine ligand groups are included inside the framework walls of the HMS (HMS-ENIS). The adsorption of Cu\(^{2+}\), Ni\(^{2+}\) and Zn\(^{2+}\) was investigated. Cu\(^{2+}\) adsorption followed Langmuir-type behavior and showed significantly higher adsorption capacity than the other ions. The Cu\(^{2+}\) adsorption characteristics, accessibility of binding sites and distribution coefficients, were compared to those of nanoporous adsorbents with ligands attached onto their pore walls. No structural change after treatment with 1M HNO\(_3\) could be confirmed by the identical XRD pattern to those before the treatment.\(^{60}\)

1.5.2. Mesoporous Anion Exchanger

Inagaki and co-worker\(^{24}\) reported ion exchange properties of mesoporous titanium phosphate molecular sieves (TCM-7, and -8). TCM-7 and -8 are amphoteric ion exchanger, which show cation and anion exchange properties. The capacity of the materials were 1.7-3.4 and 4.7-5.4 mmol g\(^{-1}\), respectively. The cation exchange site was P-O-H\(^+\)
Table 1.4. Amino- or thiol- modified mesoporous materials.

<table>
<thead>
<tr>
<th>parent</th>
<th>function</th>
<th>adsorbates</th>
<th>References</th>
</tr>
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<tbody>
<tr>
<td>MCM-41</td>
<td></td>
<td>Fe$^{3+}$, Co$^{2+}$, Ni$^{2+}$, Cu$^{2+}$ (removal of SeO$_4^{2-}$)</td>
<td>56,61</td>
</tr>
<tr>
<td>HMS</td>
<td></td>
<td>Cu$^{2+}$, Ni$^{2+}$, Co$^{2+}$, Cd$^{2+}$, Cr(VI)</td>
<td>57</td>
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<tr>
<td>SBA-15, HMS, PMO</td>
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<td>ReO$_4^-$</td>
<td>62</td>
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<tr>
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<td>ATP</td>
<td>59</td>
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<tr>
<td>MCM-41, SBA-1</td>
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<td>As(V), Cr(VI)</td>
<td>54</td>
</tr>
<tr>
<td>MCM-41</td>
<td></td>
<td>Hg$^{2+}$</td>
<td>43</td>
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### Chapter 1.

<table>
<thead>
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<th>Material</th>
<th>Structure</th>
<th>Metals Adsorbed</th>
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<tr>
<td>Hybrid mesoporous silica (BTSE)</td>
<td><img src="image1" alt="Structure" /></td>
<td>Cu$^{2+}$, Ni$^{2+}$, Zn$^{2+}$</td>
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</tr>
<tr>
<td>SBA-15</td>
<td><img src="image2" alt="Structure" /></td>
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<tr>
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<td>Hg$^{2+}$, Cd$^{2+}$, Pb$^{2+}$, Zn$^{2+}$, Co$^{3+}$, Fe$^{3+}$, Cu$^{2+}$, and Ni$^{2+}$</td>
<td>40,41</td>
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<td>Mesoporous silica</td>
<td><img src="image5" alt="Structure" /></td>
<td>As(V), Cr(VI)</td>
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</tr>
<tr>
<td>Mesoporous silica</td>
<td><img src="image6" alt="Structure" /></td>
<td>M(II)=Cu$^{2+}$, Zn$^{2+}$</td>
<td>63,65</td>
</tr>
</tbody>
</table>

34
Xu et al.\textsuperscript{68} reported that anion exchange reaction occurred on metal-cation-mediated bridged hybrid mesoscopic material (MBH) prepared from Zn(NO\textsubscript{3})\textsubscript{2}. The chemical formula was Zn\textsuperscript{2+} \((\text{H}_2\text{NCH}_2\text{CH}_2\text{CH}_2\text{SiO}_{1.5})_4\) (SDS\textsuperscript{−})\textsubscript{2}. Large anion surfactant (SDS\textsuperscript{−}) was ion exchanged with CH\textsubscript{3}COO\textsuperscript{−}, NO\textsubscript{3}\textsuperscript{−} in solution. The ion exchange capacity of the MBH was 3.9 mmol g\textsuperscript{−1}. The hexagonal structure of parent MBH changed to the lamellar phase during ion exchange;

