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Probing the Electrochemical Properties of Exclusion Zone Water

(Menyelidiki Sifat Elektrokimia Zon Air Pengecualian)

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ABSTRACT

The presence of solute-free zones, called the exclusion zones (EZ) near water interfaces, is a phenomenon that has been extensively studied. While the EZ is shown to be negatively charged, it is balanced by an adjacent region that is rich in positive ions. This paper investigates this twin characteristic of EZ on electrical conductivity. We show that EZ has a strong inhibitory effect on electrical flow, especially the negative current, providing additional evidence to its structural integrity and rigidity. More interesting is the positive ion-rich region adjoining the EZ, we show this region is a highly conductive surface that facilitates the flow of negative current.

Keywords: Electrochemistry; exclusion zone; water conductivity

ABSTRAK

Kehadiran zon bebas bahan terlarut, dipanggil zon pengecualian (EZ) berhampiran air antara muka, merupakan fenomena yang telah dikaji secara meluas. Walaupun EZ ditunjukkan bercas negatif, ia diimbangi oleh kawasan bersebelahan yang kaya dengan ion positif. Kertas ini mengkaji ciri kembar EZ ini pada kekonduksian elektrik. Kami menunjukkan bahawa EZ mempunyai kesan perencatan yang kuat pada aliran elektrik, terutamanya arus negatif, memberikan bukti tambahan kepada integriti dan ketegaran strukturnya. Lebih menarik ialah kawasan yang kaya dengan ion positif yang bersebelahan dengan EZ, kami menunjukkan rantau ini adalah permukaan yang sangat konduktif yang memudahkan pengaliran arus negatif.

Kata kunci: Elektrokimia; kekonduksian air; zon pengecualian

INTRODUCTION

The presence of solute-free zones, called the exclusion zones (EZ) near water's interfaces is a phenomenon that is being extensively studied experimentally and theoretically in recent years (Chai, Mahtani & Pollack 2012; De Ninno 2017; Elton et al. 2020; Zheng & Pollack 2006, 2003; Zheng et al. 2006). This solute-free zone typically spans several hundred microns and is observed in a variety of hydrophilic surfaces; including natural and artificial hydrogels, the water-air interface, biological surfaces, hydrophilic polymers and metallic surfaces. Electrical measurements show large negative potential within the zone, of the order of 100 mV, and pH measurements show a large concentration of H^+ beyond the zone (Zheng & Pollack 2006). EZ has also shown to absorb radiant energy to build and maintain its structure at both the UV and infrared spectrum, with peak absorption at the 270 nm wavelength in the UV region (Chai, Yoo & Pollack 2009; Shen et al. 2024; Wang & Pollack 2021) while mid-infrared radiation could expand the EZ by a ratio of 1.41 (Wang & Pollack 2021). NMR spectroscopy shows that it is highly restricted and dynamically more stable (Yoo, Paranji & Pollack 2011). It can also assert mechanical forces measurable using a laser tweezers system (Chen et al. 2012). Refractive index measurement shows a 10% increase in the EZ (Bunkin et al. 2013).

With its large span, ubiquitous presence and enormous separation of charges, the effect EZ has on conductivity in the water body has yet to be investigated. Some studies have shown that conductivity is reduced within the EZ region (Chai, Yoo & Pollack 2009; Zheng et al. 2006). More interesting is a study by Kundacina, Shi and Pollack (2016) that showed the anesthetics inhibit the formation of EZ, which implies that EZ could be a key factor in the transmission of electrochemical signals in the body. Kundacina's experiment is the starting point of investigation in this paper.

The EZ has an interesting twin characteristics, the negatively charged EZ is accompanied by an abundance of positive ions in the aqueous regions adjacent to it (Chai, Mahtani & Pollack 2012; Litman et al. 2024; Zheng & Pollack 2006), which is the focus of the investigation in this paper. We confirm that EZ has a strong inhibitory effect on electrical flow, especially the negative current, which provides additional evidence to its structural integrity and rigidity. More interesting is the adjacent positive ions-rich region, we show this region is a fluid and highly conductive surface that facilitates the flow of negative current.

Our experiments are conducted on simple electrochemical setup with asymmetrical electrodes configurations with one electrode at the water surface and another submerged deeper at varying depth. For probing the conductivity of EZ we have the surface electrode just breaking the water surface, at 1 mm depth. For probing the ions-rich region below the EZ, the surface electrode was submerged at 2 mm depth but with the top 1 mm section shielded with PVC insulation. By alternating the polarity of the voltage supply on the asymmetrical electrodes' placements, we were able to do self-controlled comparisons of the charging currents. Our experiments show that electrical conductivity is measurably higher when the near surface electrode is the anode. Reversing the electrical polarity of the electrodes can result in conductivity variance by as much as 13%.

As demonstrated by Yee et al. (2019), electrical conductivity in water is associated with the macro-scale formations in the water body that carry electrical charges. The negative charge zones with excess of OH^- ions are alkali while the positive charge zones have excess H_3O^+ ions are acidic; this allows us to use universal indicator solution to observe the path of current flow. We use timelapse video to obtain visual evidence that the water surface is indeed an electrically active region. We observed that the alkali negative charge zone forms and fans out from the anode below the water surface, indicating a high conductivity region for the flow of electrons. What is remarkable is that even when the electrode was submerged at a depth of 25 mm, the electrons will climb up along the insulation shaft and spread out on the water surface.

