

# Synthesis of meso-SAPO-37 materials using neutral surfactant

**Rekha Yadav** PhD

Research scholar, University of Delhi, Delhi, India

**Arvind Kumar Singh** PhD

Research scholar, University of Delhi, Delhi, India

**Ayyemperumal Sakthivel** PhD\*

Associate professor, Department of Chemistry, University of Delhi, Delhi, India; Associate professor, Department of Chemistry, Central University of Kerala, Nileshevar, India

The electrostatic interaction between the inorganic precursor and the organic surfactant plays a major role in the assembly of mesoporous materials; hence, the authors believe that the use of a neutral surfactant against a cationic surfactant might help to produce long-range-ordered mesoporous materials with better thermal stability. The current work focuses on the synthesis of mesoporous silicoaluminophosphate-37 materials using a neutral surfactant (hexadecyl amine) by a two-step procedure. The synthesised materials were characterised by various spectroscopic and analytical techniques, namely Fourier transform infrared spectroscopy (FT-IR) powder X-ray diffraction (XRD), scanning electron microscopy (SEM) and nitrogen (N<sub>2</sub>) sorption. The mesoporosity of the samples was evident from XRD and nitrogen sorption analysis. FT-IR spectra support the presence of microporous structural building units on the mesoporous surface. The SEM images showed the presence of a flower-like morphology with hexagonal petals. The resulting material showed promising activities for *trans* alkylation of naphthalene using 1,3-diisopropylbenzene.

## 1. Introduction

Microporous molecular sieves, including zeolite and zeolite-like molecular sieves,<sup>1</sup> are important class of materials for various applications, such as catalysis,<sup>2–6</sup> sorption<sup>7,8</sup> and ion exchange<sup>9,10</sup> in petrochemical and fine chemical industries. Aluminophosphate materials, analogous to zeolite, possesses a flexible framework where different metal ions can be incorporated.<sup>11–15</sup> However, the small pore opening of microporous materials restricts their applications involving bulkier molecules due to pore blocking and molecule diffusion limitations. The limitation of microporous molecular sieves has been overcome through discovery of silicate-based mesoporous materials, such as the M41s family,<sup>16</sup> SBA-15<sup>17,18</sup> and hexagonal mesoporous silica (HMS).<sup>19</sup> Mesoporous molecular sieve materials have been shown as promising alternative materials for various bulk molecular transformations.<sup>20</sup> However, these mesoporous molecular sieves possess lower thermal and hydrothermal stabilities at elevated temperatures. These drawbacks were addressed partially by the synthesis of meso-/microcomposites on silicate-based materials. The design, synthesis and applications of mesoporous aluminophosphate-based molecular sieves, derived from microporous precursors, is of current interests in framework materials research due to their flexible frameworks. Conversely, molecular sieve materials, derived from silicoaluminophosphate (SAPO), have great difficulty in synthesis due to the existence of aluminium (Al) in both tetrahedral and octahedral environments.<sup>18</sup>

In addition, synthesis of mesoporous materials by using a cationic surfactant (S<sup>+</sup>) and anionic inorganic precursors leads to strong electrostatic forces, which play a major role in self-assembly.<sup>21</sup> The presence of strong interaction between inorganic precursor and surfactant leads to difficulties in removing surfactant, and quite often, materials collapse during calcinations. On the other hand, the use of neutral surfactant does not exhibit electrostatic interaction; hence, the authors believe that it can help to produce long-range-ordered mesoporous materials with better structure stability.

Thus, the objective of the current work is to synthesise a series of mesoporous SAPO (meso-SAPO)<sup>22–27</sup> from the microporous precursors of SAPO-37 using a neutral surfactant. The developed material was studied for the *trans* alkylation of naphthalene (NP) by using 1,3-diisopropyl naphthalene as a model reaction.

## 2. Results and discussion

The Fourier transform infrared spectroscopy (FT-IR) spectra and the corresponding powder X-ray diffraction (XRD) patterns for microporous SAPO-37 precursors, obtained at different durations, are shown in Figure 1. The FT-IR spectra of the samples prepared for 8 h and longer showed a band around 560 cm<sup>-1</sup>, which is characteristic of the secondary building units (SBUs) of SAPO-37. A broad band in the region of 1087 cm<sup>-1</sup> corresponds to asymmetric stretching frequency of T–O–T (where T = silicon (Si), aluminium, phosphorus (P)).<sup>22,23</sup> The complete crystallisation

\*Corresponding author e-mail address: sakthiveldu@gmail.com; asakthivel@chemistry.du.ac.in