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Phosphor Synthesis Methods: A Comprehensive Review of Conventional, Wet-Chemical, Assisted, Mechanochemical and Emerging Routes

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Abstract

Phosphors are functional luminescent solids in which host lattices, activator ions, sensitizers and defects cooperate to convert absorbed energy into ultraviolet, visible or near-infrared emission. The synthesis route is therefore not merely preparative: it governs phase formation, crystallinity, particle size, morphology, dopant distribution, oxidation state, surface chemistry and defect populations, all of which can alter radiative and non-radiative relaxation. This review critically examines conventional solid-state and flux-assisted reactions, sol–gel and Pechini processing, co-precipitation, hydrothermal/solvothermal synthesis, solution combustion, microwave-assisted synthesis, mechanochemistry, spray pyrolysis, thermal decomposition, electrospinning and vapor-phase deposition. For each route, the governing principles, key process variables, advantages, limitations, scalability and influence on luminescence are discussed. Particular attention is given to activator concentration, defect engineering, reaction atmosphere, post-annealing and sustainability. Recent literature shows a movement toward lower-temperature, faster, solvent-minimized and morphology-controlled processing, while computational and data-driven approaches are beginning to accelerate host/activator discovery and process optimization. A comparative framework is proposed to help researchers select a synthesis route according to application requirements rather than treating any single method as universally superior.

Keywords: Phosphors, synthesis methods, luminescence, solid-state reaction, sol–gel, co-precipitation, hydrothermal synthesis, combustion synthesis, mechanochemistry, microwave synthesis, rare-earth activators, nanophosphors.

1. Introduction

Phosphors are inorganic luminescent materials that emit electromagnetic radiation after excitation by light, electrons, X-rays, γ-rays, electrical energy or other stimuli. Their technological importance spans solid-state lighting, displays, scintillation, radiation dosimetry, optical sensing, security marking,

bioimaging and energy conversion [1–4]. In many inorganic phosphors, a structurally robust host lattice accommodates a relatively small concentration of optically active ions such as Ce^{3+}, Eu^{2+}/Eu^{3+}, Tb^{3+}, Dy^{3+}, Sm^{3+}, Er^{3+} and Pr^{3+}. The host determines the local crystal-field and phonon environment, while the activator supplies accessible electronic states for absorption and emission [1, 5].

The development of phosphor-converted light-emitting diodes (pc-LEDs) has intensified the search for host lattices that combine strong excitation in the near-UV/blue region, high quantum efficiency, suitable emission colour, thermal stability and chemical durability [3–5]. Reviews of solid-state lighting show that composition alone does not determine performance; local structure, bonding, rigidity, defects and thermal behaviour are equally important [5, 6].

A central issue is therefore the synthesis–structure–property relationship. The same nominal composition prepared by different routes can display different crystallinity, crystallite size, morphology, dopant distribution, surface defect density and activator oxidation state. These differences can alter emission intensity, spectral bandwidth, decay lifetime and thermal quenching. Consequently, synthesis should be regarded as an integral part of phosphor design rather than a final processing step [7–9].

This review presents an application-oriented comparison of the principal synthesis strategies. It links processing variables to structure and luminescence, evaluates scale-up and sustainability, and identifies emerging opportunities in mechanochemistry, microwave processing, continuous aerosol routes and data-driven materials discovery.

2. Review Scope and Approach

This narrative review focuses on inorganic phosphors, with emphasis on rare-earth-activated oxides, phosphates, silicates, aluminates, vanadates, tungstates and related nanophosphors. Literature was screened using publisher and bibliographic records for foundational references, major reviews and representative synthesis studies. Priority was given to sources for which bibliographic metadata and DOI information could be independently checked. The review incorporates recent method-focused literature through 2025, including reviews of rare-earth nanophosphors, microwave processing and mechanochemical luminescent materials [7–12].

3. Fundamental Design of an Inorganic Phosphor

A generic phosphor composition can be written as **Host:** $_xA^{m+}$, $_yS^{n+}$

Where, A is the principal activator and S is an optional sensitizer. Energy may be absorbed directly by the activator or by the host followed by energy transfer to the activator. A sensitizer can strengthen absorption and transfer excitation to the emitting centre [1, 8].

