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Two-dimensional Metal–Organic Layers as A Bright and Processable Phosphor for Fast White-Light Communication

Xuefu Hu[a], Zi Wang[a], Bangjiang Lin*[b], Cankun Zhang[a], Lingyun Cao[a], Tingting Wang[a], Jingzheng Zhang[a], Cheng Wang*[a], Wenbin Lin*[a,c]

Abstract: Metal-organic layer (MOL) is a new type of 2D material that is derived from metal-organic frameworks (MOFs) by reducing one dimension to a single layer or a few layers. We assembled tetraphenylethylene-based tetracarboxylate ligands (TCBPE) with aggregation-induced emission property into the first luminescent MOL by linking with $Zr_6O_4(OH)_6(H_2O)_2(HCO_2)_6$ clusters. The emissive MOL can replace the lanthanide phosphors in white light emitting diodes (WLEDs) with remarkable processability, color rendering, and brightness. Importantly, the MOL-WLED exhibited a physical switching speed three times that of commercial WLEDs, which is crucial for visible-light communication (VLC), an alternative wireless communication technology to Wifi and Bluetooth by using room lighting to carry transmitted signals. The short fluorescence lifetime (2.6 ns) together with high quantum yield (50%) of the MOL affords fast switching of the assembled WLEDs for efficient information encoding and transmission.

Metal-organic layer (MOL) is a new type of 2D material that is derived from metal-organic frameworks (MOFs) by reducing one dimension to a single layer or a few layers.[1] Similar to MOFs, MOLs serve as a versatile platform to organize functional molecular building blocks.[2] Introducing fluorescent ligands into MOLs can afford luminescent material that is more processable than their MOF counterparts for LED lighting[3] and other fluorescence-related applications.[4] The network structure of MOL immobilizes fluorescent struts, which not only prevents their self-aggregation but also restricts their internal motions, both of which enhance quantum yields and suppress unwanted photochemistry. The MOLs as ultrathin nano-sheets also alleviate the scattering and non-uniform absorption of back light by bulk solid phosphors in LED construction.

Luminescent MOLs assembled from fluorescent molecules are particularly suitable for constructing white light emitting diodes (WLEDs) for visible-light communication (VLC). VLC is a wireless communication technology that uses everyday lighting for information transmission and is motivated by the possible fast switching of WLEDs to encode signals.[5] As compared to radio-frequency based technologies such as Wi-Fi and Bluetooth, VLC can transmit information with higher speed, higher security, lower radio interference, lower cost, more bandwidth and more desirable bio-friendliness.[5b]

Although the theoretical upper limit of modulation frequency for data transmission in the visible-light range is up to hundreds of THz, commercial WLEDs have limited physical response frequency[6] of only hundreds of kHz as a result of slow response of the lanthanide phosphors such as Ce-doped yttrium aluminum garnet (YAG-Ce).[7] The luminescence of YAG-Ce has an emitting lifetime of 200 ns, leading to an intrinsic modulation frequency of less than 0.8 MHz.[8] Even with such a slow response rate, the modulation frequency in VLC based on commercial WLEDs has been pushed to hundreds of MHz by resorting to regions of small signals using advanced detection and signal equalizing circuits. The transmission rate can be further boosted to over Gbit/s by using spectrally efficient modulation techniques. [9] We envision that if we can improve the intrinsic physical response frequencies of WLEDs, we will be able to further increase the capability of the VLC technology.

Molecular phosphors exhibit fluorescence lifetimes down to ns with high quantum yields, and are thus suitable replacements for YAG-Ce in fast VLCs. MOLs provide an ideal platform to organize the molecular phosphors into luminescent materials with theoretical response frequency of hundreds of MHz, and can thus remove the rate limit imposed by the phosphor in current WLEDs.

In this work, we assembled a MOL-based WLED and achieved an intrinsic physical modulation frequency of 1.7 MHz at 3dB attenuation of the received oscillating signal, as compared to 0.6 MHz for a commercial WLED. The frequency response of the MOL-based WLED is the same as that of the underlying LED circuits, demonstrating the ability to overcome the limitation of phosphor response rate by using a novel fluorescent MOL.

