

# Phosphors for LED-based Solid-State Lighting

by Anant A. Setlur

The efficacy of solid-state lighting (SSL) based upon InGaN LEDs has improved by >10x over the past decade: the efficacy of cool white LEDs surpasses linear fluorescent lamp (LFLs) efficacies (>100 lm/W) and warm white 1W LEDs surpasses compact fluorescent lamps (CFLs) efficacies (>60-70 lm/W). The U.S. DOE has set a 2015 efficacy target of 138 lm/W for warm white packages, a significant technical achievement that would lead to SSL market penetration in many lighting product segments. In LED-based SSL, violet, blue, and green LEDs are based upon InGaN semiconductors, while the red and amber LEDs are based upon AlInGaP semiconductors.<sup>1-4</sup> Both of these semiconductor systems tend to have much lower efficiencies in the green, yellow, and amber spectral regions. Many aspects regarding the progress in LED chip efficiency, such as fundamental causes for the lower green and amber LED efficiency, and the impact of LED-based SSL on lighting energy consumption have been discussed elsewhere<sup>2-5</sup> and will not be addressed here. The limitations in InGaN and AlInGaP efficiency make it necessary to use phosphor downconversion (in spite of the inherent Stokes losses) to generate green and yellow light for high efficacy LED packages, lamps, and fixtures. In addition, since the maximum efficiency of blue and violet InGaN LEDs appears to be higher than the maximum efficiency for red ( $\lambda_{\text{max}} = 600\text{-}630\text{ nm}$ ) AlInGaP LEDs,<sup>2</sup> the potential efficacy for a system that uses phosphor downconversion of InGaN LEDs (termed pcLEDs in this article) for the entire white spectrum could be greater than systems using AlInGaP LEDs to generate red light, further motivating the development of LED phosphors across the visible spectrum.

There has been extensive research and development for phosphors in LFLs/CFLs, cathode-ray tubes (CRTs), and X-ray films,<sup>6</sup> but most of these traditional phosphors are not suitable for pcLEDs. This is usually because these phosphors do not strongly absorb violet or blue LED radiation, leading to LED package losses from scattering. Also, many traditional phosphors use  $\text{Eu}^{3+}$ ,  $\text{Tb}^{3+}$ , or  $\text{Mn}^{2+}$  activators whose transitions are forbidden with long decay times (>1 ms), causing phosphor quenching due to saturation from the high LED radiation flux on the phosphor.<sup>7,8</sup> Using  $\text{Ce}^{3+}$  and  $\text{Eu}^{2+}$  phosphors with  $5d^1 \rightarrow 4f^1$   $\text{Ce}^{3+}$  or  $4f^6 5d^1 \rightarrow 4f^7$   $\text{Eu}^{2+}$  emission transitions that have decay times of <100 ns and <3  $\mu\text{s}$ , respectively, prevents this

saturation-based quenching. Finally, the phosphor temperature in LED packages can be >150°C, and many LFL/CFL and CRT phosphors have strong quenching at these temperatures. These additional requirements make it necessary to develop new phosphors specifically for pcLEDs.

The challenges and additional requirements for LED phosphors are balanced by larger potential composition spaces for LED phosphors versus that for LFL/CFL phosphors. For example, many silicate phosphors darken in the Hg-plasma due to Hg-adsorption,<sup>9</sup> generally preventing their use unless they are coated with a protective layer to prevent these reactions. These reactions are not present in LEDs, opening up many potential phosphor compositions. In addition, fluorescent lamp phosphor suspensions are water-based, preventing the use of phosphors that decompose in water. Again, these restrictions are less of an issue in LEDs since many packaging protocols avoid aqueous processing conditions. However, while processing issues might be alleviated, there are potential issues with phosphor stability at high temperatures and high humidity conditions (e.g. 85°C and 85% relative humidity) since pcLEDs are not necessarily hermetically sealed.

Many of the needs for new LED phosphors have been met by the discovery and development of new phosphors over the past 10 years. During this time, the field of LED phosphors has moved from a single family of phosphor compositions—the  $\text{Ce}^{3+}$ -doped aluminate garnets—to a variety of silicate, aluminate, nitride, oxynitride, sulfide, and fluoride compositions, leading to commercial LEDs that cover a full range of white CCTs (Fig. 1). This article will briefly

discuss these various LED phosphors with some of their advantages and drawbacks.

