

The Efficiency of Light-Emitting Electrochemical Cells

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We report on the efficiency behavior of light-emitting electrochemical cells (LECs) fabricated from a methyl-substituted ladder-type poly(*p*-phenylene) (mLPPP) that was blended with a crown ether based solid state electrolyte. Unlike organic light-emitting diodes (oLEDs) utilizing mLPPP as an active layer, the LECs suffer from a loss of efficiency at elevated current densities. From scan rate dependent studies we deduce that this efficiency drop is not only due to device decomposition upon high voltage operation and we also reveal the intrinsic mode of LEC operation. The decreasing width of the intrinsic region between the *p*- and *n*-type doped zones upon ongoing *pin*-junction formation causes distinct (either field or electrode induced) luminance quenching effects.

1. Introduction

Within the last century electronic devices have largely improved the standard of living. Like the emergence of classic electronics (which is based on inorganic materials), the emergence of organic electronics, e.g. organic light-emitting diodes (oLEDs),¹ within the last couple of decades has the potential to revolutionize everybody's life once again. The first applications such as a car radio display by Pioneer or an electronic shaver display by Philips have found their way to commercial applications, and others are on the way to replace established electronics by a flexible one. The ease of processing and deposition, the flexibility, and the low weight of these organic components promise new and cost-effective preparation technologies coming along with new and revolutionary device designs.

To further boost organic displays toward commercial applications, the availability of oLEDs that operate with high efficiencies at low driving voltages for all emission colors is indispensable. On the other hand, blue-light emission requires the utilization of wide band gap polymers as emissive layers. A fundamental problem associated with the intrinsic mode of oLED operation with such polymers is the high number of mismatches between their HOMOs and their LUMOs and the work-functions of the metal electrode materials.² Although this disadvantage can be overcome by the choice of appropriate charge transporting layers,^{3,4} the fabrication of such a multilayer setup involves several cost intensive deposition steps.

A very interesting approach for a cost-effective solution of the above-mentioned problem is the light-emitting electrochemi-

cal cell (LEC).^{5–7} LECs benefit from artificially added ionic species that support charge injection at low bias voltages for all emission colors. LECs therefore require only a single layer setup (a blend of the conjugated polymer, an inorganic salt, and an ion transporting component to dissolve the salt) that can be deposited by facile spin-coating or inkjet printing processes on substrates comprising interdigital electrodes (since the electrode spacing of LECs may be as large as several mm⁸), without the need of top metal electrode evaporation. Thus, one can avoid time- and money consuming vacuum chamber production steps.

However, it is not only the appropriate model of LEC operation (*pin*-junction formation^{5,6} vs space charge assisted charge injection^{9,10}) that is still a matter of debate; also an appropriate scheme for the high efficiency operation of LECs is still in doubt. On one hand, for LECs operated in the dynamic junction mode (the system is relaxed and the ionic species have to accumulate at their counter-electrodes upon applying a voltage), the temporal evolution of the device efficiency is comparable slow and the turn-on voltage depends on the scan speed because of the low ionic conductivity. Furthermore, high voltage operation of the LECs implicates complex electrochemical processes that in the end may result in saturation and overdoping effects.^{11–13}

Studies that in particular address the problem of LEC efficiency^{12–16} generally report on an efficiency drop with increasing bias voltages. Suggested explanations for this behavior comprise increased ionic contributions on the overall current because of enhanced ionic conductivity upon self-heating of the devices,¹⁴ formation of microshorts, or the decomposition of the active layer.^{13,15,16} In the following we report on a detailed study to further shed some light on the efficiency behavior of LECs.