Host selection depends on ionic-size compatibility, charge compensation, crystal symmetry, band structure, phonon energy and chemical/thermal stability. Low phonon energy is generally desirable where multiphonon relaxation must be suppressed, while suitable band alignment and local coordination are required to position activator levels favourably [5, 7].

4. Major Phosphor Synthesis Methods

Figure 2 summarizes a generalized sequence shared, in different forms, by most synthesis routes.

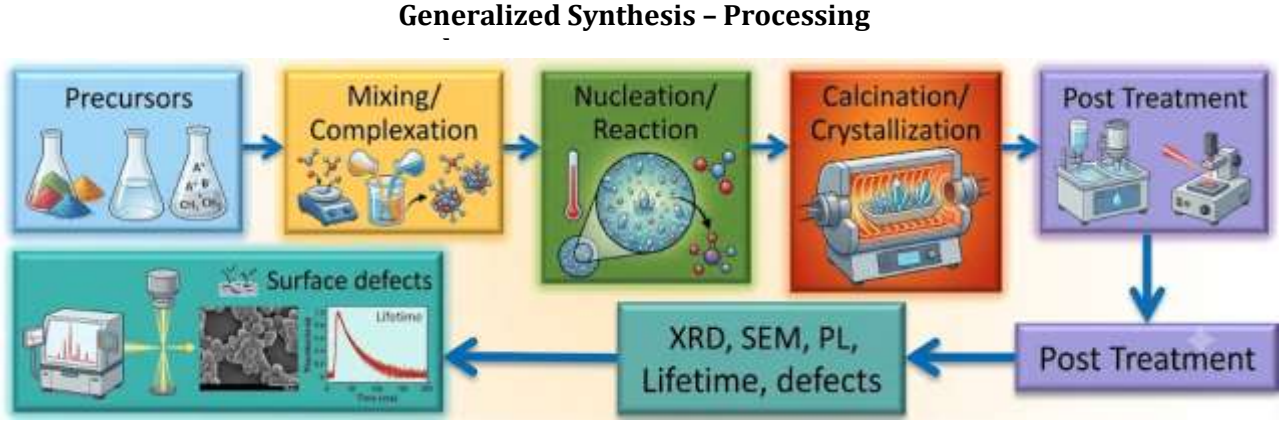

Figure 1. Generalized synthesis–processing pathway for inorganic phosphors.

4.1 Conventional Solid-State Reaction

Conventional solid-state processing is the reference route for bulk phosphors because it is simple, robust and readily scalable. Stoichiometric oxides, carbonates, nitrates or other thermally decomposable precursors are mixed, ground and heated until the target phase forms. A simplified representation of the reaction is

$$A_xO_y + B_xO_y \rightarrow A_xB_yO_z + \text{gaseous products}$$

The principal driving force is solid-state diffusion across reacting interfaces. Repeated grinding and calcination are often used to shorten diffusion lengths and eliminate residual precursor phases [2, 7, 14].

High phase purity, high crystallinity, high yield and industrial scalability are major strengths of this synthesis route. The disadvantages are high temperature, long reaction time, particle coarsening and limited morphology control. The route is therefore particularly suitable where thermal robustness and crystallinity dominate over nanoscale dimensions [3, 4].

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4.2 Flux-Assisted Solid-State Synthesis

Flux-assisted processing modifies the solid-state route by introducing a low-melting component that enhances ion mobility and mass transport. Borates, fluorides, chlorides and related salts can serve as fluxes, depending on the composition of the host. The general reaction scheme can be represented as:

$$\text{Reactant}_1(s) + \text{Reactant}_2(s) + \text{flux (l)} \rightarrow \text{Product (s)} + \text{Flux (l/s)}$$

This method can yield better crystallization and reduce repeated high-temperature treatments, but residual flux, volatilization and impurity incorporation must be controlled. It is especially attractive when large and well-crystallized particles are desired.