The charge zones in ionic solutions have an intriguing characteristic that zones carrying opposite electrical charges were drawn together by electrical attractions, but they are shown to be able to maintain the charge separation for extended length of time. The formation of the charge zones also corresponds to a large increase in conductivity, by as much as 250% (Yee et al. 2019). With the findings presented in this paper, we propose a model that can give rise to these two characteristics of the charge zones.

The test station used in our experiments is a computer-controlled electrolysis setup with two electrodes and a DC power source. The schematic diagram of the control system is shown in Figure 1(a). Switch S1 is a three-poles switch that allows switching the circuit to charge, open circuit, or discharge mode. In Charge mode the electrodes are connected to the power source. In Open Circuit mode the power source is disconnected, for observing the charges stored in the water body after the charging phase. The Discharge mode will short circuit the electrodes to discharge the stored charges. The polarity toggle switch is to control the polarity of the voltage applied to the electrodes, so the Charge mode is further divided into Forward Charging and Reverse Charging.

The voltage and current are measured with a programmable Rigol DM3068 6½ digits Digital Multimeter (DMM). Switch S2 is to momentarily break the circuit and route the current to the DMM for current measurement. The DC power source is a generic brand adjustable 0-60V/0-3A power supply, with a measured ±60 mV fluctuation within our range of operation. We construct the control system using USB relay modules. A software written in C#.NET controls the switches and logs the data into the database. The experiments are cyclic in nature; the software will repeat a program of forward/reverse charging for a preset number of times or until it is stopped manually. Data sampling rate is one sample every 20 s.

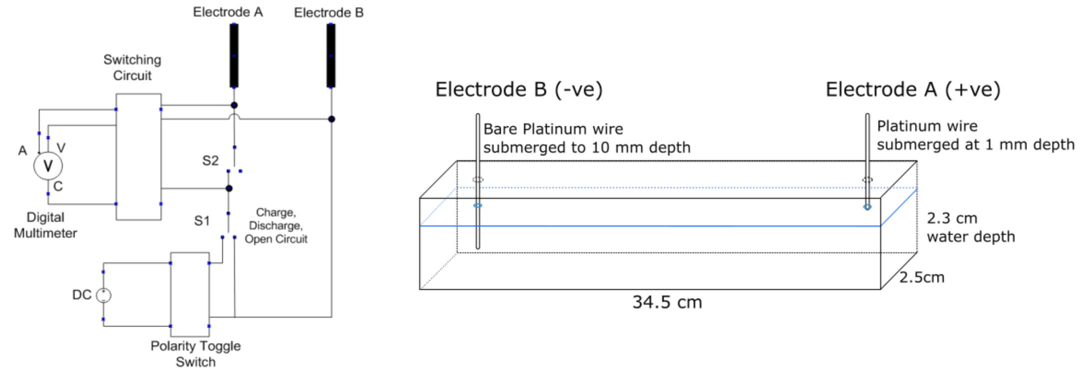
THE EFFECT OF EZ ON CONDUCTIVITY

This section investigates the electrochemical property of EZ on water surface via an asymmetrical electrochemical setup where one electrode is placed at the EZ, just breaking the water surface. As shown in Figure 1(b), this setup consists of a long acrylic container of base dimension 34.5 cm × 2.5 cm. We use 200 mL liquid volume, which gives a water depth of 23 cm. The electrodes are 26-gauge (0.405 mm diameter) 99.95% purity bare platinum wires. The left electrode (the anode during forward charging) is submerged to 10 mm depth. The right electrode (the cathode during forward charging) was submerged at 1 mm depth.

Figure 2(a) shows the charging current of 80 mM Na_2SO_4 at 5V. The current is obviously lower during the reverse cycle, when the short electrode at the EZ is the anode (-ve). This shows that the negatively charged EZ is a rigid structure that inhibits the flow of electrons. The analysis is qualitative in nature because the current is highly influenced by charge zone formation. Yee et al. (2019) has done extensive experiments that show the large changes in charging current can happen within a charging cycle. The current also varies substantially from one cycle to the next because the charge zones formed will leave residues in the water body.

Figure 2(b) shows the charging current of the solution of the same molar concentration at 6V. The difference in forward and reverse charging is not as pronounced as Figure 2(a). The voltage differential caused by the EZ is around 200 mV (Zheng et al. 2006), the 1 V increase in charging voltage would be expected to counter the resistance of the EZ significantly. The variation in current within a charging cycle is less pronounced than in