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2. Experimental Methods

The LECs were prepared by spin-casting a co-solution of methyl-substituted ladder-type poly(*p*-phenylene) (mLPPP), dicyclohexano18crown6 (DCH18C6), and lithium triflate (LiCF_3SO_3) (ratio 20:20:4 by weight) in cyclohexanone on ITO-coated glass substrates that were thoroughly treated by a wet chemical cleaning process before. Finally, an Al top electrode was deposited in a Balzers MED010 vacuum coating unit. The overall optoelectronic characteristics of LECs of such a composition are already reported.^{17–19} For low scan speeds (almost steady state conditions) the turn-on is basically determined by the single particle energy gap of mLPPP divided by the elementary charge e (2.7 V), both for the forward and the reverse directions. To highlight the differences in the underlying mode of device operation and to prove that the discussed characteristics are actually a consequence of LEC operation, oLEDs with mLPPP as active layer were investigated for comparison. The oLEDs were prepared by spin-casting PEDOT:PSS (Baytron B) on the ITO-coated glass substrates. The PEDOT layers were dried according to the specifications of Bayer Inc. under inert conditions. mLPPP films were spin-cast on top of PEDOT from a chloroform solution (thickness ~ 150 nm). Finally, top metal electrodes (Ca covered by an Al layer) were thermally deposited in the Balzers MED010 vacuum coating unit. In the case of the oLED, the thickness of the active mLPPP layer is approximately 150 nm and therefore largely equals the mean thickness of the active layer of the LEC (for the LEC the active layer has a rather pronounced surface roughness²⁰ so that only a mean thickness can be given). PEDOT and Ca were chosen as electrode materials in order to minimize the barriers for charge injection. In comparison with oLEDs composed of ITO/mLPPP/Al, the turn-on voltage for light emission therefore largely reduces to about 4.5 V (for ITO/mLPPP/Al oLEDs of such a thickness one can expect the turn-on at about 9 V,²¹ while the oLEDs discussed in the following already reach a luminance of about 150 cd/m^2 at that voltage).

The oLEDs and the LECs were finally mounted in a home-made sample holder to allow characterization under inert conditions. Current/voltage (I/V) spectra were recorded with a Keithley 236 source and measure unit, the luminance/voltage (L/V) spectra with a calibrated photodiode that is attached to an integrating sphere.

3. Results and Discussion

Generally, the steady-state I/V characteristics of LECs operated in the dynamic junction mode include three regimes:^{13,22} (1) a background current below the threshold for light-emission that can be related to ionic transport, charging of the cell^{23,24} and barrier lowering for charge injection,²² and (2) an exponential increase of the current after the turn-on of light emission that (3) abates at higher voltages. Upon such high-voltage operation, the optoelectronic characteristics are prone to be affected by saturation effects¹¹ and irreversible device degradation because of complex electrochemical processes including overoxidation of the conjugated polymer¹³ or irreversible reduction of the anionic constituent.^{15,16}

Nevertheless, concepts can be envisaged to overcome such sources for device failure. For example, Yang et al.²⁵ have reported a redox shuttle concept with an additional electroactive species as charge consumer to prohibit the over-oxidation of the conjugated component.

Edman et al.¹⁵ and Kervella et al.¹⁶ concluded that the instability of the anionic species also plays a decisive role for device decomposition at higher voltages. Otherwise, as dem-

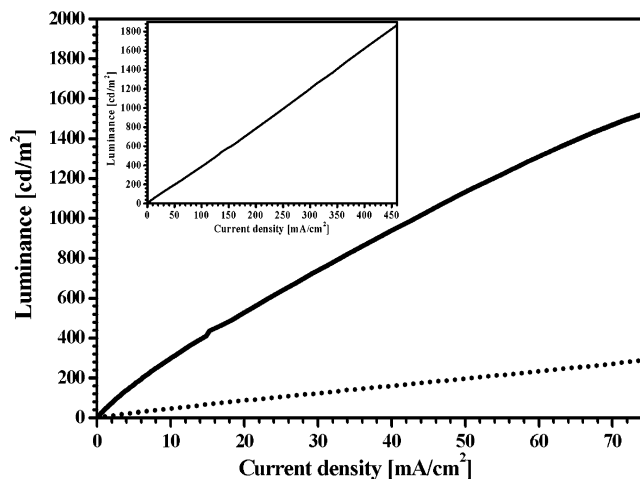


Figure 1. Luminance/current density characteristics of an oLED (ITO/PEDOT/mLPPP/Ca/Al), lower dotted line, and an LEC (mLPPP:DCH18C6:Litriflate, weight ratio 20:20:4), solid line. The inset shows the characteristic of the oLED up to higher current densities.

onstrated recently, decomposition of lithium salt anions can be largely prevented by the addition of activated acidic or neutral Al_2O_3 filler particles.²⁶ The strong hydrogen bond between the OH surface groups of such activated Al_2O_3 particles and lithium salt anions causes a distinct increase of the electrochemical stability upon cell application.