4.3 Sol–Gel Synthesis

Sol–gel processing converts molecular or ionic precursors into a homogeneous gel through hydrolysis and condensation, followed by drying and calcination. Representative reactions are

$$M(OR)_n + xH^2O \rightarrow M(OH)_x(OH)_{n-x} + xR - OH$$

And $$M-OH + HO-M \rightarrow M-O-M + H_2O$$

The main advantage of the sol-gel synthesis method is intimate mixing of constituent cations before crystallization. pH, solvent, precursor concentration, water/alkoxide ratio, complexing agents and aging strongly influence the gel network and final particle characteristics [9, 15]. Sol–gel methods can yield homogeneous nanophosphors, although drying shrinkage, agglomeration and solvent use remain limitations [9, 16].

4.4 Pechini / Polymer-Complex Method

The Pechini method is a polymeric-precursor route in which metal ions are chelated, commonly by citric acid, and immobilized in a polymeric network using a polyol. The resin maintains cation-scale homogeneity during decomposition and calcination. This is valuable for multication phosphors. Experimental studies have demonstrated well-dispersed rare-earth phosphors and tunable emission, although organic residues must be removed carefully during thermal treatment [17, 18].

4.5 Co-Precipitation

Co-precipitation forms host and activator-containing precursors simultaneously from solution. For a hydroxide route, the reaction is represented as

$$xM^{n+} + yN^{m+} + (n+m)\,OH^- \rightarrow M_xN_y(OH)_{m+n}\downarrow$$

The precipitate is filtered, washed, dried and calcined. Low-temperature precursor formation and good dopant distribution are the major advantages of this method [7, 19]. Critical variables include pH, addition rate, complexation, solubility of products, stirring, aging and washing. Different cations can precipitate at different rates, causing compositional segregation if the chemistry is not controlled.

4.6 Hydrothermal and Solvothermal Synthesis

Hydrothermal synthesis uses water at elevated temperature and autogenous pressure, while solvothermal synthesis uses non-aqueous solvents. These methods enable nucleation and crystal growth under conditions that can produce well-defined morphologies at lower temperatures than many conventional ceramic reactions. Surfactant-controlled hydrothermal processing has produced nanosized rare-earth-activated lanthanum phosphate phosphors [20]. Comparative studies also show that sol–gel and hydrothermal routes can yield single-phase nanophosphors with different structural and optical characteristics [21]. Pressure-rated equipment, batch size and scale-up remain important limitations.

4.7 Solution Combustion Synthesis

Solution combustion synthesis (SCS) uses an oxidizing precursor mixture and a fuel such as urea, glycine or citric acid. After ignition, a self-propagating exothermic reaction generates the product rapidly. The process can be represented as

$$\text{Fuel} + \text{Oxidizer} \rightarrow \text{Oxide phosphor} + \text{gaseous products} + \text{heat}$$

SCS provides molecular-level mixing and rapid formation of fine powders. The final morphology and crystallinity depend strongly on fuel type, fuel-to-oxidizer ratio, pH, precursor chemistry, atmosphere and ignition conditions [10, 22]. Combustion-derived phosphors may require post-annealing to improve crystallinity and optical output. Agglomeration, residual carbon and uncontrolled local temperatures remain limitations.

4.8 Microwave-Assisted Synthesis

Microwave heating couples electromagnetic energy to the reaction medium and can provide rapid volumetric heating with reduced thermal gradients. Reviews of luminescent materials report benefits including rapid synthesis, high compositional homogeneity, diverse morphology and accelerated screening of phosphor compositions [22]. Microwave processing can be combined with hydrothermal, sol–gel or combustion chemistry. Scale-up, field uniformity and the microwave absorption characteristics of the reaction mixture remain challenges.

4.9 Mechanochemical Synthesis

Mechanochemistry employs mechanical energy, typically through high-energy ball milling, to induce bond breaking, defect generation, intimate mixing and solid-state reaction. The process can be solvent-free or solvent-minimized and can activate reactions at lower external temperatures. A 2024 review identifies mechanochemistry as an emerging sustainable route for luminescent materials used in optoelectronics, sensing, biomedicine and photocatalysis [11]. Milling-media contamination, mechanical strain, agglomeration and independent control of crystallinity and defects remain challenges.