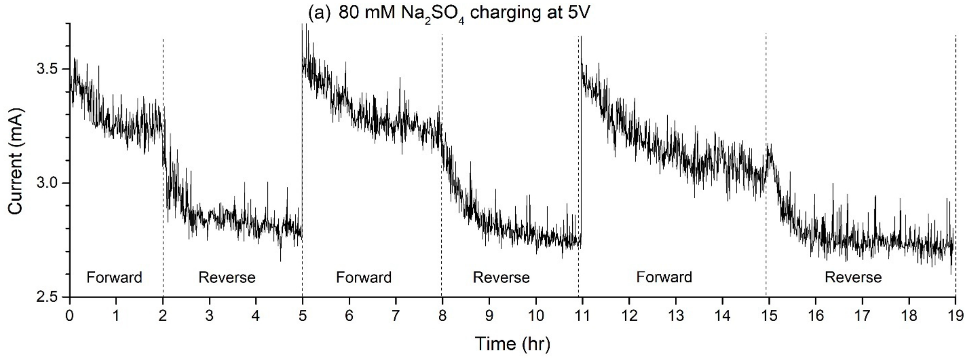
Experimental Setup and Methodology



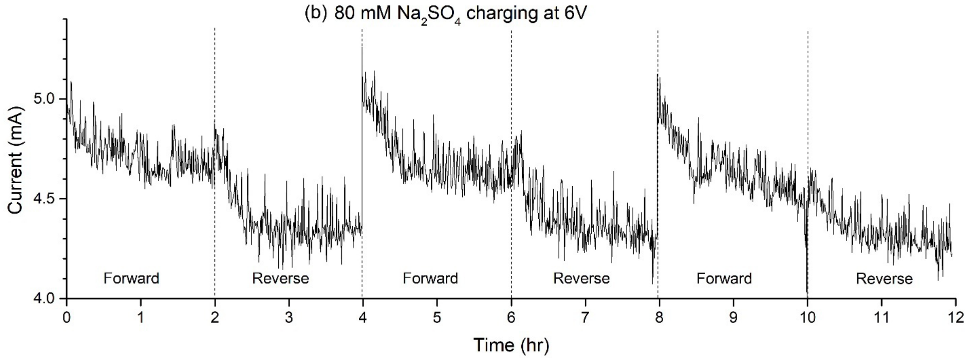
(a) Control Circuit (b) Electrochemical Setup

FIGURE 1. Control circuit and electrochemical setup

(a) 80 mM Na_2SO_4 charging at 5V



(b) 80 mM Na_2SO_4 charging at 6V



(c) 160 mM Na_2SO_4 charging at 6V

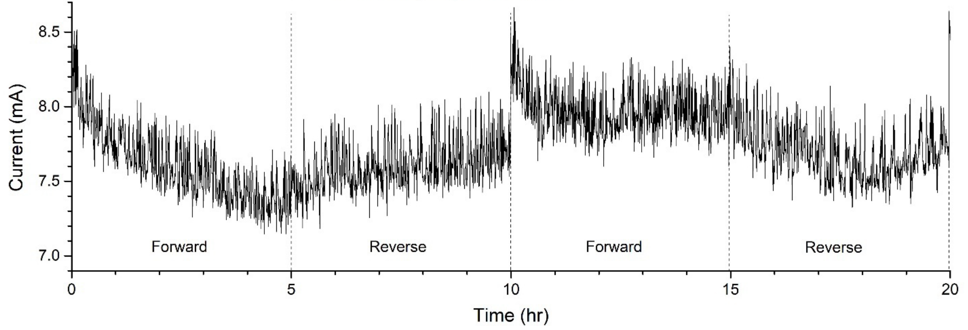


FIGURE 2. Charging current of Na_2SO_4 at different molar concentrations and voltages

Figure 2(a) too. The voltage difference between the positive and the negative charge zones is around 500 mV (Yee et al. 2019), so similarly the higher charging voltage would reduce the effects of the charge zones.

What is the effect of molar concentration in charging current? Figure 2(c) shows the charging of 160 mM Na_2SO_4 at 6 V. Compared to the previous figures, the asymmetry in forward vs. reverse charging is much less pronounced. This is due to two factors, the first being that the higher solute concentration diminishes the EZ (Zheng & Pollack 2003), especially the formation and extent of EZ water are shown to be highly sensitive to solute presence, with even low concentrations (10-40 mM) having a significant impact (Zheng, Wexler & Pollack 2009). The second factor being that the high solute concentration facilitates conductivity thus diminishes the effect of the EZ.

CONDUCTIVITY OF H^+ RICH REGION ADJACENT TO THE EZ

The following experiments use a glass cup as container with 26 gauge (0.405 mm diameter) 99.95% purity platinum wires as electrodes. The platinum wires are mounted on electrical connectors, and the assembly mounted on a rounded perplex piece that fits exactly over a glass cup (Figure 3(a)). The wires are 3.5 cm apart. The cup has a 170 mL capacity, and our experiments use a standard volume of 165 mL of solution.

In the previous experiment, the electrodes were far apart separated by a long narrow body of water which gives a lot of room for charge zone residues to be left behind in the water body as documented in Yee et al. (2019), causing unpredictable variations in charging current both within and between the charging cycles. With the current electrochemical setup, the shorter distance between the electrodes surrounded by a large body of water reduces the transient effect of the charge zones, making it more amendable for quantitative analysis.

As with the previous experiment, the following experiments feature a short electrode at the water surface against another long-shielded electrode submerged at different depths. We shall call former the surface electrode and the latter, the deep electrode. The wires are generally shielded with PVC insulation leaving a 1 mm exposed section at the lower tip (Figure 3(b)). When say we specify a 5 mm shielded electrode, we mean an electrode that is submerged at 5 mm depth, with an exposed region from 4 mm to 5 mm. The insulation materials are PVC jackets of 0.5 mm single core wires.

The surface electrode dips into the solution at a fixed 2 mm depth, so that it is at the H^+ rich region just below the EZ. In the first series of experiments, the top 1 mm section is shielded (Figure 3(c-A), so that the exposed section is 1 mm below the water surface. The shielding is to prevent the electrode from direct contact with the EZ. The second series of experiments is a continuation of the first, with the shielding removed from the surface electrode (Figure 3(c-B), so that we have a fully exposed 2 mm section spanning the EZ and the positive ions-rich region below. The deep electrodes are submerged at varying depths, 2 mm, 5 mm, 15 mm, and 25 mm, respectively. The full experimental parameters are given in Figure 3(d).