Concepts to facilitate high voltage operation of LECs are therefore conceivable by all means. On the other hand, LEC operation at high voltages is generally characterized by a distinct decrease of efficiency. Therefore, the question arises whether this efficiency drop at elevated bias voltages is primarily caused by the above-mentioned complex electrochemical processes (this means can be overcome by the utilization of appropriate active layers) or if there are additional sources for an efficiency decrease that directly relate to the intrinsic mode of LEC operation (with the efficiency drop as a fundamental consequence thereof).

Figure 1 compares the luminance (L)/current density (j) characteristics of the oLED and the LEC up to a current density of 75 mA/cm^2 . This upper limit is determined by the highest current density the LEC was operated at in order to prevent the ascendancy of a distinct device degradation provoked by the high voltage operation in combination with the long duration of the measurement (in consequence of the very slow increase of the voltage increment to ensure steady-state conditions and (almost) no influence of ionic contributions on the overall current).

As affirmed in Figure 1, the L/j relationship of the oLED is characterized by an almost linear dependence (the inset in Figure 1 shows that for the oLED this behavior remains the same up to notably higher current densities of more than 400 mA/cm^2). Contrary to the oLED, the luminance behavior of the LEC exhibits a clearly sublinear dependence on the current density. This means that, in comparison with the oLED, the luminance of the LEC drops with increasing current densities, giving reason for the efficiency (defined as Cd/A) vs current density plots shown in Figure 2.

However, operating LECs with slow increment up to high voltages always bears the risk of interference by irreversible device degradation. In most cases a linear behavior is reported for the L/j characteristics of oLEDs. Heeger et al. have shown that this is also the case for LECs operated in the frozen junction mode (the *pin* junction is prefixed at elevated temperatures (above the glass transition temperature of the ionically conduct-

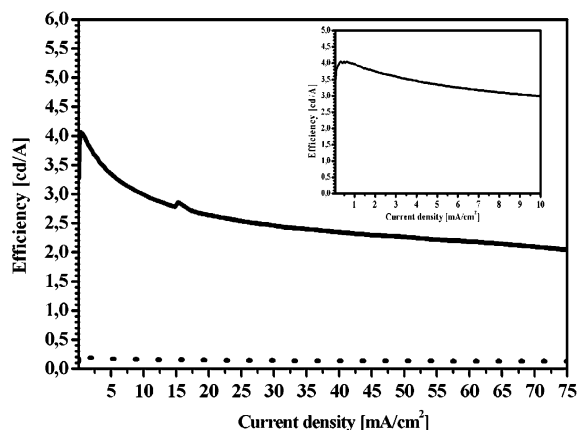


Figure 2. Corresponding efficiency (given in cd/A)/current density plots (see Figure 1) of the oLED (lower dotted line) and the LEC (solid line). The inset shows a magnification of the LEC behavior for the first few mA/cm².

ing component) and the device operated well below the glass transition temperature afterward), while for LECs operated in the dynamic junction mode the characteristic becomes sublinear upon driving voltage increase.¹² This discrepancy was attributed to irreversible device degradation for the latter mode of LEC operation.^{12,13} Care has to be taken regarding such comparisons. In the frozen junction mode the LEC is prebiased at elevated temperatures until the junction has formed and cooled well below the glass transition temperature of the solid state electrolyte subsequently to freeze out ionic motion.

As a consequence thereof, the ion distribution of LECs operated in the frozen junction mode basically accords with the one defined by the prebias voltage for all bias voltages (the corresponding redistribution of the ionic species is hindered because of the immobilization of the ionic species).

