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Wireless Electronic Light Emission: An Introduction to Bipolar Electrochemistry

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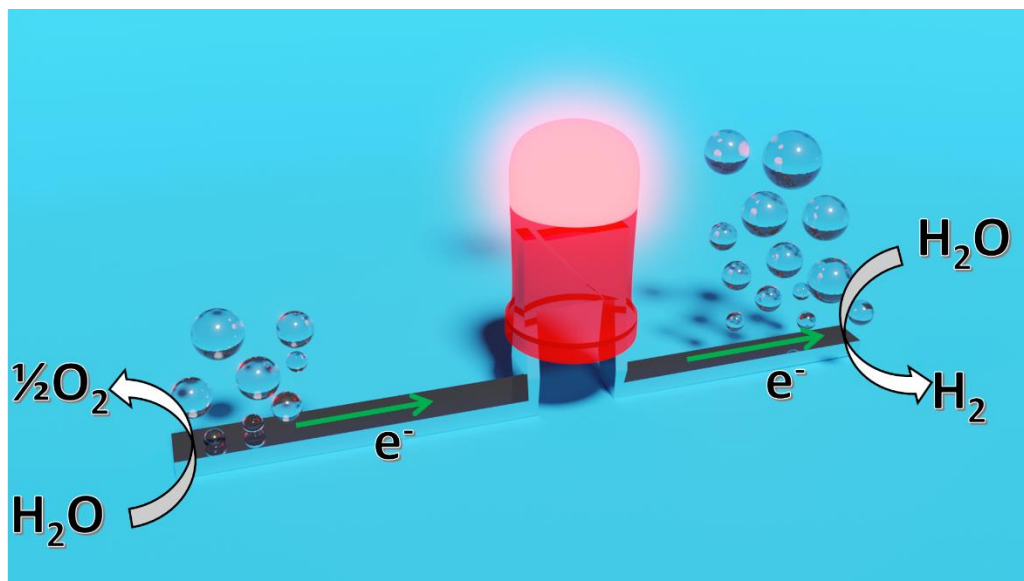
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ABSTRACT

Electrochemistry is taught in most undergraduate chemistry programs. Although this topic is important for students due to its broad interest in industry (energy, diagnostics, car industry, etc.), they often find it difficult, because it is based on a combination of various physical concepts such as electric fields, interfacial processes or charge and mass transports. Among electrochemical concepts, bipolar electrochemistry is of special interest and might be easier to teach due to a very simple general set-up. In this case, an oxidation and a reduction reactions occur at the two ends of a single conductive object exposed to an electric field in solution. Such an object is therefore called a bipolar electrode. The nature of the electrochemical reactions and their amplitude can be tuned by playing with the electric field. The fundamental concepts of bipolar electrochemistry are introduced here with a series of basic experiments designed to be carried out in a standard teaching laboratory. These simple and affordable experiments illustrate the key-parameters driving electrochemical reactions at a bipolar electrode. Their influence can be readily visualized using a cheap, commercially available light emitting diode (LED) acting as the bipolar electrode, which illuminates when the current generated by the electrochemical reactions flows through it. The concept of bipolar electrochemistry with eye-catching experiments enables a good introduction to general

electrochemistry. It points out the importance of fundamental aspects such as the electric field, or the necessity of an electrolyte and a counter-reaction.

GRAPHICAL ABSTRACT



KEYWORDS

Second-Year Undergraduate, Demonstrations, Hands-On Learning / Manipulatives, Bipolar Electrochemistry.

INTRODUCTION

Electrochemistry is at the center of many research areas. For example, to develop the use of renewable energy, or to study energy storage devices, knowledge of electrochemistry is required. Unfortunately, teaching electrochemistry to undergraduate students can be difficult. Therefore, original ways of teaching electrochemistry are crucial from a pedagogic point of view, and several unusual approaches are already available in the literature.¹⁻⁵ We propose in this contribution a complementary playful way of teaching some important notions of electrochemistry, using experiments based on so-called bipolar electrochemistry (BPE). BPE differs from conventional electrochemistry in terms of both, concept and applications.⁶⁻⁷ It is a wireless process in which asymmetric electrochemical reactions occur simultaneously at the two extremities of a single conductive object called a “bipolar electrode”.⁷⁻¹⁰ These reactions are remotely controlled by varying, across an electrolytic solution, the electric field to which the bipolar electrode is exposed. In the absence of any physical contact with an external electrical circuit, it is possible to trigger the desired reactions simultaneously at

