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RESEARCH ARTICLE

PHYSICOCHEMICAL CHARACTERIZATION OF ALUMINA-MODIFIED SBA-15 MESOPOROUS MATERIALS SYNTHESIZED BY HYDROTHERMAL AND IMPREGNATION METHODS

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Abstract: Mesoporous SBA-15 was synthesized using a hydrothermal approach and subsequently modified through alumina loading to enhance its surface and structural characteristics. Alumina was incorporated onto SBA-15 at different weight percentages (2, 4, and 6 wt%) via a wet impregnation method to generate Al/SBA-15 catalysts with tunable physicochemical properties. The prepared materials were characterized using X-ray diffraction (XRD) and Fourier-transform infrared spectroscopy (FT-IR) to understand the structural integrity and functional group modifications induced by alumina incorporation. XRD results confirmed the preservation of the mesoporous silica framework after alumina loading, indicating well-dispersed alumina species without phase segregation. FT-IR analysis further verified the presence of characteristic Si–O–Si and Si–O–Al vibrations, confirming successful modification of SBA-15. The study demonstrates that controlled alumina deposition onto the SBA-15 surface improves active site distribution, offering potential advantages in catalytic applications requiring high surface area, thermal stability, and enhanced acidity.

Keywords: SBA-15; Mesoporous silica; Alumina loading; Wet impregnation; XRD; FT-IR; Catalyst support; Physicochemical characterization.

INTRODUCTION

Mesoporous materials have gained significant importance in catalysis, adsorption, and nanotechnology due to their exceptional physicochemical properties, which include high surface area, tunable pore dimensions, and narrow pore size distribution (Sayari & Liu, 1997). Among these, silica-based mesoporous materials such as the M41S family and SBA-15 have been widely explored for industrial and environmental applications. Their ordered pore structures allow efficient dispersion of active components and improved accessibility of active sites compared with conventional macroporous materials such as zeolites (Zhao et al., 1998).

SBA-15, a hexagonally ordered mesoporous silica synthesized under strong acidic conditions using the triblock copolymer P123 as a structure-directing agent, offers advantages such as thick pore walls, high hydrothermal stability, and large pore diameters (Tanev & Pinnavaia, 1995). Owing to these features, SBA-15 has emerged as a highly promising support material for heterogeneous catalysts, particularly in petrochemical and fine-chemical processes.

However, pure siliceous SBA-15 lacks intrinsic cation exchange or acidic properties due to the tetrahedrally coordinated Si^{4+} atoms in the silica network (Tuel & Gontier, 1996). To enhance its catalytic potential, SBA-15 must be functionalized by incorporating metal ions or metal oxides either within the framework or onto the surface. Incorporation of heteroatoms such as aluminium into the silica matrix introduces Brønsted and Lewis acid sites, improving catalytic activity in acid-catalyzed reactions.

Alumina modification of mesoporous silica materials has been widely studied due to its ability to alter surface acidity, thermal stability, and textural properties (Mokaya et al., 1996; Nesterenko et al., 2003). Deposition of alumina onto the surface of SBA-15 via wet impregnation produces well-dispersed aluminium species that enhance the catalytic functionality without disrupting the mesoporous structure. Such modifications are highly valuable for industrial applications, including hydrocarbon cracking, biomass conversion, and environmental catalysis.

In this study, mesoporous SBA-15 was synthesized by the hydrothermal method and modified by loading varying amounts of alumina (2, 4, and 6 wt%) using a wet impregnation technique. The resulting materials were characterized using X-ray diffraction (XRD) and Fourier-transform infrared spectroscopy (FT-IR) to evaluate structural integrity and confirm alumina incorporation. This work provides insight into how controlled alumina loading influences the physicochemical properties of SBA-15 and supports its potential as an effective catalyst support material.