## Phosphors for LED-based Lighting

**$\text{Ce}^{3+}$ -doped garnets.**—The main yellow phosphor (with compositional modifications) used in pcLEDs,  $\text{Y}_3\text{Al}_5\text{O}_{12}:\text{Ce}^{3+}$  (YAG:Ce),<sup>10</sup> was reported in 1967<sup>11</sup> with its primary use (prior to pcLEDs) in CRTs. In pcLEDs, YAG:Ce absorbs blue LED radiation through the allowed  $4f^1 \rightarrow 5d^1$  transition and emits yellow light via the reverse  $5d^1 \rightarrow 4f^1$  transition (Fig. 2). The emission from the lowest excited  $5d^1$  level is to the spin-orbit split  $4f^1$  ground states, leading to an extremely broad emission band with a FWHM > 100 nm. The yellow emission from YAG:Ce and the blue radiation that “bleeds” through a YAG:Ce coating combines to give white light with a daylight-like color temperature (CCT > 4000 K) and reasonable color rendering (CRI ~70-80), enabling pcLEDs to be used in many applications where color quality is not a key requirement, including backlights for portable displays and indicators. Since these markets dominate the total pcLED market, current phosphor usage in pcLEDs is heavily skewed toward YAG:Ce. The properties of YAG:Ce also represent a benchmark for other LED phosphors. First, the absorption and emission transitions are parity and spin allowed, giving strong absorption of blue LEDs and a fast decay time that prevents saturation quenching. The quantum efficiency (QE) of YAG:Ce under blue LED excitation is >85%, even at 200°C,<sup>12</sup> and there are no indications that YAG:Ce degrades under blue LED excitation or moisture. Also,

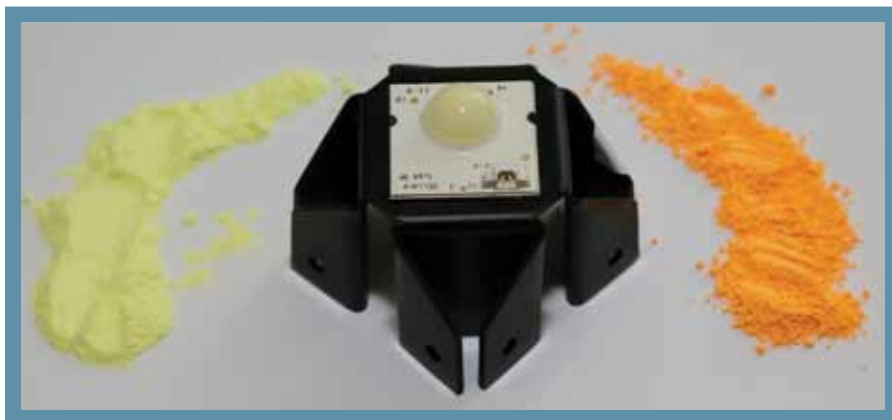


Fig. 1. Picture of GE Lumination Vio™ LED package that is based upon phosphor downconversion of 405 nm LEDs with phosphor powders.

the synthesis of YAG:Ce is relatively straightforward and uses high-purity precursors ( $Y_2O_3$ ,  $Al_2O_3$ ,  $CeO_2$ ) that have been qualified for use in traditional CFL/LFL/CRT phosphors.