several bipolar electrodes (from small arrays up to large-scale ensembles of thousands of electrodes).¹¹⁻¹² Not only macro- but also micro- and nanoscale electrodes can be used.¹³⁻¹⁵ This is especially interesting for materials modification, with the particularity that it leads to an asymmetric modification of the objects.¹⁶ The resulting modified materials are called “Janus objects”.^{9, 11-12, 17} BPE can also be used for the selective and sensitive electrochemical detection of targeted analytes,¹⁸⁻¹⁹ by combining the recognition reaction at one side of the bipolar electrode with a detectable and quantifiable reaction at the other side. The second reaction can be for example an electrochemiluminescence reaction,^{13-14, 20-22} a fluorogenic electrochemical reaction,²³⁻²⁷ an electrodisolution process,²⁸ or even the actuation of a polymer.²⁹ Another interesting application of BPE is the possibility to induce directed motion of bipolar objects, for example thanks to reactions that produce bubbles at one side of the bipolar electrode. Macro- and micro-propellers can therefore be designed and/or actuated.^{21, 30-31}

This illustrates that BPE is of broad interest for students due to the numerous applications listed above, but also from a purely conceptual point of view. Several fundamental aspects are indeed addressed and experimentally illustrated like the electric field, the electrochemical potential, the ohmic drop, the influence of the supporting electrolyte, and the electromotive force of a given reaction. With BPE, students will become familiar with several aspects that are often ignored, or at least underestimated in conventional electrochemistry, like the need for an opposite reaction at the counter electrode or the necessity for a minimal energy (or minimal voltage input) to trigger electrochemical reactions.

The experiments described here are specifically designed to introduce students to the concept of BPE. This particular kind of set-ups has been chosen for their visual aspect, which allows students to acquire a better understanding of electrochemistry in general, and in particular of BPE. It will help them to retain more easily the various electrochemical concepts. Herein, the occurrence of the bipolar electrochemical reactions is evidenced by the switching on of a simple light-emitting diode (LED),³² demonstrating that electrons are circulating across the diode as a result of the two coupled interfacial electron transfer reactions occurring simultaneously at both poles.³³ The electrical current, which turns on the LED is the result of an oxidation reaction releasing electrons at the anodic pole, and of a simultaneous

reduction reaction consuming electrons at the cathodic pole.^{32, 34-35} Students will also experience in this way that in the absence of specific electroactive species, reactions that involve solvent decomposition such as oxygen or hydrogen evolution can readily take place.^{9,}

21, 32

In the following experiments, students will get a deeper insight into the fundamental principle of BPE, and explore the key-parameters influencing the amplitude of the electrochemical reactions occurring at the poles of the bipolar electrode: the minimal required voltage, the influence of the size and orientation of the bipolar object with respect to the electric field lines. Finally, they will study the influence of a parameter specific to LEDs and linked to their intrinsic color, namely the diode's threshold voltage.

Principle of Bipolar Electrochemistry

Conventional electrochemical measurements rely on a three-electrode set-up. The reactions of interest occur at a first electrode, called “working electrode”, whose potential is controlled with respect to the known and constant potential of a “reference electrode”. A third electrode at which the opposite redox reaction occurs (*i.e.* an oxidation, if a reduction occurs at the working electrode, and *vice-versa*) is used to close the electrical circuit and to avoid current flow through the reference electrode.³⁶ On the contrary, the principle of BPE, which is quite different, is illustrated in figure 1A.