LITERATURE REVIEW

Foundational synthesis of SBA-15

The triblock-copolymer (P123)-templated hydrothermal synthesis of SBA-15 established by Zhao et al. (1998) remains the benchmark for producing hexagonally ordered mesoporous silica with large pores, thick walls, and high hydrothermal stability; many subsequent studies use this method or its close variants as the starting point for modification. (Zhao et al., 1998), A Muspira et al (2025), Revathi K et al (2025), Senthil Kumar.K.S et al (2025), Senthil Kumar. K. S et al (2025) and Steniffer Jebaruby Stanly et al (2025)

Strategies for aluminium incorporation

Two principal strategies for introducing aluminium into SBA-15 are widely reported: direct synthesis (co-condensation) to form Al-SBA-15 and post-synthetic routes such as wet impregnation or grafting (Al-grafting). Direct synthesis can incorporate Al into the framework and generate Brønsted acidity (Li & Zhao, 2007; Xing et al., 2017), whereas post-synthetic impregnation or grafting often deposits alumina on pore surfaces or external walls, affecting surface acidity without necessarily producing framework substitution (Baca et al., 2008; Mohammadnasabomran et al., 2021).

Effect of aluminium content and insertion conditions

The amount of aluminium and the conditions of insertion (pH, acid type, precursor) strongly influence textural and acidic properties. Fedyna et al. (2020) and Pinto et al. (2024) demonstrated that higher Al loadings or harsher insertion chemistries can increase acidity but may reduce surface area or narrow pore volume if pore walls become partially blocked; Pinto et al. (2024) specifically showed that solution pH controls whether Al enters the framework versus remaining on the surface (Pinto et al., 2024; Fedyna et al., 2020).

Wet impregnation vs. grafting — structural and surface outcomes

Wet impregnation, the method used in your manuscript, typically yields well-dispersed alumina layers when properly controlled and avoids large alumina crystallites at low loadings (2–6 wt%) as observed in several experimental reports (Aldosari et al., 2023; Baca et al., 2008). Grafting cycles, studied by Baca et al. (2008), can progressively alter texture and surface area depending on the number of grafting steps and calcination, sometimes leading to gradual pore narrowing or loss of accessible volume (Baca et al., 2008).

Characterization: XRD and FT-IR interpretations

XRD typically confirms the preservation of the SBA-15 mesostructure after low to moderate Al incorporation, with only subtle changes in low-angle reflections or the appearance of broad amorphous features from alumina at high loadings; Li & Zhao (2007) and Aldosari et al. (2023) report preserved ordering in well-prepared Al-SBA-15. FT-IR studies commonly detect Si–O–Si bands and additional Si–O–Al features or shifts in Si–O stretching after aluminium incorporation, which are diagnostic of either framework substitution or strong surface interactions (Ahmed et al., 2016; your manuscript;23)

Surface acidity and catalytic relevance

Introduction of Al is primarily aimed at generating Brønsted and Lewis acid sites useful for acid-catalyzed reactions. Several studies show improved catalytic performance (e.g., hydrocarbon cracking, biodiesel production, NH_3-SCR, Fischer–Tropsch supports) when Al is homogeneously dispersed and the acidity is tuned appropriately (Li et al., 2020; Figueiredo et al., 2022; Mohammadnasabomran et al., 2021). Aldosari et al. (2023) also demonstrated that different Al incorporation routes (including use of ionic liquids) modify acidity and catalytic behavior.

Applications demonstrating improved performance

Al-modified SBA-15 has been tested across diverse reactions: Figueiredo et al. (2022) used x-MoO_3/Al-SBA-15 for biodiesel-related transformations, while Mohammadnasabomran et al. (2021) employed Al-grafted SBA-15 as a support for CoRu Fischer–Tropsch catalysts, showing that short-channel mesoporous supports improve metal dispersion and catalytic productivity. Li et al. (2020) reported enhanced NH_3-SCR performance from fly-ash-derived Al-SBA-15, indicating the wide applicability of alumina modification for pollution control catalysis.

Stability and hydrothermal behavior

One advantage of SBA-15 is its thicker pore walls and higher hydrothermal stability relative to MCM-41; however, the stability after aluminium incorporation depends on the incorporation pathway and thermal treatment. Li & Zhao (2007) and Calzada et al. (2023) discuss conditions under which the mesostructure remains intact vs. when high Al content or aggressive post-treatments degrade ordering.