4.10 Spray Pyrolysis

Spray pyrolysis converts a precursor solution into aerosol droplets that undergo solvent evaporation and thermal decomposition in a heated zone. Pneumatic and ultrasonic atomizers are common. Final particle morphology depends on droplet size, precursor concentration, solvent properties and furnace temperature [23, 24]. The method is attractive for continuous production, spherical particles and coatings without vacuum and has been applied to numerous rare-earth- and transition-metal-doped luminescent oxides and sulfides [23].

4.11 Thermal Decomposition and Colloidal Routes

Thermal decomposition of molecular precursors in coordinating solvents can yield crystalline nanoparticles with controlled size and morphology. Ligands regulate nucleation and growth, making the approach useful for colloidal nanophosphors and upconversion particles [7, 25]. Precursor cost, solvent use, ligand removal and scale-up are significant considerations.

4.12 Electrospinning and Template-Assisted Synthesis

Electrospinning is primarily a morphology-control technique in which an electric field draws a precursor/polymer jet into ultrafine fibres. Subsequent calcination removes the polymer and crystallizes the inorganic phase. It is useful for one-dimensional phosphor fibres and flexible luminescent mats, but additional processing and relatively low throughput limit large-scale production.

4.13 Vapor-Phase and Thin-Film Deposition

Chemical vapor deposition, physical vapor deposition, sputtering and pulsed-laser deposition are important when phosphors are required as thin films. They provide thickness and substrate control and are relevant to electroluminescent devices and integrated photonics. Their disadvantages include capital cost, vacuum requirements for many configurations and process complexity [23].

5. Synthesis Conditions Governing Luminescence

5.1 Reaction Temperature

Temperature controls phase formation, crystallinity, defect populations, dopant diffusion and grain growth. Insufficient thermal energy can leave amorphous or metastable material; excessive heating can cause coarsening, secondary phases or oxidation-state changes. Thus, the optimum temperature is host-specific [2, 7].

5.2 Activator Concentration and Concentration Quenching

Increasing activator concentration initially increases emitting centres, but beyond an optimum level energy migration between neighbouring activators can increase non-radiative loss. The intensity trend is therefore commonly non-monotonic. Concentration quenching should be analysed together with site occupancy and energy-transfer distance [3, 5].

5.3 Reaction Atmosphere and Oxidation State

Atmosphere can determine the valence state of activators such as Eu and Ce. For Eu^{2+}/Eu^{3+} and Ce^{3+}/Ce^{4+} systems, reducing, inert and oxidizing conditions can produce substantially different absorption and emission. Atmosphere must therefore be reported explicitly.

5.4 Defect Engineering

Vacancies, antisites, interstitials and substitutional defects can serve as luminescence centres, charge traps or non-radiative centres. In persistent-luminescence and dosimetric phosphors, the defect/trap distribution may be as important as activator concentration [12, 25].

5.5 Post-Annealing

Post-annealing can remove residual organics and hydroxyl groups, improve crystallinity, redistribute dopants and modify oxidation states. It is frequently essential for wet-chemical and combustion-derived phosphors [10, 22].