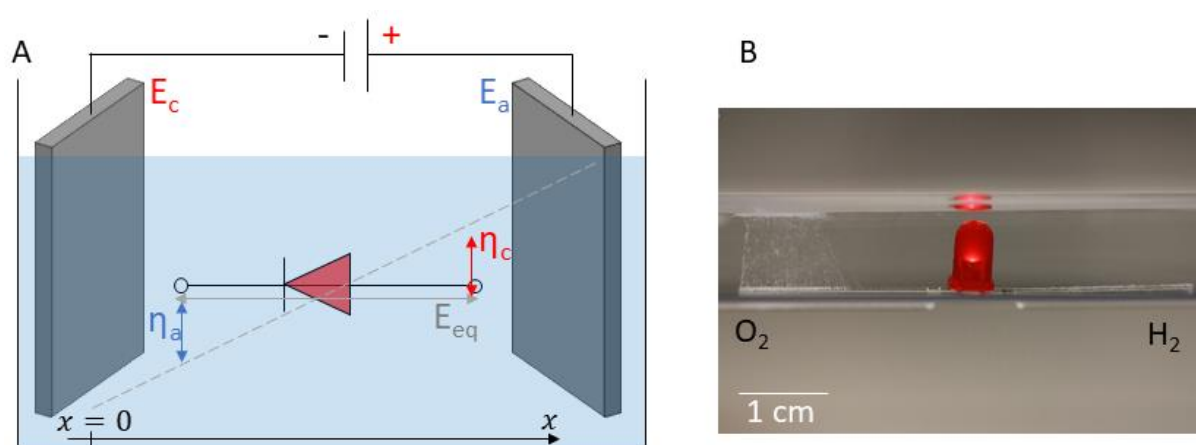


Figure 1: Principle of bipolar electrochemistry (BPE). A) Scheme of the BPE set-up with a diode used as bipolar electrode. E_a and E_c are the anodic and cathodic potentials of the feeder anode and cathode, respectively, while η_a and η_c are the voltage biases at the anodic and cathodic poles of the bipolar electrode, respectively. B) Photograph of the behavior of a diode as a bipolar electrode submitted to an appropriate electric field. The diode is switched on without electrical contact with the external

circuit. O₂ and H₂ bubbles are produced at the extremities of the diode by H₂O oxidation (left side) and reduction (right side), respectively.

In BPE, the reaction of interest and the opposite reaction take place at the two extremities of the same electrode, which is therefore called a “bipolar electrode” (a LED in our case).⁷⁻¹⁰ In this configuration, the bipolar electrode is immersed in an electrolyte between two electrodes called “feeder” or “driving” electrodes (displayed as two grey rectangles in Figure 1A), which are used to apply a voltage and therefore generate an electric field in the electrolytic solution. The bipolar electrode is at an equilibrium potential that depends on the solution composition, and the value of this potential is not controlled. However, the potential drop in the solution is controlled by the potential difference applied between the two feeder electrodes. It results in a potential gradient established along the bipolar electrode. This gradient drives the electrochemical reactions on the bipolar electrode. The bipolar electrode can be any conductive object (even semi-conductors have been used, however their behavior is slightly different). As such, it remains equipotential (at the equilibrium potential E_{eq}) when placed in an electric field, while the electrical potential of the surrounding electrolyte varies linearly with the distance x to the feeder electrode according to (Eq. 1):

$$E(x) = E(0) + \|\vec{E}\| \times x = E(0) + \frac{\Delta E}{d} \times x \quad (1)$$

where d is the distance between the two feeder electrodes, and ΔE is the difference between the potentials of the anodic and cathodic feeder electrodes. Therefore, voltage biases versus the solution appear at the extremities of the bipolar electrode. A negative bias η_c occurs at the extremity facing the feeder anode with higher electric potential, while a positive bias η_a appears at the opposite extremity, which faces the feeder cathode with lower electric potential (Figure 1). This difference in polarization potential $\Delta\eta$ is the electromotive force of the electrochemical reactions occurring at the bipolar electrode. The voltage depends on the electric field $\|\vec{E}\|$ and on the lateral dimension l of the bipolar electrode, and is given by the equation below (Eq. 2).

$$\Delta\eta = \|\vec{E}\| \times l = \frac{\Delta E}{d} \times l \quad (2)$$

with l being the length of the bipolar electrode along the electric field lines. In conventional electrochemistry, an oxidation (or a reduction) theoretically occurs at the working electrode as soon as the electrode potential exceeds (or goes below) the thermodynamic Nernstian potential E of the involved redox couple. In BPE, opposite electrochemical reactions theoretically occur at the poles as soon as $\Delta\eta$ becomes sufficiently high, that means at least equal to or higher than the difference between the thermodynamic Nernstian potentials E_1 and E_2 of the two involved redox couples (Eq. 3).

$$\Delta\eta \geq E_1 - E_2 \quad (3)$$

Of course, overpotentials due to kinetic limitations also have to be considered here, like in conventional electrochemistry. Therefore, the voltage required to start the reactions can be corrected by a term $\eta_{kinetics}$ to consider the electron transfer kinetics at the two poles (Eq. 4).

$$\Delta\eta = E_1 - E_2 + \eta_{kinetics} \quad (4)$$

An oxidation takes place at the positive pole while a reduction occurs at the negative pole. Both reactions develop the same current intensity, and the reduction current is equal to the oxidation current (in absolute values). This results in a flux of electrons across the bipolar electrode, which in our case is evidenced by the brightness of the light emitted by the LED, that is in a first order approximation correlated with the current flow (Figure 1B).