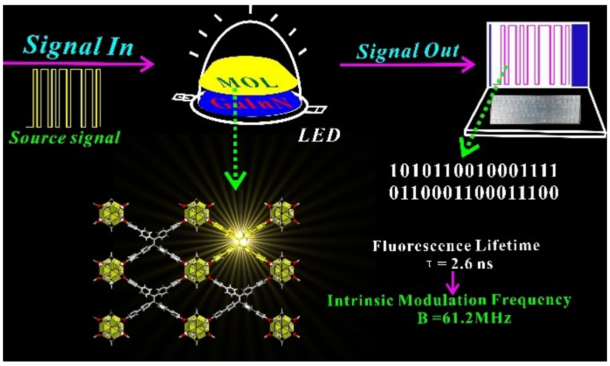

Figure 1. Schematic showing the construction of visible-light communication (VLC) devices using a fluorescent MOL as the phosphor.

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We utilized a tetraphenyl ethylene-based ligand 4',4'',4''',4''''-(ethene-1,1,2,2-tetrayl)tetrakis([1,1'-biphenyl]-4-carboxylic acid) (H_4TCBPE)[10] (Figure S1-S4) as the fluorescent phosphor with the aggregation-induced emission (AIE) property.[11] Li[10] and other groups[12] have used this ligand and related derivatives to construct MOFs for LEDs and WLEDs with remarkable color rendering and brightness. We successfully obtained a fluorescent 2D MOL (Zr-TCBPE-MOL) based on $[Zr_6O_4(OH)_6(H_2O)_2(HCO_2)_6]^{4+}$ secondary building units (SBUs) by reacting H_4TCBPE with $ZrCl_4$ in the presence of formic acid and water in DMF (Figure 2). The MOL was obtained as a result of SBU super-saturation, a strategy we previously adopted for the synthesis of 2D MOLs based on tricarboxylate ligands.[1f]

Both the TCBPE ligands and SBUs are 4-connected in Zr-TCBPE-MOL. Each carboxylate on the ligand bridges two Zr^{4+} ions on the same SBU. The four benzoate arms of the same ligand lie on the same plane with 120° and 60° angles between them. These directions match four of the twelve connections on the $[Zr_6O_4(OH)_6(H_2O)_2(HCO_2)_6]^{4+}$ SBU, leading to 4,4-connected 2D MOL with a sql topology (Figure 2c). The rest of the Zr sites on the SBUs are capped by formate groups that are added as a modulator in the synthesis.

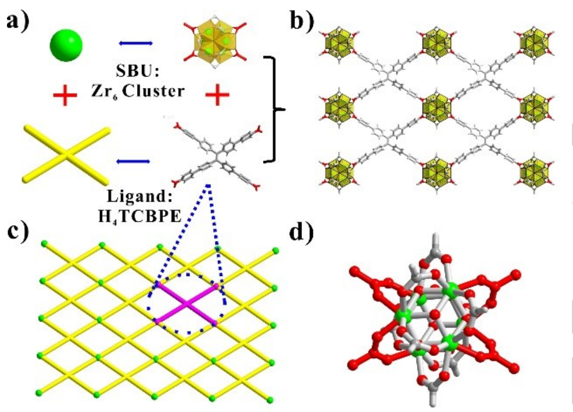

Figure 2. a) Structrues of the H_4TCBPE ligand and Zr_6 clusters; b) The structure of Zr-TCBPE-MOL; c) The sql topology of Zr-TCBPE-MOL; d) Ball-and-stick model of the Zr_6 cluster.

This 2D structural model of Zr-TCBPE-MOL was confirmed by a number of experimental evidences. First, the Bragg peaks in the powder X-ray diffraction (PXRD) pattern of Zr-TCBPE-MOL could all be indexed to (hk0) reflections, which is a unique feature for 2D materials (Figure 3a top).[1f] The experimental PXRD pattern also matched the simulated one using the 2D atomic model of Zr-TCBPE-MOL (Figure 3a bottom).

Second, the arrangement of the SBUs in two dimensions as revealed in scanning transmission electron microscope high-angle annular dark-field (STEM-HAADF) images (Figure 3d) nicely fit the structural model of the sql topology (Figure 2c). The distances between adjacent SBUs in the model (2.46 nm) were corroborated by both the distances between adjacent white dots of 2.50 nm in STEM-HAADF images (Figure 3d) and the distances between the lattice fringes of 2.38 nm in High Resolution Transmission electron microscopy (HRTEM) images (Figure 3b). Fourier-transformed maps of these images also gave an averaged value of 2.44 nm which matched these distances (Figure 3b). The TEM images of larger area show pleated films of 300 nm to 500 nm in size for the MOL (Figure 3c, S5). This pleat feature is typical for ultrathin 2D materials.