Since fundamental device parameters, such as the junction width, strongly depend on the redistribution of the ionic species,²⁷ these two modes of LEC operation are not fully comparable and the frozen junction characteristics may not reflect the intrinsic characteristics of LECs operated in the dynamic junction mode at higher voltages.

We have therefore chosen a different approach to further investigate the luminance drop of LECs operated in the dynamic junction mode by recording scan sweeps^{23,24} from 0 to 6 V and back to 0 V again. To keep device stressing at the heightened bias voltages as low as possible, the voltage increment was increased (decreased) comparably fast (0.1 V/s) so that the optoelectronic characteristics of the LEC are by far no more in steady-state conditions. However, even for this operating condition some additional information on the efficiency behavior can be gained by comparing the j/V characteristics with the L/V characteristics both for the forward and the backward scans, respectively.

In Figure 3 the current density and the luminance are drawn against the number of scan steps for the above-described scan sweep (with ITO wired as a cathode). For the first 60 steps the voltage is increased with an increment of 0.1 V (forward scan); this means scan step 60 corresponds to a voltage of 6 V. At this voltage the sweeping direction is reversed and the bias voltage decreased with the same increment (backward scan), scan step 61 therefore corresponds to 5.9 V, scan step 63 to 5.7 V until after scan step 120 0 V is reached again. Due to the non steady state conditions of the experiment, for the forward scan the device turns on at a voltage of about 3.3 V, which is about 0.6 V higher than the turn-on voltage for steady-state

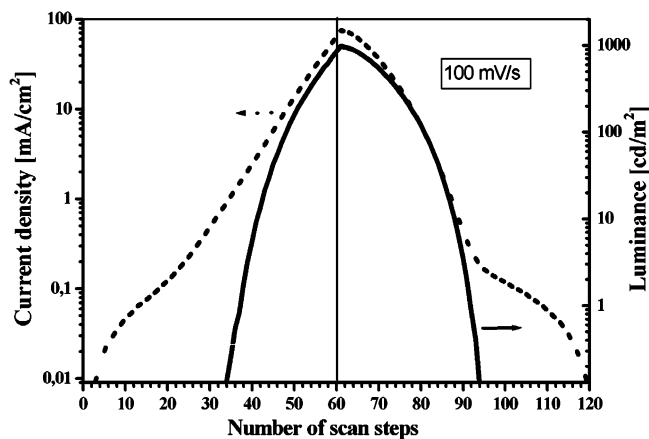


Figure 3. Semilogarithmic representation of the current density (dashed line) and luminance (solid line) vs number of scan steps characteristics of the LEC. The device was operated with a voltage increment of 0.1 V from 0 to 6 to 0 V. The vertical line indicates the turning point of the scan sweep.

conditions (about 2.7 V, corresponding to a band gap of about 2.7 eV for mLPPP) as a consequence of the delay in junction formation because of the low ionic mobility. Upon successive voltage increase, the junction largely forms so that for the backward scan the corresponding current density and luminance curves are asymmetrically shifted to higher scan step numbers (lower voltages). For the backward scan luminance can be detected up to about 93 scan steps (corresponding to a voltage of about 2.7 V). The current density shows an exponential increase up to the turning voltage of 6 V. The interplay of the specific conditions for the bias voltage increase with the operation at rather high voltages (up to 6 V) gradually arouses junction formation so that for the backward scan besides the exponential regime also the sub-exponential regime can be observed.

Contrary to the current density, the luminance behavior does not show such pronounced differences concerning the forward and backward scan. Luminance drops at higher voltages of the forward with a similar behavior in the backward scan (there is only a slight asymmetry due to the shift to lower voltages because of a more distinct junction formation). This means that, although the current density keeps an exponential increase for the whole forward scan, it is the luminance that already drops at the higher bias voltages and thereby reveals the presence of a mechanism different from complex electrochemical processes responsible for the efficiency drop of LECs.