One deficiency for pLEDs that use only YAG:Ce is that they are limited to high CCTs and lower CRIs, due to a lack of a red spectral component. It is possible to redshift the  $Ce^{3+} 5d^1 \rightarrow 4f^1$  emission through  $Gd^{3+}$  substitution of  $Y^{3+}$ ,<sup>13</sup> or  $Mg^{2+}$ - $Si^{4+}$  substitution for  $Al^{3+}$ (octahedral)- $Al^{3+}$ (tetrahedral).<sup>14</sup> This redshift comes at the cost of efficiency, especially at high temperatures.<sup>14,15</sup> The limits for red  $Ce^{3+}$ -doped aluminate garnets (as well as intellectual property concerns in using  $Ce^{3+}$ -doped aluminate garnets) led to further investigation of alternate garnet compositions. One route was to investigate silicate garnets with the caveat that the composition space for silicate garnet synthesis at ambient pressure is smaller than that for aluminate garnets. In spite of these limitations, there has been progress finding silicate garnet phosphors with  $Ce^{3+}$  emission maxima ranging from 505-605 nm with room temperature QEs that are comparable to YAG:Ce phosphors<sup>16-18</sup> that also give initial structure-property relationships for  $Ce^{3+}$  emission in garnets.<sup>19</sup> It has also been shown that replacing  $Al^{3+}$ (tetrahedral)- $O^{2-}$  with  $Si^{4+}$ - $N^{3-}$  in the garnets leads to an additional red component in the emission spectra due to  $Ce^{3+}$  ions coordinated by  $N^{3-}$ .<sup>20</sup> However, any orange-red garnet still has stronger high temperature quenching compared to YAG:Ce. Since this additional quenching is relatively independent of composition, it may be intrinsic and points to a limitation for  $Ce^{3+}$ -doped garnets for orange-red phosphors.

**Nitride and oxynitride phosphors.**—The energy position of  $Eu^{2+}/Ce^{3+} 4f^N-15d^1$  level  $4f^N \rightarrow 4f^{N-1}5d^1$  transitions in inorganic hosts is modified by the covalency and polarizability of  $Eu^{2+}/Ce^{3+}$ -ligand bonds.<sup>21</sup> Therefore, ligands with a lower electronegativity compared to  $O^{2-}$  ( $\chi(O) \sim 3.4$ ), such as  $S^{2-}$  ( $\chi(S) \sim 2.6$ ) and  $N^{3-}$  ( $\chi(N) \sim 3.0$ ) will lower the energy of the  $4f^N-15d^1$  levels making it more likely for  $Ce^{3+}/Eu^{2+}$  absorption of InGaN LED radiation. There are efficient  $Ce^{3+}/Eu^{2+}$ -doped sulfide phosphors, but there are drawbacks that lead many pLED manufacturers to move away from sulfide phosphors. First, sulfide phosphor synthesis could require toxic  $H_2S$  atmospheres or may create  $H_2S$  as a by-product. Second, many sulfide phosphors are moisture sensitive, degrading in high humidity conditions unless phosphor particles are coated with a moisture barrier. Unlike sulfides, little work was done on the luminescence of  $Eu^{2+}/Ce^{3+}$  in nitrides until Hintzen and co-workers discovered efficient  $Eu^{2+}$  luminescence

in  $Ca^{2+}$ - $\alpha$ -SiAlON<sup>22</sup> and  $M_2Si_5N_8$  ( $M = Ca^{2+}, Sr^{2+}, Ba^{2+}$ )<sup>23</sup> (Fig. 3) that could be excited with violet or blue LEDs. Many of these new phosphors match the room and high temperature QE of YAG:Ce. In addition, these new nitrides do not degrade under high temperature/high humidity conditions due to the ability of  $N^{3-}$ -containing  $Si(O,N)_4$  tetrahedra to form condensed and cross-linked tetrahedral networks.<sup>24</sup> The potential for high efficiency and stable pLED phosphors within nitride compositional spaces has led to extensive investigation of a variety of (oxy)nitride compositions for potential pLED phosphors. One family of efficient phosphors are the  $MSi_2O_2N_2:Eu^{2+}$  ( $M = Ca^{2+}, Sr^{2+}, Ba^{2+}$ ) compositions whose emission ranges from  $\sim 498$  nm for  $BaSi_2O_2N_2:Eu^{2+}$  to  $\sim 560$  nm to  $CaSi_2O_2N_2:Eu^{2+}$ ,<sup>25</sup> with QE > 85% at  $T > 200^\circ C$  for the green ( $\lambda_{max} \sim 540$  nm)  $SrSi_2O_2N_2$  phosphor.<sup>26</sup>  $Eu^{2+}$ -doped  $\beta$ -SiAlON has also been reported to be an efficient green phosphor.<sup>27</sup> For orange and red phosphors, another important family of materials are based upon  $Eu^{2+}$ -doped  $CaAlSiN_3$  hosts with a distorted wurtzite structure.  $CaAlSiN_3:Eu^{2+}$  phosphors have a  $\sim 650$  nm emission peak and QEs > 85% beyond  $200^\circ C$  (Fig. 3),<sup>28</sup> and the emission maximum can be tuned down to 620 nm by  $Sr^{2+}$  substitution by making phosphors using high pressure nitriding of arc-melted alloys.<sup>29</sup>