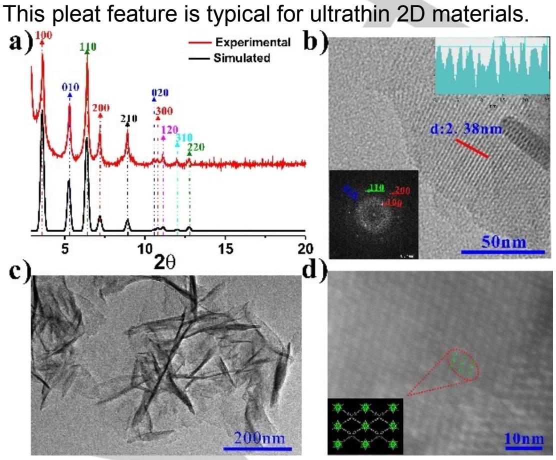

Figure 3. a) Experimental (red line at the top) and simulated (black line at the bottom) PXRD patterns of Zr-TCBPE-MOL; b) HRTEM and corresponding FFT images of Zr-TCBPE-MOL; c) TEM image of Zr-TCBPE-MOL; d) HAADF image of Zr-TCBPE-MOL.

Third, the thickness of the Zr-TCBPE-MOL was measured by atomic force microscopy (AFM) (Figure 4) to be 2.43 nm, corresponding to the van der Waals thickness of a bilayer of the proposed structure. Thicker films with averaged thickness of 5.0 nm were also observed, consistent to four layers of the proposed structure. (Figure S6).

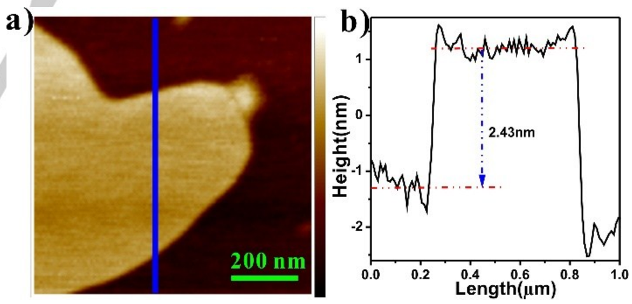

Figure 4. a) AFM image; b) the height profile of Zr-TCBPE-MOL.

Fourth, the chemical composition of Zr-TCBPE-MOL was determined by a combination of thermogravimetric analysis (TGA) and NMR spectroscopy. TGA gave the metal/ligand ratio (Figure S7) whereas ^{1}H-NMR of the digested sample afforded the ratio between the capping formates and the TCBPE ligand (Figure S8). The resultant formula of $[Zr_6O_4(OH)_6(H_2O)_2(HCO_2)_6(TCBPE)]$ is consistent with that of the structural model shown in Figure 2. Hydroxides and water molecules in the formula were assigned based on the charge balance requirement.

Fifth, we used preformed $[Zr_6O_4(OH)_4]^{12+}$ clusters capped by methacrylates as the Zr source in the synthesis, and obtained

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the same Zr-TCBPE-MOL based on PXRD patterns (Figure S9). The direct assembly of Zr_6 SBU to MOL structure further supports our assignment of the SBUs.

Photophysical property of Zr-TCBPE-MOL was measured before assembling WLEDs. Zr-TCBPE-MOL gives yellow fluorescence with a broad emission spectrum centered at 560 nm [full width at half maximum (FWHM) = 100 nm] when excited at 450 nm, the peak wavelength of commercial blue LED based on GaInN (Figure 5a). This fluorescence spectrum closely represents that of YAG-Ce[13] and corresponds to a coordinate of (0.42, 0.54) on the Commission Internationale de L'Eclairage (CIE) map (Figure 5b). This result is very similar to MOFs built from the same ligand reported by Li and coworkers.[10] The blue emission of the GaInN LED can be drawn at (0.15, 0.05) on the CIE map (Figure 5b). Any combination of these blue and yellow emissions should fall on the line connecting the two end points on the CIE map. The white light point (0.33, 0.33) locates right on the line, indicating the possibility of constructing WLEDs using Zr-TCBPE-MOL and GaInN LED.

