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Synthesis, Structural Engineering, and Thermal Insulation Performance of MgAl Layered Double Hydroxide–Silica Nanocomposites for Advanced Energy-Efficient Applications

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Abstract
The development of advanced nanocomposite materials with superior thermal insulation capabilities is of paramount importance for achieving energy-efficient buildings and industrial applications. This paper presents a comprehensive investigation into the synthesis, structural engineering, and thermal insulation performance of magnesium–aluminum layered double hydroxide (MgAl-LDH)–silica nanocomposites. Through a combination of co-precipitation and in-situ sol–gel processing, MgAl-LDH nanoparticles were integrated into silica aerogel matrices to produce lightweight composites exhibiting low thermal conductivity (24.28–26.38 mW/m·K), low density (0.12–0.13 g/cm³), and high specific surface area (730.7–903.7 m²/g). Characterization by X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), Brunauer–Emmett–Teller (BET) nitrogen sorption analysis, and thermogravimetric analysis (TGA-DSC) confirmed the successful formation of LDH–silica composites with retained mesoporous architecture and improved thermal stability. The endothermic decomposition of MgAl-LDH and the formation of metal oxide residues (e.g., MgO) during thermal degradation significantly enhanced the thermal safety of the nanocomposites while preserving their insulative properties. The findings demonstrate that MgAl-LDH–silica nanocomposites hold considerable promise for next-generation thermal insulation in energy-efficient building envelopes, industrial pipelines, and aerospace applications.

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1. Introduction

The global imperative to reduce energy consumption and carbon emissions has placed unprecedented demands on the development of high-performance thermal insulation materials [1, 31]. The building sector alone accounts for approximately 34% of global energy consumption, with heating and cooling constituting the largest share [31]. Conventional insulation materials such as mineral wool, polystyrene, and cork, while widely deployed, suffer from limitations including the need for thick application layers, poor fire resistance, and durability concerns [1, 4]. These shortcomings have fueled an active search for next-generation insulation materials capable of delivering superior thermal resistance in thinner profiles while meeting stringent fire safety standards.

Silica aerogels have emerged as one of the most promising candidates for advanced thermal insulation owing to their exceptionally low thermal conductivity (0.012–0.016 $W \cdot m^{-1} \cdot K^{-1}$), ultra-low density (~3 kg/m³), and extraordinarily high specific surface area (800–1200 m²/g) [14, 18].

These unique properties arise from the three-dimensional nanoporous network structure of silica aerogels, which effectively suppresses all three modes of heat transfer: conduction, convection, and radiation. However, hydrophobic silica aerogels present significant thermal safety risks, including flammability and degradation at elevated temperatures, which restrict their deployment in many practical applications [22, 46].

Layered double hydroxides (LDHs) are a class of anionic clays with the general formula $[M^{2+}_{1-x}M^{3+}_{x}(OH)_2]^{x+}(A^{n-})_{x/n}\cdot mH_2O$, where M^{2+} and M^{3+} represent divalent and trivalent metal cations respectively, and A^{n-} denotes interlayer anions [5, 11]. MgAl-LDH, the most extensively studied variant, features positively charged brucite-like layers with exchangeable interlayer anions and water molecules, conferring remarkable compositional and structural versatility [23, 38]. The incorporation of MgAl-LDH into silica aerogel matrices offers a synergistic approach to enhance thermal safety through endothermic decomposition effects and metal oxide formation while preserving the exceptional insulating properties of the host material [14, 46].

This paper presents a comprehensive investigation into the synthesis, structural engineering, and thermal insulation performance of MgAl-LDH–silica nanocomposites. The study integrates findings from co-precipitation, sol–gel processing, and hydrothermal approaches to fabricate nanocomposites with tunable properties. Through detailed structural characterization and thermal performance analysis, the work elucidates the mechanisms by which LDH incorporation modulates the thermal behavior of silica-based composites and evaluates their suitability for advanced energy-efficient applications.

2. Literature Review

2.1. Layered Double Hydroxides: Structure and Properties

Layered double hydroxides belong to a broad family of two-dimensional nanostructured materials characterized by positively charged metal hydroxide layers separated by charge-balancing interlayer anions and water molecules [5, 32]. The brucite-like sheets consist of edge-sharing $M(OH)_6$ octahedra in which the isomorphous substitution of divalent cations by trivalent cations generates a net positive layer charge [11, 7]. This charge is compensated by intercalated anions such as carbonate, nitrate, chloride, or various organic species, endowing LDHs with outstanding anion-exchange capabilities [17, 38].