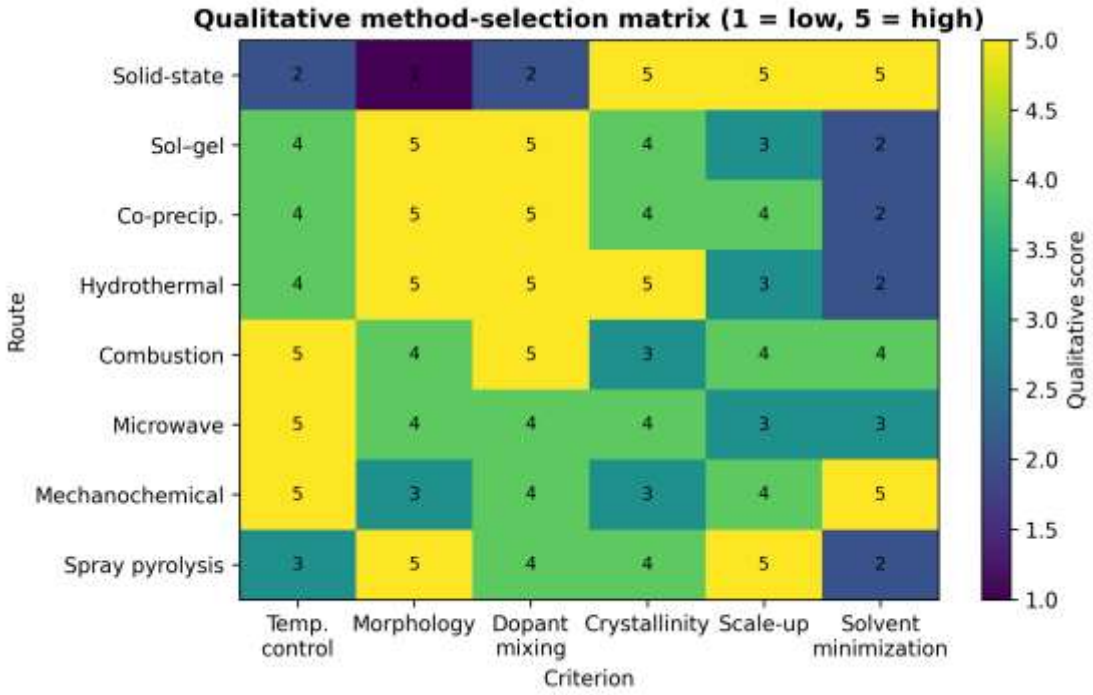

Figure 2. Qualitative comparison matrix for synthesis-method selection. Scores are conceptual indicators, not experimental measurements.

6. Comparative Assessment of Synthesis Routes

Method	Thermal demand	Morphology	Dopant mixing	Crystallinity	Scale-up	Key limitation
Solid-state	High	Low–moderate	Moderate	High	High	Large particles; high T

Flux-assisted	Moderate –high	Moderate	Moderate	High	High	Residual flux
Sol–gel	Low–moderate + anneal	High	High	High after anneal	Moderate	Drying/shrinkage
Co-precipitation	Low–moderate + anneal	High	High if controlled	High after anneal	High	Precipitation chemistry
Hydro/solvothermal	Moderate	High	High	High	Moderate	Pressure equipment
Combustion	Low external T; exothermic	Moderate	High	Moderate–high	Moderate –high	Agglomeration
Microwave	Low–moderate	High	High	High	Moderate	Scale-up/field uniformity
Mechanochemical	Low–moderate	Moderate	High mixing	Moderate–high	Moderate –high	Milling contamination
Spray pyrolysis	Moderate –high	High	High	High	High	Atomization control
Vapor deposition	Moderate –high	High	High	High	Moderate	Capital/vacuum cost

Table 1. Comparative assessment of major phosphor synthesis methods; categories are qualitative and intended for process selection.

7. Characterization of Synthesized Phosphors

7.1 X-ray Diffraction

XRD establishes phase identity, crystallinity and lattice parameters. Crystallite size is often estimated from peak broadening using $D = \frac{k\lambda}{\beta \cos \theta}$, where D is coherent crystallite dimension, k is a shape factor, λ is X-ray wavelength, β is corrected peak breadth(FWHM) in radians and θ is the Bragg angle. Scherrer size should not automatically be interpreted as particle size.

7.2 Electron Microscopy and Composition

SEM reveals morphology and agglomeration, while TEM can resolve particle size and lattice fringes. EDS provides elemental screening but may be insufficient for accurate quantification of low-level activators.

7.3 Photoluminescence

PL excitation and emission spectra identify absorption bands, activator transitions and emission maxima. FWHM, chromaticity, intensity and spectral overlap are useful comparative descriptors. Quantum yield is preferable to raw intensity when absolute efficiency is being compared.

7.4 Lifetime and Thermal Stability

For a single exponential decay, $I(t) = I_0 \exp(-t/\tau)$, where τ is the emission lifetime. Multi-exponenti

al decay can indicate multiple sites or coupled radiative/non-radiative channels. Temperature-dependent PL is required to evaluate thermal quenching.

7.5 Defect and Surface Analysis

XPS, EPR, thermoluminescence, Raman spectroscopy and diffuse-reflectance measurements can provide evidence for oxidation states, vacancies, traps, lattice vibrations and optical transitions. A strong synthesis study should connect these measurements mechanistically.