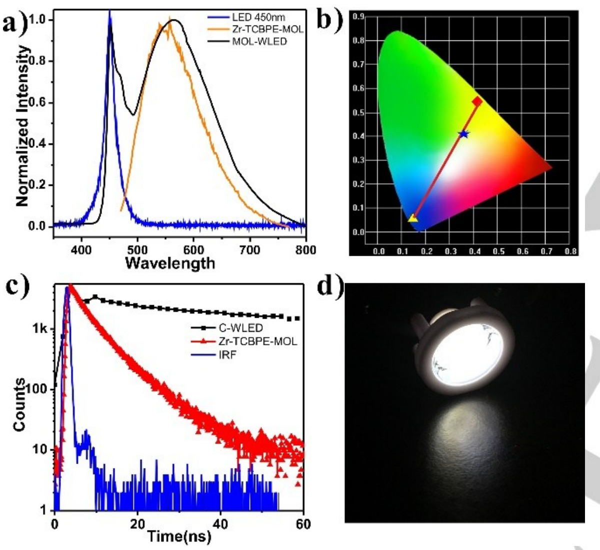

Figure 5. a) Emission spectra of Zr-TCBPE-MOL (orange line) and 450 nm LED (blue line) and the MOL-WLED (black line); b) The coordinates of the MOL-WLED on CIE map (red square: Zr-TCBPE-MOL, yellow triangle: 450 nm LED, blue star: MOL-WLED); c) The fluorescence decay curves of Zr-TCBPE-MOL (line and triangles) and commercial WLED (line and squares); d) The photo of the MOL-WLED.

The fluorescence lifetime of Zr-TCBPE-MOL was determined to be 2.6 ns as shown in Figure 5c, significantly shorter than that of YAG-Ce (Figure 5c, S10). We thus hypothesize that Zr-TCBPE-MOL can give a WLED that is turned on/off much faster than commercial ones. This fast response rate can support an on/off frequency of over 60 MHz in VLC. The quantum yield of Zr-TCBPE-MOL is as high as 50%, suitable for the construction of efficient LEDs.

To assemble the WLED, a suspension of Zr-TCBPE-MOL was filled into an optical cavity just above the GaInN LED chip and dried in air. (Fig. S11) The yellow emission from Zr-TCBPE-MOL and the transmitted blue light were redistributed by the optical head to give a uniform white light field. The emission power of this WLED exhibited a linear dependence on the working current (Figure S12), a desirable feature for VLC. The spectrum of the white emission from the MOL-WLED (Figure 5b) corresponds to (0.37, 0.41) on the CIE coordinates, representing a warm-white light that is comfortable to human eyes (Figure 5d, S13).The MOL-WLED was continuously operated over 168 hours to test its stability. A 30% drop of the output power was detected at the end of the test (Fig. S14), possibly due to decomposition of the TCBPE ligand under intense light in the presence of oxygen. We believe that this stability can be further improved by sealing the optical cavity in air-tight assemblies.

A VLC system was constructed to test the frequency response of the WLED in communication. An arbitrary waveform generator (AWG) was used to generate a high frequency signal to drive the WLED in a coupled mode of alternating current (AC) and direct current (DC). The WLED received a voltage signal with a DC background of 3.3 V that drove the device and an AC overlay of 3 V that modulated the light flux at high frequency. This intensity-modulated light was received by a PIN photodiode and then converted back to electric signals which were analyzed by an oscilloscope.

The amplitudes of the received AC signals were recorded at different input frequencies with the same input driving voltage. When the input frequency was too high, the system could not fully follow the oscillation, and the transmitted signal dropped in amplitude. The frequency at which the transmitted signal drops to -3dB with respect to the amplitude at zero frequency is defined as the maximum usable frequency in communication. As shown in Figure 6a, the normalized transmission amplitude of the MOL-WLED showed a linear dependence over the frequency range of 0-4 MHz, with an intrinsic modulation frequency of 1.7 MHz at -3dB. In comparison, a similar system constructed from a commercial WLED gave an intrinsic modulation frequency of only 0.6 MHz.

This VLC system was further examined in data transmission tests. A random binary signal was encoded with on-off keying (OOK) by the AWG and transmitted at different bit rates. The bit error rate (BER), defined as the number of error bits divided by the total number of transmitted bits, is a measure of the reliability of the system at the specific bit rate. A BER of over $1{\times}10^{-3}$ is regarded unsuitable for wireless communication. The MOL-WLED could transmit the testing signal at a speed of 3.5 Mbps with a nearly negligible BER (Figure 6b), while the commercial one (c-WLED) only worked below 1.2 Mbps. This bit rate difference of ~three times between the MOL-WLED and the c-WLED is similar to their difference in intrinsic modulation frequency.