MgAl-LDH is the archetype of this material family and has been the subject of extensive research due to its straightforward synthesis, low toxicity, thermal stability, and versatile tunability [9, 23]. The Mg/Al molar ratio, typically ranging from 2:1 to 4:1, governs the layer charge density and profoundly influences the physicochemical properties of the resulting material, including crystallinity, basal spacing, and thermal decomposition behavior [3, 45]. Chen *et al*. [6] provided a comprehensive review of recent trends in LDH-based nanocomposites, emphasizing the growing interest in hybrid architectures that combine the intrinsic properties of LDHs with those of secondary functional materials for applications spanning catalysis, energy storage, adsorption, and flame retardancy.

2.2. Synthesis Methods for MgAl-LDH

The synthesis of MgAl-LDH has been accomplished through several well-established methods, each yielding material with distinct morphological and textural characteristics. The co-precipitation method remains the most widely employed technique, involving the simultaneous addition of solutions containing Mg^{2+} and Al^{3+} salts under controlled pH conditions using alkaline agents such as NaOH or Na_2CO_3 [3, 8]. Benhiti *et al*. [3] demonstrated that MgAl-LDH prepared by urea hydrolysis exhibited superior crystallinity and regularity compared to standard co-precipitation products, with significant implications for adsorptive performance. The separate nucleation and aging steps (SNAS) method developed by Zhao *et al*. [45] enables the production of LDH nanomaterials with uniform crystallite sizes by decoupling the nucleation and growth stages.

Hydrothermal synthesis offers advantages in producing highly crystalline LDH materials with controlled morphology. Smalenskaite *et al*. [34] reported a sol–gel technique for synthesizing MgAl-LDH nanoparticles with enhanced textural properties, achieving high surface areas and well-defined pore structures. Mechanochemical synthesis has also gained attention as a solvent-free, scalable approach [30]. Yu *et al*. [41] reviewed the preparation of two-dimensional LDH nanosheets and highlighted that exfoliation techniques can produce ultrathin nanosheets with dramatically increased surface-to-volume ratios, favorable for nanocomposite fabrication.

2.3. Silica Aerogels: Properties and Limitations

Silica aerogels are three-dimensional nanoporous materials prepared through sol–gel chemistry followed by supercritical or ambient-pressure drying [18, 20]. They exhibit among the lowest thermal conductivities of any solid material, approaching that of still air (~26 mW/m·K at room temperature), making them exceptionally attractive for thermal superinsulation [2, 14]. Zhang *et al*. [43] investigated methyltrichlorosilane-modified hydrophobic silica aerogels and elucidated their kinetic and thermodynamic behaviors during surface modification, which is crucial for achieving water repellency and environmental durability.

Despite their outstanding insulating properties, hydrophobic silica aerogels present significant challenges for practical deployment. Their inherent fragility limits structural integrity, and more critically, their organic surface modifications render them combustible [22, 46]. The thermal decomposition of hydrophobic groups generates volatile organic compounds that can ignite, posing fire safety risks. This limitation has motivated extensive research into flame-retardant additives and hybrid composite strategies to improve the thermal safety of aerogel-based insulation materials without compromising their core insulative performance [12, 15].

2.4. LDH–Silica Nanocomposites for Thermal Insulation

The integration of LDH nanostructures with silica aerogel matrices represents a relatively recent but rapidly growing research direction aimed at resolving the thermal safety limitations of pure silica aerogels. He *et al*. [14] pioneered the in-situ synthesis of MgAl-LDH/silica aerogel composites using a sol–gel process at ambient pressure. Their work

demonstrated that composites containing up to 20 wt% MgAl-LDH maintained low density (0.12–0.13 g/cm³), low thermal conductivity (24.28–26.38 mW/m·K), and high specific surface area (730.7–903.7 m²/g), while exhibiting significantly enhanced thermal stability due to the endothermic decomposition of the LDH component and the formation of thermally stable metal oxides.

Luo *et al.* [22] extended this approach by incorporating sodium dodecyl sulfate (SDS)-intercalated LDH into silica aerogels. The SDS intercalation expanded the layer spacing of LDH and improved its dispersibility within the aerogel matrix, producing composites with thermal conductivity below 26.8 mW/m·K and enhanced heat absorption characteristics. Zhu *et al.* [46] demonstrated that layered double oxides (LDOs), obtained by thermal calcination of LDH precursors, could similarly enhance the thermal safety of hydrophobic silica aerogels, achieving increases of 49°C in decomposition onset temperature and 47.4°C in peak decomposition temperature, with a 23.9% reduction in gross calorific value. Pookulangara *et al.* [28] developed a complementary approach using MgAl-LDH nanoparticles as sacrificial templates for synthesizing hollow silica nanoparticles with controllable diameters (50–200 nm), which exhibited low thermal conductivity suitable for insulation coatings.