<table>
<thead>
<tr>
<th>Mesoporous silica</th>
<th>HS\textsuperscript{−}H\textsubscript{2}N Si</th>
<th>Hg\textsuperscript{2+}</th>
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<tr>
<td>MCM-41</td>
<td>(\text{H}_2\text{N}\text{−}\text{HN}\text{−}\text{HN}\text{−}\text{HN}\text{−}\text{Si})</td>
<td>Co\textsuperscript{2+}</td>
<td>66</td>
</tr>
<tr>
<td></td>
<td>(\text{H}_2\text{N}\text{−}\text{HN}\text{−}\text{HN}\text{−}\text{HN}\text{−}\text{Si})</td>
<td>(-)</td>
<td>45</td>
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<tr>
<td></td>
<td>(\text{H}_2\text{N}\text{−}\text{HN}\text{−}\text{HN}\text{−}\text{HN}\text{−}\text{Si})</td>
<td>(-)</td>
<td>67</td>
</tr>
</tbody>
</table>

\[\text{H}_2\text{N}\text{−}\text{HN}\text{−}\text{HN}\text{−}\text{HN}\text{−}\text{Si}\]

\[\text{H}_2\text{N}\text{−}\text{HN}\text{−}\text{HN}\text{−}\text{HN}\text{−}\text{Si}\]

\[\text{H}_2\text{N}\text{−}\text{HN}\text{−}\text{HN}\text{−}\text{HN}\text{−}\text{Si}\]
however, the lamellar structure returned to the hexagonal phase by introducing SDS$^-$ anion.

Zhang et al.$^{17}$ synthesized similar MBH type material with Cd and Ni salt as inorganic precursor. Kron et al.$^{69}$ reported the removal of organic dye and chromate on mesoporous aluminophosphate. This material showed anion exchange property. Anion exchange site in aluminophosphate is sodium dodecyl sulfate (SDS), which is templating agent. The aluminophosphate was prepared by treating a salt composed of Al$_{13}$ polyoxometalate clusters (AlO$_4$Al$_{12}$(OH)$_{24}$(H$_2$O)$_7^+$) and an anionic surfactant (sodium dodecyl sulfate, SDS) with a phosphate buffer. Anion exchange capacity was approximately 1.3-1.6 meq g$^{-1}$. They concluded the removal process was occurred electrostatic interaction because sorption was not occurred MCM-41 (silica, neutral) as adsorbent and non-ionic dye.

Ordered nonlamellar alumino- and galloalumino-phosphate mesoporous anion exchangers were first synthesized with AlO$_4$Al$_{12}$(OH)$_{24}$(H$_2$O)$_7^+$ and GaO$_4$Al$_{12}$(OH)$_{24}$(H$_2$O)$_7^+$ Keggin-like polyoxocation by Holland.$^{30}$ Naphthol yellow (NYS$^2$) in aqueous solution could be successfully removed and the removal capacities of alumino- and galloalumino-phosphate were 1.6 meq g$^{-1}$ and 1.4 meq g$^{-1}$, respectively.
1.5.3. Removal of Organic Pollutant with Mesostructured Silica

Denoyel and coworker\textsuperscript{70} used non-calcined mesostructured silica for removal of organic pollutant from an aqueous solution. The removal process was often termed as “adsolubilization”. The sorption isotherms of 3-chlorophenol were obtained by using mesostructured silica, which were synthesized with different alkyl chain length of surfactant. As the alkyl chain length of surfactant increased, the affinity and sorption capacity were increased.

Nagy and coworker\textsuperscript{71} removed trichloroethylene and tetrachloroethylene with surfactant containing MCM-41. Sorption properties of as-synthesized MCM-41 are comparable to those of HDTMA-modified clays. There is disadvantage that Si and surfactants dissolved into the solution from the as-synthesized MCM-41 during sorption.

1.6. Removal of Selenium

Selenium is a byproduct metal usually associated copper. Primary selenium was produced domestically as byproducts of copper refining in 2001.\textsuperscript{72} Japan produced the largest amount of selenium in the world.

The end use of selenium demand in 1999 are as follows: glass, 25 %; metallurgical applications, 23 %; micscellaneous chemical uses, 14 %; electronics, 11 %; pigments, 8 %, and other uses, including agricultural feed additives, 19 %.