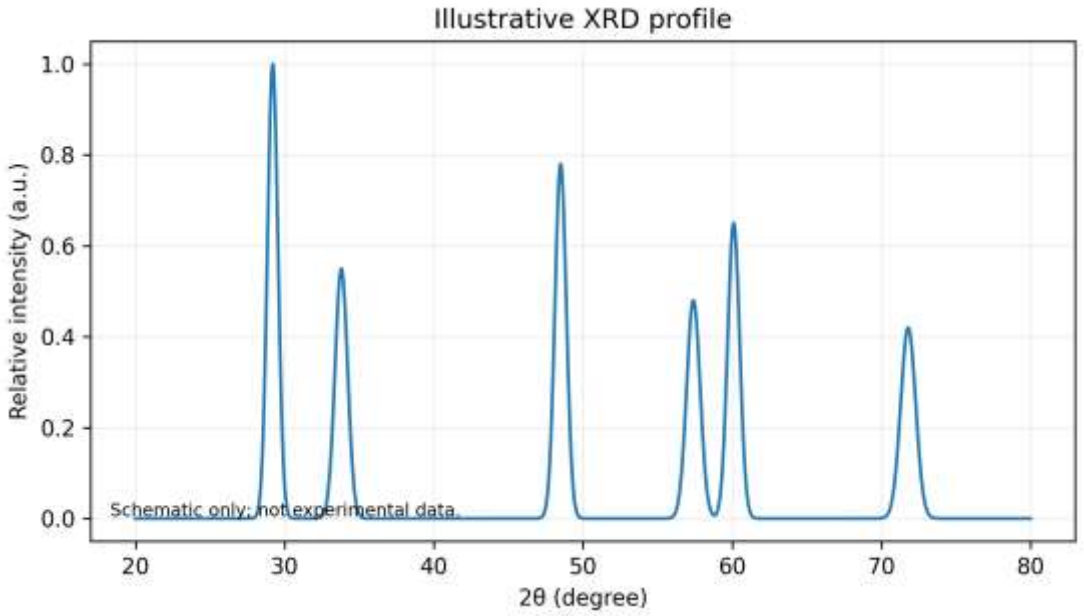

Figure 3. Illustrative XRD profile for explaining phase identification and crystallinity; peak positions are schematic.

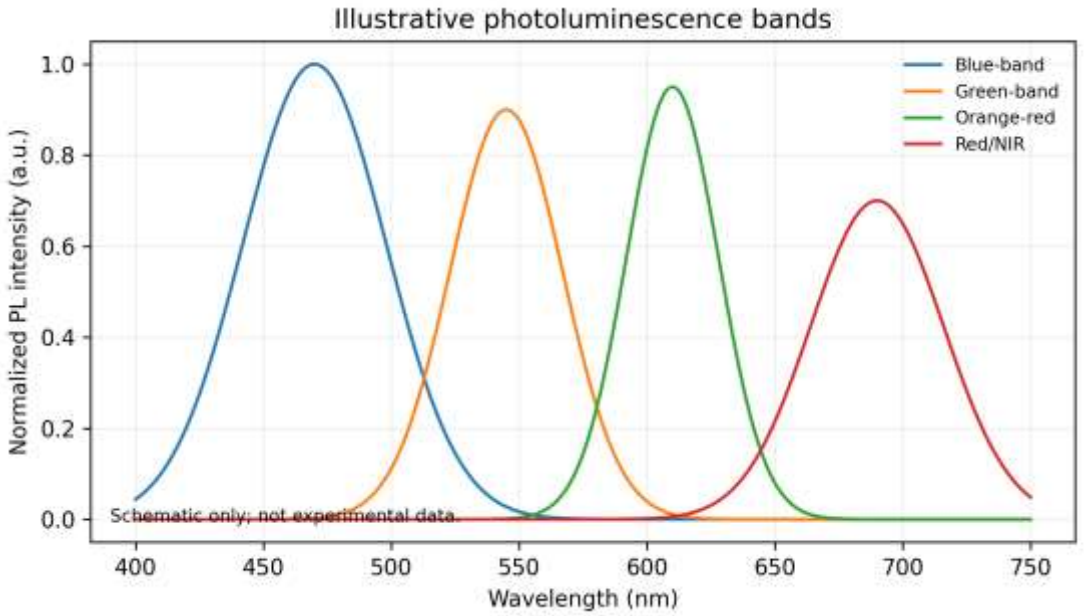

Figure 4. Illustrative photoluminescence bands for discussing spectral analysis; curves are schematic and not experimental data.