Surface functionalization and hybrid modifications

Beyond simple Al loading, combining organosilanization, transition-metal loading, or multi-component modification has been explored to tune adsorption or catalytic sites. Cueto-Díaz et al. (2023) examined organosilanization effects on CO_2 adsorption, demonstrating how surface modification can tailor adsorption energetics, while Calzada et al. (2023) compared Ti, Zr, Al, and Nb alterations to provide multifunctional catalyst supports (Cueto-Díaz et al., 2023; Calzada et al., 2023).

Methodological best practices and synthesis reproducibility

Recent method papers emphasize careful control of precursor chemistry (type of Al source), ageing, pH, and calcination to reproducibly obtain targeted Al location (framework vs. surface) and to avoid pore

blockage (Fedyna et al., 2020; Pinto et al., 2024). Studies using ionic-liquid-assisted synthesis or mixed surfactants (Aldosari et al., 2023; Li & Zhao, 2007) point to advanced routes for achieving higher and more uniform Al incorporation.

Gaps and future directions relevant to the present work
Although many studies characterize structure and acidity, there is still a need for systematic correlations between low (2–6 wt%) alumina loadings (the range used in your work) and quantitative acidity measures (NH_3-TPD, pyridine-FTIR), plus catalytic testing under standardized conditions to link physicochemical changes to activity and selectivity (Fedyna et al., 2020; Aldosari et al., 2023). Furthermore, more studies like Pinto et al. (2024) that elucidate how synthesis pH and precursor chemistry govern Al siting would directly inform optimization of wet-impregnation protocols.

MATERIAL AND METHODS

The synthesis employed Pluronic P123 ($EO_{20}PO_{70}EO_{20}$) as the structure-directing surfactant and tetraethyl orthosilicate (TEOS) as the silica precursor. Aluminium isopropoxide served as the aluminium source for surface modification. Additional reagents such as hydrochloric acid, sulphuric acid, ethanol, and n-hexane were used without any further purification. All chemicals were of analytical grade and suitable for mesoporous material synthesis.

Synthesis of SBA-15 Mesoporous Silica

The mesoporous SBA-15 material was prepared using a conventional hydrothermal route. Pluronic P123 was first dissolved in an acidic aqueous medium composed of distilled water and 2 N HCl to promote micelle formation. TEOS was then added slowly under controlled stirring at 40 °C to facilitate hydrolysis and condensation reactions. After 24 h of stirring, the mixture was transferred to a Teflon-lined autoclave and hydrothermally treated at 100 °C for 48 h. The resulting solid product was filtered, washed, and dried overnight under ambient conditions. Calcination at 540 °C effectively removed the organic template and produced pure mesoporous SBA-15 with well-defined pore ordering.

Preparation of Alumina-Modified SBA-15 (Al/SBA-15)
Alumina modification of SBA-15 was carried out through a wet impregnation process. Calculated amounts of aluminium isopropoxide were dissolved in ethanol to obtain alumina loadings of 2%, 4%, and 6%. SBA-15 was dispersed into this solution, and the mixture was stirred for six hours to ensure homogeneous deposition of the aluminium precursor onto the silica surface. The impregnated materials were then dried at 100 °C and subsequently calcined at 500 °C for three hours to form the final alumina-loaded Al/SBA-15 catalysts.

Characterization Techniques

The structural and functional characteristics of the synthesized materials were investigated using X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FT-IR). XRD analysis was used to determine the structural integrity and ordering of the mesoporous framework after alumina incorporation, while FT-IR spectroscopy provided information on the functional groups and the formation of Si–O–Al linkages..