In the absence of specific electroactive species in the electrolyte, the oxidation-reduction reactions at the poles involve solvent decomposition. In the case of an aqueous electrolyte, water oxidation to O_2 occurs at the anodic pole of the bipolar electrode, while water reduction into H_2 occurs at the cathodic pole. This is clearly evidenced by the formation of bubbles, which can be easily distinguished in Figure 1B. Interestingly, the difference of stoichiometry between the two reactions is revealed by the larger amount of bubbles produced in the case of H_2 compared to O_2 .

EQUIPMENT AND CHEMICALS

Bipolar electrochemistry experiments:

A voltage generator is used to establish the electric field in the electrolyte solution. The feeder electrodes can be any conductive objects of a sufficient size (like the counter electrode must have a sufficient size not to limit the electrochemical reactions in conventional electrochemistry, the current flowing across the electrochemical cell must not be limited by the size of these feeder electrodes). In our experiments, we use two rectangular graphite sheets of approximately $1 \times 2 \text{ cm}^2$. The electrical connection is ensured by electrical wires connected to the graphite sheets with copper tape. The electrolyte solution is an aqueous solution containing a dissolved inert salt, like in conventional electrochemistry. However, it is noteworthy that the electrolyte must not be too concentrated so that the current preferentially flows through the bipolar electrode, and to ensure a sufficient potential drop in the solution to be able to trigger the electrochemical reactions. For that, a 5 mM sodium chloride solution was selected. When

the LED is used as a bipolar electrode, the two connection tips are folded so that they point into opposite directions in order to form a rather long bipolar electrode (typically several cm).

We would like to point out that no specific material is actually necessary and that these experiments can be performed with everyday items, even in primary school classes. For instance, instead of a voltage generator, the voltage can also be applied by using two 9 V-batteries in series. Similarly, the feeder electrodes can be any type of conducting object: in a recent tutorial demonstration, for example, a knife and a fork made out of steel were used ([tutorial](#)).

Characterization of the light-emitting diode

The LEDs were characterized in two different ways. First, the current-potential behavior of the diodes was determined. For that, we used a potentiostat (CHI 900A) in Linear Sweep Voltammetry (LSV) mode, and connected one side of the diode to the “Working electrode” outlet, and the other side to both the “Reference electrode” and “Counter electrode” outlets. In this way, the current flowing through the diode as a function of the applied voltage between the two extremities of the diode can be recorded. The voltage was swept linearly between 0 and 2 V vs. Ag/AgCl at a constant scan rate of 100 mV s⁻¹.

Second, the electrochemical behavior of the material constituting the diode wire was characterized. The wire of one side of the diode was cut and used directly as a working electrode in a conventional three-electrode set-up. The counter electrode was a platinum wire and the reference electrode an Ag/AgCl reference electrode. The electrolyte was 100 mM NaCl. The potentiostat was used again in LSV mode and the potential was varied linearly between -1.8 and 1.2 V vs. Ag/AgCl at a constant scan rate of 100 mV s⁻¹.

HAZARDS

No particular chemical hazard has to be mentioned since only sodium chloride solutions are used. Caution has to be taken when manipulating a voltage generator, to avoid an electric-shock hazard. The maximum current measured was 40 mA.

RESULTS AND DISCUSSION

1- Diode characterization

A LED was chosen as bipolar electrode, because it provides a visual evidence of current flowing through the electrode and therefore of the coupling of the electrochemical reactions occurring at its two poles.^{32, 34-35, 37} We will not go here into the technical details of the principle of a LED, but just explain some basic points that will be useful in the following sections. A

LED is a polarized dipole very similar to a PN-junction diode, *i.e.* it relies on the contact between a P and a N semi-conductor. Current flows through the diode only if the voltage is applied in the convenient direction, allowing electrons from the N-doped part to combine with holes in the P-doped part. A minimum voltage is required to allow current flow through the diode. This “threshold voltage” depends on the materials used in the junction, and is therefore linked to the LED color. To better understand this behavior, we performed some basic characterization experiments (*i.e.* conventional electrochemistry measurements) of the diodes, before using them as bipolar electrodes. First, the current-voltage behavior of three LEDs of different colors (Figure 2A) was recorded. The LSV mode of a potentiostat was used and one side of the LED was connected to the “working electrode” connection, while the other side was connected to both the “reference electrode” and “counter electrode” connections. In this way, it was possible to vary linearly the voltage applied between the two extremities of the diode and to measure the resulting current flowing across the diode.