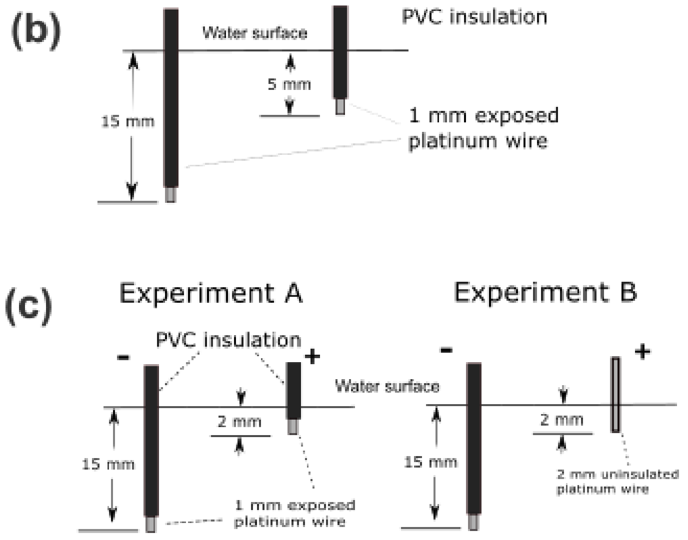
The charging current can vary significantly with the building up of charge zones (Yee et al. 2019), and our configuration took 4 to 5 cycles for the current to settle into a relatively stable pattern (Figure 4). We allowed 10 cycles for the current to be settled, and took the data for the next 4 cycles for experiment A. Experiment B is an immediate continuation of experiment A with the shielding removed on the surface electrode. We allowed for a further 2 cycles for the current to settle then, took the data of the next 4 cycles. Table 1 shows the average charging current for experiments A and B.

Herewith are some observations: The conductivity (on both forward and reverse charging) decreased from samples 1 to 2 then, went up again in sample 3 and 4, despite the monotonic increase in distance between the electrodes from sample 1 to sample 4. Conductivity in an ionic solution is not a function of distance; it is highly dynamic and is governed by the location of the charge zones relative to the electrodes. We will elaborate more on this in the next section.

There is a clear asymmetry between forward charging and reverse charging. Reverse charging, i.e., negative polarity on the surface's electrode has significantly higher conductivity. That the water surface with a negatively charged EZ would facilitate negative charge dispersion seems counter intuitive at first glance. While the previous section established that the negatively charged EZ is tightly bound and non-conductive, it induces a self-replenishing collection of positive ions - a mixture of H_3O^+ and Na^+ - underneath, creating a conductive surface for the negative charges.

Experiments A and B are not directly comparable because experiment B has a larger conducting surface on the surface electrode. As expected, B showed higher conductivity, however, the increase in current is not proportional with respect to forward and reverse charging. The difference is significant; reverse charging shows an increment of 46% vs. 28% for forward charging. Experiment A and B differs only in the contact with the surface 1 mm of water, and our results indicate that significant number of negative charges are dispersed within this 1 mm layer.

Figure 5 shows the current during discharge phase for experiment A and B. This is another surprising result. The discharge current for experiment B on the reverse cycle almost disappeared completely. This is an indication that a sizable positive zone has formed near the water surface just below the EZ, and the unshielded 2 mm electrode is in contact with it leading to rapid internal discharge with the negatively charged EZ.



(d)

Voltage: 4 V

Cycles: 10 min forward charging, 5 min open circuit, 20 min discharge. The cycle is repeated with reverse polarity. Forward charging is the surface electrode as anode (positive polarity).

Solution: 1 ml of 2 M NaCl in 164 g deionized water (12 mM molar concentration)

Electrode configurations

Experiment A (shielded 2 mm)

Sample 1: shielded 2 mm

Sample 2: shielded 5 mm

Sample 3: shielded 15 mm

Sample 4: shielded 25 mm

Experiment B (unshielded 2 mm)