While many of these nitride phosphors were based on known (oxy)nitride compositions, there has also been investigation of nitride and oxynitride phase diagrams to discover

new phosphors. In many respects, the discovery of new (oxy)nitrides required some degree of guesswork and perseverance since the formation of condensed tetrahedral networks with  $N^{3-}$  makes it difficult to draw analogies to silicate or aluminate solid-state chemistry. Investigation of nitride and oxynitride compositional spaces has led to the discovery of more new nitride phosphors (and hosts) such as the green  $Sr_5Al_{5+x}Si_{21-x}N_{35-x}O_{2+x}:Eu^{2+}$  (with  $x \sim 0$ ),<sup>30</sup> the orange-red  $SrAlSi_4N_7:Eu^{2+}$ ,<sup>31</sup> the yellow  $Ba_2AlSi_5N_9:Eu^{2+}$ ,<sup>32</sup> and the green  $Ba_3Si_6O_{12}N_2:Eu^{2+}$ .<sup>33</sup> Further work will likely give more new materials that also could be of interest in pLEDs.

The efficient yellow, orange, and red  $Eu^{2+}$  emission from these nitride phosphors can be combined with YAG:Ce in warm white LEDs for lamps and fixtures that would normally use incandescent lamps or CFLs. For example, phosphor blends of YAG:Ce and  $CaAlSiN_3:Eu^{2+}$  combined with blue LEDs can be used for high CRI, warm white lamps. In addition, it is also possible to use all-nitride phosphor blends to make pLEDs that cover a full range of CCTs at CRI > 90.<sup>34,35</sup> However, while (oxy)nitrides have significant potential, there are some possible drawbacks to (oxy)nitride phosphors. First, the synthesis of these materials can be more difficult than for typical oxide phosphors. The refractory nature of  $Si_3N_4$  often requires high temperature reactions ( $> 1500^\circ C$ ), and many of the precursor materials, such as alkaline-earth nitrides or  $Si(NH_2)_2$ ,<sup>24</sup> require glove-box handling due to reactions

