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Supportive Information

High-Gradient Magnetic-Separation of Compact Fluorescent Lamp Phosphors: Elucidation of the Removal Dynamics in a Rotary Permanent Magnet Separator

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Supporting Information 1. Time-resolved Laser-induced Fluorescence Spectroscopic quantification: method validation

For the development of the quantification model, a standard curve of each phosphor was obtained by analyzing samples that contained 0.01 $g.L^{-1}$, 0.05 $g.L^{-1}$ and 0.10 $g.L^{-1}$ of one individual phosphor as presented in Section 3.3. This was followed by data acquisition of 13 samples containing all five phosphor particles with randomly predetermined concentrations for the validation of the analysis approach in these more complex systems. The assigned concentrations of these mixed samples are shown in Table S1. In PARAFAC, these samples were analyzed together with the standard curve samples. Thus, the same emission spectra and luminescence decays were obtained. Furthermore, distributions from the distinct YOX emissions ($^5D_0 \rightarrow {}^7F_j$ and $^5D_1 \rightarrow {}^7F_j$) were summed up for simplification. The component distributions and the linear correlations between the assigned concentrations and the calculated intensities of the mixed samples are presented in Figure S1. For each phosphor, a coefficient of determination of 0.9943, or higher, could be obtained. Hence, the deconvolution of the TRLFS data using PARAFAC worked excellently to obtain a linear correlation between the phosphors' concentrations and the intensities of their PARAFAC components. Hereby, we consider the approach to combine TRLFS with PARAFAC for phosphor quantification to be validated and applicable for quantification of the mixed samples of the HGMS experiment.

Table S1. Concentrations of the synthetic mixtures with randomly predetermined concentrations of each phosphor in $g.L^{-1}$.

	LAP	CAT	BAM	YOX	HAP
1	0.030	0.005	0.055	0.040	0.050
2	0.025	0.000	0.075	0.045	0.075
3	0.045	0.035	0.005	0.030	0.075
4	0.030	0.000	0.025	0.045	0.030
5	0.015	0.025	0.040	0.050	0.060
6	0.025	0.020	0.025	0.090	0.075
7	0.025	0.015	0.065	0.050	0.025
8	0.050	0.015	0.030	0.035	0.025
9	0.025	0.035	0.045	0.085	0.035
10	0.020	0.010	0.045	0.065	0.090
11	0.090	0.000	0.010	0.060	0.100
12	0.100	0.000	0.040	0.070	0.010
13	0.000	0.070	0.010	0.080	0.020

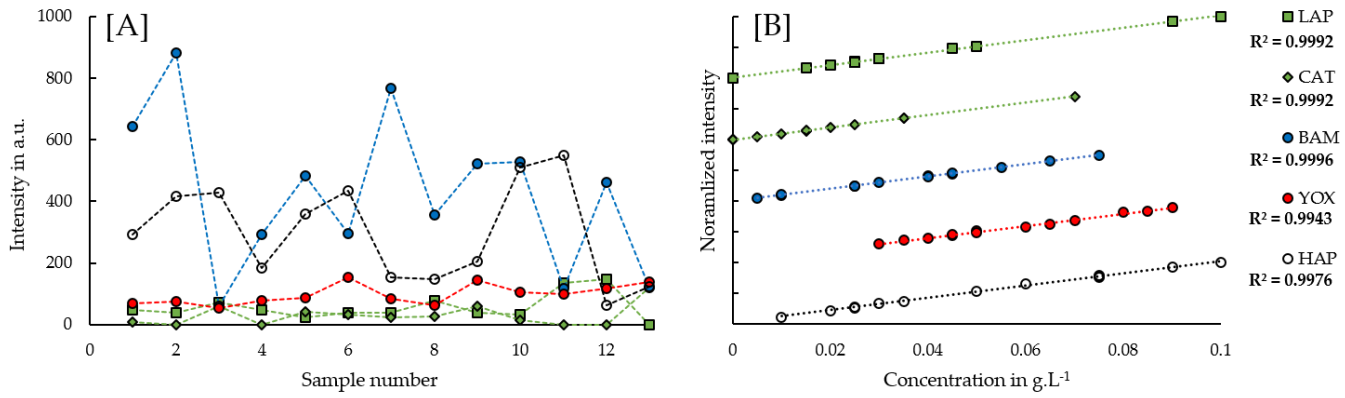
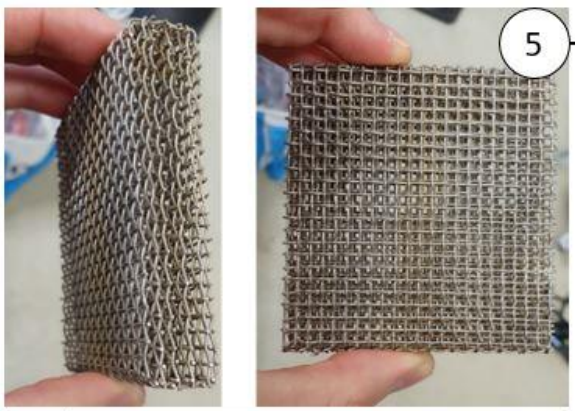


Figure S1. PARAFAC analysis of the TRLFS measurements of the synthetic mixtures with randomly predetermined concentrations of each phosphor. **[A]** Distributions of the single PARAFAC components. For YOX, the components of the two Eu^{3+} emittive levels were summed to a single value. **[B]** Linear regression analysis of the component intensities in function of the assigned concentrations of the five phosphors.