8. Application-Oriented Selection

For bulk solid-state lighting, conventional or flux-assisted synthesis remains attractive because high crystallinity, thermal robustness and production yield can dominate. For nanophosphors, sol–gel, co-precipitation, hydrothermal and thermal-decomposition routes provide stronger size and morphology control [7, 15]. For persistent luminescence and dosimetry, synthesis must be optimized for trap populations and defect chemistry rather than PL intensity alone [12]. For thin films, spray pyrolysis, sputtering and vapor deposition enable substrate-integrated materials [23, 24]. Mechanochemistry is especially promising where solvent minimization and fewer processing steps are priorities [11].

A useful decision rule is therefore: choose the method that best controls the property limiting the intended application. High crystallinity and scale-up favour solid-state routes; nanoscale morphology favours wet-chemical routes; rapid screening favours combustion or microwave routes; solvent minimization favours mechanochemistry; and continuous particle/film production favours aerosol or deposition methods.

9. Sustainability, Scale-Up and Reproducibility

Environmental assessment should include precursor toxicity, solvent use, energy consumption, furnace residence time, washing requirements, gaseous emissions, waste treatment and product yield. A low reaction temperature does not automatically imply a green route if large volumes of organic solvent or repeated purification are required. Mechanochemistry offers a solvent-minimized pathway, while spray pyrolysis offers a continuous-processing opportunity [11, 23].

Scale-up changes heat and mass transfer. Combustion reactions can develop gradients in larger reactors; microwave fields can become non-uniform; and precipitation can become mixing-limited. Scale-up should therefore preserve relevant reaction and transport conditions rather than simply increasing batch mass.

Reproducibility requires reporting precursor purity, stoichiometry, pH, solvent composition, atmosphere, heating rate, dwell time, cooling rate, washing and post-treatment. For activator-containing phosphors, the oxidation state and actual dopant concentration should be verified whenever they are central to the proposed luminescence mechanism.

10. Emerging Directions

10.1 Hybrid Processing

Combining wet chemistry with microwave heating, combustion with post-annealing, or co-precipitation with hydrothermal crystallization can exploit complementary advantages.

10.2 Defect- and Site-Selective Synthesis

Future routes will increasingly target specific activator sites and controlled vacancy populations rather than phase purity alone, especially for persistent luminescence, upconversion and dosimetry.

10.3 Continuous Manufacturing

Spray pyrolysis and other aerosol approaches can move phosphor synthesis toward continuous manufacturing and potentially improve batch-to-batch uniformity [23, 24].

10.4 Mechanochemistry and Green Processing

Mechanochemical synthesis can eliminate or reduce solvents and activate reactions at lower external temperatures. Its development depends on better control of contamination, milling energy, crystallinity and scale-up [11].

10.5 Data-Driven Discovery

The number of possible host/activator combinations makes empirical trial-and-error inefficient. Modern discovery frameworks combine crystal-structure databases, computed descriptors, high-throughput synthesis and machine learning. Recent work emphasizes comprehensive host/activator screening and structure–property reasoning [6].