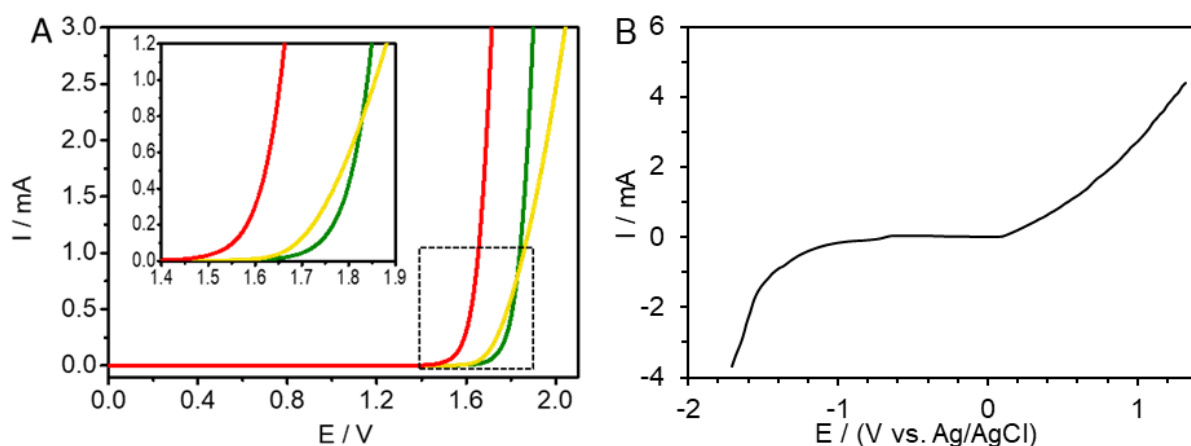
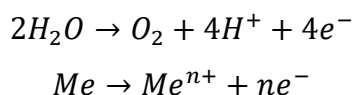


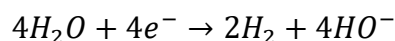
Figure 2: Characterization of the LEDs. A) Current-voltage behavior for three different diode colors: red, green, and yellow, respectively. The inset is a magnification of the zone indicated by a dashed frame. B) LSV curve with a diode tip used as working electrode, to characterize the kinetics of electrochemical reactions at this interface.

As one can easily see in Figure 2A, and consistently with what is explained above, the current is null until the threshold voltage is reached. Starting from this voltage and beyond, current flows through the LED, which then switches on. The LED does not behave like a linear resistance and current increases exponentially with increasing voltage. The threshold voltage is approximately 1.4 V for the red LED, and approximately 1.6 V for both the green and the yellow LEDs. It is noteworthy that diodes also have a maximum voltage they can withstand before they break down. It is usually around 3 V (we do not show it here) and is referred to as “Peak reverse voltage” or “Peak inverse voltage” in the diode specifications. Similarly, LEDs must not be crossed by a too high current to avoid irreversible damages.

The diode wire was also used as a working electrode in a conventional electrochemistry set-up to characterize the kinetics of the electrochemical reactions necessary to allow current flowing through the diode in BPE (Figure 2B). Indeed, the threshold voltage, which is needed to promote the reactions and allow current flowing through the diode, is the sum of the thermodynamic voltage and the overpotentials due to kinetic limitations, as shown in Eq. 4. Thus, the difference between the potentials at which the oxidation and reduction reactions actually start at a given electrode material will give the real (i.e. experimental) required voltage. On the linear sweep voltammogram, an oxidation process starts around 0.2 V *vs.* Ag/AgCl and increases with increasing potentials. This potential is lower than the thermodynamic potential of H₂O oxidation, which means that another oxidation reaction takes place. In the absence of any diffusing electroactive specie, the only possibility is the oxidation of the LED wire. The latter is a metallic alloy of unknown composition (and may vary depending on the supplier). When going to higher potentials, a second process corresponding to H₂O oxidation can be observed. Although these two processes cannot be clearly resolved on the I-E curve, they can be easily visualized on the electrode during the experiment. First, the metal color changes, indicating the oxidation of the electrode material, then bubbles are released, which reveals water oxidation to form O₂. The equations of the anodic reactions can be written as (Me is the metallic alloy and n the number of electrons released):



In the cathodic part of the LSV curve, H₂O reduction starts around -1.0 V *vs.* Ag/AgCl and increases with decreasing potential. The onset potential of H₂O reduction is characteristic of a slow reaction kinetics at the electrode. The corresponding reaction equation is:



The required voltage difference for driving the two redox reactions simultaneously is therefore approximately 1.2 V, *i.e.* it is smaller than the threshold voltage for all LEDs. We can therefore expect that the reactions will start at the two extremities of the LED when the applied voltage $\Delta\eta$ exceeds the threshold voltage of each LED.

2- Bipolar electrochemistry experiments.