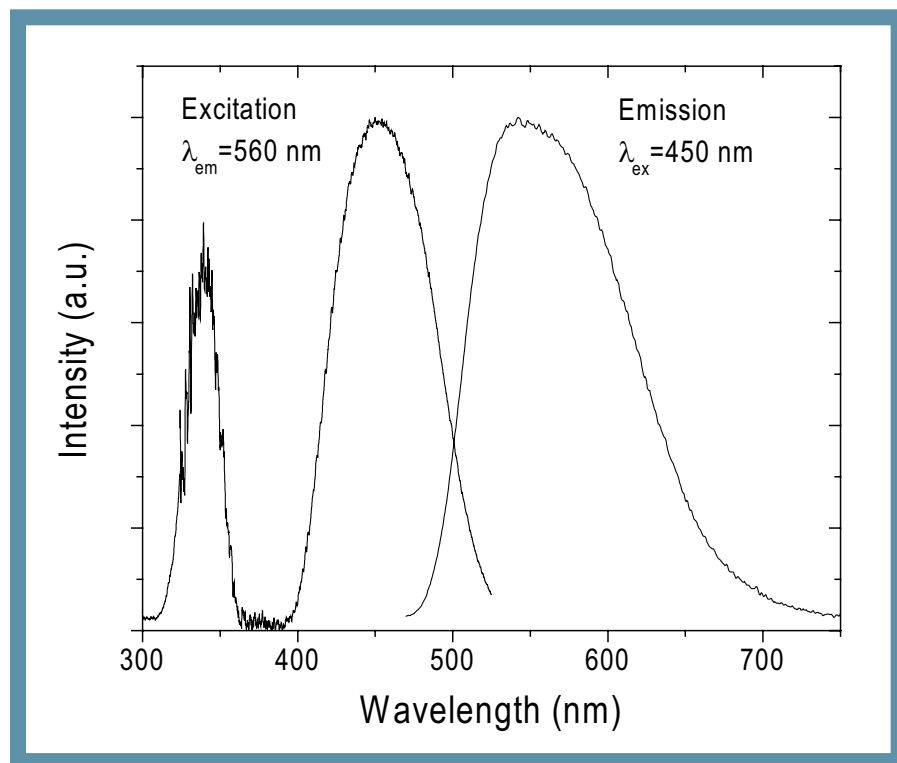


Fig. 2. Emission ( $\lambda_{ex} = 460$  nm) and excitation ( $\lambda_{em} = 560$  nm) spectra of  $Y_3Al_5O_{12}:Ce^{3+}$  (3%) phosphor typically used in LED-based lighting.

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with air or moisture. There has been progress in using air-stable precursors to make nitride phosphors,<sup>29,36</sup> so this issue could be alleviated. However, there have also been reports in the patent literature that some of these  $\text{Eu}^{2+}$ -doped nitride phosphors may lose efficiency over time in pcLED packages.<sup>37</sup> As with traditional CRT/LFL/CFL phosphors, it is possible that improved synthesis processes will improve the stability of some nitride phosphors in pcLEDs.

**Oxide, oxyhalide, and halide phosphors.**— Apart from the newer nitride phosphors, there has also been progress in the development of LED phosphors based upon more traditional oxide and oxyhalide host lattices. The main set of non-garnet, oxide phosphors for pcLEDs the alkaline earth silicates,  $\text{M}_2\text{SiO}_4:\text{Eu}^{2+}$  ( $\text{M} = \text{Ba}^{2+}, \text{Sr}^{2+}, \text{Ca}^{2+}$ ) that were originally studied in the 1960s.<sup>38,39</sup> The emission of  $\text{Ba}_2\text{SiO}_4:\text{Eu}^{2+}$  peaks at  $\sim 505$  nm, and  $\text{Sr}^{2+}/\text{Ca}^{2+}$  substitution leads to a redshift in the emission until  $\sim 585$ – $590$  nm with the excitation generally peaking in the violet and deep blue (Fig. 4). The excitation of the yellow phosphors in this family extend into the blue spectral region, making these phosphors an alternative to YAG:Ce. However, while these phosphors are efficient at room temperature, their main drawback is the relatively strong quenching at elevated temperature where  $\text{QE}(150^\circ\text{C})/\text{QE}(\text{RT}) = 60$ – $65\%$ . Another potentially interesting system is based upon hosts with the  $\text{Cs}_3\text{CoCl}_5$  structure or variants of that structure.  $\text{Eu}^{2+}$ -doped materials in this family, such as  $(\text{Sr},\text{Ca},\text{Ba})_3\text{SiO}_5:\text{Eu}^{2+}$ , have a yellow-orange emission,<sup>40</sup> while  $\text{Ce}^{3+}$ -doped materials, such as  $\text{Sr}_2\text{LaAlO}_5:\text{Ce}^{3+}$ ,  $\text{Sr}_3\text{SiO}_5:\text{Ce}^{3+}$ , or their solid solutions,<sup>41</sup> have a yellow-green emission similar to YAG:Ce. As is the case for the  $\text{M}_2\text{SiO}_4:\text{Eu}^{2+}$  phosphors, many of these phosphors have strong high temperature quenching, but perhaps the compositional flexibility in this system can lead to better pcLED phosphors. There also have been reports of various oxyhalide phosphors doped with  $\text{Eu}^{2+}$ ,<sup>42</sup> but further work is also necessary to determine their utility. As more oxide systems are shown to be suitable for pcLEDs, there could be more emphasis on (re)searching these compositional spaces for new phosphors.