The j/V and L/V characteristics of Figure 3 give reason for the efficiency (defined as cd/A)/voltage (number of scan steps) characteristic as shown in Figure 4. The efficiency curve contains two maxima provoked by a distinct drop of efficiency at the highest voltages applied. This new increase of efficiency upon bias voltage lowering in the course of the backward scan largely argues that the efficiency drop at the highest driving voltages is not simply caused by irreversible degradation but reveals an intrinsic property of LEC operation.

In comparison with the backward scan, for the forward scan the efficiency maximum is not that pronounced and is slightly shifted to higher voltages. This can be primarily attributed to the uncompleted junction formation in the forward direction because of the non steady state conditions of the experiment (in this case the electronic current is still comparably low and the ionic fraction therefore forms a large part of the overall current but does not contribute to luminance generation). The slight shift of the efficiency minimum (observed at 63 scan steps

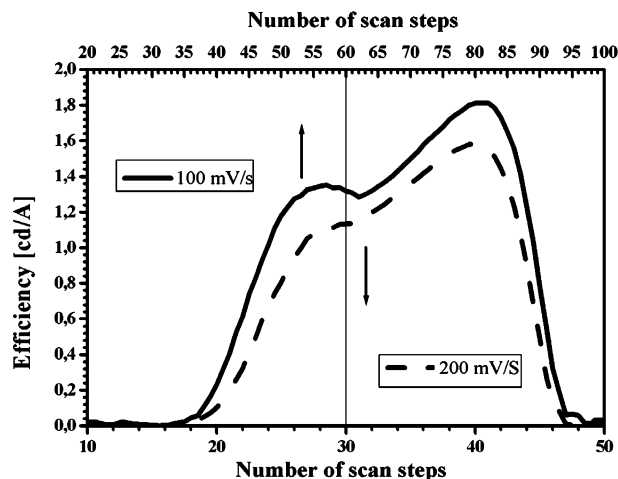


Figure 4. Efficiency (cd/A) vs number of scan steps characteristic of the LEC operated with a scan speed of 0.1 V/s from 0 to 6 to 0 V, solid line. For comparison, the characteristic of the same LEC operated with a scan speed of 0.2 V/s from 0 to 6 to 0 V is also shown. The vertical line indicates the turning point of the scan sweep.

corresponding to 5.7 V of the backward scan) from the turning point (60 scan steps) can be explained by the fact that even at these high voltages the particular ionic redistribution (magnitude of junction formation) still slightly lags behind the corresponding voltage value because of the non steady state conditions.

To further illustrate the scan speed dependence, the same characteristic is also shown for a scan sweep recorded with an increased scan speed of 0.2 V/s. Although the overall efficiency is lower and the turn-on occurs at even higher voltages (number of scan steps) (because of fortified non steady state conditions), the overall shape of the efficiency/voltage characteristic remains the same. A similar behavior was also observed for other scan speeds investigated: the overall device efficiency increases the lower the voltage increment but the characteristics of the efficiency/voltage dependence as shown in Figure 4 largely remain the same.

From Figure 4 one could deduce that the efficiency maximum of LECs emerges at bias voltages clearly higher than the turn-on voltage. However, care has to be taken regarding such a direct interpretation of the data since with receding bias voltage the electronic contribution to the overall current also largely decreases while the ionic portion comparatively increases and thereby feigns a drop of efficiency (since the ionic portion does not contribute to luminance generation) for bias voltages close to the turn-on voltage. Contrarily, one can envisage that for steady state conditions (no more ionic contribution) the maximum of the efficiency shifts to lower voltages, in full accordance with the efficiency plot recorded for the LEC under steady state conditions (see Figure 2).

From the inset of Figure 2 one can evaluate that the maximum efficiency emerges well below 1 mA/cm². In fact, compared with Figure 2, the exact value may be even shifted to petty lower values of the current density since we cannot fully exclude that, despite the decelerated conditions of device operation, marginal contributions of an ionic portion on the overall current are still present because of the basically low ionic conductivity.