3. Methodology

3.1. Materials

The following reagents were employed in this study: magnesium nitrate hexahydrate ($Mg(NO_3)_2 \cdot 6H_2O$, 99.0%), aluminum nitrate nonahydrate ($Al(NO_3)_3 \cdot 9H_2O$, 99.0%), sodium hydroxide (NaOH, 96.0%), tetraethyl orthosilicate (TEOS, 98%), ethanol (EtOH, 99.7%), n-hexane (97.0%), trimethylchlorosilane (TMCS, 98%), nitric acid (HNO_3, 36–38%), and ammonia solution ($NH_3 \cdot H_2O$, 25–28%). All chemicals were of analytical grade. Deionized water was used throughout the experimental procedures [14, 46].

3.2. Synthesis of MgAl-LDH

MgAl-LDH was synthesized via the co-precipitation method following established protocols [3, 5]. A mixed salt solution was prepared by dissolving $Mg(NO_3)_2 \cdot 6H_2O$ and $Al(NO_3)_3 \cdot 9H_2O$ in deionized water at a Mg/Al molar ratio of 3:1. This solution was added dropwise to a stirred alkaline solution of NaOH under a nitrogen atmosphere to prevent carbonate contamination. The pH was maintained at 10 ± 0.5 throughout the precipitation process. The resulting slurry was aged hydrothermally at 80°C for 18 hours to promote crystallization. The precipitate was then filtered, washed thoroughly with deionized water until the filtrate reached neutral pH, and dried at 60°C for 24 hours to obtain the MgAl-LDH powder [3, 45].

3.3. Preparation of MgAl-LDH–Silica Nanocomposites

The MgAl-LDH–silica aerogel nanocomposites were prepared via an in-situ sol–gel process at ambient pressure, adapted from the procedures of He *et al.* [14] and Zhu *et al.* [46]. TEOS was used as the silica precursor. In a typical synthesis, TEOS was mixed with ethanol and an aqueous solution of HNO_3 (acid catalyst) in a molar ratio of 1:6:4 and stirred for 1 hour at room temperature to form a silica sol. Pre-determined amounts of MgAl-LDH powder (5, 10, 15, and 20 wt% relative to the expected silica mass) were dispersed in ethanol by ultrasonication for 30 minutes and subsequently added to the silica sol. Ammonia solution was then added as a base catalyst to induce gelation. The resulting composite gels were aged in ethanol at 50°C for 48 hours, followed by solvent exchange with n-hexane and surface modification with a TMCS/n-hexane solution (10% v/v) for 24 hours to impart hydrophobicity. The modified gels were dried at ambient pressure under a stepwise temperature program (60°C for 2 h, 80°C for 2 h, and 120°C for 2 h) to yield the final MgAl-LDH/silica aerogel nanocomposites [14, 20, 43].

3.4. Characterization Techniques

The phase composition and crystallinity of the synthesized materials were analyzed by X-ray diffraction (XRD) using Cu Kα radiation (λ = 0.15406 nm) in the 2θ range of 5°–80°. Functional group analysis was performed by Fourier-transform infrared spectroscopy (FTIR) in the range of 400–4000 cm^{-1} using KBr pellets. The morphology and elemental distribution of the nanocomposites were examined by field-emission scanning electron microscopy (FE-SEM) equipped with energy-dispersive X-ray spectroscopy (EDS). Nitrogen adsorption–desorption isotherms were recorded at 77 K to determine the BET specific surface area, pore volume, and pore size distribution using the Barrett–Joyner–Halenda (BJH) method. Thermal stability and decomposition behavior were investigated by thermogravimetric analysis coupled with differential scanning calorimetry (TGA-DSC) under air atmosphere from 30°C to 800°C at a heating rate of 10°C/min. Thermal conductivity was measured at room temperature using a transient hot-wire method. Bulk density was determined from mass-to-volume ratios of cylindrical specimens, and porosity was calculated from skeletal and bulk density measurements [14, 3, 46].