Sample 1: shielded 2 mm

Sample 2: shielded 5 mm

Sample 3: shielded 15 mm

Sample 4: shielded 25 mm

FIGURE 3. Experimental setup and parameters

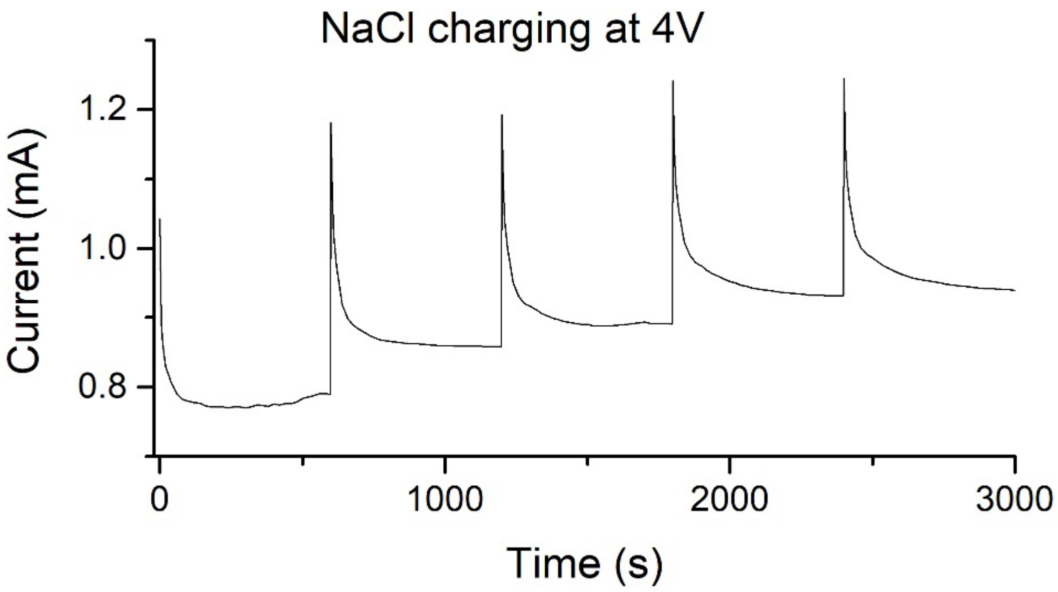


FIGURE 4. Charing current of HCl at 4V. It takes 4 to 5 cycles for the current to settle into a stable pattern

TABLE 1. Probing the effect of surface EZ on conductivity. Experiment A has an electrode insulated from the top 1 mm of water. Experiment B is with the insulation removed. Forward charging is defined as having the near surface electrode as the cathode (positive polarity). The result shows that the water surface has a significant impact on negative charge dispersion

	Sample 1	Sample 2	Sample 3	Sample 4
Deep electrode depth	2 mm	5 mm	15 mm	25 mm
Experiment A				
Forward charge current (mA)	3.78	3.69	3.71	4.12
Reverse charge current (mA)	3.75	3.68	3.90	4.35
Variance (forward vs. Reverse)	-0.8%	-0.3%	5.1%	5.6%
Experiment B				
Forward charge current (mA)	4.84	4.41	4.44	4.66
Reverse charge current (mA)	5.47	4.70	4.92	5.25
Variance (forward vs. Reverse)	13%	6.6%	11%	13%

B vs. A	Note: A and B are not directly comparable, the asymmetry with respect to the increase in current between forward charging and reverse charging is of interest here			
Increase in forward charging current	28%	20%	19%	13%
Increase in reverse charging current	46%	28%	26%	21%

VISUALIZING THE CHARGED ZONES DYNAMICS USING UNIVERSAL INDICATOR SOLUTION

In this experiment, we use universal indicator solution to show the charge zones dynamics in NaCl solution. The electrochemical configuration is as with the previous experiments, with the electrodes dip down to a depth of 15 mm. The experiment parameters are given in Table 2. Experimental parameters for using universal indicator solution and time-lapse video to visualize the charge zones dynamics.

The timelapse video is uploaded in YouTube, refer to the following link: https://youtu.be/aNMrYM62vbc. The first few seconds are particularly interesting, we suggest playing the video at 0.25 speed. Figure 6 are snapshots of progression through the first positive charging cycle. The anode (positive electrode) is on the left. Photos (a)-(d) show the charge buildup during the charging phase; (e)-(f) show what happened after the voltage source is cut off. Herewith are our observations, and some interpretations.

(a) the blue (negative) zone forms and spreads out from the water surface and rains down from there. This is a very surprising feature. It is true even for sample 2 where there the electrodes contact the solution 14 mm below the water surface. For sample 1 we do not see any sign of blue forming on the length of the electrode, so it seems that the blue zone is formed almost exclusively at the water surface. For sample 2, there is a streak of blue dropping down from the exposed tip, but it is small compared to the massive blue zone forming at the water surface. This indicates that the negative current traverses and disperses along the EZ. This is true even with sample 2, as the PVC insulator on the electrode is surrounded by a layer of EZ that continues to the water surface. The Blue (negative) zone appeared to be denser and less mobile, and it sunk to the bottom. The orange (positive) zone spread from the anode towards the cathode. It appears to have higher mobility and occupies the upper part of the cup.

(b), (c), (d) the blue zone is pushed to the far side of the cup away from the anode. There is counter intuitive and intriguing. The blue and the red zones can remain separated for hours; they do not seem to neutralize each other despite being drawn together by electrical attractions. This indicates the presence of an insulation layer surrounding the blue zone maintaining its integrity and insulates it from the surrounding negative charge zones. What is the nature of this interfacial layer? We conjecture that it is EZ, as it shows characteristics typical of EZ as described in the literature. The negative charge zone with the surrounding positive charge zone is seen as having a net positive charge, and the ensemble is being pushed away from the anode. (d) towards the end of 15 min charging, we can see a misty light-blue diffusing from the bottom upwards towards the positive electrode on the left.