2a- Different LED colors

A unique advantage of BPE is the possibility to trigger electrochemical reactions simultaneously at several bipolar electrodes using a single pair of driving electrodes. This is easily shown, for example, in this experiment and the following one, in which several LEDs are switched on simultaneously and the gas bubbles on each of them can be observed (Figures 3 and 4).

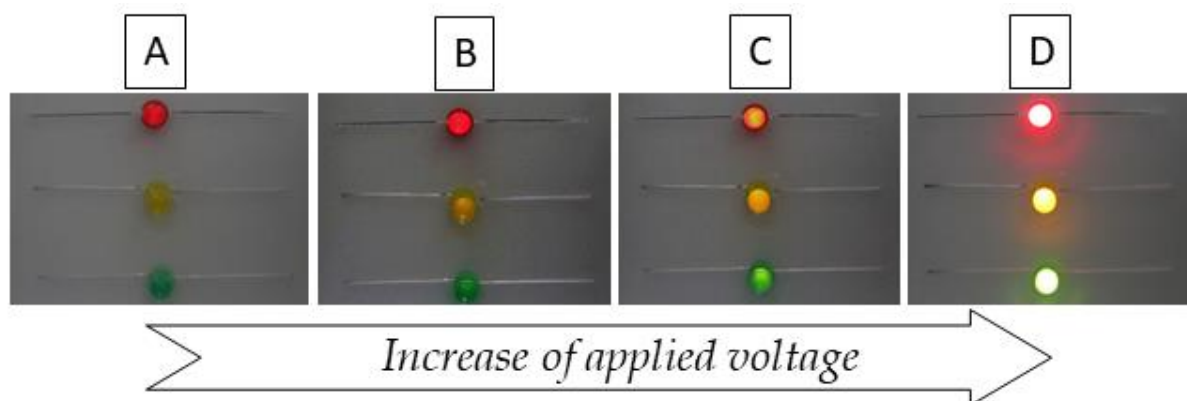


Figure 3: Influence of an increasing voltage on LED lighting and brightness for the same diode length but different colors. The applied voltage is 18 V (A), 19 V (B), 20 V (C) and 24 V (D), respectively. Electrolyte solution: 5 mM NaCl. Distance between the feeder electrodes = 10 cm. The length of these LED is 5.2 cm.

As underlined above, the element doping the semi-conductor that constitutes the LED determines both its color and its threshold voltage. Here, this feature is used to selectively trigger the reaction and thus selectively light-up the different diodes by varying the applied voltage. This is demonstrated with the three LEDs having different colors and which have already been characterized above. While applying an increasing voltage, we first address the red LED with the lowest threshold voltage, then the yellow and green LEDs with a slightly higher threshold voltage (Figure 3). Quite interesting is also the intensity of the emitted light, which directly reflects the current intensity, *i.e.* the electron flux, which is itself a direct measure of the rate of the electrochemical reactions at the two poles of the bipolar electrode. All parameters except the voltage are kept constant. The feeder electrodes are separated by 10 cm, the electrolyte concentration is fixed at 5 mM and the total diode length is 5.2 cm. Diodes are positioned parallel to the electric field lines (*i.e.* perpendicular to the feeder electrodes). When 18 V are applied between the two feeder electrodes, which corresponds to an electric field of 1.8 V cm^{-1} , only the red LED is slightly illuminated while the yellow and green LEDs remain switched off. Considering that the total diode length is 5.2 cm, equation 2 allows calculating that the voltage between the extremities of the diode is 9.4 V. Reactions should therefore occur at least at the two extremities. However, a minimum current intensity is also necessary to switch on the diode (usually $\sim 1\text{-}2 \text{ mA}$). In our case, such an intensity is not obtained at lower voltages.

Even if the voltage should be sufficient, the number of recombined charges is too weak and the light intensity is too low to be observed with the naked eye. The use of a photomultiplier tube can be envisioned to detect photons emitted at lower voltages. The applied voltage, and thus the resulting electric field is then increased. At 19 V, the light intensity of the red LED slightly increases and light can now also be seen at the yellow and green LEDs. The three LEDs are clearly switched on at 20 V, and the light intensity becomes very bright at 24 V.