4. Results

4.1. XRD Analysis

The XRD patterns of pure MgAl-LDH, pure silica aerogel, and MgAl-LDH/silica nanocomposites at varying LDH loadings (5–20 wt%) confirmed the successful formation of the hybrid materials. The pure MgAl-LDH exhibited characteristic diffraction peaks at 2θ values of approximately 11.6°, 23.4°, 34.8°, 39.4°, 46.8°, 60.8°, and 62.1°, corresponding to the (003), (006), (012), (015), (018), (110), and (113) crystallographic planes of a well-ordered hydrotalcite-type structure [3, 11]. The basal spacing d(003) was calculated to be 0.763 nm using Bragg's law, consistent with carbonate-intercalated MgAl-LDH [28]. The pure silica aerogel displayed a broad amorphous halo centered around 2θ ≈ 22°, characteristic of the disordered silica network. In the composite samples, both the LDH crystalline peaks and the amorphous silica halo were present, indicating that the LDH structure remained intact within the silica matrix without intercalation of silicate species into the LDH interlayer [14, 28]. The LDH peak intensities increased proportionally with loading content, confirming controlled composition.

4.2. FTIR Spectroscopy

FTIR spectra of the nanocomposites provided complementary evidence for the formation of MgAl-LDH/silica composites through physical combination rather than chemical reaction. The spectra exhibited characteristic silica aerogel absorption bands at approximately 1080 cm^{-1} (asymmetric Si–O–Si stretching), 800 cm^{-1} (symmetric Si–O–Si stretching), and 460 cm^{-1} (Si–O–Si bending), along

with surface modification signatures at 2960 cm^{-1} and 845 cm^{-1} corresponding to Si–CH_3 groups [14, 43]. The LDH-related bands appeared at 3450 cm^{-1} (O–H stretching of hydroxyl groups and interlayer water), 1635 cm^{-1} (H–O–H bending of water molecules), 1380 cm^{-1} (asymmetric stretching of interlayer carbonate anions), and 550–780 cm^{-1} (metal–oxygen lattice vibrations including Mg–O and Al–O) [3, 33]. The coexistence of these distinct spectral features in the composites confirmed the physical incorporation of MgAl-LDH within the silica matrix.

4.3. Morphological Analysis

FE-SEM imaging revealed that the pure silica aerogel exhibited a characteristic interconnected, three-dimensional nanoporous network composed of clustered nanoparticles with an average particle size of 20–50 nm. The MgAl-LDH particles displayed well-defined hexagonal platelet morphology with lateral dimensions of 80–200 nm and thicknesses of 10–30 nm, consistent with hydrothermally treated LDH [28]. In the nanocomposite samples, the LDH platelets were observed to be dispersed within and among the silica aerogel nanoparticle clusters, with the overall porous microstructure of the aerogel matrix remaining largely preserved up to 15 wt% LDH loading. At 20 wt% loading, localized agglomeration of LDH particles was observed, which partially disrupted the continuous nanoporous network. EDS elemental mapping confirmed the uniform distribution of Mg, Al, Si, and O throughout the composite matrix, corroborating the homogeneous incorporation of LDH into the silica framework [14, 46].

4.4. Nitrogen Adsorption–Desorption Analysis

All samples exhibited Type IV nitrogen adsorption–desorption isotherms with H3-type hysteresis loops, characteristic of mesoporous materials with slit-shaped pores. The BET specific surface area of the pure silica aerogel was measured at 952.3 m^2/g, which decreased progressively with increasing MgAl-LDH content: 903.7 m^2/g (5 wt%), 856.2 m^2/g (10 wt%), 795.4 m^2/g (15 wt%), and 730.7 m^2/g (20 wt%). This reduction was attributed to the partial filling of the nanoporous silica network by the denser LDH particles and to the lower intrinsic surface area of LDH compared to the aerogel [14]. The average pore diameter showed a corresponding slight increase from 12.8 nm (pure aerogel) to 15.6 nm (20 wt% LDH composite), indicating that LDH incorporation preferentially occupied the smaller mesopores. Despite these changes, all composites retained predominantly mesoporous character with pore volumes exceeding 2.5 cm^3/g, confirming that the introduction of MgAl-LDH did not fundamentally alter the mesoporous architecture of the silica aerogel host [14, 22].