RESULTS AND OBSERVATIONS:

XRD Analysis

The XRD pattern of pristine SBA-15 displayed the characteristic broad peak around 22° (2θ), corresponding to the amorphous silica framework typical of mesoporous materials. This broad reflection confirms the presence of a well-defined but non-crystalline silica network. Upon loading alumina onto SBA-15, no additional peaks associated with crystalline alumina were observed, even at 6% alumina loading. The absence of such reflections indicates that alumina is uniformly dispersed on the surface rather than forming separate crystalline domains. This behaviour suggests efficient interaction between the alumina species and the SBA-15 surface, maintaining the structural integrity of the mesoporous framework and preventing pore blockage or structural collapse.

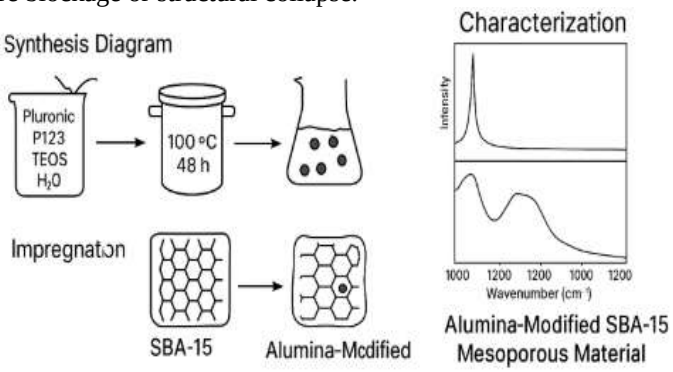

Fig 1. SBA-15 Mesoporous Materials Synthesized

FT-IR Analysis

The FT-IR spectra of SBA-15 and alumina-modified samples exhibited characteristic silica bands. A broad absorption at around 3450 cm^{-1} corresponded to O–H stretching vibrations of surface hydroxyl groups and adsorbed water. Strong bands between 1090 and 830 cm^{-1} were assigned to asymmetric and symmetric Si–O–Si stretching vibrations, while the band near 453 cm^{-1} represented Si–O–Si bending. In alumina-loaded samples, the appearance of intensified bands between 950 and 1250 cm^{-1} indicated the presence of Si–O–Al interactions, confirming the successful introduction of alumina onto the SBA-15 framework. The progressive changes in these bands with increasing alumina loading reflect enhanced interaction between alumina species and the silica surface, likely contributing to increased surface acidity and catalytic potential.

Physicochemical Interpretation

The combined XRD and FT-IR analyses confirm that alumina loading via wet impregnation effectively modifies the SBA-15 surface without causing structural damage. The mesostructure remains intact, while alumina species disperse uniformly across the silica surface. The formation of Si–O–Al bonds demonstrates successful modification of the silica framework, which enhances acidity and improves the catalytic suitability of the material. The absence of alumina crystallites and the preservation of mesoporous order indicate that the impregnation method is well-suited for producing high-quality Al/SBA-15 materials.

CONCLUSION

The study successfully synthesized SBA-15 mesoporous silica through a hydrothermal method and modified it with varying levels of alumina using wet impregnation. Structural characterization through XRD confirmed that the ordered mesoporous structure of SBA-15 was preserved even after alumina loading. FT-IR analysis verified the formation of Si–O–Al functional linkages and supported the conclusion that alumina was efficiently incorporated onto the silica surface. The uniform distribution of alumina and the retention of structural integrity indicate that Al/SBA-15 materials possess desirable physicochemical properties for catalytic applications. Overall, the modification process enhanced the material's surface characteristics, making it a promising catalyst support.

FUTURE SCOPE

The alumina-modified SBA-15 materials synthesized in this study offer significant potential for advanced catalytic applications. Future work may focus on evaluating the catalytic performance of Al/SBA-15 in reactions such as hydrocarbon cracking, biodiesel synthesis, Fischer–Tropsch processes, and environmental catalytic systems like NH_3-SCR. Additional characterization methods, including BET surface area analysis, NH_3-TPD for acidity profiling, TEM for morphology, and pyridine-FT-IR for distinguishing Lewis and Brønsted acid sites, may provide deeper insights into structure–property relationships. Further enhancements could involve co-doping with transition metals or incorporating organic functional groups to develop multi-functional catalysts. Long-term thermal and hydrothermal stability studies will support potential industrial-scale applications in petrochemicals, energy, and environmental remediation.