2b- Influence of the bipolar electrode length

For a constant electric field, the voltage between the two poles of a given bipolar electrode depends linearly on the length of the object (Eq. 2). Controlling the object's length is easy with diodes since the tips that determine the size of the object can be cut at will. Figure 4 presents a visual evidence of the influence of the bipolar electrode length. Three red LEDs are cut at different lengths ($l = 2.2, 3.4$ and 5.2 cm, respectively) and dipped in the electrolyte between the feeder electrodes. The longer the diode, the higher the voltage drop between its two poles; and therefore the higher the current flow, as evidenced by the increasing intensity of emitted light. The results shown are obtained for three different applied voltages between the feeder electrodes. At 20 V, which corresponds to an electric field of 2 V cm^{-1} , only the longer LED is switched on. At 25 V, the light becomes brighter at the longer diode, while the LED of intermediate length is slightly illuminated, and the shorter LED remains switched off. Finally, at 32 V, all three LEDs are switched on, which shows that electrochemical reactions are occurring at the two poles of all three bipolar electrodes.

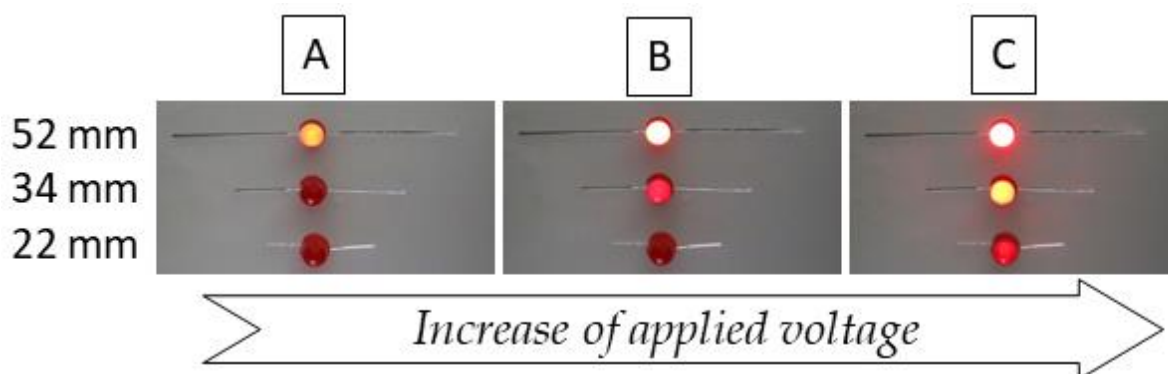


Figure 4 : Influence of different voltages on the light intensity for the same diode color but different effective lengths. The applied voltage is 20 V (A), 25 V (B) and 32 V (C), respectively. Electrolyte solution: 5 mM NaCl. Distance between feeder electrodes = 10 cm.

2c- Angle between the bipolar electrode and the electric field lines

This experiment deals with a complementary aspect of the previous one, demonstrating the importance of the alignment of the bipolar electrode with the direction of the electric field. A given bipolar electrode, which corresponds to the longest LED used in the previous experiment (i.e. $l = 5.2$ cm), is placed in a constant electric field. Only the angle between the LED's main axis and the electric field direction is varied. The electric field is orthogonal to the feeder electrodes. In a first experiment, like in all experiments presented above, the LED is positioned parallel to the electric field. Under these conditions, the brightest intensity is observed. However, it must be noted that due to the intrinsic directionality of the diode, placing it in the opposite direction would not switch it on for whatever voltage below the breakdown voltage (data not shown). The light intensity decreases with an increasing angle and is null when the LED's main axis is perpendicular to the electric field (Figure 5).