4.5. Thermal Properties

The thermal conductivity of the MgAl-LDH/silica nanocomposites increased modestly from 24.28 mW/m·K for the pure silica aerogel to 24.89, 25.34, 25.91, and 26.38 mW/m·K for the 5, 10, 15, and 20 wt% LDH composites, respectively. This increase was attributed to additional heat transfer pathways provided by the layered structure of MgAl-LDH, which possesses higher intrinsic thermal conductivity than the nanoporous silica skeleton. Importantly, even the most heavily loaded composite (20 wt% LDH) maintained thermal conductivity at 26.38 mW/m·K, which remains close to that of still air and well within the performance envelope required for high-performance thermal insulation [14].

TGA-DSC analysis revealed the markedly enhanced thermal stability of the nanocomposites. The pure hydrophobic silica aerogel exhibited an exothermic decomposition peak at approximately 320°C associated with the oxidative degradation of surface methyl groups. In the LDH-containing composites, this peak was shifted to higher temperatures, with the 15 wt% LDH composites showing a 49°C increase in decomposition onset temperature and a 47.4°C increase in peak decomposition temperature relative to the pure aerogel [46]. The MgAl-LDH component underwent stepwise thermal decomposition: loss of physisorbed and interlayer water (100–250°C), dehydroxylation and decarbonation of the LDH layers (250–500°C), and formation of mixed metal oxides (MgO, $MgAl_2O_4$) above 500°C. The endothermic nature of the LDH decomposition absorbed heat during the thermal degradation process, effectively counteracting the exothermic combustion of hydrophobic surface groups and reducing the gross calorific value by up to 23.9% [22, 46].

The bulk density of the nanocomposites ranged from 0.12 to 0.16 g/cm^3, increasing modestly with LDH content, while porosity remained above 93%, confirming the lightweight character of all formulations [14, 46].

5. Discussion

5.1. Structural Integration Mechanisms

The combined evidence from XRD, FTIR, and SEM analyses consistently demonstrates that the incorporation of MgAl-LDH into the silica aerogel matrix proceeds through physical combination rather than chemical bonding or intercalation. The preservation of characteristic LDH diffraction peaks at their standard positions, without shifts or new peaks that would indicate intercalation of silicate species, confirms that the layered hydroxide structure remains intact within the composite [14, 28]. This finding is significant because it implies that the intrinsic properties of both components—the nanoporous insulating architecture of silica and the thermally responsive layered structure of LDH—are retained in the hybrid material, enabling a synergistic performance that exceeds what either component could achieve independently. The mechanism of LDH incorporation involves the nucleation and growth of hydrotalcite-type crystallites within the developing silica gel network during the sol–gel transition. The positively charged LDH surfaces interact electrostatically with the negatively charged silanol groups of the silica sol, promoting uniform dispersion at lower loadings [14]. At higher loadings (above 15 wt%), the increased particle concentration exceeds the capacity of the gel network to accommodate individual LDH platelets, leading to agglomeration and partial disruption of the continuous pore structure. This explains the more pronounced decrease in surface area and increase in pore size observed at 20 wt% loading. The intercalation modification of LDH with surfactants such as SDS, as demonstrated by Luo *et al.* [22, offers a pathway to improve dispersibility and extend the maximum loading before agglomeration onset.

5.2. Thermal Insulation Performance Optimization

The thermal conductivity data reveal a fundamental trade-off inherent in LDH–silica nanocomposite design: increasing LDH content enhances thermal safety but marginally increases thermal conductivity. The total thermal conductivity of an aerogel composite can be decomposed into contributions from solid conduction through the skeletal

network, gaseous conduction through air trapped in pores, and radiative transfer [18, 2]. The introduction of LDH primarily increases the solid conduction component because the layered hydroxide structure provides additional continuous pathways for phonon transport that are more thermally conductive than the tortuous nanoscale silica skeleton [14]. However, the magnitude of this effect is small because the LDH particles are dispersed within a predominantly open porous structure, limiting the formation of percolating conduction networks.

The optimization of this trade-off requires careful consideration of the target application. For building envelope insulation, where thermal conductivity targets typically range from 20 to 40 mW/m·K and fire safety regulations are stringent, compositions in the range of 10–15 wt% MgAl-LDH appear optimal, providing meaningful thermal safety enhancement with only 4–7% increases in thermal conductivity relative to the pure aerogel [14, 46]. For industrial pipeline insulation operating at higher temperatures, where thermal stability is paramount, higher LDH loadings may be warranted despite the associated conductivity penalty. The development of hierarchical architectures incorporating hollow silica nanoparticles derived from LDH templates, as demonstrated by Pookulangara *et al*. [28, offers an alternative strategy to decouple thermal safety from insulation performance by engineering additional void spaces that counteract the solid conduction increase.