We close this discussion by noting that the phosphors discussed to this point are  $\text{Eu}^{2+}$  or  $\text{Ce}^{3+}$  broadband emitters (FWHM  $> 70$  nm). There are advantages to use narrow-line phosphors (FWHM  $< 10$  nm) in the red spectral region since this can simultaneously optimize color rendering and lumens.<sup>2</sup> Within LFL/CFL phosphors, there are two main activators used to give efficient, red-line emission,  $\text{Mn}^{4+}$  and  $\text{Eu}^{3+}$ . There have been many literature reports of potential

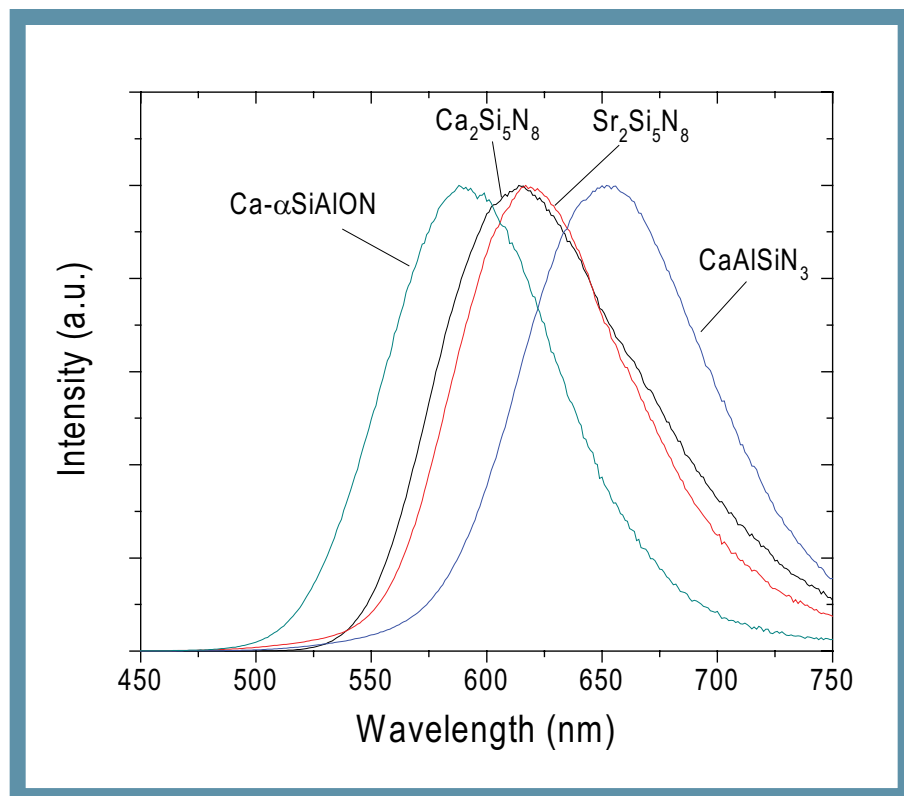


Fig. 3. Emission spectra ( $\lambda_{\text{ex}} = 405$  nm) of  $\text{Eu}^{2+}$ -doped nitride and oxynitride phosphors.

$\text{Eu}^{3+}$ -pcLED phosphors where the only excitation bands overlapping with violet or blue LED radiation are the forbidden  $\text{Eu}^{3+}$  4f-4f transitions. Since these transitions have low oscillator strengths ( $< 10^{-6}$ ), the absorption of InGaN LED radiation will be weak and micron-sized powders will strongly scatter incident LED radiation. In addition, these 4f-4f absorption transitions are sharp, making the phosphor excitation sensitive to the LED wavelength. There has been recent progress in developing high efficiency,  $\text{Eu}^{3+}$ -doped nanophosphors that could significantly reduce phosphor scattering,<sup>44</sup> so it will be interesting to see their impact on pcLED SSL.  $\text{Mn}^{4+}$ -phosphors have the spin-allowed, parity forbidden  ${}^4\text{A}_2 \rightarrow {}^4\text{T}_2$  transition that has good absorption of blue and violet LED radiation.<sup>45</sup> For example, the commercial LFL phosphor Mg-fluorogermanate: $\text{Mn}^{4+}$  can improve the color rendering of pcLEDs based on violet LEDs, and there are various  $\text{Mn}^{4+}$ -doped fluoride phosphors that have high efficiency under blue LED excitation.<sup>45</sup> It is important to note that  $\text{Eu}^{3+}$  and  $\text{Mn}^{4+}$  have slow decay times ( $\sim 1$  ms for  $\text{Eu}^{3+}$ ,  $> 3$  ms for  $\text{Mn}^{4+}$ ), so they are best used in remote phosphor packages that reduce the incident flux on the phosphor.<sup>43</sup> Whether this limits the potential market for lamps and packages that use these phosphors is also an open question.