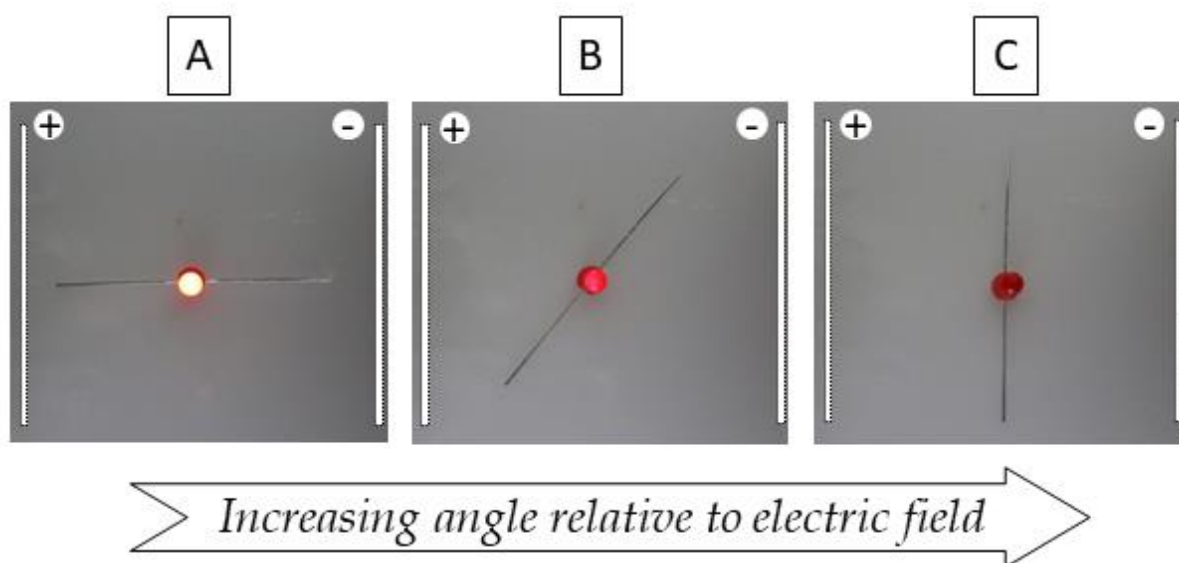
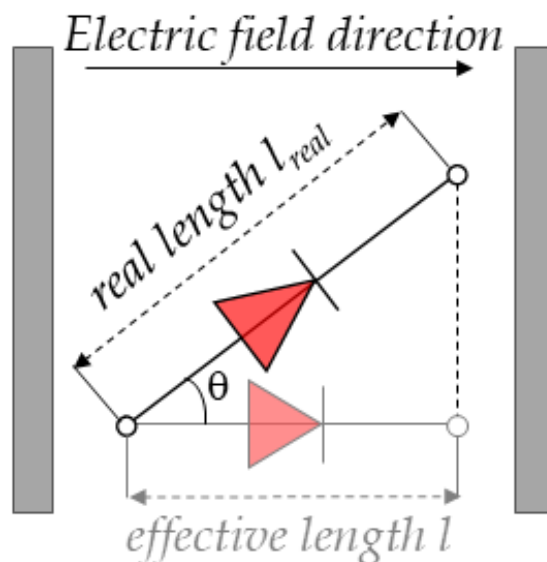


Figure 5: Influence of the angle between the diode and the electric field lines for a given diode color (red) and a constant voltage of 25 V. The angle is: 0° (A), 45° (B) and 90° (C), respectively. The position of the feeder electrodes is schematically indicated by the white rectangles at the borders of the images. Electrolyte solution: 5 mM NaCl. Distance between feeder electrodes = 10 cm (this distance is not drawn at scale).

The explanation is quite simple: the effective length l of the bipolar object is not its actual absolute length l_{real} , but its projection in the direction of the electric field, which is given by Eq. 5 (Scheme 1):

$$l = l_{real} \times \cos \theta \quad (5)$$

where θ is the angle between the bipolar electrode and the electric field. With this equation, it is obvious that the effective length is zero when the electrode is perpendicular to the electric field (i.e. $\theta = 90^\circ$), and therefore the polarization potential difference between the two electrode extremities is also zero. This is clearly evidenced in our case by a complete switch-off of the LED.



Scheme 1: Effective length of the LED in the direction of the electric field. The LED is orientated with an angle θ with respect to the electric field direction. The grey rectangles are the feeder electrodes (top view).

CONCLUSION

In conclusion, the concept of BPE is illustrated here in a very visual way and some simple experiments to underline the main parameters influencing the involved phenomena are described. A LED has been chosen as a bipolar electrode, which switches on, without any physical contact to a power supply, due to the bipolar electrochemical reactions triggered by the electric field that is applied across the electrolyte solution. The electrolyte does not need to contain any specific redox probes; the involved reactions being essentially water oxidation and reduction. We believe that this straight-forward visualization of the main parameters governing BPE is interesting for both students and teachers for several reasons. From the teacher's viewpoint, an important aspect is the low cost of these experiments, due to their simplicity in terms of set-up and the cheap material and chemicals that are involved. The absence of specific chemicals also results in avoiding any chemical hazard. Finally, the experiments are extremely easy to implement and to control. From the student's viewpoint, the very visual aspect of these experiments will help gaining an intuitive understanding of the involved phenomena since "seeing is believing". This will in turn allow them understanding also several aspects of conventional electrochemistry that are often underestimated in usual laboratory classes. Last

but not least, these LED-based electrochemical systems also have applications in real life such as in analytical chemistry.³⁸

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available on the ACS Publications website at DOI: 10.1021/acs.jchemed.XXXXXXX.

SI contents : description of learning objectives; materials needed in the laboratory; hazards; lab organization; details of conducted experiments; materials needed to reproduce the experiments at home; and pedagogical benefits of the lab experiments.

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