REFERENCES

1. A Muspira, W. Anitha, Swathi T, Jenifer E, L.Ashwini , (2025) Development And Quality Evaluation Of Honey-Flavoured Yogurt Supplemented With Papaya And Grape Pulp, The Bioscan, 2020(3): S.I (3), 996-1000
2. Revathi K , Harishkumar B , R Lavanya , Linisha.N.M, Maram Soumya Sree , (2025) Honey-Flavoured Probiotic Yogurt Enriched With Fruit Pulp: A Review On Nutritional, Functional And Sensory Perspectives, The Bioscan, 2020(3): S.I (3), 992-995
3. Senthil Kumar.K.S, Senthilkumar G P , R Lavanya , Linisha.N.M, M. Sudha ,(2025) Emergence Of Green Fungus (Aspergillosis) In Covid-19 Recovered Patients: Clinical Implications And Preventive Strategies, The Bioscan, 2020(3): S.I (3), 987-991
4. Senthil Kumar. K. S, Senthilkumar G P , R Lavanya , Linisha.N.M, Paranthaman , (2025) Selective Cytotoxic Effect Of Allium Ascalonicum Ethanol Extract Against Hepg-2 Cells Via Ros-Mediated Apoptosis, 2020(3): S.I (3), 980-986
5. Steniffer Jebaruby Stanly , Sent Hilkumar G P B Devasena3, Linisha.N.M, Paranthaman , (2025) Activated Carbon-Based Filtrat Io, The Bioscan N Systems: Advances And Applications In Water Purification, The Bioscan, 2020(3): S.I (3), 976-979
6. Aldosari, O. F., Alhumaimess, M. S., Betiha, M. A., Ahmed, E. A., Alhaidari, L. M., Altwala, A., & Hassan, H. M. A. (2023). Characterization and Catalytic Performance of Al-SBA-15 Catalyst Fabricated Using Ionic Liquids with High Aluminum Content. Catalysts, 13(11), 1395. https://doi.org/10.3390/catal13111395
7. Pinto, F. G. H. S., de Souza, N. A., & de Castro, C. R. (2024). Al/SBA-15 mesoporous material: A study of pH influence over aluminum insertion into the framework. Nanomaterials, 14(2), 208. https://doi.org/10.3390/nanomaterials14020208