(e) - (f) with the voltage source disconnected the blue zone began to drift and diffuse gradually. The electrodes are short circuited at t=20 (e). The discharge currents drop down

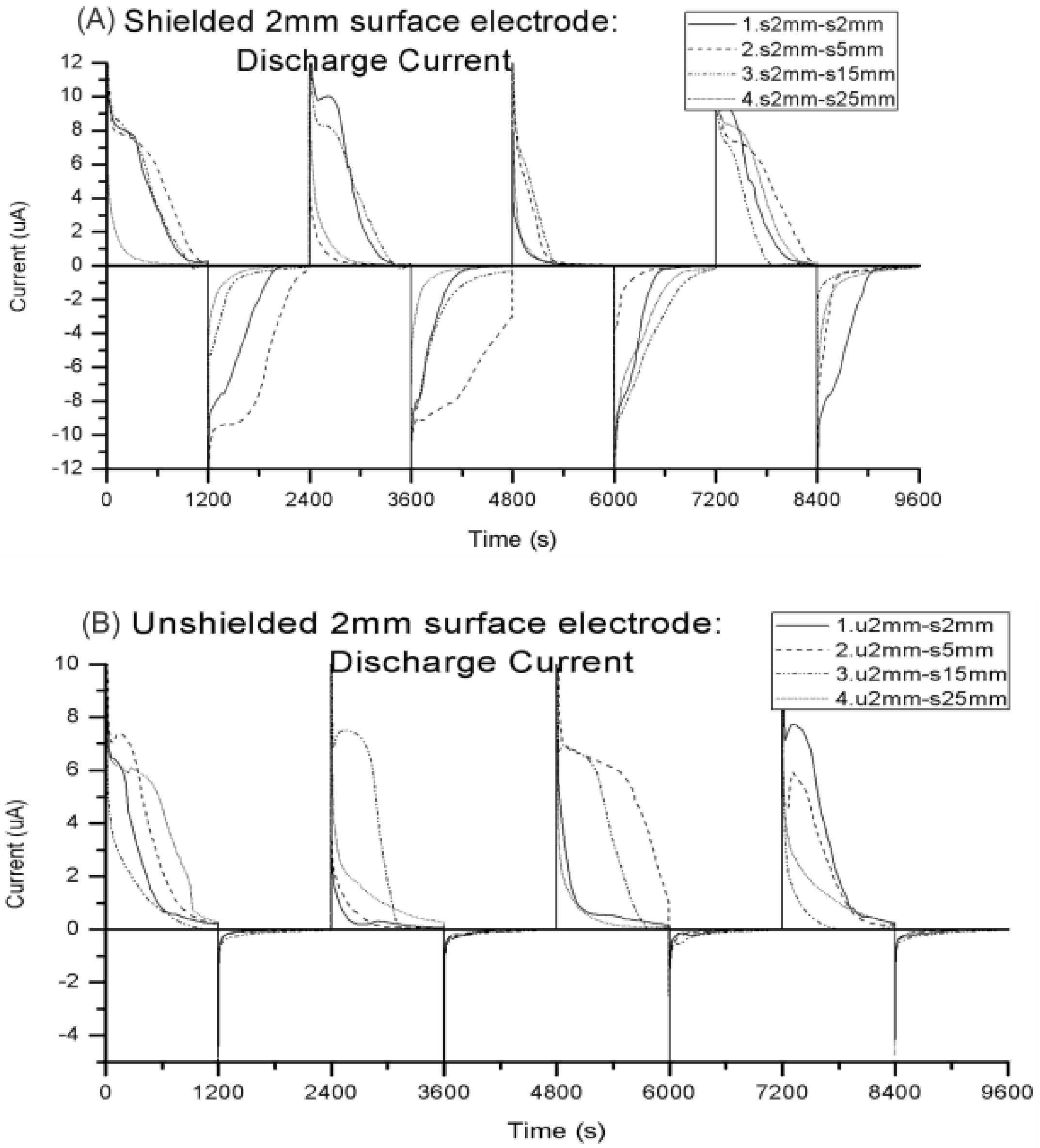


FIGURE 5. The discharge current graphs of experiments A and experiments B

TABLE 2. Experimental parameters for using universal indicator solution and time-lapse video to visualize the charge zones dynamics

Voltage	4 V
Cycles	15 min charge, 5 min open circuit, 40 min discharge. Repeat with inverse polarity
Solution	2 mL of 2 M NaCl in 161 g of deionized water + 2 mL of universal indicator. NaCl ion concentration is 24.2 mM, the universal indicator is an ionic solution of unknown composition
Electrodes	(Configuration 1) two 15 mm platinum wires fully exposed (Configuration 2) two 15 mm shielded platinum wires with 1 mm exposed tip

very quickly - and we can see visually that the charged zones were diffusing in a rather chaotic manner. (f) 30 min into the discharge phase. For sample 1, it looks like there is a concentration of blue surrounding the positive electrode on the left, but the zone was mostly at the back portion of the cup behind the electrode. The 2D projection of a 3D happening can be misleading. The discharge current at that point was -0.2 μA, when we shifted the electrode into the blue zone the discharge current increased to -0.8 μA.

The charge zone's location and movements can vary greatly with different conditions and/or solutes. Figure 7 shows stable patterns that formed approximately 20 min into the discharge stage with two different solutes – NaCl and Na_2SO_4. It took a few charge/discharge cycles for the patterns to settle down, and once it is stabilized the pattern will repeat through several cycles, but eventually another pattern will emerge, probably due to changes in chemical composition caused by the electrolytic process.

The left photo shows two samples of sodium chloride solutions, 2 mL of 2 M NaCl in 161 g of deionized water + 2 mL of universal indicator. The ionic concentration for NaCl is 24.2 mM, the universal indicator is an ionic solution of unknown composition. Left cup: 2 platinum wires fully exposed. Right cup: 2 platinum wires shielded with PVC jacket except for 1 mm length at the lower tip. Cycle: 15 min charge, 5 min open circuit, 40 min discharge, 4 V charging voltage. The photo is a snapshot of the open circuit phase after the sixth charging cycle (a forward charging followed by a reverse charging is counted as 2 cycles).

