Summary and conclusions

The objective was to carry out synthesis of SBA-15 and transition metal incorporated SBA-15 materials and to characterize the ordered mesoporous SBA-15 materials and find the applicability of the transition metal modified SBA-15 in catalytic applications. One of the objectives was also to introduce organic functionality into the mesoporous silica materials and to study the applications of functionalized materials as catalysts in organic transformations or as adsorbents.

It is possible to investigate many of the physical properties of bulk materials, and to gain information about the order and morphology of a material with the help of techniques like XRD, \( \text{N}_2 \) adsorption desorption, Electron Microscopy, FT-IR, TGA, UV-Visible spectroscopy, etc. These methods collectively give much information about the surface properties like pore size, pore structure, and pore size distribution. They also provide information about changes occurred to the SBA-15 materials due to surface modification and metal loading. A survey of literature provided information about the different synthetic strategies, applications, and structural aspects of ordered mesoporous materials.

Ordered mesoporous silica, SBA-15 were successfully synthesized by hydrothermal process under acidic conditions using triblock co-polymer P123 and Tetraethylorthosilicate as the silica source. The synthesized materials were characterized using low angle powder XRD which shows reflections indexed to (100), (110) and (200) planes which are in good agreement with literature reported mesoporous silica having hexagonal symmetry \( P6_{3}mm \). The wide angle powder XRD pattern shows peak at 2\( \theta \) value of around 23° which is due to amorphous silica. The morphological features were studied by Transmission Electron Microscopy (TEM) which shows ordered structures of mesoporous materials and electron diffraction typical of amorphous silica. The materials synthesized have high surface area of 615 m\(^2\)/g as estimated using BET method by \( \text{N}_2 \) adsorption desorption study at liquid nitrogen temperature. The micropore volume and the pore size were estimated by t-plot and BJH method. The SBA-15 materials were loaded with a model drug molecule aspirin to study the controlled drug delivery property of the mesoporous silica materials and found to be a good candidate for drug support and release under biological pH.

We have successfully incorporated Platinum, Nickel, TiO\(_2\) and Silver into SBA-15. The in-situ synthesized Pt-SBA-15 materials show characteristic XRD peaks indexed to (100), (110)
and (200) reflections in low angle powder XRD patterns similar to pure SBA-15 along with a peak at $2\theta = 23^\circ$ in the wide angle powder X-ray diffractogram implying the retention of ordered hexagonal pore symmetry $P6mm$. Ni, Ag and TiO$_2$ incorporation was achieved by post synthetic route. Although the surface area of the SBA-15 materials decreased somewhat all the synthesized materials have high surface area and thus have potential catalytic activity. All materials also have micropore surface area confirmed by the t-plot method. The catalytic activity of Pt-SBA-15 materials were tested for the model reactions of 4-nitrophenol reduction by NaBH$_4$ and electron transfer between hexacyanoferrate (III) and thiosulfate ions which were monitored using UV-Vis spectrophotometer and the kinetics study showed to follow the Langmuir-Hinshelwood model. Ni loading was also achieved via post synthetic method. Ni-SBA-15 and Ag-SBA-15 materials were also efficient in catalyzing the reduction of 4-NP to 4-AP. TiO$_2$ loaded SBA-15 materials showed good catalytic activities towards photochemical degradation of rhodamine B and methylene blue dyes under UV irradiation. Ag loading was achieved via a simple sonochemical method where a previously synthesized Ag nanoparticles (AgNPs) were loaded onto SBA-15. The AgNPs were synthesized using a new “green” route where the leaf extracts of Hibiscus Sabdariffa L. plant were used as the reducing and stabilizing agent for the synthesis of silver nanoparticles under room temperature. The AgNPs were characterized by UV-Visible using the surface plasmon band and transmission electron microscopy. The synthesized nanoparticles had various shapes and sizes but larger percentage of spherical shapes are generated. The average particle size was calculated to be 26.5 nm and the standard deviation 6.9 nm. Ag-SBA-15 materials show good catalytic activity towards the electron transfer. The high surface area, pore symmetry and benign nature of the SBA-15 materials make it a good candidate for metal nanoparticle loading makes it a good candidate for catalytic applications.

The amine functionalization of SBA-15 using 3-aminopropyltriethoxysilane (APTES) was achieved by post synthetic process. The functionalized materials show XRD patterns similar to amorphous SBA-15 implying the retention of ordered porous structure after functionalization with APTES. The hexagonal porous nature was also confirmed from the TEM images. The BET surface area is high but somewhat lower than the parent SBA-15 materials and the samples have micropore surface area which is lower than the pristine SBA-15. The
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surface characteristics are comparable to SBA-15 and those reported in literatures. The pore size distribution by BJH method shows that the pores get expanded after functionalization process. The amine functionalization was confirmed using FT-IR spectroscopy and referring to available literatures. Thermal properties were studied using thermogravimetry. The functionalized materials were successfully used as catalyst in Knoevenegal condensation between benzaldehyde and ethyl cyanoacetate under microwave and solvent free conditions. The materials also catalyzed transesterification of butyl cinnamate to methyl cinnamate with microwave irradiation. The materials were able to remove methylene blue from aqueous solutions partially.