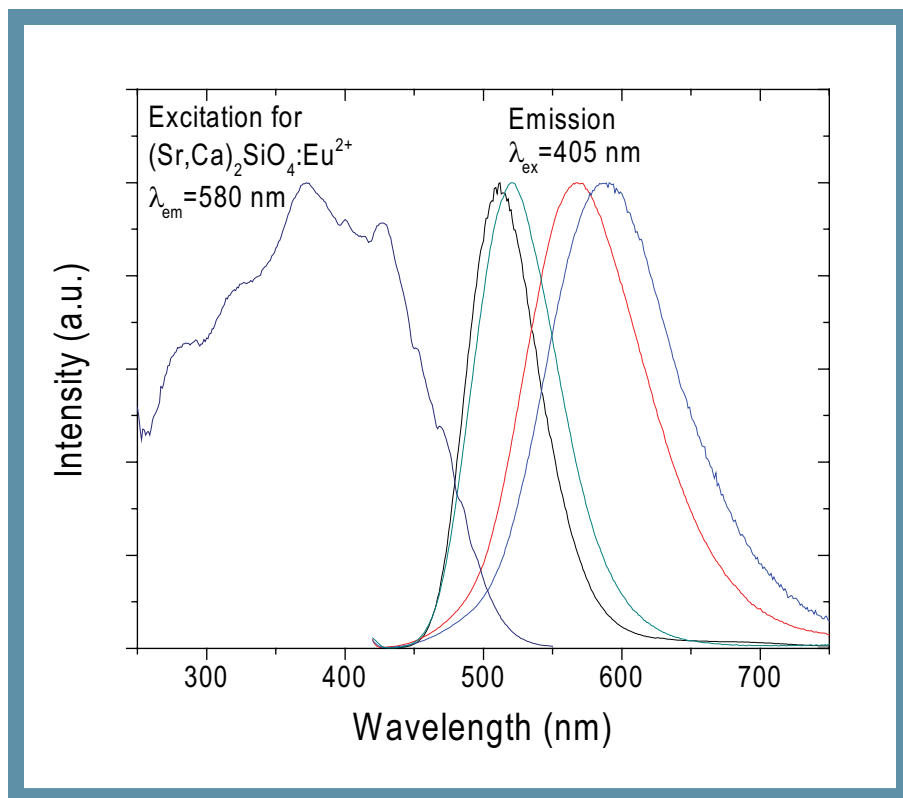
## Summary and Future Outlook

This article is a brief summary of the different pcLED phosphors that have been developed over the past ten years. The discovery and optimization of these materials has involved significant work by the various chemists, physicists, and materials scientists that are developing these materials. However, while there has been progress, there are still many challenges and new phosphors to be discovered. It is also important to note that the discovery of new phosphor compositions is one step toward pcLEDs implementation with extensive process optimization required to maximize conversion efficiencies. These aspects include various powder processing techniques and/or other ceramic processing methods such as sintering<sup>46</sup> or glass-ceramic formation<sup>47</sup> to make uniform phosphor parts and plates. These aspects are not covered here but are necessary to fully optimize pcLED efficacy.

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**Fig. 4.** Emission spectra ( $\lambda_{\text{ex}} = 405 \text{ nm}$ ) of  $\text{Eu}^{2+}$ -doped  $\text{M}_2\text{SiO}_4$  ( $\text{M} = \text{Ca}^{2+}$ ,  $\text{Sr}^{2+}$ , and/or  $\text{Ba}^{2+}$ ) phosphors and excitation spectra ( $\lambda_{\text{em}} = 580 \text{ nm}$ ) for  $(\text{Sr,Ca})_2\text{SiO}_4:\text{Eu}^{2+}$ .

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