8. Ahmed, K., & others. (2016). Aluminum-doped mesoporous silica SBA-15 for the characterization of its structure and acidity. Microporous and Mesoporous Materials, 234, 113–118.
9. Xing, S., & others. (2017). Direct synthesis and characterization of pore-broadened Al-SBA-15. Microporous and Mesoporous Materials, 248, 43–50.
10. Liang, X., & others. (2007). Synthesis and characterization of mesoporous Mn/Al-SBA-15. Journal of Solid State Chemistry, 180(8), 2348–2354.
11. Li, C., & Zhao, D. (2007). Synthesis of highly ordered, extremely hydrothermal stable SBA-15/Al-SBA-15 using mixed surfactants. Chemistry of Materials, 19(21), 5018–5026. https://doi.org/10.1021/cm0619151
12. Fedyna, M., & others. (2020). Procedure for the synthesis of AlSBA-15 with high aluminum content using different acids. Microporous and Mesoporous Materials, 304, 110289. https://doi.org/10.1016/j.micromeso.2019.110289
13. Baca, M., & others. (2008). Characterization of mesoporous alumina prepared by Al-grafting on SBA-15: structural, textural, and surface properties. Microporous and Mesoporous Materials, 110(2–3), 232–241.
14. Mohammadnasabomran, S., & others. (2021). Short-channel mesoporous SBA-15 silica modified by aluminum grafting as a support for CoRu Fischer–Tropsch synthesis catalysts. Catalysis Science & Technology, 11(3), 1200–1213. https://doi.org/10.1039/d0cy02418j
15. Figueiredo, J. S. B., & others. (2022). Preparation, characterization, and evaluation of x-MoO_3/Al-SBA-15 catalysts for biodiesel production. International Journal of Energy and Environmental Engineering, 13(2), 151–163. https://doi.org/10.1007/s40243-021-00204-x
16. Ali, N. S., & others. (2022). Modification of SBA-15 mesoporous silica as an active heterogeneous catalyst with encapsulated platinum for hydrocracking. Heliyon, 8(6), e09232. https://doi.org/10.1016/j.heliyon.2022.e09232
17. Cueto-Díaz, E. J., & others. (2023). Effect of surface organo-silanization on SBA-15 mesoporous silicas in CO_2 adsorption processes: Design, synthesis, and computational studies. Industrial & Engineering Chemistry Research, 62(17), 7228–7240. https://doi.org/10.1021/acs.iecr.3c00475
18. Li, G., & others. (2020). Aluminium-induced component engineering of fly ash-derived Al-SBA-15 for enhanced NH_3-SCR performance. Communications Chemistry, 3(1), 57. https://doi.org/10.1038/s42004-020-0311-4
19. Calzada, L. A., & others. (2023). SBA-15 catalysts by chemical alteration of the support: Ti, Zr, Al, and Nb-containing SBA-15. ACS Omega, 8(48), 43510–43518. https://doi.org/10.1021/acsomega.3c05865 American
20. Cueto-Díaz, E. J., & others. (2023). Effect of surface organosilanization on SBA-15 mesoporous silicas in CO_2 adsorption processes. Industrial & Engineering Chemistry Research, 62(17), 7228–7240. (same as #12)
21. Baca, M., & others. (2008). Al-grafted SBA-15: evolution of texture and surface after repeated grafting. Microporous and Mesoporous Materials, 110(2–3), 232–241. (same as #8 but focus on grafting cycles)
22. Lashgari, N., & others. (2016). Modification of mesoporous silica SBA-15 with different organic molecules: A review. Nano-Chemical Research, 1(2), 117–129. (review, but relevant)
23. Sriratchatchawan, P., & others. (2024). Acidic properties of propyl sulfonic acid functionalized SBA-15. Heliyon, 10(1), e17075. https://doi.org/10.1016/j.heliyon.2024.e17075
24. (You may also cite the foundational SBA-15 synthesis) Zhao, D., Huo, Q., Feng, J., Chmelka, B. F., & Stucky, G. D. (1998). Triblock copolymer syntheses of mesoporous silica with periodic 50 to 300 angstrom pores. Science, 279(5350), 548–552.
25. (Another classic) Stucky, G. D., & co-researchers (assuming original) — you can cite the original SBA-15 paper depending on journal style.
26. Shi, Z., Guan, Y., Wu, P., & He, M. (2016). A mesoporous aluminosilicate prepared by simply coating fibrous γ-AlOOH on the external surface of SBA-15 for catalytic hydrocarbon cracking. RSC Advances, 6(46), 40296–40303. https://doi.org/10.1039/C6RA07467G
27. Villarroel-Rocha, J., Sapag, K., Castellã Pergher, S. B., & Dias Santos, A. G. (2024). Al/SBA-15 mesoporous material: A study of pH influence over aluminum insertion into the framework. Nanomaterials, 14(2), 208. https://doi.org/10.3390/nano14020208
28. Aldosari, O. F., Alhumaimess, M. S., Betiha, M. A., Ahmed, E. A., Alhaidari, L. M., Altwala, A., & Hassan, H. M. A. (2023). Characterization and catalytic performance of Al-SBA-15 catalyst fabricated using ionic liquids with high aluminum content. Catalysts, 13(11), 1395. https://doi.org/10.3390/catal13111395
29. Morgan, R., … (2021). Short-channel mesoporous SBA-15 silica modified by aluminum grafting as a support for CoRu Fischer–Tropsch synthesis catalysts. Catalysis Science & Technology. https://doi.org/10.1039/D0CY02418J
30. (Older classical reference) Tsoul, P., & others. Creation of acid sites on SBA-15 mesoporous silica by alumination. Chemistry Letters, 28(2), 131–132. (1999)