Supporting Information 2. High-Gradient Magnetic-Separation: setup and control experiments

A picture of the HGMS setup during operation in a removal experiment is depicted in Figure S2. For a thorough explanation of the working principle of the rotary permanent magnet based separator, we refer to Hoffmann *et al*. [1]. For this study, a 430 grade stainless steel wire matrix, provided by Haver & Boecker OHG (Oelde, Germany), was introduced into the separation cell. The matrix consisted of 6 mesh layers with approximately 300 wires, having diameters of 0.67 mm. Approximately half of the wires were arranged in a transversal, whereas the other half were arranged in an axial direction.



1	Mixed phosphor suspension
2	Peristaltic pump
3	Two inlet tubes
4	Rotary permanent magnet
5	Stainless steel wire mesh
6	Separation cell
7	Two outlet tubes
8	Pneumatic ball vibrator
9	Overhead stirrer

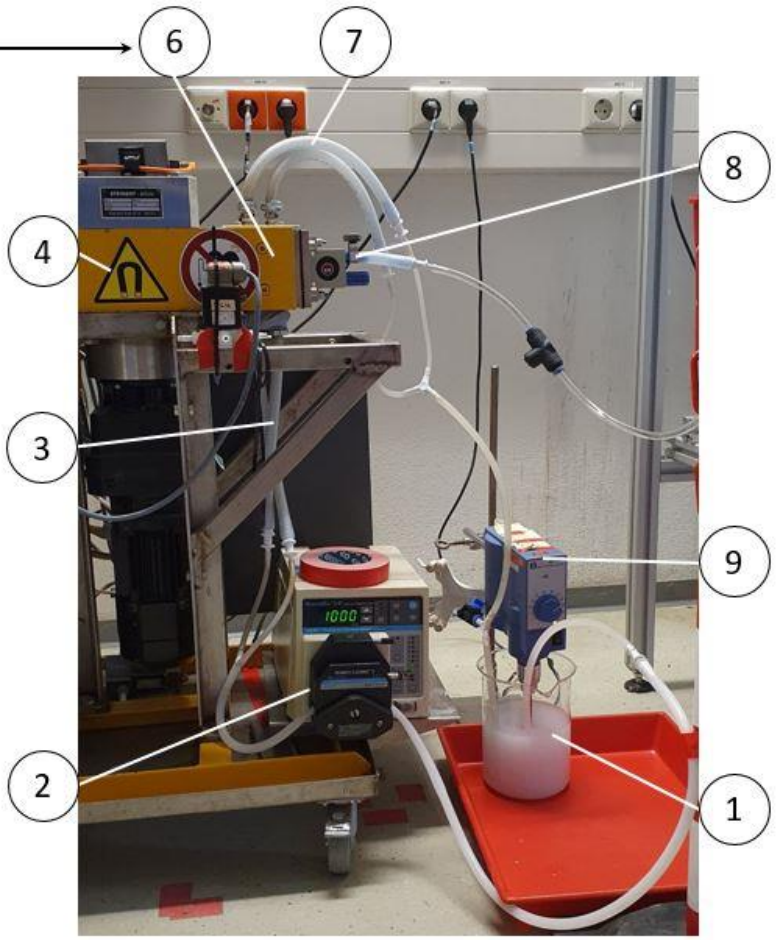


Figure S2. Picture of the HGMS setup during operation in a removal experiment. During normal operation, the mixed phosphor suspension was pumped in an upward direction, through the separation cell filled with the wire mesh. As shown in (8), a K-10 type (Aldak GmbH, Troisdorf, Germany) pneumatic ball vibrator was attached to the separation cell, as proposed by Kampeis *et al*. for particle recovery from the separation cell [2]. However, the pneumatic ball vibrator was not used in this study, as recovery was succesfuly achieved with an intermittent air-water flow in the downward direction with the device in the "field off" mode.