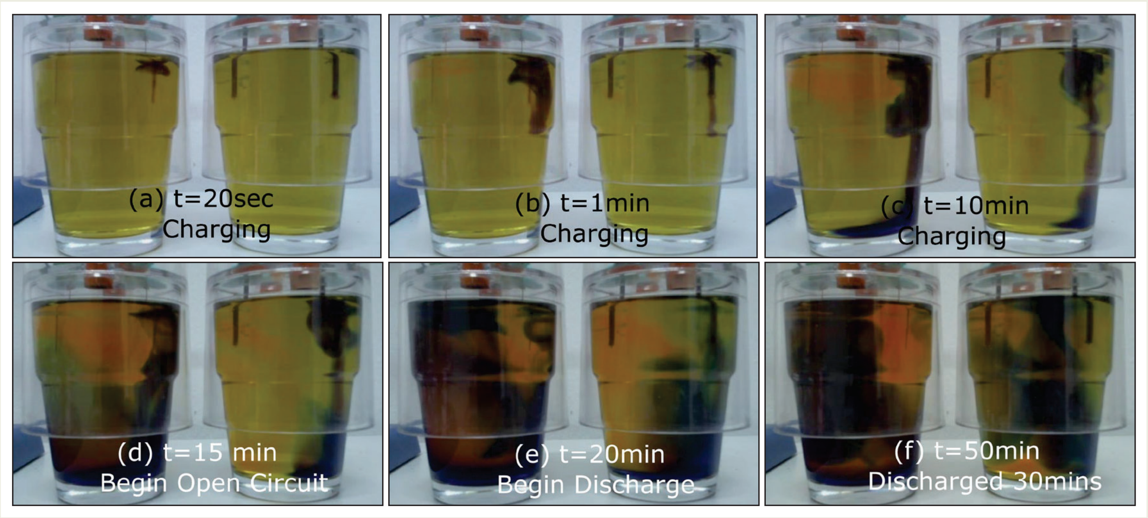


FIGURE 6. Using universal indicator solution to visualize charge zone dynamics in NaCl solution. (a)-(f) shows the progression through the first positive charging cycle. Sample 1 (left) has two 15 mm fully exposed platinum wires, sample 2 (right) has shielded electrodes with 1 mm exposed tip at the bottom

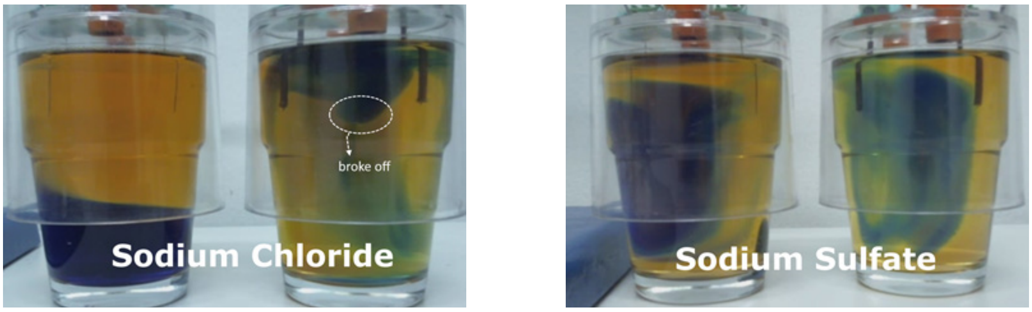


FIGURE 7. The charge zones locality and movements vary greatly with different conditions and/or different solutes. The photos show stable patterns that form approximately 20 min into the discharge stage. It usually takes a few charge/discharge cycles for a pattern to settle down, and once it is stabilized the pattern will repeat through many cycles

Sample 1 (left) has fully exposed wires thus a higher rate of electrolysis. We see here there is a massive layer of blue settled at the bottom. Sample 2 with shielded wires created much lower volume of the blue zone – and it was drawn to the water surface. We also see a bulge dangling at the bottom, which eventually broke off, fell downwards and became neutralized. The mechanism that drew the negative zone towards the negatively charge EZ is probably the like-likes-like principle (Zhao, Coult & Pollack 2010), where the negatively charged EZ creates a concentration of positive ions underneath, which in turn assert an attraction on the negative zones. We can see the interplay between the electrical forces and gravity here. The negative charge zone has higher density and would sink to the bottom, but at the same time the EZ induced electrical forces is attracting it upwards – so when the zone is below a certain size it will get lifted to the water surface.

The right photo shows two samples of sodium sulfate solution, 2 mL of 2 M Na_2SO_4 in 161 g of deionized water + 2 mL of universal indicator. The ionic concentration of Na_2SO_4 is 24.2 mM, the universal indicator is an ionic solution of unknown composition. Left cup: 2 platinum wires fully exposed. Right cup: 2 platinum wires shielded with PVC jacket except for 1 mm length at the lower tip. Cycle: 15 min charge, 5 min open circuit, 40 min discharge, charging voltage 4 V. The photo is a snapshot of the open circuit phase after the fifth charging cycle. Here in both samples the blue zones are suspended in the center of the cup, showing that there is no obvious disparity in the relative density of the different zones.