Current densities of about 0.3–0.5 mA/cm² (efficiency maximum for the steady state condition) are characteristic values that we observe coincidentally with the turn-on of the devices. This infers that the efficiency of LECs is the highest in the range of the turn-on voltage but already starts to drop upon marginal increase of the driving voltage. This observation also renders some other interpretations of the fundamental mechanism for

the efficiency loss less likely. Heeger et al. suggested that at high current densities self-heating of the devices increases ionic conductivity and thereby the ionic portion on the overall current.¹⁴ However, as shown here, efficiency starts to drop even at low current densities, and it is not very likely that self-heating causes temperatures exceeding 56 °C (the melting temperature of the DCH18C6/LiTf complex²⁸) already at these low current densities. Also, the formation of microshorts²⁹ that can be envisaged at higher voltages (small junction width) is not that likely to occur at these early stages of junction formation when the intrinsic layer still forms an appreciable part of the electrodes spacing. In fact, the commencement of a maximum efficiency with the turn-on of the device and the decrease of device efficiency with ongoing *pin*-junction formation (the widths of the p-type and n-type doped zones increase while that of the intrinsic region between decreases) upon voltage increase lets one construe a more coherent explanation based on the intrinsic mode of LEC operation itself (model of electrochemical doping).

For driving voltages below junction formation, the ionic species accumulate at their counter-electrodes and thereby screen the internal field. Once the threshold voltage is achieved, electrochemical doping starts and gives reason for the formation of Ohmic contacts. We note that a maximum efficiency of about 4cd/A even slightly exceeds the highest values reported by Bliznyuk³⁰ performing electrode engineering on mLPPP-based oLEDs to enhance device efficiency. Since balanced charge injection is one of the base requirements to achieve highly efficient devices, such behavior upon the in-situ formation of Ohmic contacts is not that amazing.³¹

Upon successive bias voltage increase, the additional potential largely drops across the junction region (as directly evidenced by electroabsorption measurements^{32,33}) with a subsequent decrease of the junction width aroused by the redistribution of the ionic species. Experimental studies assisted by theoretical calculations²⁷ have shown that the junction width may be as thin as about 15 nm (for a 150 nm thick device) for a current density of 0.1 A/cm². Extrapolating the data to higher voltages, a high potential drop across a very thin junction gives reason for a very high field strength that may reach several MV/cm. This indeed can cause pronounced loss of device efficiency since such high electric fields are prone to abate light emission upon field-assisted quenching of the luminance. For mLPPP, detailed studies have revealed almost no effect of the electric field strength on the photoluminescence intensity up to about 1 MV/cm (field strengths up to which the oLEDs could be operated within this study before breakdown because of the high current densities).

However, further increase of the electric field strength has a drastic impact on the luminance behavior: A decrease of the photoluminescence intensity by about 50% for fields reaching only 2 MV/cm^{34,35} was observed. Such high field strengths are conceivable in LECs upon high voltage operation by all means because of the consecutive decrease of the junction width. Additionally, such a distinct variation of the field strength may also considerably influence the individual field dependences of the positive and negative charge carrier mobilities and thereby their recombination kinetics.³⁶

Despite such field-induced effects, a junction width (non doped region where recombination takes place) of only about several tens of nanometers will additionally affect the luminance behavior by electrode-induced effects, a phenomenon also known for oLEDs with rather thin active layers.³⁷ Taking an exciton diffusion length in mLPPP of about 15 nm,³⁸ luminance quenching because of exciton diffusion toward the nearby p-

and n-doped zones, which act as distinct quenching centers,³⁹ will be an additional source for an efficiency decrease.

4. Conclusion

We have shown that, despite complex electrochemical processes (which in the end may lead to device degradation), high-voltage operation of light-emitting electrochemical cells excites additional sources for an efficiency decrease which can be related to the intrinsic mode of LEC operation: the formation of the *pin* junction and the decrease of the width of the intrinsic region upon successive driving voltage increase cause the presence of large electrical fields across the junction width. On one hand, the results presented here further support the model of electrochemical *pin*-junction formation in LECs. On the other hand, they also point out some limitations regarding the high voltage operation of LECs: even if it would be possible to prevent complex electrochemical processes, high-voltage operation of LECs is not favorable because of the related luminance quenching effects that give reason for a distinct decrease of device efficiency at elevated driving voltages.

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