A scheme of the HGMS setup during normal operation and during backwashing is shown in Figure S3. During the removal experiment, the phosphor mixture was pumped in a loop through the separation cell in the "field on" mode in an upward direction at a flow rate of 1000 mL.min^{-1} for 30 minutes. Subsequently, the recovery of the removed phosphors from the separation cell was investigated by backwashing. For this purpose, a second beaker with deionized water was first pumped in a loop through the separation cell in a downward direction at a high flow rate of 2500 mL.min^{-1}. Finally, an intermittent air-water flow was pumped through the cell to create a high turbulence in a downward

direction at a flow rate of 2500 mL.min^{-1} by periodically removing the suction tube out of the beaker, in order to improve the recoverability.

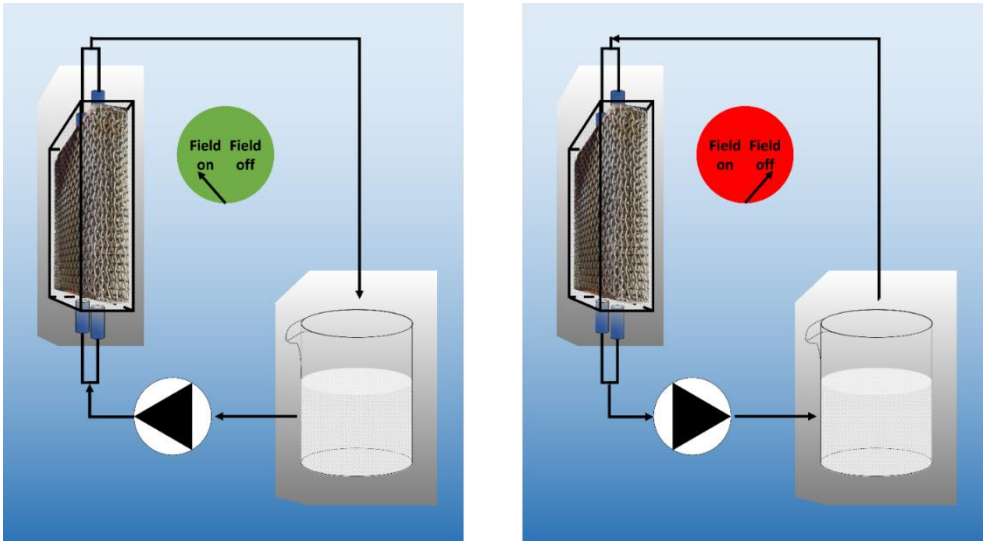


Figure S3. Scheme of the HGMS setup during normal operation (left) and recovery of the removed phosphors from the separation cell by backwashing (right).

As shown in Figure S4, the phosphors' concentrations had a slight decrease when the suspension was pumped through the separation cell during a control experiment with the device in the "field off" mode. During the 30 minutes of the experiment, the concentration of each phosphor in the suspension decreased 25-35%. This indicates, that besides a magnetic removal of the phosphors, a mechanical removal of the particles e.g. by trapping on the wire mesh or sedimentation in "dead zones" of the separation cell may have occurred. The removed fractions of the individual phosphors during the control experiments were not clearly distinguishable from eachother.

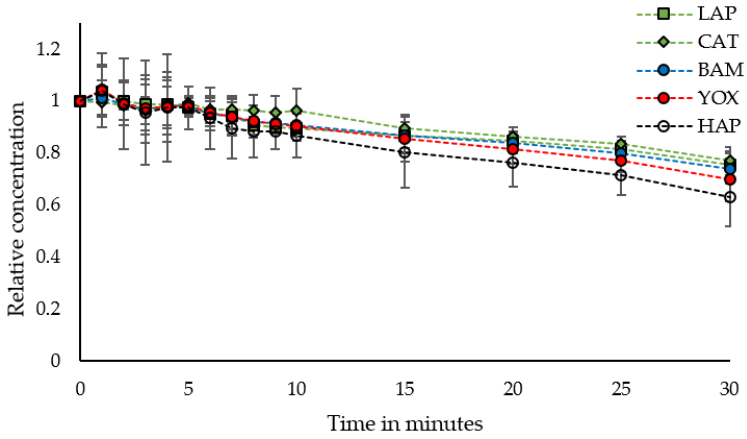


Figure S4. Evolution of the phosphors' relative concentrations during the control experiments with the HGMS, in which the device wasn't switched to the "field on" mode at the experiment's starting point. The error bars depict one standard deviation.

References

1. Hoffmann, C.; Franzreb, M.; Holl, W.H. A novel high-gradient magnetic separator (HGMS) design for biotech applications. *IEEE Transactions on Applied Superconductivity* **2002**, *12*, 963-966, doi:10.1109/TASC.2002.1018560.
2. Kampeis, P.; Bewer, M.; Rogin, S. Einsatz von Magnetfiltern in der Bioverfahrenstechnik Teil 1: Vergleich Verschiedener Verfahren zum Rückspülen der Magnetfilter. *Chem. Ing. Tech.* **2009**, *81*, 275-281, doi:10.1002/cite.200800121.