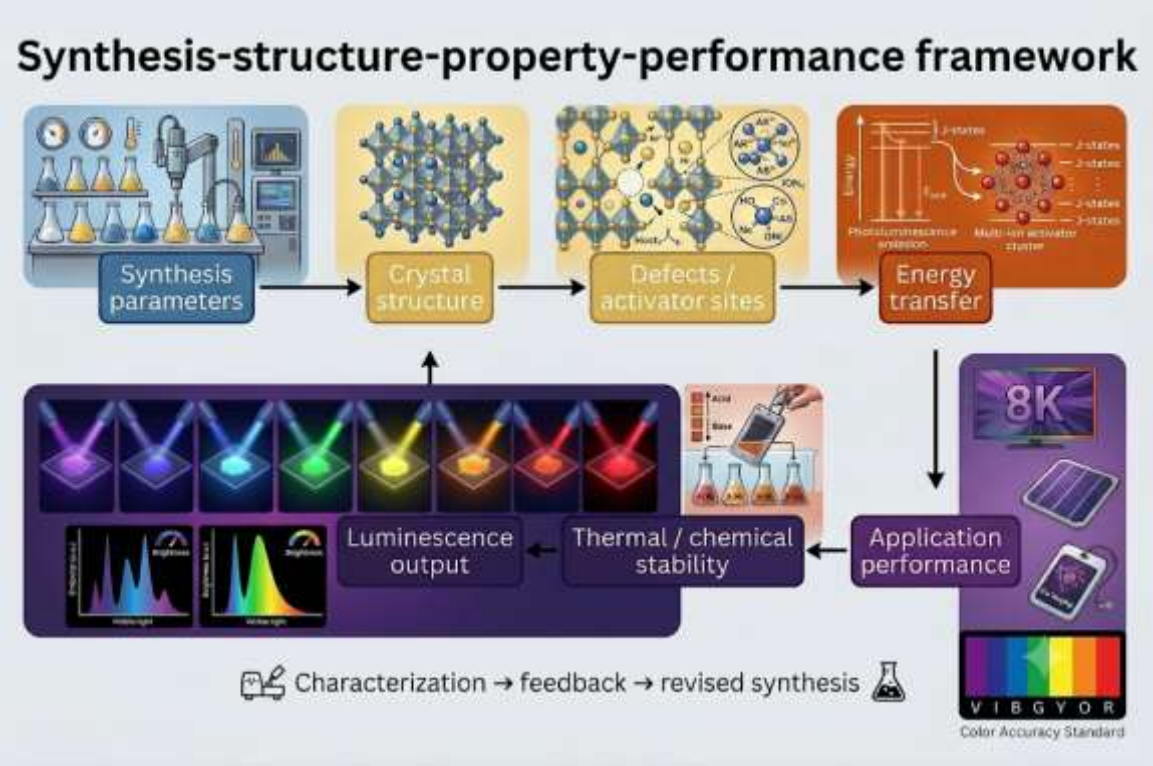

Figure 5. Proposed synthesis–structure–property–performance framework for rational phosphor development.

11. Research Gaps and Future Perspective

Direct side-by-side comparisons of several synthesis routes for the same host–activator system remain relatively uncommon. Such studies are needed because comparisons across different compositions confound chemistry and processing effects.

The relationship between synthesis-induced defects and luminescence also remains incompletely resolved. Defects can act as sensitizers, traps or quenching centres, and the same nominal defect concentration can have different effects depending on spatial distribution and local coordination. Combining XPS/EPR/thermoluminescence with time-resolved PL and computational modelling should improve mechanistic understanding.

Future research should report energy input, solvent use, yield and waste alongside optical metrics such as quantum yield, lifetime and thermal quenching. This will make meaningful comparison of sustainable synthesis possible rather than relying on qualitative claims.

12. Conclusions

Phosphor synthesis is a controlling variable in the development of high-performance luminescent materials. Conventional solid-state processing remains indispensable for scalable, highly crystalline bulk

phosphors, whereas sol–gel, co-precipitation and hydrothermal routes provide stronger compositional and morphological control for many nanoscale systems. Combustion and microwave-assisted methods reduce processing time and can enhance homogeneity; Pechini chemistry is valuable for complex multication hosts; spray pyrolysis supports continuous particle and film processing; and mechanochemistry offers a promising solvent-minimized alternative. No method is universally optimal. The appropriate route must be selected by balancing host chemistry, activator oxidation state, desired particle architecture, defect population, optical performance, scalability, cost and environmental burden. The next generation of phosphor research will increasingly combine synthesis engineering with defect control, advanced characterization, computational screening and data-driven optimization. Such integration offers a credible path toward reproducible, scalable and sustainable phosphors for lighting, displays, sensing, dosimetry, scintillation and biomedical technologies.

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