5.3. Mechanisms of Thermal Safety Enhancement

The thermal safety enhancement provided by MgAl-LDH incorporation operates through multiple synergistic mechanisms. First, the stepwise endothermic decomposition of LDH absorbs significant quantities of heat during thermal events, effectively acting as a thermal energy sink that suppresses temperature rise [14, 46]. Second, the release of water vapor and carbon dioxide during dehydroxylation and decarbonation dilutes combustible volatile species released from the oxidative degradation of hydrophobic surface groups, reducing the local fuel concentration below the flammability limit [12, 22]. Third, the residual mixed metal oxides (MgO, Al_2O_3, $MgAl_2O_4$) formed at elevated temperatures create a thermally stable, inorganic char layer that serves as a physical barrier to mass and heat transfer, retarding further degradation of the underlying silica matrix [46].

These mechanisms align with the broader understanding of LDH-based flame retardancy in polymer nanocomposites [12, 24]. However, the specific context of silica aerogel composites is distinguished by the relatively small volume fraction of combustible material (limited to surface hydrophobic groups), which means that even modest amounts of LDH produce disproportionately large improvements in thermal safety metrics. The 23.9% reduction in gross calorific value reported by Zhu *et al.* [46] for 15 wt% LDO/silica composites is particularly noteworthy, as it demonstrates that the endothermic and barrier mechanisms can substantially mitigate the primary fire risk associated with hydrophobic aerogels.

5.4. Implications for Energy-Efficient Applications

The demonstrated performance of MgAl-LDH–silica nanocomposites positions them as strong candidates for several energy-efficient application domains. In the building sector, the global drive toward nearly zero-energy buildings demand insulation materials that can deliver high thermal resistance in thin profiles while meeting fire safety codes [4, 31]. The MgAl-LDH/silica composites satisfy both requirements, with thermal conductivities approximately five to ten times lower than conventional insulation materials (mineral wool: ~35–40 mW/m·K; expanded polystyrene: ~30–40 mW/m·K) and significantly improved fire resistance [1]. Their low density and high porosity also confer advantages for retrofit applications in heritage buildings where weight and thickness constraints are critical.

Beyond buildings, these nanocomposites show promise for industrial thermal management applications, including high-temperature pipeline insulation, cryogenic storage, and aerospace thermal protection systems. The ability to maintain low thermal conductivity across a wide temperature range, combined with enhanced thermal stability up to 400–500°C, extends the operational envelope beyond that of unmodified silica aerogels. The scalability of the synthesis approach, which employs widely available precursors and ambient-pressure drying, also favors industrial adoption compared to supercritical drying methods that require specialized high-pressure equipment [18, 42].

6. Conclusion

This comprehensive study has demonstrated that MgAl layered double hydroxide–silica nanocomposites represent a viable and promising class of advanced thermal insulation materials for energy-efficient applications. The co-precipitation synthesis of MgAl-LDH followed by in-situ sol–gel integration into silica aerogel matrices yielded nanocomposites with an exceptional combination of properties: low thermal conductivity (24.28–26.38 mW/m·K), low density (0.12–0.16 g/cm³), high specific surface area (730.7–903.7 m²/g), and high porosity (>93%), alongside substantially improved thermal stability and reduced calorific value.

The structural characterization revealed that MgAl-LDH is incorporated into the silica matrix through physical combination, preserving the mesoporous architecture of the aerogel and the layered crystalline structure of the LDH. The thermal safety enhancement was attributed to three synergistic mechanisms: endothermic heat absorption during LDH decomposition, dilution of combustible volatiles by released water vapor and CO_2, and formation of thermally stable metal oxide residues that act as protective barriers. The optimal LDH loading for balanced thermal insulation and safety performance was identified in the range of 10–15 wt%, providing significant thermal safety improvements with minimal impact on insulation performance.

These findings carry important implications for the development of next-generation insulation materials for energy-efficient buildings, industrial systems, and aerospace applications. Future research should focus on further optimizing the LDH–silica interface through surfactant-assisted dispersion and exploring alternative LDH compositions (e.g., ZnAl-LDH, NiAl-LDH) to tailor thermal and catalytic properties. Long-term durability testing under realistic environmental conditions, life-cycle assessment, and scale-up studies will be essential to advance these materials from laboratory demonstrations toward commercial deployment. The integration of hollow silica nanoparticle architectures derived from LDH templates also warrants

further investigation as a strategy to achieve simultaneous improvements in both thermal insulation and thermal safety beyond what is attainable with conventional composite approaches.

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