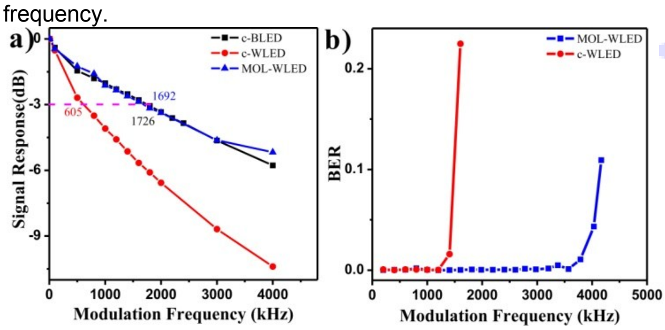

Figure 6. a) The modulation frequency of the VLC system (line and squares for the c-BLED, line and dots for the c-WLED, line and triangles for the MOL-WLED); b) The BER of the data transmission (line and dots for the c-WLED and line and squares for the MOL-WLED).

In summary, we have synthesized a new 2D MOL based on an AIE ligand and Zr_6 cluster. This MOL with thickness down to a bilayer exhibits intense yellow fluorescence and remarkable processibility. A WLED constructed using this new phosphor is at least three times faster than the commercial ones in visible-light communication, thanks to the much shorter fluorescence lifetime of the MOL as compared to lanthanide emitters in commercial WLEDs. The MOL-WLED achieved three times data transmission speed over commercial WLEDs with simple OOK modulation in VLC. This work illustrates the opportunities in designing and assembling new functional molecules into hybrid nanomaterials for advanced information technology.

Experimental Section

Synthesis of 2D Zr-TCBPE-MOL: H_4TCBPE (2 mg 0.0025 mmol) was dissolved in 0.2 mL of DMF, and $ZrCl_4$ (3.5 mg 0.015 mmol) was dissolved in a mixed solvent of 0.3 mL of DMF, 0.1 mL of formic acid and 0.2 mL of H_2O. The two solutions were combined and kept at 120 °C for 48 h to obtain a white suspension. The Zr-TCBPE-MOL solid was obtained through centrifugal separation.

Acknowledgements

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Keywords: metal-organic layers (MOLs); white light emitting diode (WLED); visible-light communication (VLC); metal-organic frameworks (MOFs); aggregation-induced emission (AIE)

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[6] For a given device, the response to oscillating input signals drop in intensity as a function of the frequency of this signal. This physical response frequency is defined as the frequency at which half the intensity of zero frequency is preserved.

[7] In commercial WLEDs, blue light from GaInN-based emitting diodes excites YAG-Ce to give yellow light from the 4f-5d transition of Ce^{3+}. The combination of blue and yellow in a proper ratio looks white to human eyes.

[8] Although short-pass filters have been installed before the receiver to remove the slow yellow light and recover fast data transmission rate,[a] the reduction in light intensity presents a challenge to the signal receiver part, especially considering that commercial Si-based PIN and avalanche photodiode (APD) detectors are usually more sensitive to yellow light. [a]: see ref [S. Wang, F. Chen, L. Liang, S. He, Y. Wang, X. Chen, W. Lu, IEEE. Wirel. Commun. 2015, 22, 61.]

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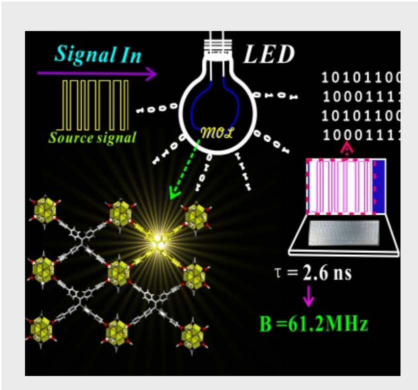
Entry for the Table of Contents (Please choose one layout)

Layout 1:

COMMUNICATION

A new two-dimensional metal-organic layer, constructed from zirconium clusters and fluorescent ligands, is used as a fast phosphor to increase the response rate of white light LED for visible-light communication. The use of molecular phosphors increases the data transfer rate by three times, illustrating the opportunity in using nano-assemblies for advanced information technology.



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