We also see here that the blue negatively charged zone is bound together with considerable cohesive force. The zone is able to hold together despite the repulsive force of a considerable concentration of negative charges, and resist being neutralized by surrounding positive zones. The negative zone is formed at the cathode, where the Na^+ ions are attracted, and OH^- is produced. We conjecture that the negative zone is a large-scale lattice structure consisting of Na^+, OH^- and H_2O molecules. The anode is where Cl^- (or SO_4^{2-}) is drawn to and H^+ (which hydrate immediately to H_3O^+) is produced, so similarly the positive zone would be a lattice structure that consists of Cl^- and $H3O^+$. The positive zone seems to has higher mobility, they are less structurally coherent, and they gather loosely around the negative zones.

CONCLUSION

While current research shows that EZ is ubiquitous on water interfaces with other substances, we propose that the EZ also appears within the water body, especially surrounding the negative charge zones. The EZ surrounding the negative charge zone acts as an insulation layer that preserves the charge separation between the positive and the negative charge zones, we posit that this is the mechanism that allows ionic solutions to store substantial number of charges (Ovchinnikova & Pollack 2009). As the negative charge zone grows in the electrochemical process, the EZ surface also grows proportionally. As shown in this paper, the EZ is a conductive layer that facilities the flow of electrons. This will explain why conductivity can increase by as much as 250% (Yee et al. 2019) as the charge zone grows. Figure 8 is a schematic diagram showing the role of ionic solutes in charge zones formation and how they are integral to electrical conductivity in water. Figure 9(a) shows the anatomy of EZ. The EZ itself is a tightly bound non-conductive structure, the adjacent positive ions-rich region, however, is a fluid and highly conductive surface. Figure 9(b) is a schematic diagram showing how the buildup of negative zones increases the conductive surfaces in the solution.

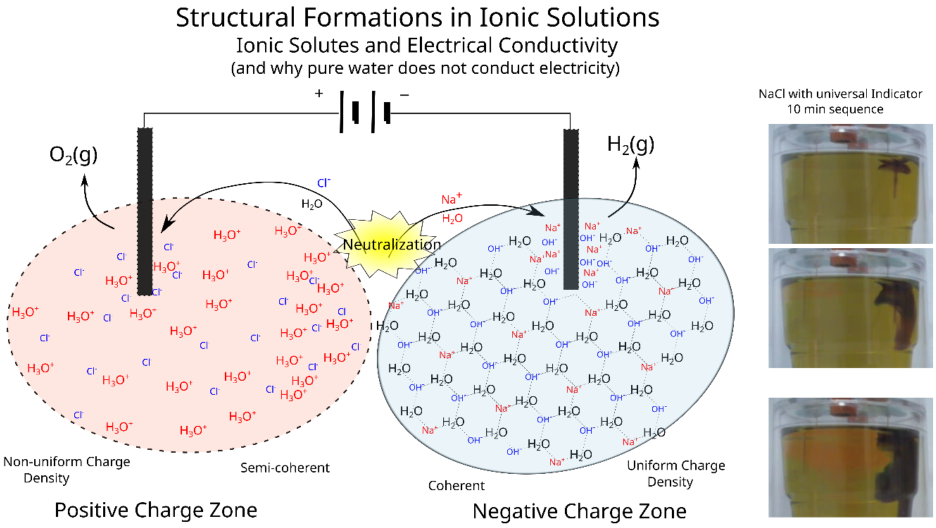


FIGURE 8. Schematic diagram showing the role of ionic solutes in charge zone formation, and how they are integral to electrical conductivity in water

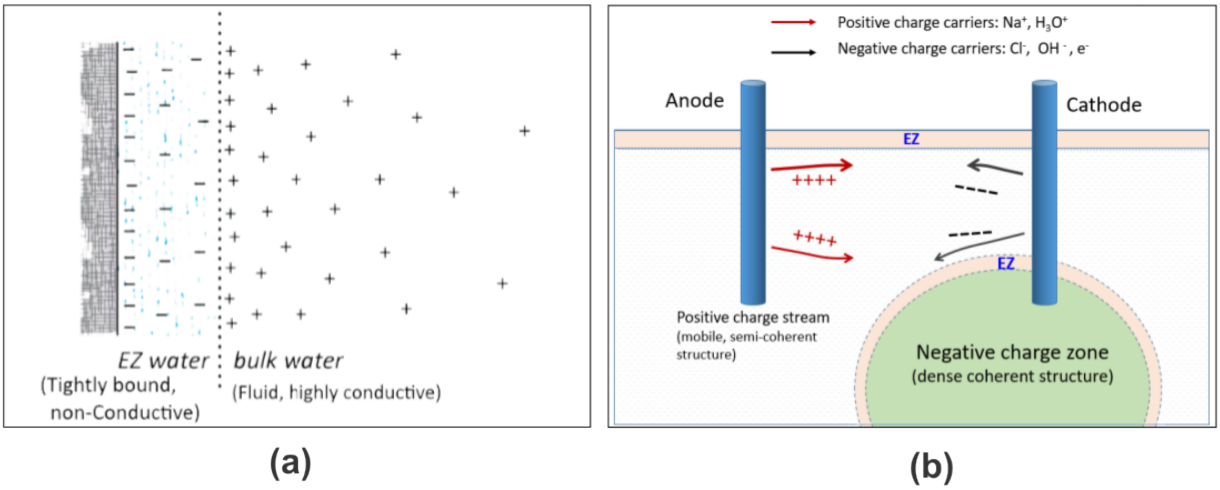


FIGURE 9. Schematic diagrams (a) the anatomy of EZ (b) we propose that EZ forms around the negative charge zones, which explains why charge zones formation can cause a dramatic increase in conductivity

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